A LABORATORY MANUAL

FOR

PHARMACEUTICAL ORGANIC CHEMISTRY

Pharmaceutical Chemistry Division
Balaji college of Pharmacy

Ananthapuramu, Andhra Pradesh – 515001

Lab manual

Pharmaceutical organic chemistry-III

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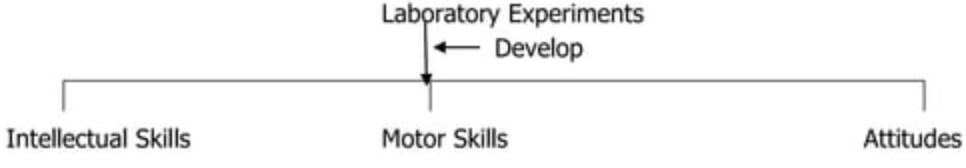
QUALITATIVE ORGANIC ANALYSIS:

Introduction

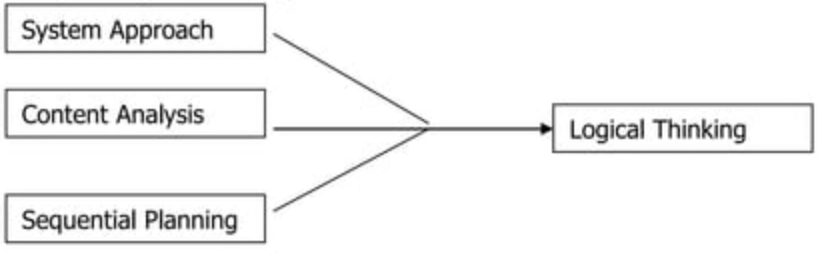
1.0 Title:
Introduction to Laboratory/Laboratory Work.

2.0 Prior concepts:
Curriculum concepts, Scope of work, Planning, Assessment.

3.0 New Concepts :
Proposal 1: laboratory Experiments
Laboratory experiments are expected to develop intellectual skills, motor skills and attitudes in the students.

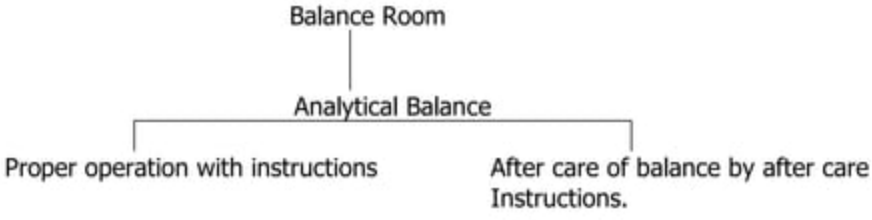


Proposal 2: Logical thinking
Logical thinking is developed in students through systems approach, content analysis and sequential planning of laboratory work.



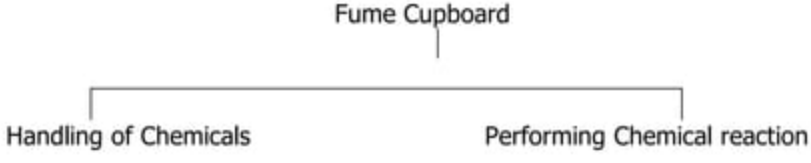
Proposal 3: Balance Room

It is a room where analysis weighing balance is kept. The operation of balance is demonstrated to the students and they handle the balance under the supervision of teacher.



Proposal 4: Fume Cupboard

It is an enclosed chamber with exhaust fan for handling dangerous chemicals and processing reactions.



4.0 Procedure:

1. Read the learning overview carefully.
2. Listen to the lecture given by teacher about importance of the subject, curriculum philosophy, graphical structure, and skills to be developed, information about equipment, instrument, procedure, and method of continuous assessment and tentative plan of work in laboratory.
3. Take the students round the laboratory, show balance room, fume cupboard and explain about the general working in laboratory.
4. Student shall observe the equipment/instrument and record the information in the following table.
5. Observe the charts and diagrams displayed in the laboratory.
6. Understand general precautions to be followed while working in the Laboratory.

Definition of Qualitative organic analysis

Qualitative organic analysis in simple words means the systematic identification of an unknown organic compound.

The student must be prepared to apply his/her knowledge of organic chemistry at all times and must be alert to the significance of any observations which may be throw light on the analysis.e t

Importance and need of qualitative organic analysis :

Qualitative organic analysis is an essential part of the training of the young organic chemist. It gives balance between structured systematic approach and the perception, which the student acquires with experience.

For the systematic identification of an unknown organic compound the students should follow the following steps without skipping to the next step.

a) Preliminary examination / Preliminary tests
b) Detection of elements /Elemental analysis
c) Detection of functional groups
d) Derivative preparation
e) Determination of physical constants
f) Separation of binary mixture

In this chapter, the students will get exact idea about what are the tests to be performed for particular

Functional group along with the choice of derivative. It will help in error free identification of an unknown organic compound. The tests are given in sequence one after the other, so as to reach near the end result and to confirm the given unknown organic compound, with out confusion.

Experiment No.1

1.0 **Title :**

To identify the given organic drug/compound D1 by Systematic Qualiative Analysis.

2.0 **Prior Concepts:**

Drug, Qualitative analysis, Organic compounds, Melting Point/Boiling point, Aromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2:- Physical constant (Melting Point/Boiling Point)

It is characteristic, distinguishing Physical Identity of the Organic compound.

Proposal: Elemental Analysis.

It is finding out of all elements present in the organic compound by some colour and precipitation reaction.

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound that renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements, groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reactions or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the Organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analyse and interpret the observations
4. To plan the experiment.

4.2 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.

3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus: 1. Glass wares: Test tubes, Beakers, Measuring Cylinder, Graduated Pipettes, Evaporating dish, Water bath, beaker, 100°C Thermometer, 360°C thermometer, wire gauze.
2. Chemicals: All general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis
2. Refer to Laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary Tests:

Sr. No	Test	Observation	Inference
a	Colour		
B	Odour		
C	Solubility behavior 0.2 ml or 4 drops of liquid/ Solid or 0.1 gm of solid + 3ml.of the solvent. Shake thoroughly. If sample does not dissolve warm gently and cool to room temperature		
I	solubility in Water		
II	Cold or hot solution, test With litmus		
III	If acidic, add a substance To 10% sodium bi-carbonate solution		
IV	If not soluble in Water, Then try in 2N NaOH		

V	If not soluble in 2 N NaOH, then try in dil. HCI		
d 1	Action of reagents Action 0.2 g/3 drops of Compound + 2 ml of Water +2ml 10% NaOH and the Mixture is shaken well		
II	Action of hot con. H_2 SO_4 0.1g solid or 2 drops of Liquid + 1ml con.H_2SO_4 Warm.		
III	Action of Na_2 CO_3 solution: 10 ml of 10% Na_2 CO_3 Solution + 0.2g of solid or 4 drops of liquid.		
IV	Action of $KMnO_4$ solution: 0.2g of solid or 4 drops of Liuq2id + 10ml Na_2 CO_3 soln + drop – by-drop $KMn0_4$ solution		
V	Action of bromine Water		
VI	Action of $FeCl_3$ soln: Substance + Water + a drop of two of $FeCl_3$ soln		
e	Heating on oxidized Copper gauze (Beilstain's test)		
f	Heating in a dry test tube		
g	Heating on a clean glass rod		
h	Heating with soda lime: Take in hard glass test Tube, 0.5 g of substance + 2g of finely powdered Soda lime + 1g of coarse Soda lime If the substance is Liquid, add 5 drops of it to a fine layer of soda lime, close the tube by a cork with a bent delivery tube, heat from top of test tube downwards neatly at first and then strongly, and collect the product		

Conclusion:- On the basis of the tests performed above and with reference to handbook the given organic drug/compound is

1) Aromatic/aliphatic
2) Saturated/Unsaturated
3) Acid/Base/Neutral/Phenol Halide
4) .....

7.2 Determination of Physical constant:

Conclusion :- The melting point/boiling – Point of the given organic compound was found to be .....

7.3 Determination of elements (Lassaigne' s Test)

Sr. No	Test	Observation	Inference
a.	Test for Nitrogen 3-4 ml filtrate + solid FESO4 ,till saturation, heat to boil for few minutes, then acidify with con. H2SO4		
b.	Test for sulphur: i)2ml filtrate +1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii)2ml filtrate + acetic acid to acidify + few drops of leads acetate solution. iii)2 ml filtrate + HCl to neutralize the soln + few drops of $Fecl_3$ solution.		
c.	Test for Halogens: i) Acidify 5ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/ or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution. ii) Chloroform layer test Filtrate, acidify with mineral acid + 1ml of chloroform + few drops of fresh chorine water, shake well and observe the colour of the chloroform		

	layer.		

Conclusion: The given organic drug/ compound found to contain _____ elements.

7.4 Determination of Functional Group/Groups (For .....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/compound was found to contain .....functional group/groups.

7.5 Result:

The given organic drug/ compound was having ..... M.p/b.p ....., elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural formula and category:

(Student shell write it from Indian Pharmacopoeia)

10.0 Questions:

Note: Write answers to the following questions

1. List out all organoleptic tests for organic compounds.

2. Which preliminary tests can be perform for finding out unsaturation in the organic compound.
3. Give one important characteristics each of aromatic and aliphatic compound.
4. Why all aromatic compounds give sooty flame on burning?
5. Give concentration of concentrated sulphuric acid used in the laboratory.

(Space for Answers)

(Space for Answers)

Experiment No.2

1.0 Title

To identify the given organic drug/compound D2 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: test tubes, Beakers, measuring cylinder, Graduated pipettes,

Evaporating dish, water bath, beaker, 100^0 C thermometer, 360^o C thermometer, Wire gauze.

2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis
2. Refer to laboratory handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr. No	Test	Observation	Inference
a	Colour		
b	Odour		
c.	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
	i. Solubility in water		
	ii. Cold are hot solution, test with litmus.		
	iii. If acidic, add a substance to 10% sodium bicarbonate solution		
	iv. If not soluble in water, then try in 2 N NaOH,		
	v. If not soluble in 2 N NaOH, then try in dil. HCl		
d	Action of reagents i. Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
	ii. Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
	iii. Action of Na_2CO_3 solution:		

	10 ml of 10% Na_2CO_3 solution + 0. G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
	iv. Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
	v. Action of bromine water:		
	vi. Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
e.	Heating on oxidized copper gauze (Beilstain's tests)		
f.	Heating in a dry test tube.		
g	Heating on a clean glass rod.		
h	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic

2. Saturated/Unsaturated
3. Acid/Base/Neutral/Phenol Halide
4. .....
5. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (lassaigne's Test)

Sr. No	Test	Observation	Inference
a.	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b.	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c.	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.5 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student shall write it from Indian Pharmacopoeia)

7.0 Questions:

Note: Write answer to the following questions

1. Which class of organic compounds are acidic to litmus but do not gives effervescence with sodium bicarbonate.
2. Arrange the following class of organic compound in the increasing order of acidic nature, carboxylic acid, phenols, Nitrophenols.
3. What is Beilstain's test?
4. Give concentration of liquor ammonia used in laboratory.

(Space for Answers)

Experiment No.3

1.0 Title

To identify the given organic drug/compound D3 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: test tubes, Beakers, measuring cylinder, Beaker Graduated pipettes, Evaporating dish, water bath, b, 100^{0}C thermometer, 360^o C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis.
2. Refer to laboratory handbook for the sequence of various tests.

7.0 Observation Table and Conclusion:

7.1 Preliminary Tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent.Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		

II	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0. G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
e.	Heating on oxidized copper gauze (Beilstain's tests)		
f.	Heating in a dry test tube.		
g.	Heating on a clean glass rod.		
h.	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic

2. Saturated/Unsaturated
3. Acid/Base/Neutral/Phenol Halide
4. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (Lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
C I	**Test for halogens**: Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		
II	**Chloroform layer test** Filtrate, acidify with mineral acid + 1		

	ml of chloroform + few drops of fresh chlorine water, shake well and observe the colour of the chlorine layer.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.5 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student shall write it from Indian Pharmacopoeia)

1.0 Questions:

Note: Write answer to the following questions

1. If a compound decolorises bromine water with formation of precipitate what is the possible compound
2. What is chemical composition of soda lime
3. Whether boiling point various from place to place? Explain
4. Give the concentration of concentrated hydrochloric acid used in Laboratory

(Space for Answers)

Experiment No.4

1.0 Title

To identify the given organic drug/compound D4 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.1 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis.
2. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution:		

	10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
d	Heating on oxidized copper gauze (Beilstain's tests)		
e	Heating in a dry test tube.		
f.	Heating on a clean glass rod.		
g	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic
2. Saturated/Unsaturated
3. Acid/Base/Neutral/Phenol Halide
4. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements)lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.5 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

11.0 Questions:

Note: Write answer to the following questions

1. What precaution you will take for detection of halogens in elemental analysis, if the compound contains "N" and/or "S"?
2. Give the test for detection of "N" and "S" together in elemental analysis?
3. Write chemical equations for detection of "N" "S" and halogens in the elemental analysis?
4. What is difference between acetic acid and glacial acetic acid?

(Space for Answers)

Experiment No.5

1.0 Title

To identify the given organic drug/compound D5 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.

4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis.
2. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid		

	or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	**Test**	**Observation**	**Inference**
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
h	Heating on oxidized copper gauze (Beilstain's tests)		
i.	Heating in a dry test tube.		
j.	Heating on a clean glass rod.		
k.	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic
2. Saturated/Unsaturated
3. Acid/Base/Neutral/Phenol Halide
4. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.5 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

10.0 Questions:

Note: Write answer to following questions

1. How neutral solution of a compound is prepared for distinguishing tests for acids?
2. Name the functional groups present in compound analysed.
3. Name any two organic compounds belonging to the same chemical class as that of compound analysed

(Space for anwers)

Experiment No.6

1.0 Title

To identify the given organic drug/compound D6 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.2 Intellectual Skills:

5. To understand concept of the experiment.
6. To understand test procedure.
7. To analysis and interpret the observations
8. To plan the experiment.

6.1 Motor Skills:

6. Ability to write systematic analytical report.
7. Ability to handle equipment, take and record observations.
8. Ability to refer standard literature/Indian Pharmacopoeia.
9. Ability to work according to the plan of the experiment.
10. Ability of group working.

7.0 Apparatus:

3. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
4. Chemicals: all general and table reagents.

8.0 Stepwise Procedure:

5. Start the conduct of Systematic Qualitative Analysis.
6. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH:		

	About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
l.	Heating on oxidized copper gauze (Beilstain's tests)		
m	Heating in a dry test tube.		
n	Heating on a clean glass rod.		
o	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda		

	lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

2. Aromatic/Aliphatic
2. Saturated/Unsaturated
7. Acid/Base/Neutral/Phenol Halide
8. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements)lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S		

	and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.5 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

12.0 Questions:

Note: Write answer to following questions

1. Define chemically Tollen's reagent.
2. Which organic compounds give silver mirror test with Tollen's reagent
3. What is 2,4-DNP?
4. Name the group which is common in aldehydes and ketones.

(Space for Answers)

Experiment No.7

1.0 Title

To identify the given organic drug/compound D7 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations

4. To plan the experiment.

4.2 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis.
2. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		

V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	**Test**	**Observation**	**Inference**
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
p	Heating on oxidized copper gauze (Beilstain's tests)		
q	Heating in a dry test tube.		
r.	Heating on a clean glass rod.		
s.	Heating with soda lime: Take in hard glass test tube, 0.5 g of		

	substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic
2. Saturated/Unsaturated
3. Acid/Base/Neutral/Phenol Halide
4. .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements)lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:**		

	Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.6 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

10.0 Questions:

Note: Write answer to following questions

1. What is Tollen's reagent? How it is prepared?
2. Name the functional groups present in compound analysed.
3. Name any two organic compounds belonging to the same chemical class as that of compound analysed.

(Space for Answers)

Experiment No.8

1.0 Title

To identify the given organic drug/compound D8 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.

2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2 Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

3. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
4. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

3. Start the conduct of Systematic Qualitative Analysis.
4. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		

IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	**Test**	**Observation**	**Inference**
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
t.	Heating on oxidized copper gauze (Beilstain's tests)		
u	Heating in a dry test tube.		
v.	Heating on a clean glass rod.		

w	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

2. Aromatic/Aliphatic

2. Saturated/Unsaturated

7.0 Acid/Base/Neutral/Phenol Halide
8.0 .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize		

	the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.7 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

11.0 Questions:

Note: Write answer to following questions

1. What are esters?
2. Why ethyl acetoacetate does not give iodoform test although it containes $O{=}C\text{-}CH_3$ group?
3. Write the hydrolytic products of ester?

(Space for Answers)

Experiment No.9

1.0 Title

To identify the given organic drug/compound D9 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|
Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

5. Start the conduct of Systematic Qualitative Analysis.
6. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		
I	Solubility in water		

II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
x.	Heating on oxidized copper gauze (Beilstain's tests)		

y.	Heating in a dry test tube.		
z.	Heating on a clean glass rod.		
a.	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

3. Aromatic/Aliphatic

2. Saturated/Unsaturated

9.0 Acid/Base/Neutral/Phenol Halide

10.0 .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside.		

	ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.8 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

5.0 Questions:

Note: Write answer to following questions

1. What are alcohols?
2. Write down the reaction of alcohol with sodium metal.
3. Write down the reaction of alcohol with ceric ammonium nitrate solution.
4. What is estetrification?

(Space for Answers)

Experiment No.10

1.0 Title

To identify the given organic drug/compound D10 by Systematic Qualitative Analysis.

2.0 Prior concepts:

Drug, Qualitative analysis, Organic compounds, melting point/Boiling point, Aeromatic compound, Aliphatic compound.

3.0 New Concepts:

Proposal 1: Preliminary Tests

These are primary tests carried out to get some idea/clue about the compound. No definite conclusion can be drawn from these tests.

Proposal 2: Physical Constant (Melting Point/Boiling Point)

It is characteristic, distinguishing physical identity of the organic compound.

Proposal 3: Elemental Analysis

It is finding out all the elements present in the organic compound by some colour and precipitation reaction

Proposal 4: Group Analysis

It is to find out different functional group present in the organic compound.

Functional group is the group of elements present in the compound

That renders characteristic chemical and physical property to the compound.

Proposal 5: Literature for reference:

It is a reference table in which compounds are classified according to elements,

Groups and physical constants.

Final identification of the compound is done with reference to this table.

Proposal 6: Confirmative Test:

These are specific colour reaction or preparation of simple derivatives and determining its M.P/B.P., which confirms identification of the organic drug.

General concept structure:

Identification of the drug
|
Confirmative Test
|
Result with reference to literature
|
Elemental analysis
|
Group analysis
|
Physical constant
(M.P./B.P.)
|

Preliminary tests

4.0 learning Objectives:

4.1 Intellectual Skills:

1. To understand concept of the experiment.
2. To understand test procedure.
3. To analysis and interpret the observations
4. To plan the experiment.

4.2Motor Skills:

1. Ability to write systematic analytical report.
2. Ability to handle equipment, take and record observations.
3. Ability to refer standard literature/Indian Pharmacopoeia.
4. Ability to work according to the plan of the experiment.
5. Ability of group working.

5.0 Apparatus:

1. Glass wares: Test tubes, Beakers, measuring cylinder, Graduated pipettes, Evaporating dish, water bath, Beaker, 100^0 C thermometer, 360^0 C thermometer, Wire gauze.
2. Chemicals: all general and table reagents.

6.0 Stepwise Procedure:

1. Start the conduct of Systematic Qualitative Analysis.
2. Refer to laboratory Handbook for the sequence of various tests.

7.0 Observation Table and Conclusions:

7.1 Preliminary tests:

Sr.No	Test	Observation	Inference
a	Colour		
b	Odour		
c	Solubility behavior: 0.2 ml or 4 drops of liquid/ solid or 0.1 gm of solid + 3 ml of the solvent. Shake thoroughly. If sample dose not dissolve warm gently and cool to room temperature		

I	Solubility in water		
II	Cold are hot solution, test with litmus.		
III	If acidic, add a substance to 10% sodium bicarbonate solution		
IV	If not soluble in water, then try in 2 N NaOH,		
V	If not soluble in 2 N NaOH, then try in dil. HCl		
d. I	Action of reagents Action of cold NaOH: About 0.2 g/3 drops of compound + 2ml of water + 2ml 10% NaOH and the mixture is shaken well		
II	Action of hot con. H_2SO_4 0.1g solid or 2 drops of liquid + 1ml con.H_2SO_4, warm		
III	Action of Na_2CO_3 solution: 10 ml of 10% Na_2CO_3 solution + 0.2 G of solid or 4 drops of liquid.		

Sr. No	Test	Observation	Inference
IV	Action of $KMnO_4$ solution: 0.2 g of solid or 4 drops of liquid + 10 ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution		
V	Action of bromine water:		
VI	Action of $FeCl_3$ soln: Substance + water + a drop or two of $Fecl_3$ soln.		
b	Heating on oxidized copper gauze		

	(Beilstain's tests)		
c	Heating in a dry test tube.		
d	Heating on a clean glass rod.		
e	Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. **If the substance is liquid**, add 5 drops of it to a fine layer of \soda lime. Close the tube by a cork with a bent delivery tube downwards gently at first and than strongly, and collect the product.		

Conclusion: On the basis of tests performed above and with reference to handbook the given organic drug/compound is

4. Aromatic/Aliphatic

2. Saturated/Unsaturated

11.0 Acid/Base/Neutral/Phenol Halide
12.0 .....

7.2 Determination of Physical Constant:

Conclusion:- The melting point /boiling point of the given organic compound was found to be .....

7.3. Determination of elements (lassaigne's Test)

Sr. No	Test	Observation	Inference
a	**Test for Nitrogen**: 3-4 ml filtrate + solid $FeSO_4$, till saturation, heat to boil for few minutes, then acidify with con.H_2So_4		
b	**Test for Sulphur**: i) 2ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium		

	nitroprusside. ii) 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii) 2 ml filtrate + HCl to neutralize the soln + few drops of $FeCl_3$ solution.		
c	**Test for halogens:** Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and/or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and $AgNo_3$ solution.		

Conclusion: The given organic drug/compound found to contain ..... elements.

7.4 Determination of Functional Group/Group (For.....Elements)

Sr. No	Test	Observation	Inference

Conclusion: The given organic drug/ compound was found to contain .....functional group/groups.

7.6 Results:

The given organic drug/compound was having ..... m.p/b.p.,..... elements and ..... functional group/groups, therefore with reference to literature, the organic drug/compound may be .....

8.0 Confirmative Test:

Sr. No	Test	Observation	Inference

9.0 Structural Formula and category:

(Student should write it from Indian Pharmacopoeia)

11.0 Questions:

Note: Write answer to following questions

1. What are nitro compounds?
2. W hat is reduction?
3. What are the different metals used for reduction ?
4. What is the confirmatory test for nitro group?

(Space for Answers)

SEPARATION OF BINARY MIXTURE

The organic chemist is frequently faced to the task of separating mixtures of compounds. Since few organic reactions give a single product, this commonly occurs in the work up stage of a reactions when it s desired to isolate a single pure compound from mixture which may contain un-reacted starting materials and reagents, inorganic materials produced from reagents, and expected – unexpected by -products.

In order to gain an understanding of the principles involved and variety of methods, which may be employed, it can be useful practice for the organic chemistry student to carry out exercises on the investigation and separation of given mixtures.

The successful separation of a mixture of the two organic substances will depend largely upon the student's general knowledge of organic chemistry and in particular upon his acquaintance with the reactions given under qualitative detection of individual compound.

The identification of the components of a mixture involves a separation into individual compounds and the characterization of each according to the qualitative analysis of compound. It is far more important, to carry out the separation in such a manner that each compound is obtained in the pure state because, the renders the individual identification much easier.

It is impossible to give more than an outline of the methods employed, as the mode of separation depends upon the actual substances present as well as the classes to which they belongs.

The general method to be adopted for the analysis of mixtures of organic compounds is to separate them into their components and to identify each component as previously described.

Following three steps caries out the separation of binary mixture:

1) Physical nature of binary mixture
2) Type determination
3) Separation of binary mixture

1) Physical Nature f Binary Mixture:
   Physically the mixture may be either one of the following type:

a) Solid – Solid [Two solids mixed together]
   e.g. Benzoic acid + α – napthol]

b) Solid – Solid [Eutectic mixture i.e. at room temperature one solid liquefies] another solid & both becomes liquid; liquefied mass become solidifies on cooling]
   e.g. Camphor + Menthol

c) Solid – Liquid [Solution: i.e. solid may dissolve in liquid & forms a solution]

e.g. Acetone + Benzoic acid; Ethanol +Salicylic acid

d) Solid – Liquid [suspension; i.e. solid is observed at the bottom in a mixture]
e.g. Toluene + Benzoic acid

e) Liquid – Liquid
e.g. Pyridine + Toluene

Identification of mixture:

a) Two solids mixed together either both are colorless or colored or one of them may be colored : Solid + Solid
b) Pour about 2.0 mL of liquid on a watch glass and place it on an ice bath and observe:
i) if complete solidification: Eutectic mixture Solid + Solid)
ii) If solidification Partially: Solid + Liquid (suspension)
iii) No solidification: Solid + Liquid (solution) or Liquid + Liquid Mixture.

2) Type determination of Binary Mixture:

The mixture may be one of the following types:
(a) Acid + Phenol
(b) Acid + Base
(c) Acid + Neutral
(d) Phenol + Base
(e) Phenol + Neutral
(f) Base + Neutral
(g) Neutral + Neutral

The type determination and method of separation of organic binary mixture only depends upon its solubility in water. Take 0.5 gm of mixture in a test tube and add 5ml water and shake well and keep it for 2minutes and observe, if one of the component of a mixture is dissolved in water then it is called as water – soluble mixture.

To make an observation of solubility of colored compound is easy because, water will take color of it.

But it is difficult to find out the solubility of colorless compound(i.e. both the solids is a mixture are colorless and one is water soluble) In this case, take 0.5 gm of compound and add 5ml 10% aqueous $NaHCO_3$, only acid will give effervescence, i.e. oxalic acid (water soluble) where as other colorless compound viz., carbohydrates, urea, thiourea, amides etc. they will solubalise and do not give effervescence and you can draw the conclusion the mixture is water soluble& follow the method for separation accordingly.

The type is detected as follows:

(i) Type determination of water – soluble compounds: Place small quantity of mixture in test tube and add 1 to 2 ml of water only. Shake for two minutes and filter it, check the filtrate with litmus paper.

Place few drops of filtrate on watch glass and evaporate, if residue is observed on watch glass then one of the compound is water – soluble.

Test	Observation	Inference
Litmus test	a) Blue litmus turns red b) Red litmus turns blue c) No change on either litmus	Acid or phenol present. Base present. Neutral substance present.

To distinguish between acid &phenol, take little amount of compound and add 2.0ml of 10% aqueous solution of $NaHCO_3$. If effervescence of CO_2 observed acid is present and if no effervescence of CO_2, Phenol is present.

(ii) Type determination of water insoluble compounds:

Test	Observation	Inference
a) Place 0.2 to 0.3 of mixture in test tube + 2 ml 10% aq. $NaHCO_3$ solution. Shake well and filter above mixture, acidify the filtrate with concentrated HCl	Effervescence of CO_2 No effervescence of CO_2 White precipitate No white precipitate.	Acid may be present Acid absent Acid confirmed Acid absent
b) Take the residue of test (a) if type (a) is present OR take the mixture if type (a) is absent+2.0ml 10% aqueous NaOH solution. Shake well and filter, acidify the filtrate with concentrated HCl and cool	Residue is partly or completely soluble White precipitate No white Precipitate	Phenol may be present. Phenol confirmed Phenol absent.
c) Take the reside of test (b)if type (b) is present OR take the mixture if type (b) is absent+ 1:1 HCl. Shake well and filter, basify the filtrate by 10% aqueous NaOH solution.	Reside is partly or completely soluble Precipitate formed No precipitate	Base may be present. Base confirmed Base absent

Note: If only one type is found in above test then other type is neutral

In case of Solid – Solid mixtures, it is necessary first to find out the type of mixture and then the compounds are separated. However in case of Solid – Liquid of Liquid – Liquid mixtures the compounds are first separated and then type of the individual compounds is determined.

3) **Separation of Binary Mixture:**

(a) Separation of Solid mixture (Water Soluble): Place all the mixture (10gm) in 30ml water in a beaker and stir it for 10 minutes and filter.

Observation	Inference
Evaporate the filtrate to get crystals of substance and analyse it, for type determination.	Residue is subjected for type determination.

(b) Separation of Solid + Solid mixture (water insoluble) :

According to the types of organic mixture three groups are made for separation.

(i) Acid – Phenol/Acid – Base/ Acid - neutral.
(ii) Phenol – Base/ phenol – neutral
(iii) Base – Neutral.

(i) Separation of Acid – Phenol/ Acid - Base/ Acid – Neutral type mixtures: Place all the mixture (10gm) in a beaker, add to it with stirring 10% aq. $NaHCO_3$, Solution, till effervescence of CO_2 stops and filter it.

Observation	Inference
Cool the filtrate, add cone. HCl Slowly till blue litmus turns red. Acid precipitates. Filter the precipitate of acid. Wash it with water, recrystallized from hot water and dry it.	Wash the residue with water, recrystallized it with ethanol or water ethanol mixture.

Reaction:

$$R\text{-}COOH + NaHCO_3 \longrightarrow R - COONa + CO_2 \uparrow + 6H_2O$$

Sodium Salt (Soluble)

$$R - COOH + HCl \longrightarrow R\text{-}COOH \downarrow + NaCl$$

(ii) Separation of Phenol – Base/Phenol – Neutral type mixtures: Place all the mixture 10gm in beaker, add to it 10% aqueous NaOH solution [till red litmus turns blue] stirs well and filter it.

Filtrate (Na – salt of phenol)	Residue (Base / Neutral)
Add cone. HCl Slowly till blue Litmus turns red Phenol precipitates. Filter the precipitate, wash it with water, recrystallise from ethanol.	Wash the residue with water, recrystallise from suitable solvent.(for Base: ethanol- water, for Neutral: benzene etc.)

Reaction:

$$Ar - OH + NaHCO_3 \longrightarrow Ar - ONa + H_2O$$

(iii) **Separation of Base – neutral type mixtures:** Place all the mixture (10 gm) in a beaker adds to it about 20ml concentrated HCl + 20ml water mixture, stir the mixture for 10 minutes and filter it.

Note: After the separation of binary mixture, each compound is subjected for its individual identification by performing qualitative analysis.

Filtrate	Residue
Cool the filtrate, add slowly 10% aqueous NaOH solution till alkaline (red litmus turns blue) Base precipitate. Filter the precipitate, wash with water, recrystallise from ethanol or ethanol water.	Wash the residue with water, recrystallise by using suitable solvent.

Reaction:

$$R - NH_2 + HCl \longrightarrow R - NH_2 \cdot Cl$$

Base Hydrochloride of base (Soluble)

$$R - NH_3^+ Cl + NaOH \longrightarrow R - NH_2 \downarrow + NaCl + H_2O$$

c) Separation of solid + liquid mixture (Solution):

Take all the mixture (20ml in a distillation flask, add one porcelein piece, attach water condenser and thermometer to the distillation flask. Heat the flask gently. Collect

volatile compound in dry receiver at constant temperature, note down the temperature, it is the boiling point of the liquid. Stop the heating when temperature starts rising above the constant temperature. Pour the remaining liquid in porcelein dish. Keep it for cooling at room temperature for solidification.

d) Separation of Solid + liquid mixture (Suspension):

This type of mixture is separated by simply filtering by using filter paper and glass funnel. The residue obtained on filter paper can be recrystallised using suitable solvent if needed.

e) Separation of liquid – liquid mixture:

Place all the mixture (20ml) in a distillation flask, add one porcelaein piece, and attach water condenser and thermometer to the distillation flask. Heart the flask gently. Collect first liquid at constant temperature in dry receiver flask, noted the temperature, it is the boiling point of the first liquid. Stop the heating when temperature starts rising above the constant temperature. Pour the remaining liquid in porcelein dish. It is the second compound.

Note: After the separation of binary mixture, each compound is subjected for its individual identification by performing qualitative analysis.

Various examples of Binary mixtures:

i) Benzoic acid + α –napthol
ii) Cinnamic acid + β –napthol
iii) Phthalic acid +Resorcinol ⟶ (Water soluble)
iv) Salicylic acid + P – toluidine
v) Acetyl salicylic acid +α - napthylamine
vi) Benzoic acid + Napthacene
vii) Cinnamic acid + Anthracene
viii) Phthalic acid+Urea / thiourea / glucose ⟶ (Water soluble)
ix) α- napthol + P - toluidine
x) Resorcinol + Napthalene ⟶ (Water soluble)
xi) β- napthol + Anthracene
xii) Aniline + - Toluene ⟶ Liquid – Liquid
xiii) O-cresol + Toluene ⟶ Liquid – Liquid
xiv) Benzoic acid + Toluene ⟶ Solid – Liquid (Suspension)
xv) Cinnamic acid + Benzaldehyde ⟶ Solid – Liquid (Suspension)
xvi) Salicylic acid + Ethanol Solid – Liquid (Solution).

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