

(iii) (2) Caustic alkali : Proceed as for acid in eyes, but in eye (serious) wash with 1% boric acid solution in place of bicarbonate solution. Do not neglect. Consult a physician.

(3) Bromine in the eye (serious) : Wash thoroughly with water and then immediately with 1% sodium bicarbonate solⁿ.

(4) Glass in the eye : Remove loose glass very gently with forceps or by washing with water in an eye cup. Call for a doctor immediately.

(D) Fire : In flammable solvents should be handled carefully.

1. Carbon tetrachloride should not be used if sodium or potassium is present, as violent explosions may result.
2. The laboratory must be well ventilated immediately.
3. For burns of oil or organic solvents, do not use water as it will spread the fire, Mixture of sand and sodium bicarbonate is very effective. Use dry powder extinguisher available in laboratory.

(E) Poisons :

Solids or liquids :—

1. In the mouth but not swallowed. Spit out at once and wash repeatedly with water.
2. IF swallowed : call a doctor immediately. In the meanwhile give an antidote according to the nature of the poison.

(a) Acid (Including oxalic acid) :— Dilute by drinking lots of water, followed by lime water or milk of magnesia.



5. Sodium on the skin :- IF any small fragment of sodium can still be seen, remove it carefully with forceps. Wash thoroughly with water, then with 1% acetic acid and finally with water.

6. Organic substances on the skin :- Wash with cotton/cloth soaked in rectified spirit, then with soap, & warm water.

B Cuts :
IF the cut is only a minor one, allow it to bleed for a few seconds, make sure that the no glass particles remain. Apply a disinfectant (rectified spirit or Dettol) and bandage. For serious cuts, consult a doctor at once, meanwhile, wash with disinfectant and check bleeding by applying pressure. Immediately above the cut. Continuous pressure should not be given, for more than 5 minutes.

C Eye accidents :
In all cases, the patient must see a doctor. IF the accident appears serious, doctor should be called immediately while first aid is applied.
1. Acid in the eye :
i) IF the acid wash the eye repeatedly with 1% sodium is dilute : bicarbonate solⁿ in eye cup.

ii) IF the acid is concentrated : First wash the eye with large amount of water and then continue with the bicarbonate solution.

5. Use a clean spatula for removing the chemical reagent from the container. Observe the instructions mentioned on the container.
6. Remove only the required amount of chemical reagent from the container. ~~Observe~~ So that there is no need to return the excess to the container. This avoids the contamination of the chemical.
7. Keep the reagent and the laboratory balances clean. Clean any spilled chemical immediately.
8. While handling hazardous or toxic chemicals use hands gloves and mouth mask.

In case of an Accident :-

(A) Burns:

1. Burns caused by dry heat (e.g. by flames, hot object etc.)
For slight burns in which the skin is not burnt, apply Burnol. For more severe burns call for medical aid.
2. Acid on the skin: Wash immediately and thoroughly with liberal quantity of water, then with saturated sodium bicarbonate solution and finally with water.
3. Alkali on the skin: Wash immediately with a large volume of water, then with 1% acetic acid and finally with water.
4. Bromine on the skin (serious): Wash the affected part immediately with cloth / cotton sponge soaked in light petroleum and then rub glycerine well into the skin. After a little while, remove the superficial glycerin and apply Burnol.



Safety in laboratory

• Safety :-

Safety in the laboratory is essential at all times. You are responsible for the safety of other person as well as your own. Many chemicals encountered in chemistry laboratories are poisonous and must be carefully handled. Extra precaution is to be taken for concentrated acids, poisons such as potassium cyanides, halogenated solvents, benzene and mercury.

Many operations involving chemical reactions are potentially dangerous and recommended procedures must be followed and obeyed. All laboratory workers/persons should familiarize themselves with local safety requirements, which may include the compulsory wearing of lab coats and safety spectacles, and the positions of first aid equipments.

* Care in Handling of Chemicals :-

1. Handling of chemicals, especially hazardous chemicals must be done with due care. Everyday working in the laboratory must follow certain rules while handling the chemicals.
2. Select required grade of chemical for analytical work.
3. Select the smallest pack available.
4. Replace the top of every container immediately after removal of reagent; do not rely on someone to do this.

iv) Explosive: These products may explode by the action of heat, sources of ignition, shock or friction. The compounds are at often packaged wet to reduce the risk of explosion; they will become dangerous if allowed to dry. Some compounds form sensitive explosive salts on contact with metals.

v) Flammable: These compounds have low flash point, and react with water or damp air to give rise to flammable gases (e.g. hydrogen) from metal hydrides. Ignition sources include Bunsen burners, a hot metal surface, electric sparks etc. Fire fighting equipments should be readily available and frequently checked.

vi) Harmful: Irritant chemicals cause inflammation of the skin, mucous membranes, or discomfort of respiratory system. All laboratory chemicals should be regarded as harmful; some are specifically harmful by skin contact, inhalation or swallowing.

require heating. The mixture should be prepared in hard borosilicate glass beaker and all safety precautions must be taken while making the chromic acid mixture as it highly corrosive and hygroscopic. It should be stored in glass stoppered bottle.

4. Glass apparatus treated with chromic acid should be subjected to prolonged rinsing with de-ionized water, as glass tends to absorb chromic acid. The mixture should not be used ^{adsorb} for cleaning of containers used for optical measurements.

5. A very effective degreasing agent is obtained by dissolving 100 g of potassium hydroxide in 50 ml of water and after cooling, making solution upto one litre with industrial methylated spirit. Handle this with great care.

6. For general cleaning of glassware, synthetic detergent cleaning solution like SLS (sodium lauryl surface) or alkaline solution such as tri-sodium phosphate may be used but requires prolonged rinsing.

7. A mixture of concentrated sulphuric acid and fuming nitric acid may be used for cleaning of greases and dirty glasswares. All glassware should be rinsed with purified water before use.

After cleaning and rinsing with distilled water, small glass apparatus may be dried by leaving it in an electrically heated oven maintained at 100-120°C for about 1 hour. If there is an urgency, then drain the apparatus completely of water and then rinse with a little industrial spirit or acetone. The wet industrial spirit or acetone should be collected in a suitably labelled Winchester bottles for future recovery by distillation.



and re-use. Apparatus rinsed in this manner dries up quickly even at room temperature.

★ Labelling of Laboratory Reagents :

Proper handling of chemicals in the laboratory is important. Therefore every student should be familiar with the symbols appearing on labels of reagents used.

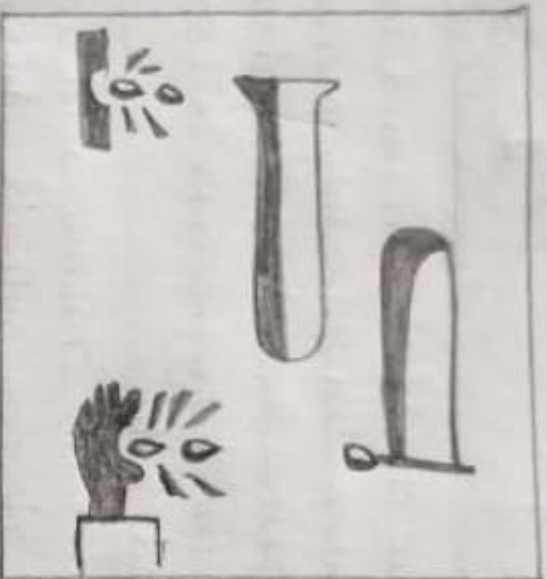
● Hazard Symbols :

legislation on packaging and labelling of dangerous substances defines hazardous chemicals under the following categories;

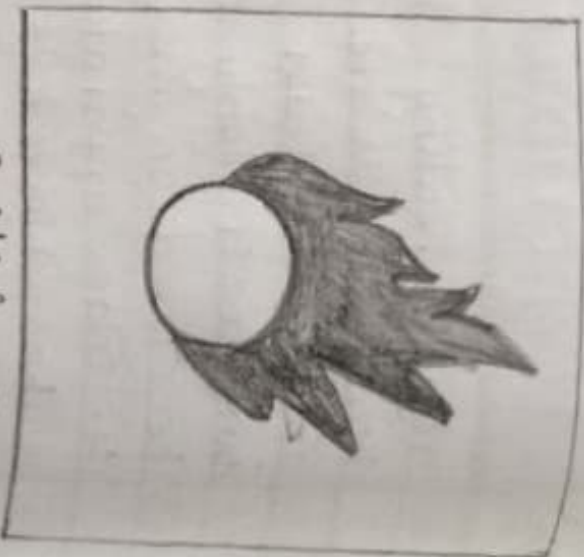
i) Corrosive : These products may destroy living tissue, eyes are particularly susceptible. Emergency showers should be available. If swallowed, plenty of water should be given after mouth rinsing.

ii) Toxic : These products can cause death or serious illness when small amount enter the body by ingestion, inhalation of vapour, fumes or dust, or by absorption through the skin; hygiene considerations should be rigorously observed.

iii) Oxidizing : These compounds may cause fire and will always assist combustion. They can produce heat on contact with organic matter and reducing agents.



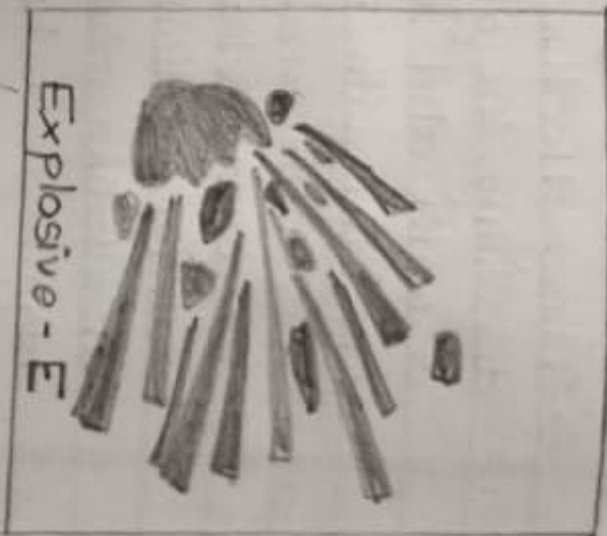
Corrosive-C



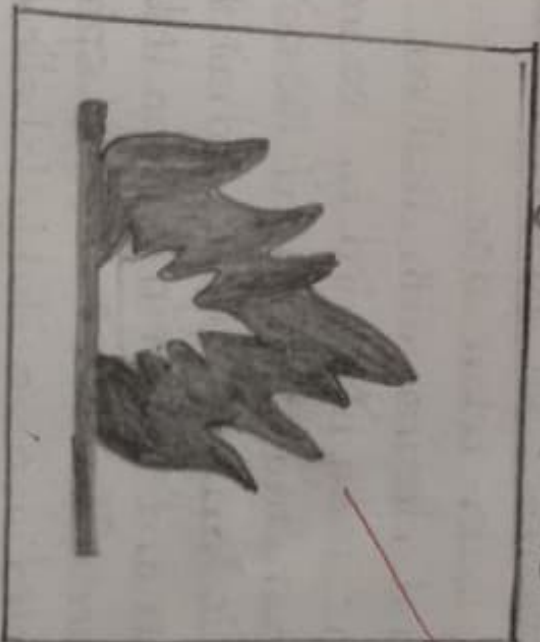
Oxidizing-O



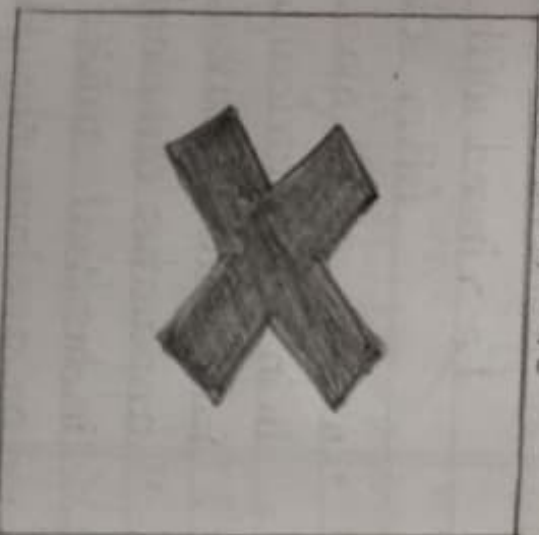
Toxic-T



Explosive-E



Highly Inflammable



Harmful Xn



Experiment No-1

PAGE NO.

1.

Introduction

Importance of the Subject :-

1. Pharmaceutical Organic chemistry is the chemistry of substances used as medicines.
2. Organic pharmaceutical chemistry is concerned with medicinal organic compounds, determination of their structure, synthesis, purification, isolation and their chemistry.

Objectives of the Subject :-

1. To provide the guidelines and procedure for systematic qualitative analysis of organic compounds. Identification of a compound plays an important role in discovery of new drug molecules or for already existing drug substances.
2. To understand the concept of purity of organic compounds to be used as medicines by various tests.
3. To understand the synthesis of organic compounds of industrial significance and also to develop skill of synthesis.

Grades and properties of chemicals :-

The reagents and chemicals which are used in the laboratory for chemical analysis are available in different grades as follows,

1. Technical or Commercial Grades: These are used when high purity is not required.
e.g. preparation of cleaning solⁿ.

2. Chemically pure (cp) Grades: These are more refined than technical grades. These chemicals are not suitable for analytical work or if, to be used, they must be tested.

3. L.R. Grade: These are laboratory grade reagents. They are normally 99% pure and can be used for the synthesis or procedures that do not require special specifications.

4. A.R. Grade: These are analytical reagent grades and have a purity above 99% and are used for procedures that are sensitive to impurities.

5. Pharmacopoeial grade chemicals: These chemicals confirm the tolerances set by pharmacopoeias (e.g. Indian pharmacopoeia and British pharmacopoeia).

* Cleaning and drying of glasswares :-

All glasswares to be used must be perfectly clean and free from grease to ensure correctness of results.

Following cleaning solutions can be used to clean the glassware

1. Use a mild detergent to wash the glassware.
2. Hot Nitric acid is very effective cleaning fluid for removing organic matters from glass.
3. Chromic acid mixture (a nearly saturated solution of powdered sodium dichromate 200g in 1000 ml of water) or potassium dichromate in concentrated sulphuric acid (1500 ml) allow to stand for several hours (preferably overnight), is a very effective cleaning fluid and it does not



Experiment No-3.

• Aim :- To determine boiling point of given sample.

• Requirements :-

• (i) Apparatus = Stand, thermometer, capillary tube, Burner, thiele's tube, fusion tube.

• (ii) Chemicals = Paraffin oil, sample etc.

Procedure :-

1] The liquid compound (0.5-1 ml) is taken in a fusion tube and capillary tube sealed at upper end is inserted in it.

2] The fusion tube is tied to a thermometer in such a way the liquid comes at the level of mercury.

3] The assembly is inserted in a Thiele's tube. The Thiele's tube is gradually heated at a uniform rate.

4] Record the temperature at which a continuous evolution of bubbles is seen from the lower end of the capillary.

• It is the boiling point of liquid.

The range of this constant should not exceed 5.0°C except for extremely high boiling compounds.

• Theory : (Boiling Point)

The boiling point of liquid (at a given pressure) is defined as 'the temperature at which its vapour pressure is equal to atmospheric pressure. Though a pure liquid has a sharp boiling point, the converse is not true. A sharp boiling point may sometimes be caused by a

after 2ml of distillation is collected, empty the graduated cylinder into a test tube, as it fills cover the test tube first fraction. (Component A)

- ④ Collect the next 10 ml distilled, again recording the temperature after every 1 ml of distilled. After the temperature been collected, empty the graduated cylinder in the test tube as it fills covers and label the rest tube sec. and fraction (Component B)
- ⑤ Determination of B.P range of 1st and 2nd fraction of the collected liquid. Identify the unknown by their B.P. using the possible boiling point of compound.

Theory :

Distillation is a process of separating the component substance from liquid mixture by the evaporation and condensation, Distillation. It may result in complete separation of liquid mixture. In industry chemistry, distillation is unit operation of practically, distillation is unit, but its phys universal importance, but it is physical separation process and not a chemical reaction.

Simple distillation is procedure by which two liquids with different boiling point can be separated.

Purified compound will boils of that turns into vapours that will forms be, ever a relative small temperature / range (20-30°C) by carefully watching the temperature. In the distillation flask, it is possible to affect a resondly good spseparation.

As distillation progresses, the concentration of lowest BP component will steadily decreases

- (b) **Caustic alkalis** : Dilute by drinking lots of water, followed by vinegar, lemon or orange juice, or a solution of lactic acid or citric acid. Milk may then be given but no emetics.
- (c) **Salts of mercury compounds** : - Give milk or egg white.
- (d) **Arsenic or Mercury compounds** : - Give an emetic immediately, e.g. one teaspoonful of mustard, or a one teaspoonful of salt or zinc-sulphate, in a cup of warm water.
- (e) **Gas** : - Remove the victim to the open air and loosen clothing at neck. To counteract chlorine or bromine fumes if inhaled in only small amounts, inhale ammonia vapour or gargle with sodium bicarbonate solution. Afterwards, the patient should suck eucalyptus oil soaked cotton swabs or drink warm dilute peppermint or cinnamon essence to soothe the throat and lungs. If breathing has stopped, apply artificial respiration, call medical aid immediately.

• Aim : To perform the recrystallization of given compound,
Recrystallize the given crude product

• Requirement : Test tube, test tube holder, funnel,
filter paper, sample.

• Procedure :

① Choice of solvent :

(i) for the judicious choice of solvent about 50 mg of the solid is taken in test tube and shaken with few drops of solvent.

(ii) The solvent is unsuitable if whole of the solid get into solution at room temperature.

(iii) If the solid dissolved upon heating and throws out maximum crystal on cooling, the solvent is suitable.

(iv) This process is repeated with solvent such as water, benzene, ether, alcohol, acetone etc. Till the most satisfactory one is started out.

② Preparation of solution :

A suitable quantity of solid substance is taken in a conical flask fitted with a reflex condenser. A small volume of the solvent selected in step (i) is also placed in the flask.

③ Filteration of HOT solution :

The hot solution above obtained is taken then filtered through a fluted filter paper placed in an ordinary glass funnel.

Eventually the temperature, within the apparatus will be to change a pure compound, is no longer being distilled, the temperature, will continue to increase until B.P of next lowest boiling compound, when the temperature again stabilized.

Another pure fraction of the distilled can be collected. This fraction of distilled will be primarily the compound that boils at the second lowest temperature. This process can be repeated until all fractions of original mixture, has been separated.

* Result :

- (i) Boiling point of 1st compound (A) observed, literature = 78°C .
- (ii) Boiling point of 2nd compound (B) observed, literature = 100°C .

* Reference : B.S. Furnis et al, Vogel's textbook of practical organic chemistry.



Experiment No-4

• Aim :- To determine boiling point of given sample.

• Requirements :-

(i) Apparatus: Stand, thermometer, capillary tube, Burner, thiele's tube.

(ii) Chemicals: Paraffin oil, Sample etc.

Procedure :

(i) Finely powdered compound is taken in a thin capillary tube (5-6 cm long and 1 mm width) sealed at one end. Capillary tube is used tied to a thermometer in such a way that the substance comes at the level of the mercury bulb of the thermometer refers fig.

(ii) The thermometer with the capillary tube is then immersed in a Thiel's tube. The oil in the Thiele's tube is then heated gradually.

(iii) The temperature range at which the compound just melts is noted. It is the melting point of compound.

✦ Theory :-

The melting point (M.P) and boiling point (B.P) of a compound are important characteristics that can be used in its identification. Also a depression in values is indicative of presence of impurities in a sample. Thus, melting point of solid boiling point of any liquid is determined.

• Procedure :-

Using Ice point and point boiling point Method:

- 1) Suspend thermometer in a slurry of ice & water. (use approximately 2:1 mix. of ice and water) in a 600ml beaker.
- 2) Stir the mixture thoroughly (If ice and water mixture is not stirred, the temperature on the bottom of beaker will be about 4°C) and wait for 5 min. Thermometer to stabilize, record the temperature reading.
- 3) Place about 75 ml of hot water in a 125ml conical flask and add about 3-4 porcelain pieces to ensure smooth boiling.
- 4) Carefully suspend the thermometer in the flask and heat the flask with bunsen burner. after water start boiling vigorously for about 5 min. record the temperature reading.
- 5) The information obtained above represents the error of thermometer in the region of 0°C and 100°C , the freezing and boiling points of water, respectively.
- 6) Correct your thermometer's readings for future determination.

• Result :-

- 1) The temperature in ice/water bath = 0°C
- 2) Thermometer error at ice/water temperature = 0°C
- 3) Temperature of boiling point (water) = 100°C
- 4) Thermometer error in boiling point of water = 0°C

- Reference : Dr. M. N. Deodhar, Mrs. J. R. Jagtap practical handbook of pharmaceutical organic chemistry, Nirali prakashan Page No- 31-32.



Caliberation of Thermometer

• Aim: To caliberate given thermometer.

• Requirements :-

(i) Apparatus: Beaker, conical flask, thermometer, Thiele's tube, capillary tube etc.

(ii) Chemicals: Ice, water, Paraffin oil etc.

• Theory :-

• Caliberation of thermometer is needed to check whether the thermometer is showing correct temperature reading or not.

• Melting points are important characteristic property of an organic compound that can aid in its identification and purity assesment. While thousands of different compounds can have coincident melting points, a melting point is useful within the context of an organic synthesis where it serves more as confirmation of identity rather than as proof of identity.

• Since the presence of impurities depresses the melting point of a compound, and broadens its range, the melting point of an organic product is a measure of purity.

• The melting points must be reported as ranges. The first number to be recorded is that temperature at which the first droplet of liquid is visible and the last temperature recorded is the temperature at which the entire sample has to liquified.

A) To check the purity of compounds: Pure substances have sharp and definite melting point/boiling point. The impure compound melts slowly. A pure liquid boils at a definite temperature. A solid substance dissolved in a liquid increases the boiling point of liquid. On the other hand, miscible liquid mixed with a liquid decreases the boiling point of the liquid.

B) To identify the organic compounds: If the M.P or B.P of the given organic compound is determined with an accuracy of $\pm 2^\circ\text{C}$ then possible compound can easily be guessed.

- Melting point :- Melting point of compound is the range of temperature at which solid phase changes to liquid. The melting point is considerably influenced by presence of other substances and is therefore an important criteria of purity. Some pure compound may not have a sharp melting point (melt entirely in a range of about 1°C) especially if they undergo decomposition such as turning dark at a near the temperature used for the melting point determination. The correct melting point can be determined by using a calibrated thermometer. The calibration is done by determining the melting or boiling point of a new selected organic compounds, such as benzoic acid (M.P. 122°C) salicylic acid (M.P. 159°C), acetone (B.P. 56°C) and water (B.P. 100°C) as reference compound.

• Note:—

It is advisable to determine the approximate melting point first. Repeat the procedure to determine the correct melting point. In the second determination, near the melting point of the compound, the rate of heating should be carefully controlled. Slow heating is recommended when the temperature of bath is within 15°C of the expected melting point because this will help in noting down the correct temperature at which the collapse of the crystals commences and the temperature at which the sample becomes liquid. The difference between these two temperatures is about 0.5 to 1°C for the pure substance so we should heat the bath slowly. If the melting point range encompasses more than 2.0°C , the compound should be recrystallized.

• Result :— The melting point of given sample is found to be $121-122^{\circ}\text{C}$.

• Reference : Dr. M. N. Deodhar, Mrs J. R. Jagtap, Practical handbook of pharmaceutical organic chemistry [Nirali prakashan] PgNo-41-42.

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3. Test for Nitrogen & sulfur:
 Take 1ml of sodium fusion extract + 5-6 drops of dil. HCl + 2-3 drops of ferric chloride solution

No blood red colour precipitation is formed

Nitrogen and sulfur may be absent

4. Test for Halogen:

Sodium fusion extract + dil. Nitric acid (HNO_3) + 1-2 drops of silver nitrate solⁿ.

No particular observation

Halogen may be absent

* Conclusion :- The given organic compound is found to contain $\boxed{\text{C, H, O}}$ elements and compound is $\boxed{\text{Acidic}}$ therefore group $\boxed{\text{Group I (A)}}$ is present.

(C) Determination of physical constant :-

The melting point / boiling point of the given organic compound was found to be $\boxed{121^\circ - 122^\circ \text{C}}$

insoluble. Then add few drop of Neutral $KMnO_4$

6) Detection of Aliphatic / Aromatic Nature

Test	Observation	Inference
@ Heating of copper gauge: Take a small coil foil and heat it in flame. Place 0.5 gm if solid or 0.1 ml if liquid on it & heat on flame.	Sooty flame	Aromatic Compound Present

* Conclusion :- On the basis of test performed above the given organic compound is,

1) Acid

2) Unsaturated

3) Aromatic

B. Determination of element {Lassaigner's Test}

* Procedure :- Take a small piece of clean dry sodium (0.5g) in a clean dry sodium fusion tube. Heat the tube slowly. Remove it from the flame and add about 0.02-0.03 gm solid compound or 2-3 drops if liquid directly to the molten sodium first heat on a low flame and then on a stronger flame until it becomes red hot. Keep it in the red hot condition for 1-2 min. (until the compound has completely reacted) and the plunge into 10-15 ml of distilled water kept in a porcelain dish. Carry out the same procedure for 2-3 separate ignition tubes. Boil the contents of porcelain dish for 2-3 minutes and filter. Use the filtrate. (Known as Lassaigne's extract)

or sodium fusion extract) for testing the extra elements. In this case, the filtrate should be water clear. If it is dark coloured, the whole fusion should be repeated.

Sr. No.	Test	Observation	Inference
1.	<p><u>Test for Nitrogen:</u> Take 2-3 ml of sodium fusion extract + 0.2-0.3 gm of powdered ferrous sulfate crystals, greenish precipitate is formed; in case sulfur is present, a black precipitate of ferrous sulphide is obtained, IF no greenish precipitate is seen then add few drops of NaOH solution, boil, add dil. H_2SO_4 and observe</p>	<p>Blue precipitate not form</p>	<p>Nitrogen may be absent</p>
2.	<p><u>Test for Sulfur:</u> (a) Take about 2 ml of sodium fusion extract + 2 ml acetic acid + few drops of lead acetate solution (b) Take 2-3 ml of sodium fusion extract + 2-3 drops of freshly prepared solⁿ of sodium nitroprusside</p>	<p>Black precipitate not form no red ppt</p>	<p>Sulphur may be absent Sulphur may be absent</p>

2. Colour	Colourless Solid	Carbohydrate, acids, amides etc. may be present
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3. Effect of heat:

A small quantity of given sample (solid) is heated in clean dry test tube

Sharp Melting

Pure organic compound may be present

4. ~~D~~ Type of Determination :-

Sample + cold water (shake) : Water insoluble/Immiscible

15 Type determination for water insoluble samples :-

Test	Observation	Inference.
(a) 10 mg sample + 10 drops of 10% NaHCO_3 sol ⁿ .	Soluble with effervescence of CO_2 & reprecipitated by con. HCl.	Acid may be present.

5. Determination of saturation / Unsaturation:

Test	Observation	Inference
(a) Action of potassium Permanganate (Baylors test): Dissolve 0.2 gm of sample if solid or 0.2 ml if liquid in 2ml of H_2O or in acetone if water	pink colour of KMnO_4 disappears	Unsaturated compound may be present

* Result :-

(i) The melting point of the crude sample was found to be - $234-235^{\circ}\text{C}$.

(ii) The melting point of the pure compound was found to be - $231-234^{\circ}\text{C}$.

* Reference :

Arun Bahl and B.S. Bahl, A textbook of organic chemistry. {Nirali publication}
Page NO - 3.1 - 3.4.

- Aim :- To identify the given organic compound by {quantitative analysis}.

- Requirements :-

- (i) Apparatus :- Test tube, beaker, stirrer, measuring cylinder, porcelain dish, water bath, Thiel's tube, thermometer, wire gauze etc.
- (ii) Chemicals :- All general and table reagents.

- Procedure :-

Identification of an organic compound involves the following step:-

A) Preliminary test :-

- Type / Nature determination
- Detection of saturation / Unsaturation
- Detection of aliphatic / aromatic

B) Detection of Elements {Elemental analysis}

C) Determination of physical constant {M.P / B.P}

D) Detection of functional groups

E) Derivative preparation.

A) Preliminary Test :-

Sr.No	Test	observation	Inference
1.	State	Solid	Compound having high molecular weight e.g. phenol, carbohydrate, may aromatic acid present

Similarly, the compound prepared in laboratory are generally mixed with products which may also have been formed during the reaction.

In order to study its properties and to determine its structure, a given substance must be obtained in state of purity.

The method employed for purification depends upon the nature of the organic compound, and the impurities present in it.

• The processes commonly used for the separation of organic compounds are:—

(i) Crystallisation.

(ii) Sublimation

(iii) Distillation

(iv) Fractional distillation

(v) Distillation under reduced pressure.

(vi) Steam distillation

(vii) Extraction with solvent.

(viii) Chromatography

→ Crystallization:—

This is most commonly employed for the purification of solid organic compounds.

The impure solid is dissolved in the minimum volume of suitable solvent.

The soluble impurities, pass into solution while the insoluble ones are left behind.

The quantity of solution is large, it takes longer and longer the crystal way from in the funnel during the filtration.

④ Crystallization:

- The hot filtrate is taken then allowed to cool undisturbed in a beaker.
- The pure solid substance separates as crystals
- When a rich crop of crystals has been obtained crystallization is complete.
- Sometimes crystallization does not occur even after cooling the filtrate for a sufficiently long time.

⑤ Separation and Drying of Crystals:

- The crystals are separated from the liquid by filtration. The filtration is generally effected under reduced pressure using a Bucher or Hirsch funnel.
- The crystals are then dried by pressing between pads or filter paper, in an oven or in vacuum desiccator.

* Theory:-

Organic compounds derived from natural sources are seldom pure.

They are often mixed with other substances which also occur with them.



constant boiling mixture (azeotropic) of two or more liquids. Distillation is recommended if the boiling point range indicates extensive contamination by a wide boiling point range, if compound is heterogenous, or if it appears to be discoloured. Distillation at reduced pressure may be necessary for compounds that show evidence of decomposition in the boiling point test.

● Result:

The boiling point of given sample is found to be ~~180°C~~ 150-152°C.

● Reference:-

Dr. M.N. Deodhar, Mrs. J.R. Jagtap practical handbook of pharmaceutical organic chemistry. {Nirali publication} Page No-42-43



Experiment No-6

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- Aim : To demonstrate the simple distillation method.

* Requirement :

- ① Apparatus - Condenser, heating mantle, conical flask, R.B.F., etc.
- ② Chemicals - Alcohol, water etc.

• Procedure :-

- ① Place 20 ml of an unknown liquid mixture (10 ml A + 10 ml B) that is to be purified by simple distillation assemble the assigned distillation apparatus.
- ② Transfer the unknown liquid to 1000 ml round bottom flask, add 2-3 porcelain piece and porcelain dish and proceed to distill the liquid into a 10 ml graduated cylinder (receiver), check the position of thermometer must be below the arm of the distillation head and make sure that the bottom of the distillation pot touching the heating surface of the heating mantle.
- ③ Securely attached a piece of condenser tubing to each condenser outlets. Surely connect the other end of 'water in' tubing to water jet in the sink place the other end of 'water out' tubing in the sink plug in the heating mantle.
- ④ Adjust the heating mantle to maintain a distillation rate of one drop per second. As the lower boiling component is distilled, the BP of the mixture in the distillation flask will increase. Record the temperature after first drop is collected and again

Sr. No.	Test	Observation	Inference.
①	<u>Test for Nitrogen :</u> Take 2-3 ml of sodium fusion extract + 0.2-0.3 gm of powdered ferrous sulfate crystals, greenish ppt is formed in case sulfur present, a black ppt of ferrous sulphide is obtained. if no greenish ppt is seen then add few drops of NaOH sol ⁿ , boil, add dil. H ₂ SO ₄ and observe	Blue precipitate form	Nitrogen may be present
②	<u>Test for sulfur.</u>		
a)	Take about 2ml sodium fusion extract + 2ml acetic acid + few drops of lead acetate sol ⁿ	No black ppt form	Sulphur may be absent
③	<u>Test for Halogen</u>		
"	IF nitrogen and sulphur absent, acidify then extract directly by HNO ₃ + 2-1 drops of silver nitrate sol ⁿ .	White ppt sol ⁿ in ammonium hydroxide sol ⁿ .	Halogen may be absent

* Conclusion :- The given organic compound is found to contain C, H, O and N and compound is Neutral therefore Group II C is present.

(C) Detection of Physical Constant :-

The melting point / Boiling point of given organic compound was found to be $129-130^{\circ}C$.

(D) Determination of functional group :-

Group II :- Compound containing C, H, O and N .

Test for group II C Neutral compound.

Amide :- $R-CO-NH_2$, Imide :- $R-\begin{matrix} \diagup C=O \\ \diagdown C \end{matrix}-N-H$

Nitriles :- $R-C \equiv N$ and anilides $\begin{matrix} O \\ || \\ ph-NH(OR) \end{matrix}$

Sr No.	test	Observation	Inference.
(a)	0.01 gm sample + 10 drops of NaOH sol ⁿ and boil.	Evolution of ammonia which turn moist turmeric paper red	An amide, imide or nitrile group present
(b)	Biuret test :- Heat 0.2 gm sample in a dry test tube till the melted compound solidifies and evolution of NH_3 causes col dissolve. The solid in 2-3 ml dil. NaOH & add 1-2 ml dil. $CuSO_4$ sol ⁿ dropwise	Formation of pink colour	Urea is confirmed.

Molecular weight = 60.06 g/mol .

Nature = Aliphatic

Functional group = NH_2

Melting point = ~~130~~ 132°C .

Derivative = Urea nitrate

* Uses :-

- 1] Use as fertilizer in agricultural field
- 2] Use as food supplement.

* Reference :-

Dr. M.N. Deodhar and Mrs. J.R. Jagtap practical handbook of pharmaceutical organic chemistry of {Nirali prakashan}, Second edition of year 2016

Experiment No-10

Aim :- To identify the given organic compound by qualitative analysis

Requirement :-

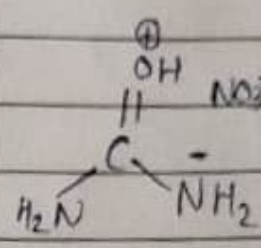
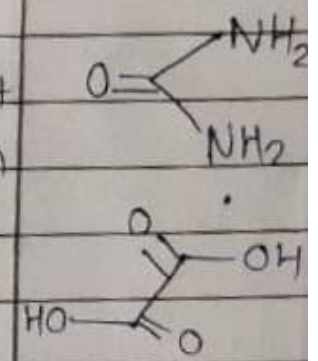
(i) Apparatus :- Test tube, beaker, stirrer, water bath, Thiel's tube, porcelain dish, thermometer, wire gauze etc;

(ii) Chemicals :- All general and table reagent.

A Preliminary Test :-

Sr. No.	Test	Observation	Inference
1	State	Solid	Compound having high molecular weight. e.g. aromatic acids, phenols, carbohydrates.
2	Colour	colourless solid	Carbohydrate acids, amides etc; may be present
3	Effect of Heat Small qty of given sample is heated in dry test tube	Sharp melting point	pure organic compound may be present

Physical Constant of Neutral compound (II C) and their derivatives.

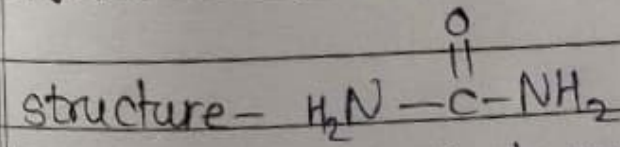
Name of Structure M.P/B.P.	Derivative preparation or confirmatory test	Structured derivative
<p>Urea</p> $\text{NH}_2 - \overset{\text{O}}{\parallel} \text{C} - \text{NH}_2$ <p>M.P = 132°C</p>	<p>Urea nitrate = 0.5 gm urea 4 ml water & add 1 ml con. HNO₃, heat with stirring & cool. filter the urea nitrate that has separated (M.P. 163°C)</p>	 <p>Urea Nitrate</p>
<p>"</p>	<p>urea oxalate :- 0.5 gm urea + 4 ml water + solⁿ of oxalic acid (0.5 gm in 8 ml water) mix and filter the solid. (M.P = 171°C)</p>	 <p>Urea oxalate.</p>

* Result :-

The given organic compound was found

to be :-

Name - Urea



IUPAC Name - Carbamide carbonyl diamide

Molecular formula - CH₄N₂O

Experiment No-9

PAGE NO.

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* Aim :- To identify the given organic compound by Quantitative analysis.

* Requirements :

(i) Chemicals : All general and table reagents.

(ii) Apparatus : Test tube, beaker, porcelain dish, wire gauze, thermometer, water bath, Thiel's tube, measuring cylinder, etc;

• Procedure :- Identification of an organic compound involves the following steps.

A) Preliminary test :-

- Type / Nature determination.
- Determination of saturation / unsaturation.
- Detection of aliphatic / aromatic.

B) Detection of elements {Elemental Analysis}

C) Determination of physical constant :- [M.P/B.P]

D) Determination of functional groups.

E) Derivative preparation.

* Procedure A) Preliminary Test :-

Sr.No.	Test	Observation	Inference
1.	State	Solid	Compound having high molecular weight e.g. aromatic acid, phenol, carbohydrate may present
2.	Colour	Colourless solid	Carbohydrates, acids, amides may be Present

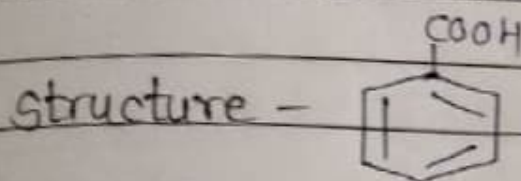
Benzoic acid
M.P. 121-122°C

cold water filter the
solid & dry (M.P. 140°C)

m-nitrobenzoic acid.

* Result :-

The given organic compound was found to be
Name - Benzoic acid



IUPAC Name - Benzene carboxylic acid

Molecular formula - $C_7H_6O_2$

Molecular Weight - 122 gm/mol

Nature - Aromatic acid

Functional group - COOH

Melting point - ~~121-122~~ 121-122°C

Name of derivative and its melting point :-

m-nitrobenzoic acid and its melting point is 140°C.

Uses :-

1] Use in perfume, dye.

2] Used as preservative agent.

Reference :- Dr. M. N. Deodhar and Mrs. J. R. Jagtap
practical handbook of pharmaceutical
organic chemistry of {Nivali prakashan}
second edition of year 2016.

6. Detection of Aliphatic / Aromatic

(a) Heating on a copper gauze :-

Take a small copper foil and heat it in the flame. Place 0.5 gm if solid or 0.1 ml if liquid on it and heat in the flame

Non sooty flame

Aliphatic compound may be present

* Conclusion :- On the basis of tests performed above, the given organic compound is

1. Neutral
2. Saturated
3. Aliphatic

3. Effect of heat :-

A small quantity of the given sample (solid) is heated in a clean, dry test tube

Sharp
Melting

Pure Organic
Compound may
be present

4. Type of determination :-

Sample + Cold water (shake) :- ① Water soluble / Miscible.

@ Type determination for water soluble sample :-

Test	Observation	Inference
Few drops of aq. sol ⁿ or of liquid on wet litmus paper	No change on litmus	Neutral Sample Present

5. Detection of Saturation / Unsaturation :-

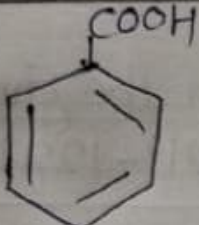
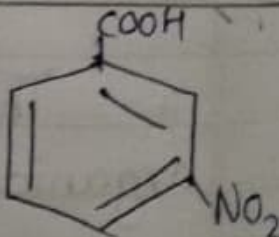
Site	Test	Observation	Inference.
@	Action of $KMnO_4$ (Bayer's test) :- Dissolve 0.2 gm of sample if solid or 0.2 ml if liquid in 2 ml water or in acetone, if water insoluble. Then add few drops of neutral $KMnO_4$	No decolourisation	Saturated Compound present

D) Determination of functional group:

Tests for Group I(A) - Carboxylic acids (R-COOH)

Sr. No.	Test	Observation	Observation	Inference
(a)	<p>NaHCO₃ test: Take 0.2-0.3 gm of compound + 2ml saturated solution of NaHCO₃</p>		Strong evolution of CO ₂ . Re-precipitated with con. HCl.	- COOH group is may be present
(b)	<p>Esterification Test: 0.02 gm sample in dry test tube + 10 drops of ethyl alcohol + 2 drops of con. H₂SO₄</p>		Fragrant smell (sweet smell of ester)	- COOH group is may be present
(c)	<p>Neutral test solution:- 0.5 gm of sample + 2-3 ml water + phenolphthalein solⁿ + Ammonia solution till just alkaline Now boil to remove excess of NH₃ (cool) + add few drops of aq. FeCl₃ solⁿ Neutral solⁿ from test (d) + CaCl₂ solⁿ</p>		Buff coloured ppt No white ppt	Benzoic acid or phthalic acid may be present Cinnamic or citric acid may be absent

E) Physical constant of carboxylic acid & their derivative:-

Sr. No.	Name & structure	Derivative prep or confirmatory test	Structure of derivative.
		<p>m-nitrobenzoic acid 0.5 gm acid + 0.5 ml con. HNO₃ + 0.5 ml con. H₂SO₄ warm and pour in ice</p>	

• Aim :- To identify the given organic compound by qualitative analysis.

• Requirement :- Test tube, beaker, Measuring cylinder, (Apparatus) :- Thermometer etc;

• Chemicals :- All general and table reagent

A Preliminary Test :-

Test	Observation	Inference
1) State	Solid	Comp. having high M.W e.g. aromatic acids, carbohydrate phenols, present
2) Colour	Colourless solid	Carbohydrate, acids, phenols may present
3) Effect of Heat	Sharp melting	Pure organic compound may Present

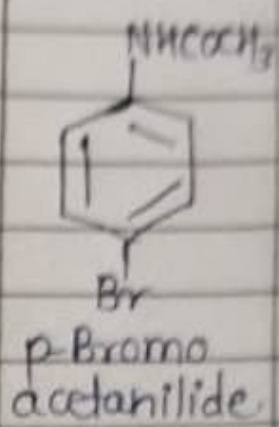
4) Type of determination

Sample + cold water → Water Soluble

I Types of determination for water soluble.

Acetanilide
 $C_6H_5NHCOCH_3$
 MP: $114^\circ C$

p-Bromoacetanilide
 Sample + glacial acetic acid, CCl_4 or $CHCl_3$. Add bromine in same solvent slowly from a burette until the colour of bromine persists on shaking. Allow it to stand for 15 min. Pour into ice cold water, if glacial acetic acid has been used, filter the product, wash with water & recrystallize from alcohol. evaporate the solvent, if CCl_4 or $CHCl_3$ has been used, wash the residue with water, recrystallize from alcohol.
 (M.P. $167^\circ C$)



* Result :- The given organic compound is found to be;

- Name :- Acetanilide
- Structure :- $C_6H_5NHCOCH_3$
- IUPAC Name :- N-phenylacetamide
- Molecular formula :- C_8H_9NO
- Molecular weight :- 135.17 g/mol

• Nature :- Neutral, Unsaturated, Aromatic

• Functional group :- Amide

• Melting point :- 115°C

• Derivative :- p-bromoacetanilide.

• Uses = Acetanilide is used as inhibition of H_2O , Decomposition and used to stabilize cellulose ester varnishes and It is also a fever reducing drug.

→ * Reference :- Dr. M. N. Deodhar and Mrs. J. R. Jagtap practical handbook of pharmaceutical organic chemistry of Nirali prakashan second edition of year 2016.

(a) Azo-dye test :-
0.01 gm sample + 10 drops of conc. HCl, boil and cool in ice water. Add few drops of NaNO_2 solution. Then pour few drops of this mix. into 5 drops of ice cold soln of β -naphthol in NaOH.

Orange dye stuff

Anilide group present

H₃

(b) Tafel's test :-
0.2 gm sample + 3 ml conc. HCl + 0.3 gm of potassium dichromate

Red or violet colour change to green

Anilide is confirmed.

(i) Hydrolysis test :-
0.3 gm of sample + 2-3 ml 70% H_2SO_4 , reflux for 10 min. Cool and dilute with water.

White ppt

anilide of aromatic acid present.

E] Physical Constants of Neutral compounds group (IIC) and their derivatives :-

Sr. No.	Name and structure (M.P./B.P)	Derivative preparation or confirmatory test	Structure of derivative
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Test	Observation	Inference
1) Test for Nitrogen	Blue ppt	Nitrogen may be present
2) Test for sulphur	Black PPT formed	Sulphur may be present

* Conclusion :- The given organic compound is found to obtain C, H, O, N, S element and compound in is Neutral therefore Group III B is present.

c) Determination of physical constant :-

" The melting point of given organic compound was found to be 178°C .

d) Determination of functional group

* Group III B - Compounds containing C, H, O, N, S

B) = Neutral

• Test for group III B Neutral Compound :-

Test	Observation	Inference
(a) lead acetate Test 0.1 gm sample + 1 ml lead acetate	Yellow ppt form	Thio (SH) group present
(b) Reac ⁿ with FeCl ₃ Melt 0.2 gm sample in test tube, cool & dissolve in 2 ml water. Add a few	Blood red colouration	Thiourea is confirmed

B Detection of Elements (Lassaigne's Test):

• Procedure :-

Take a small piece of clean dry sodium (0.5 gm) in a clean, dry sodium fusion tube. Heat the tube slowly. Remove it from the flame and add about 0.02 - 0.03 gm solid compound or 2-3 drops if liquid directly to the molten sodium. First heat on a low flame and then on a stronger flame until it becomes red hot. Keep it in the red hot red^n for 1-2 minutes (until the compound has completely reacted) and then plunge into 10-15 ml of distilled water kept in a porcelain dish. Carry out the same procedure for 2-3 separate ignition tubes. Boil the contents of porcelain dish for 2-3 min. & then filter. Use the filtrate for testing extra elements. Filtrate should be water clear.

Sr. No.	Test	Observation	Inference.
1.	<p><u>Test for Nitrogen:-</u> Take 2-3 ml of sodium fusion extract + 0.2-0.3 gm of powdered ferrous sulfate crystals, greenish precipitate is formed; if no greenish precipitate is seen then add few drops of NaOH solⁿ, boil, add dil. H_2SO_4 and observe</p>	<p>Blue precipitate or Blue colour is formed</p>	<p>Nitrogen may be present</p>

2. Test for Sulphur :-
Take about 2ml of sodium fusion extract + 2ml acetic acid + few drops of lead acetate solution

Black ppt does not form

Sulphur is may be absent.

3. Test for Halogen.
Acidify 1ml of sodium fusion extract with dil. HNO_3 and boil for 2-3 minutes, cool and dilute with equal volume of water + 0.5ml of silver nitrate solution.

No white ppt

Chlorine is absent

No pale yellow ppt

Bromine is absent.

Yellow ppt insoluble in NH_3OH

Iodine is absent

Halogen may be absent

* Conclusion :-

The given organic compound is found to contain C, H, O and N elements and compound is Neutral therefore group II-C is present.

C) Determination of physical constant :-

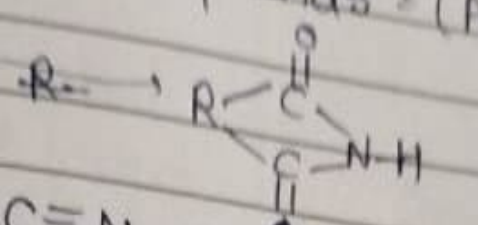
The melting point of the given compound was found to be 115°C .

D) Determination of functional group :

Group II-C = Compound containing C, H, O and N.
Compound is Neutral.

• Tests for Neutral compounds = (Amide = $R-CO-NH_2$)

Imide =



Nitriles = $RC \equiv N$ and anilides = $Ph-NHCO R$

Nitro = $-NO_2$,

Sr. No.	Test	Observation	Inference
a)	0.01 gm sample + 10 drops of NaOH solution and boil	Evolution of ammonia which turns moist turmeric paper red.	An amide, imide or nitrile group may be present
b)	Hydroxamic acid test 0.2 gm sample + 2ml hydroxylamine hydrochloride (2M in ethanol) + Boil on a water bath for 5min. Cool and add few drops of K_2FeCl_4 .	No Bluish red colour.	Aliphatic amide is not confirmed
c)	Nitrous acid test:- 0.2 gm sample + 2ml dilute HCl + 2ml $NaNO_2$ solution If no effervescence observed the boil off N_2 from the above test	white ppt	Amide of aromatic acid is confirmed

4) Type of Determination :-

Sample + Cold water \Rightarrow Water Insoluble / Immiscible
(shake)

ii) Type determination for water insoluble samples:-

a) 10 mg sample + 10 drops of 10% NaHCO_3 solution	Soluble with effervescence of CO_2 & reprecipitated by conc. HCl	Acid is absent
b) 10 mg sample + 10 drops of 10% NaOH, shake	completely soluble & reprecipitated with conc. HCl.	Phenol is absent
c) 10 mg sample + 10 drops of 1:1 HCl, shake	Completely soluble & reprecipitated with 10% NaOH	Base is absent
d) Here, all above tests are negative		Neutral sample may be present

5) Detection of saturation / Unsaturation =

a) Action of KMnO_4 (Bayer's test) Dissolve 0.2 gm of sample if solid or 0.2 ml if liquid in 2ml water or acetone, if water insoluble add few drops of neutral KMnO_4	Pink colour of KMnO_4 disappear	Unsaturation compound may be present
--	--	--------------------------------------

④ Action of bromine water :

Treat 0.1 gm of sample if solid or 0.1 ml if liquid with 2ml water in test tube. Add 2ml of bromine water. Shake well

Brown colour of bromine disappears

Unsaturated compound present

⑤ Action of bromine in chloroform :-

Dissolve 0.2 gm (solid) or 0.2 ml (liquid) with 2ml of chloroform. Treat it with bromine in chloroform. Shake and warm

Brown colour of bromine disappears

Unsaturated compound is present

⑥ Detection of Aliphatic / Aromatic Nature :-

① Heating on a copper gauze

Take a small copper foil and heat it in the flame. Place 0.5 gm if solid or 0.1 ml if liquid on it and heat in the flame.

Sooty flame

Aromatic compound may be present

* Conclusion : On basis of tests performed above, the given organic compound is,

1) Neutral

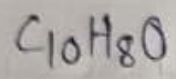
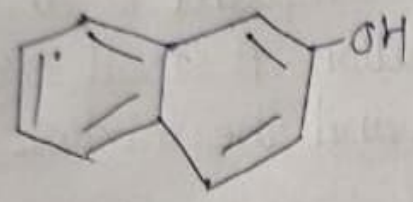
2) Unsaturated

3) Aromatic

* Calculations:

(i) Theoretical Yield :-

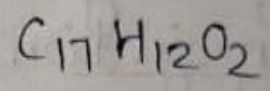
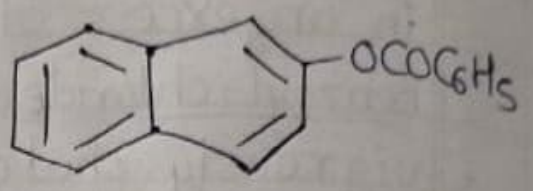
1 mole of β -Naphthol \equiv 1 mole of β -Naphthyl benzoate



$$12 \times 10 + 1 \times 8 + 16 \times 1$$

$$120 + 8 + 16$$

$$144$$



$$12 \times 17 + 1 \times 12 + 16 \times 2$$

$$204 + 12 + 32$$

$$248$$

$$5 \text{ gm} \equiv \frac{5 \times 248}{144} = 8.61 \text{ gm}$$

\therefore Theoretical yield = 8.61 gm.

(ii) Practical Yield = 8 gm

$$(iii) \text{ Percent yield} = \% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{8}{8.61} \times 100$$

$$= 92.9\%$$

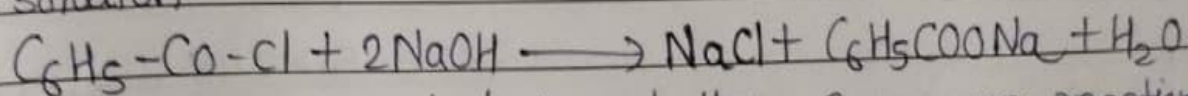


known as benzoylation. The reagent used is benzoyl chloride in presence of NaOH.

(iv) In the Schotten-Baumann reaction method of benzoylation, the hydroxyl or amino compound is dissolved in an excess of 10% NaOH solution. A small excess of benzoyl chloride is then added and the mixture is vigorously shaken.

(v) Benzoylation proceeds smoothly under these conditions and the solid benzoyl compound being insoluble in water separates out.

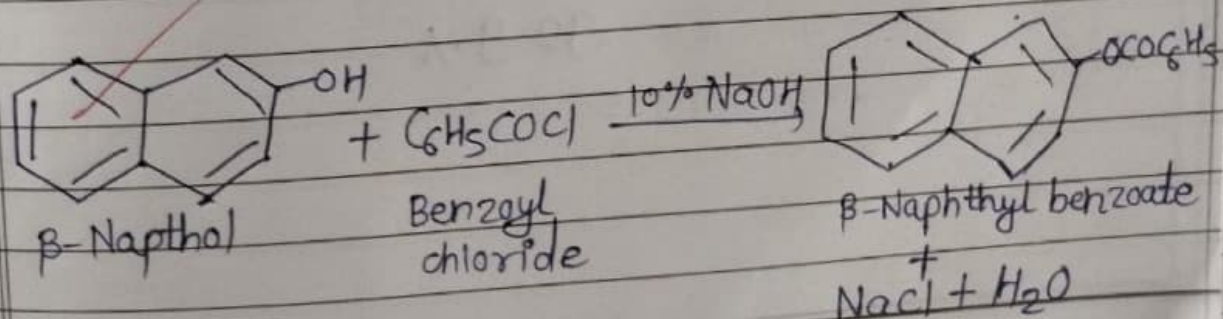
(vi) The NaOH then hydrolyzes the excess of benzoyl chloride giving sodium chloride and benzoate which remains in solution.



(vii) Phenol when subjected to Schotten-Baumann reactions first dissolves in NaOH to give sodium phenoxide which then undergoes benzoylation to give phenyl benzoate.

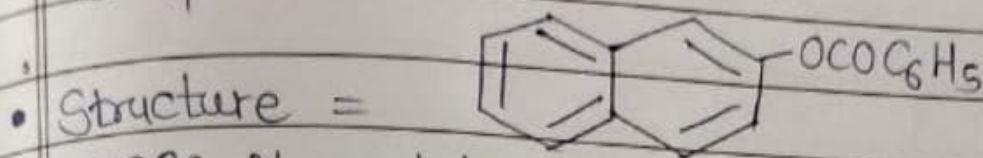
(viii) Benzoylation is a nucleophilic substitution reaction where the phenoxide nucleophile attacks the carbonyl carbon of benzoyl chloride to get substituted by removal of the chloro group.

Reaction:-



Result:

- Name of the compound = β -Naphthyl benzoate



- IUPAC Nomenclature = β -Naphthyl benzoate.

- State, colour and odour = colourless solid with pungent smell.

- Molecular formula = $C_{17}H_{12}O_2$

- Molecular weight = 248

- Theoretical yield = 8.61 gm

- Practical yield = 8 gm

$$\% \text{ yield} = 92.9\%$$

- Melting point = (i) Crude = 104°C

$$(ii) \text{ Recrystallized} = 108^\circ\text{C}$$

- M.P. (Literature) = $107-110^\circ\text{C}$.

- Uses = Hardening agent for paraffin, antiseptic (intestinal).

Reference:

Dr. M. N. Deodhar and Mrs. Jagtap
practical handbook of pharmaceutical
organic chemistry of Nirali Publication.
Page No- 14.1 - 14.3

Experiment No-15

PAGE NO

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- Aim :- Synthesis of β -Naphthyl benzoate from β -Naphthol

- Requirements :-

(i) Apparatus = Iodine flask, conical flask, beaker, stirrer, funnel etc.

(ii) Chemicals = β -Naphthol (5 gm), benzoyl chloride (4 ml), NaOH (5%) solution (25 ml) etc.

- Procedure \Rightarrow

① Dissolve 5 gm (0.035 mol) of β -naphthol in 25 ml of 5% sodium hydroxide solution kept in a 250 ml iodine flask.

② To it add 4 ml (4.9 gm, 0.035 mol) of benzoyl chloride and shake vigorously off after closing the mouth of the flask by cork for more than 15 minutes or until the smell of benzoyl chloride disappears.

③ Filter the separated solid at temp the pump, wash with cold water and dry.

④ Recrystallize from rectified spirit and report M.P and % yield.

Theory and Mechanism :-

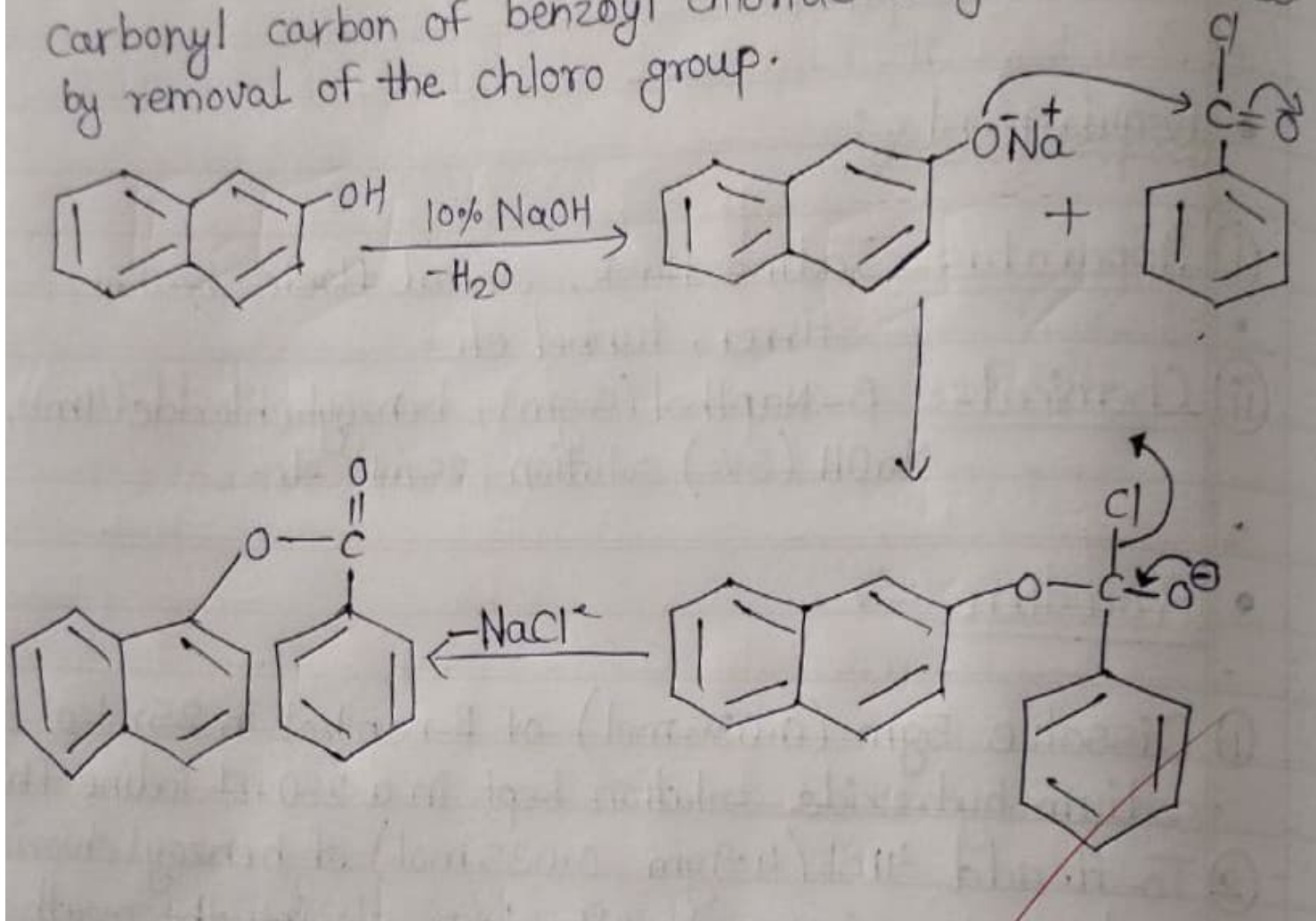
(i) β -Naphthyl benzoate is prepared by Schotten-Baumann reaction.

(ii) Benzoylation in the presence of NaOH is known as Schotten-Baumann reaction.

(iii) The introduction of benzoyl group (C_6H_5CO-) in place of the active hydrogen of $-OH$, $-NH_2$ or $-NH$ group is

Mechanism:

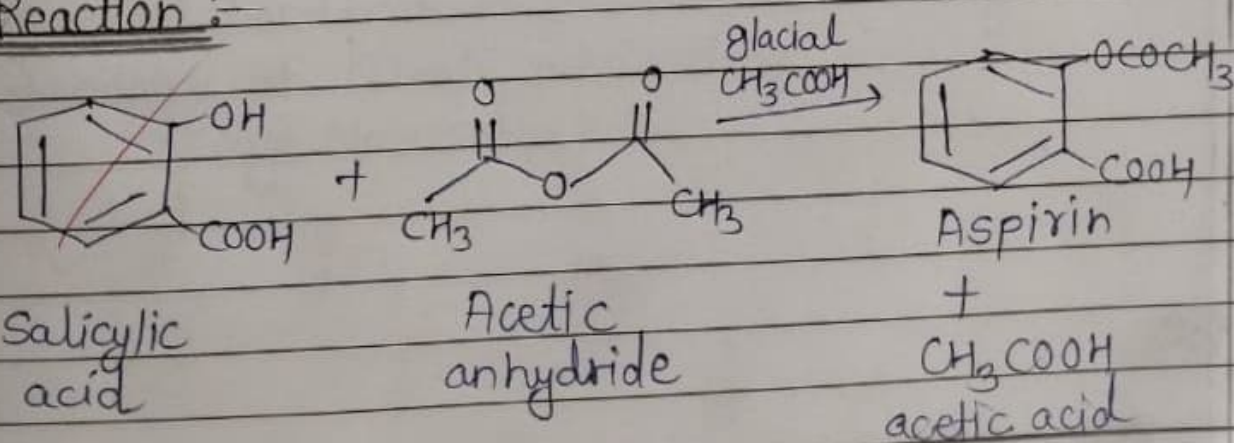
The phenoxide Nucleophile attacks the carbonyl carbon of benzoyl chloride to get substituted by removal of the chloro group.



Theory and Mechanism:

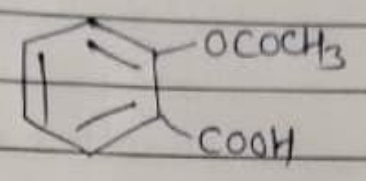
- (i) Synthesis of aspirin is an esterification reaction.
- (ii) The hydroxyl group of salicylic acid reacts with acetic anhydride to form an ester.
- (iii) Commercially, aspirin is synthesized using a two-step process. First, phenol (generally extracted from coal tar) is treated with a sodium base which generates sodium phenolate, which is then reacted with carbon dioxide under high temp. and pressure to yield salicylate, which is acidified, yielding salicylic acid. Salicylic acid is then acylated using acetic anhydride, yielding aspirin and acetic acid as a byproduct.
- (iv) Aspirin can also be synthesized from salicylic acid using acetyl chloride. However, in this case the byproduct formed is hydrochloric acid which is corrosive and environmentally hazardous. Thus, acetic anhydride is said to be a better acylating agent, as the byproduct is acetic acid which does not have the unwanted properties of hydrochloric acid.

Reaction:



Result :-

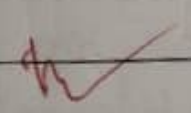
- Name of the compound = Aspirin
- Structure =



- TUPAC Nomenclature = 2-Acetoxy benzoic acid / Acetyl Salicylic acid.
- State, colour and odour = Colourless, odourless and solid crystals.
- Molecular formula = $C_9H_8O_4$
- Molecular Weight = 180
- Theoretical yield = 7.83 gm
- Practical yield = 7 gm
- % yield = 89.39 %
- Melting point = (i) Crude = $133^\circ C$
(ii) Recrystallized = $131^\circ C$

Reference :-

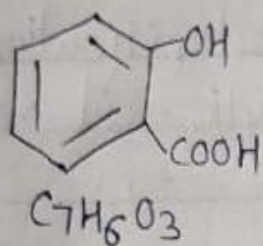
Dr. M. N. Deodhar and Mrs. Jagtap
practical handbook of pharmaceutical organic
chemistry of Nirali publication.
Page No - 8.1 - 8.3



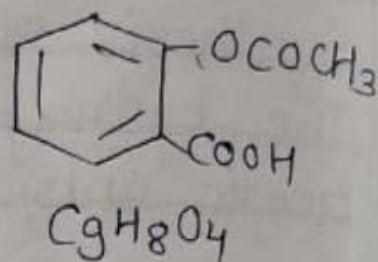
* Calculations :-

(i) Theoretical yield :

1 mole of salicylic acid \equiv 1 mole of Aspirin



\equiv



$$12 \times 7 + 1 \times 6 + 3 \times 16$$

\equiv

$$12 \times 9 + 1 \times 8 + 4 \times 16$$

$$84 + 6 + 48$$

\equiv

$$108 + 8 + 64$$

$$138$$

\equiv

$$180$$

$$6 \text{ gm}$$

\equiv

$$\frac{6 \times 180}{138} = 7.83 \text{ gm}$$

\therefore Theoretical yield = 7.83 gm

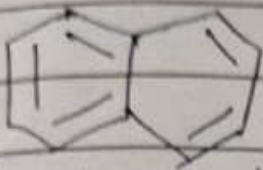
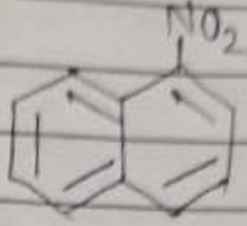
(ii) Practical yield = 7 gm

(iii) Percent yield = $\% = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$

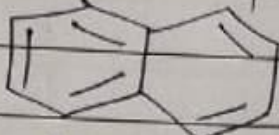
$$= \frac{7}{7.83} \times 100$$

$$= 89.39\%$$

• Physical constant of hydrocarbon and their Derivatives

Name of structure	Derivative preparation	Structure of derivative
Napthalene  Melting point 80°C	1 nitronapthalene 4ml + HNO_3 + Shaking 1gm of Sample, shake at room temp. for 15-20 min. Pour mixture in cold water filter & dry. (Melting point 61°C)	 1-nitronaptha- lene

→ Result :- Name :- Napthalene

• Structure :- 

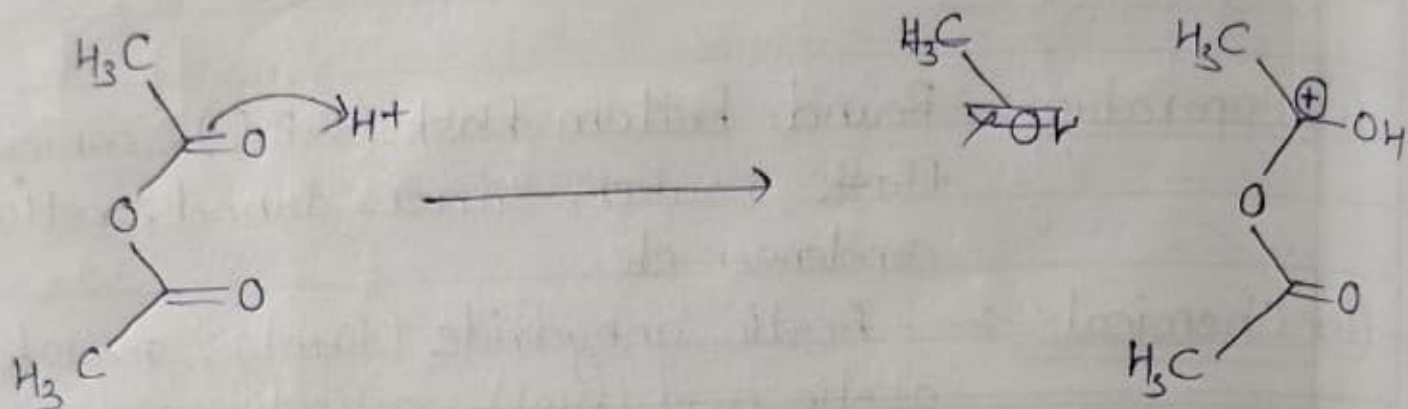
- Molecular formula = C_{10}H_8
- Molecular Weight = 128.1705 g/mol .
- IUPAC Name = Bicyclodeca-1,3,5,7,9-pentaene.
- functional group = Polycyclic aromatic hydrocarbons
- Melting point = 80°C .
- Derivative = 1-Nitronapthalene.
- Uses = (i) Used in dyes.
(ii) Used in manufacturing of resins, plastic, fuel etc;

→ Reference :-

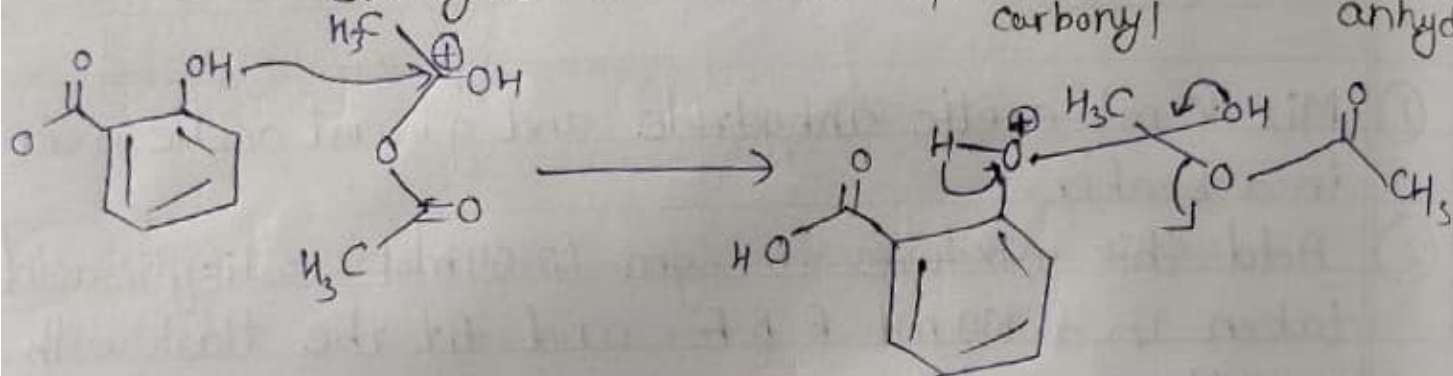
Dr. M. N. Deodhar and Mrs. Jagtap Practical
 handbook of Pharmaceutical organic chemistry.
 { Nirali publication } Page No = 101-110^a.

Mechanism :-

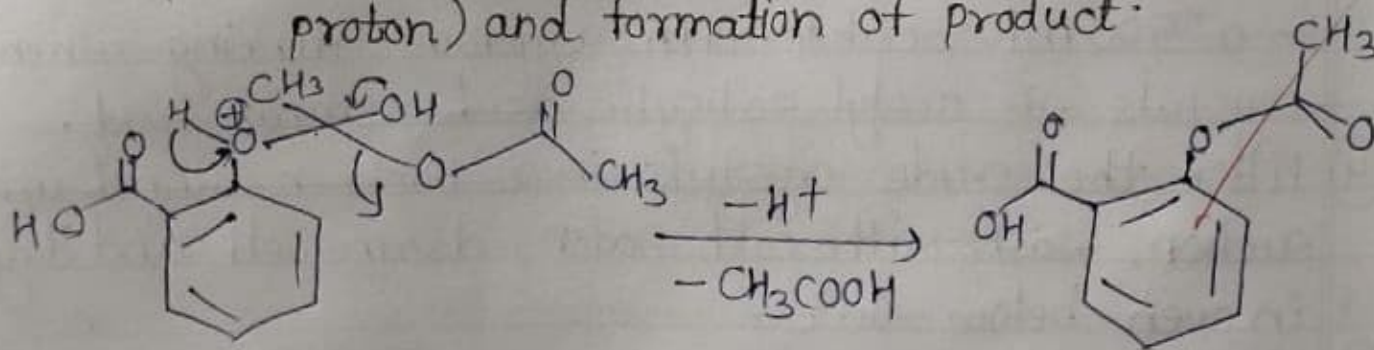
(i) Step-1 = Protonation of carbonyl oxygen of acetic anhydride.



(ii) Step-2 = The attack of nucleophilic hydroxyl group of salicylic acid on electrophilic carbonyl carbon of acetic anhydride.



(iii) Step-3 = Loss of acetic acid (loss of carboxylate ion and proton) and formation of product.



Experiment No-14.

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- Aim :- Synthesis of acetyl salicylic acid (Aspirin) from salicylic acid.

- Requirements :-

(i) Apparatus :- Round bottom flask (R.B.F), conical flask, beaker, stirrer, funnel, reflux condenser etc.

(ii) Chemicals :- Acetic anhydride (10 ml), glacial acetic acid (10 ml), salicylic acid (6 gm) etc.

- Procedure :-

- ① Mix 10 ml acetic anhydride and glacial acetic acid in a beaker.
- ② Add this mixture to 6 gm (0.04 mol) salicylic acid taken in a 100 ml R.B.F. and fit the flask with a reflux condenser.
- ③ Boil the reaction mixture for about 40 min. and then pour the hot solⁿ in 100 ml cold water contained in a 500 ml beaker with vigorous stirring when crystals of acetyl salicylic acid separate out.
- ④ Filter the crude crystals in a Buchner funnel with suction, wash with cold water, drain well and dry in oven below 100°C.
- ⑤ Recrystallize from hot ethanol.
- ⑥ Report the % yield and Melting point.

* Conclusion :- The given organic compound is found to contain C, H, O elements and compound is acidic so group I(A) present.

c) Determination of physical Constant =
Melting point of given organic compound is 101°C .

D) Determination of functional group :-

Group IA = Test for Group IA - Carboxylic acid
[R-COOH]

Test	Observation	Inference
1) NaHCO_3 Test 0.2-0.3 gm of compound + 2ml saturated sol ⁿ	Strong evolution of CO_2 reprecipitation with conc. HCl.	-COOH group present
2) Esterification Test :- 0.02 gm sample into a dry T.t + 10 drops of ethyl alcohol + con. H_2SO_4	Fragrant Smell	-COOH group is present
3) Neutral sol ⁿ from test + CaCl_2 solution.	White ppt insoluble in acetic acid but soluble in HCl.	Oxalic acid Present



Test	Observation	Inference
1) Test for Nitrogen	Blue ppt not form	Nitrogen may be absent
2) Test for Sulphur	Black ppt not form	Sulphur may be absent
3) Test for Halogen	No particular observation	Halogen may be absent

* Conclusion :- The given organic compound is found to contain C, H, O element and compound is neutral therefore (group IC) present.

* Determination of Physical Constants:-

The melting point of given organic compound was found to be 80°C.

* Determination of functional group:-

Test	Observation	Inference
a) Jodine test = 0.2gm sample dil sol ⁿ of iodine in benzene	A violet solution	Hydrocarbon may present
b) Test with chloroform Heat 0.5gm AlCl ₃ + sample + chloroform	bright colour	Aromatic hydrocarbon present

B) Determination of Elements {Lassaigne's Test} :-

Take a small piece of clean dry sodium (0.5 gm) in a clean, dry sodium fusion tube. Heat the tube slowly. Remove it from the flame and add abt 0.02 - 0.03 gm solid compound or 2-3 drops if liquid directly to molten sodium. First heat on a low flame and then on a stronger flame until it becomes red hot. Keep in hot condition for 1-2 minutes (until the compound has completely reacted) and then the plunge into 10-15 ml of distilled water kept in a porcelain dish. Carry out the same procedure for a 2-3 separate ignition tubes. Boil the contents of porcelain dish 2-3 minutes and filter. Use filtrate (known as Lassaigne's extract or sodium fusion extract) for testing the extra elements. In this case, the filtrate should be water clear. If it is dark coloured, the whole fusion should be repeated.

b) 10mg sample + 10 drops of 10% NaOH shake	Sample Not soluble	phenol is absent
c) 10mg sample + 10 drops of HCl	Sample not soluble	Base is absent
d) All the tests are Negative	—	Neutral sample is present

e) Detection of saturation / Unsaturation :-

Test	Observation	Inference
a) Action of $KMnO_4$		
15 dissolve 0.2 gm of solid sample in water if insoluble dissolve in acetone add few drop $KMnO_4$	Pink colour of $KMnO_4$ disappears	Unsaturated compound may be present

f) Detection of aliphatic / aromatic nature :-

Test	Observation	Inference
Heating on a copper	Sooty flame	Aromatic compound present

* Conclusion :- On the basis of above test performed given organic compound

- is
- a) Neutral
 - b) Unsaturated
 - c) Aromatic

Aim:- To identify the given organic compound by qualitative analysis.

Apparatus:- Test tubes, Measuring cylinder, Tripod stand, Bunsen burner, fult fusion tube, porcelain dish, wize gauze, thermometer etc;

Chemicals = All general and Table reagent.

A Preliminary Test:-

Test	Observation	Inference
a) State	Solid	Compound having high molecular weight eg. aromatic acids, carbohydrates, phenols may be present.
b) Colour	Colourless solid	Carbohydrates, acids, phenols may be present.
c) Effect of Heat	Sharp Melting	Pure organic compound may be present.

d) Type of determination:-

Cold water + sample = Insoluble.

Test	observation	Inference.
a) 0.1mg sample + 10 drops of 10% NaHCO_3	Sample Not Soluble	Acid is not Present

E) Physical Constants of COOH and their derivatives:-

Name and Structure (M.P./B.P)	Derivative preparation test	Structure of derivative
Oxalic acid COOH COOH M.P. 101°C	Urea oxalate = Prepare 1 ml saturated sol ⁿ of urea & 1 ml sat. sol ⁿ of oxalic acid. Mix both sol ⁿ , crystals of urea oxalate separate (M.P. 171°C)	Urea oxalate

Result :-

- (i) Name = Oxalic acid.
- (ii) Structure = $\begin{matrix} \text{COOH} \\ | \\ \text{COOH} \end{matrix}$
- (iii) IUPAC Name = Ethanedioic acid
- (iv) Molecular formula = $\text{C}_2\text{H}_2\text{O}_4$
- (v) Molecular Weight = 90.03 g/mol .
- (vi) Functional group = Carboxyl group
- (vii) Melting point = 101°C
- (viii) Derivative = Urea oxalate
- (ix) Uses = Removing rust and stains on objects and metals.

Reference :- Dr. M. N. Deodhar and Mrs. T. R. Jagtap practice handbook of pharmaceutical organic chemistry of Nirali publication, Edition of year 2016. Page No - 5.10.

5) Detection of saturation / unsaturation :-

Test	Observation	Inference
a) Action of $KMnO_4$ Dissolve 0.2gm solid in water if water insoluble add few drop of Neutral $KMnO_4$	No decolourisation	Saturated compound may be present.

5) Detection of Aliphatic / Aromatic Nature :-

Test	Observation	Inference
a) Heating on a copper	Non-sooty flame	Aliphatic compound may be present

* Conclusion:- On the basis of test performed above is organic compound's

1) Acid

2) Saturated

3) Aliphatic

B) Determination of Elements [Lassaigne's Test] :-

Test	Observation	Inference
1) Test for Nitrogen	No blue ppt or blue colour.	Nitrogen may be absent
2) Test for Sulphur	No black ppt	Sulphur may be absent
3) Test for Halogen	No particular observation	Halogen may be absent

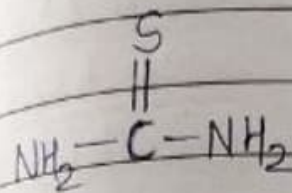
Physical Constants of Neutral Compounds (III.B) and their derivatives.

Thioamides

Name of structure
M.P./B.P.

Derivative test

Structure of derivative

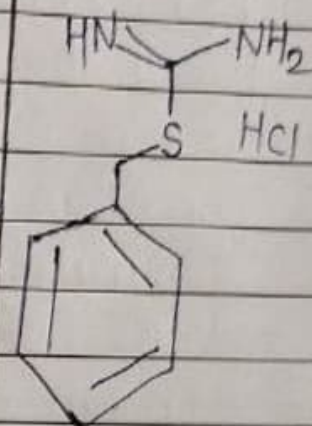


M.P. = 180°C

Thiourea

Colour reaction:—

0.2 gm sample + 1 ml dil. NaOH solution, heat till ammonia evolves. Add 5 ml water and a drop of FeCl₃ solution. Blood red colour due to formation of ferric thiocyanate.



S-Benzyl iso-thiuronium chloride

Result :- Name = Thiourea

Structure = $\text{NH}_2 - \overset{\text{S}}{\parallel} \text{C} - \text{NH}_2$

IUPAC Name = Thiocarbamide

Molecular weight = 76.12 g/mol.

Molecular formula = CH₂N₂S

Nature = Neutral, Unsaturated, aliphatic

functional group = Carbonyl group.

Melting point = 180°C.

Derivative = S-benzyl isothiuronium chloride.

Uses :- Manufacture of flame.

Reference :- Dr. M. N. Deodhar and Mrs. J. R. Jagtap practical

handbook of pharmaceutical organic chemistry of Nirali publication. Edition of year 2016. Pg. No = 5.73-5.74.

Experiment No-12

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Aim :- To identify the given organic compound by qualitative analysis

Requirements :-

(i) Apparatus :- Test tubes, Measuring cylinder, stirrer, Bunsen burner, wire gauze, test tube holder, fusion tubes, thermometer, capillaries

(ii) Chemicals :- All general and Table reagent.

A) Preliminary Test :-

Test	Observation	Inference
1) State	Solid	Comp. having high molecular weight. e.g. aromatic acid, carbohydrate may be present.
2) Colour	Colourless solid	Carbohydrates, amides may be present
3) Effect of Heat	sharp melting point	Pure organic compound may be present

4) Type of determination :-

[shake]
Sample + cold water = water soluble / Miscible.

i) Type determination of water soluble samples

Test	Observation	Inference
1) Litmus Test	Blue litmus turns red	Acid may be Present

B) Determination of Elements [Lassaigne's Test] :-

Take a small piece of clean dry sodium (0.5g) in a clean, dry sodium fusion tube. Heat the tube slowly. Remove it from the flame and add about 0.02 - 0.03 gm solid compound or 2-3 drops if liquid directly to molten sodium. First heat on a low flame and then on a stronger flame until it becomes red hot. Keep it in red hot condition for 1-2 minutes (until the compound has completely reacted) and then plunge into 10-15 ml of distilled water kept in a porcelain dish. Carry out the same procedure for 2-3 separate ignition tubes. Boil the contents of porcelain dish for 2-3 minutes and filter. Use the filtrate (known as Lassaigne's extract or sodium fusion extract) for testing extra elements. In this case, the filtrate should be water clear. If it is dark coloured the whole fusion should be repeated.

a) Litmus Test	No change	Neutral sample Present
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5) Detection of saturation / Unsaturation :-

Test	Observation	Inference
a) Action of $KMnO_4$ Dissolve 0.2 gm of sample if solid in 2ml water or acetone, if water insoluble. Then add few drops of neutral $KMnO_4$	pink colour of $KMnO_4$ disappears	Unsaturated compound present

6) Detection of aliphatic / aromatic compound :-

Test	Observation	Inference
a) Heating on a copper gauze	Non-sooty flame	Aliphatic compound Present

• Conclusion :-

On the basis of test formed above the given organic compound is

- 1) Neutral
- 2) Unsaturated
- 3) Aliphatic

B) Determination of elements [Lassaigne's Test]:

Take a small piece of clean, dry sodium (0.5 gm) in a clean, dry sodium fusion tube. Heat the tube slowly. Remove it from the flame and add about 0.02-0.03 gm solid compound or 2-3 drops if liquid directly to molten sodium. First heat on a low flame and then on strong flame until it becomes red hot. Keep it in red hot condition for 1-2 minutes (until the compound has completely reacted) and then plunge into 10-15 ml of distilled water kept in porcelain dish. Carry out same procedure for 2-3 separate fusion tubes. Boil contents of porcelain dish for 2-3 minutes and filter. Use the filtrate (known as Lassaigne's extract or sodium fusion extract) for testing extra elements. In this case, the filtrate should be water clear. If it is dark coloured, the whole fusion should be repeated.