Umm Alqura University
Faculty of Applied Sciences
Department of Chemistry

Practical Organic Chemistry

Prepared by:

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Organic Chemistry (1) Organic Chemistry (2)
Organic Chemistry (3) Organic Chemistry (4)

By

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Practical Organic Chemistry (1)

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Safety in Chemical Laboratories

In case of fire

- If your clothing catches fire, immediately drop to the floor and roll to smother the flames and call for help.
- If a compound or solvent catch on fire, *if you can*, quickly cover the flames with a piece of glassware
- If it is feasible, use a fire extinguisher to put the fire out.
- Do not put water on an organic chemical fire because it will only spread the fire.
- If the fire is large, do not take chances: evacuate the lab and the building immediately and tell your TA or the Coordinator what has happened
- If no one in authority is available, pull the fire alarm in the hallway
- If no one in authority is available, call the Department, Faculty and University Administration.
- If the fire alarm sounds for any reason, leave the room immediately and exit the building

This picture illustrates the 3 different fire extinguishers found in the teaching labs. The 2 on the left are dry chemical; the one on the right is a CO_2 extinguisher. The CO_2 one has a large nozzle and is usually the best choice in the case of a chemical fire.

<u>If you inhale vapors</u>

Leave the area immediately - at least into the hallway. Tell your TA or the Coordinator; they will take you outside into the fresh air, and if necessary provide first aid or take you to get medical attention.

<u>If you spill a chemical on yourself</u>

Immediately rinse the affected area with lots of water. Use soap if you wish, but never try to "treat" the spill with another solvent or chemical unless directed to do so by your TA. If the affected area remains more than slightly red after the rinsing period, seek for medical attention.

<u>If you spill a corrosive on yourself</u>

Immediately rinse the affected area with lots of water. Use soap if you wish, but never try to "treat" the spill with another solvent or chemical unless directed to do so by your TA. If the affected area remains more than slightly red after the rinsing period, seek medical attention.

If you cut or burn yourself

If you cut yourself, wash the wound immediately with large amounts of cold water. If it is your neighbor who has been hurt, be prepared to help them if they are unable to help themselves. Apply direct pressure to stop the bleeding as necessary. If the bleeding is profuse, elevate the affected limb. Watch for evidence of shock and contact TA or the Lab Coordinator as necessary.

Thermal burns are treated by covering the affected area with cold water or ice. After a while, you can apply a pain-relieving cream. If the burn looks like it is more than just a reddening of the skin, seek medical attention.

Laboratory equipments

Graduated cylin	Beaker	Erlenmeyer flask

Round-bottom flask

Y-adaptor

Vacuum adaptor

laboratory balance

Condenser

Thermometer adaptor

Claisen adaptor

Beaker

Erlenmeyer flask

Side-arm flask

Buchner funnels

Thermometer

Stemmed funnels

Separatory funnel

Watch glass

Stir rod

Vials

Graduated cylinder

Pasteur Pipet

Volumetric Flasks

Filtering Flask

Crystallizing dish

Versatile clamp

3-pronged clamp

Ring clamp

Spatula

Scoopula

Forceps

Keck clip

Stir bar

Heating mantle

Stir motor

water bath

Stirring Hotplate

Ring stand

Tubing

Recrystallization

When solid organic compounds isolated from organic reactions, they are usually contaminated with small amounts of other compounds (impurities) which are produced with the desired product during the chemical reaction. Each chemist must master the technique of recrystallization to become proficient in the laboratory workup.Impurities are excluded from the crystalline compounds by recrystallisation from a suitable solvent or mixture of solvents depending upon differences in their solubility, in its simplest form, the crystallization process consists of:

(i) Dissolving the impure substance in some suitable solvent at or near the boiling point.
(ii) Filtering the hot solution from particles of insoluble material and dust.
(iii) Allowing the hot solution to cool thus causing the dissolved substance to
crystallize out.
(iv) Separating the crystals from the supernatant solution (or mother-liquor). The resulting solid, after drying, is tested for purity (usually by a melting point determination, by spectroscopic methods, or by thin-layer chromatography), and if found impure is again recrystallized from fresh solvent.
The most desirable characteristics of a solvent for recrystallization are as follows:

1- A high solvent power for the substance to be purified at elevated temperatures and a comparatively low solvent power at the laboratory temperature or below.

2- It should dissolve the impurities readily or to only a very small extent.
3- It should yield well-formed crystals of the purified compound.
4- It must be capable of easy removal from the crystals of the purified compound, i.e. possess a relatively low boiling point.

It is of course, assumed that the solvent does not react chemically with the substance to be purified. If two or more solvents seem to be equally suitable for recrystallization, the final decision will depend on some factors as ease of manipulation, toxicity, flammability and cost.

Common solvents for recrystallization

Solvent	b.p. (^{o}C)	
Water (distilled)	100	To be used whenever suitable
Methanol	64.5	Flammable; toxic
Ethanol	78	Flammable
Industrial spirit	77-82	Flammable
Rectified spirit	78	Flammable
Acetone	56	Flammable
Ethyl acetate	78	Flammable
Acetic acid (glacial)	118	Not very flammable, pungent vapors
Dichloromethane (methylene chloride)	41	Non-flammable; toxic
Chloroform	61	
Diethyl ether	35	Non-flammable; vapor toxic
Benzene	80	Flammable, avoid whenever possible
Dioxane	101	
Carbon tetrachloride	77	Flammable, vapor highly toxic
Light petroleum	40-60	Flammable, vapor toxic

Cyclohexane	81	Non-flammable, vapor toxic Flammable Flammable

Filtration

After completion of a reaction filtration of a mixture will often be necessary either to isolate an organic solid which has separated out or to remove insoluble impurities or reactants, in which case the desired product remains in solution. The two types of filtration commonly used in organic chemistry laboratories are gravity filtration and vacuum or suction filtration.

Gravity Filtration

Gravity filtration technique is used to remove solid impurities from an organic liquid. Gravity filtration can be used to collect solid product, although generally vacuum filtration is used for this purpose because it is faster.

Vacuum Filtration

Primarily to collect a desired solid, for instance, the collection of crystals in a recrystallization procedure. When considerable quantities of a solid are to be filtered from suspension in a liquid, a vacuum filtration is used. It uses a Buchner funnel of suitable size and a **side-arm flask**.

Solvent Removal

Removal of solvent from a solution to recover either a solid or a high-boiling liquid is often necessary. There are several ways to do this.

Open-Dish Evaporation

The evaporation of solvent can be effected by placing the solution in an open container (an Erlenmeyer, evaporating dish, beaker, vial). The container is put on a heat source (steam bath, hot plate, heating mantle, sand bath) and the solvent boiled off. (If the solvent is water, use a heat source other than a steam bath.) The problem with open-dish evaporation is that the solvent is released into the air (pollution). Open-dish evaporation should always be completed in a hood if the solvent is anything other than water. If the solvent is a hazardous compound (for instance, methylene chloride, chloroform etc…), it is probably better to choose another method of solvent removal.

Reduced-Pressure Evaporation

Complete removal of solvent can be done quickly by placing it in a side-arm flask and then applying vacuum. Under vacuum (reduced pressure) liquids vaporize and boil off at lower temperatures; effectively, the solvents removed faster under vacuum rather than at atmospheric pressure.

Rotary Evaporators: Rotary evaporators, or rotovaps, are standard equipment in most organic chemistry research labs. These evaporators are designed to remove solvent rapidly from solutions. The motor in the rotovap turns the flask rapidly, providing a greater surface from which evaporation can occur and speeding up the process. Cooling coils in the rotovap condense the vapors and drop them into a collection flask so that they can be recycled or properly disposed. The rotovap is connected to a vacuum source, and again, this speeds up the evaporation process.

Melting Point Determination

Organic compounds possess distinguishing physical properties which enable their identification. In many situations, a careful determination of the physical properties of a given chemical can be used for its identification. The melting of a substance refers to the temperature at which the solid and liquid states are in equilibrium. The melting point is the temperature at equilibrium when starting in the solid state and going to the liquid state.

Melting points of pure substances occur over a very narrow range and are usually quite sharp. The criteria for purity of a solid is the narrowness of the melting point range and the correspondence to the value found in the literature. Impurities will lower the melting point and cause a broadening of the range. For example, pure benzoic acid has a reported melting point of 122.13^{o}C; benzoic acid with a melting point range of 121–123°C is considered to be quite pure.

Digital Melting Point Apparatus

Distillation

Distillation is the process of heating a liquid until it boils, then condensing and collecting the resultant hot vapors. Mankind has applied the principles of distillation for thousands of years. Distillation was probably first used by ancient Arab chemists to isolate perfumes. Vessels with a trough on the rim to collect distillate, called *diqarus*, date back to 3500 BC.

In the modern organic chemistry labs, distillation is a powerful tool, both for the identification and the purification of organic compounds. The boiling point of a compound is one of the physical properties used to identify it. Distillation is used to **purify a compound** by separating it from a non-volatile or less-volatile material. When different compounds in a mixture have different boiling points, they separate into individual components when the mixture is carefully distilled.

Distillation Guide

What's distillation used for?

Distillation is a laboratory technique used for separating and purifying liquids.

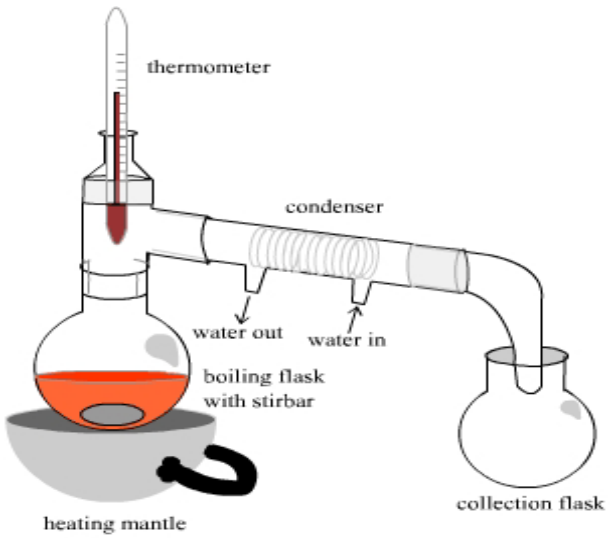
The two main kinds of distillation are *simple distillation* and *fractional distillation*, and both are used widely.

What is simple distillation?

Simple distillations are used frequently in the organic chemistry teaching labs. They are useful in the following circumstances:

- the liquid is relatively pure to begin with (e.g., no more than 10% liquid contaminants).
- the liquid has a non-volatile component, for example, a solid contaminant.
- the liquid is contaminated by a liquid with a boiling point that differs by at least 70°C.

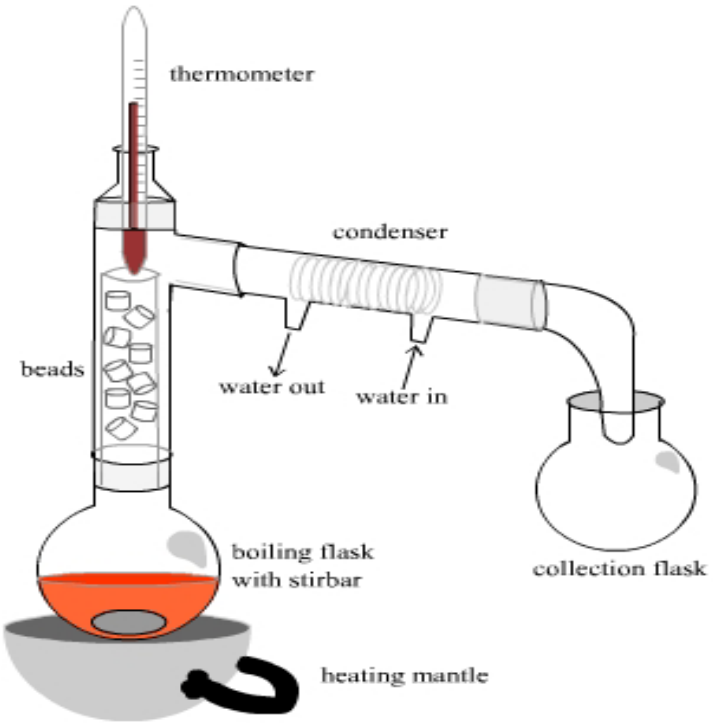
A simple distillation apparatus (shown below) consists of a boiling flask (round-bottom flask) attached to an adapter holding a thermometer (to determine the boiling temperature of the liquid). The adapter connects to a condenser into which cold water is constantly passed through. The condenser leads into a collection flask for the purified liquid.



Simple distillation.

What is fractional distillation?

Mixtures of liquids whose boiling points are similar (separated by less than 70°C) cannot be separated by a single simple distillation. In these situations, a fractional distillation is used. Fractional distillation is essentially the same as simple distillation except that a fractionating column is placed between the boiling flask and the condenser. The most excellent separation between the liquids is obtained by fractional distillation because the glass beads in the fractionating column provide "theoretical plates" on which the refluxing liquid can condense, re-evaporate, and condense again, essentially distilling the compound over and over. The more volatile liquids will tend to push towards the top of the fractionating column, while lower boiling liquids will stay towards the bottom, giving a better separation between the liquids.



Fractional distillation.

So, simple or fractional?

The choice of whether to use fractional distillation or simple distillation depends on the two liquids being separated. Typically, using simple distillation is preferable because the apparatus is, well, simpler, and a simple distillation typically goes faster than a fractional distillation (and requires less energy). On the other hand, fractional distillation gives better separation between the liquids. The choice of whether to use simple or fractional distillation, then, depends usually on the difference in boiling temperatures between the two liquids. If there is a large difference in the boiling points (>70°C) between the two liquids then simple distillation is probably the best option. On the other hand, if there is only a small temperature difference between the two liquids a fractional distillation is the preferable option.

	Simple distillation	**Fractional distillation**
Advantages	• simpler setup than fractional • faster distillation times • consumes less energy than fractional distillation	• much better separation between liquids than simple distillation • can more readily purify complex mixtures than simple distillation
Disadvantages	• requires the liquids to have large boiling point differences (>70°C) • gives poorer separation than	• more complicated setup than simple distillation • takes longer for liquids to distill • consumes more

	fractional distillation • only works well with relatively pure liquids	energy than simple distillation
Best used for:	separating relatively pure liquids with large boiling differences or liquids with solid impurities	Separating complex mixtures of liquids with smaller boiling point separations.

Vacuum Distillations

Vacuum distillation is distillation at a reduced pressure. Since the boiling point of a compound is lower at a lower external pressure, the compound will not have to be heated to as high a temperature in order for it to boil. Vacuum distillation is used to distill compounds that have a high boiling point or any compound which might undergo decomposition on heating at atmospheric pressure. The vacuum is provided either by a water aspirator or by a mechanical pump.

Sublimation (phase transition)

Sublimation is the change of a substance directly from the solid to the gas phase without passing through an intermediate liquid phase. Sublimation is an endothermic phase transition that occurs at temperatures and pressures below a substance's triple point in its phase diagram. The reverse process of sublimation is de-sublimation, or deposition.

At normal pressures, most chemical compounds and elements possess three different states at different temperatures. In these cases, the transition from the solid to the gaseous state requires an intermediate liquid state. Note, however, that the pressure referred to here is the *partial*

pressure of the substance, not the *total* (e.g., atmospheric) pressure of the entire system (e.g., water ice just below 0 °C).

Extraction

Liquid-liquid extractions using a separator funnel are essentially the only kind of extraction performed in the organic teaching labs. Liquid-liquid means that two liquids are used in the extraction procedure. The liquids must be immiscible: this means that they will form two layers when added together, like oil and water. Some compounds are more soluble in the organic layer (the "oil") and some compounds are more soluble in the aqueous layer (the "water").

The photo above illustrates how two liquid layers separate. The red layer is simply red food coloring in water. Water is immiscible with the other liquid, which is methylene chloride. Methylene chloride is heavier (denser) than water, therefore, the clear methylene chloride layer is under the red, aqueous food coloring layer.

Qualitative Analysis of Organic Compounds

The analysis and identification of unknown organic compounds constitutes a very important aspect of experimental organic chemistry. There is no definite set procedure that can be applied overall to organic qualitative analysis. Qualitative tests that require substantial quantities of a number of (often hazardous) chemicals to be stocked in the lab for experimental use are frequently being phased out of organic chemistry in favor of modern spectroscopic techniques (IR, MS and NMR).

General Scheme of Analysis

A. Preliminary Test

Note physical characteristics: solid, liquid, color, and odor. Compounds that are yellow to red in color are often highly conjugated. Amines often have a fish-like odor, while esters usually have a pleasant fruity or floral smell. Acids have a sharp, biting odor. Some compounds can have corrosive vapors or make you feel nauseous.

B. Physical Constants

Determine the boiling point or melting point. Distillation is recommended in case of liquids. It serves the dual purpose of determining the boiling point as well as purifying the liquid for subsequent tests.

C. Solubility Tests

The solubility of the unknown in the following reagents provides very useful information. In general, about 1 mL of the solvent is used with 0.1 g or 0.2 mL (2-3 drops) of the substance.

D. Group Classification Tests

From the previous tests it is often possible to deduce the functional groups present in the unknown compound.

FUNCTIONAL GROUP ANALYSIS

Below are listed 24 chemical tests that could be used to help identify an unknown. The tests are listed in numerical/alphabetical order.

Introduction to qualitative tests

1. 2,4-dinitrophenylhydrazine (for aldehydes and ketones)
2. Acetyl chloride (for acidic hydrogen compounds such as alcohols)
3. Basic hydrolysis (for amides, esters and nitriles)
4. Beilstein test (for halogenated compounds)
5. Benedict test (for aldehydes and reducing sugars)
6. Bromine in carbon tetrachloride (unsaturation for alkenes and alkynes)
7. Ceric nitrate (for alcohols and phenols)
8. Chromic acid (for aldehydes, primary and secondary alcohols)
9. Combustion test (for flammable or combustible compounds)
10. Ferric chloride (for phenols)
11. Ferric hydroxamate (for esters, acid chlorides and acid anhydrides)
12. Ferrous hydroxide (for nitro compounds)
13. Hinsberg test (to distinguish primary, secondary and tertiary amines)
14. Hydroxylamine hydrochloride (for aldehydes and ketones)
15. Iodoform test (for methyl carbonyl compounds)
16. Lucas test (to distinguish primary, secondary and tertiary alcohols of six carbons or less)
17. Nitrous acid (to distinguish primary, secondary and tertiary amines)

18. pH in ethanol/water (to distinguish low molecular weight acidic or basic compounds)
19. Potassium permanganate (for compounds that can be oxidized)
20. Silver nitrate in ethanol (for S_N1 reactions of alkyl halides)
21. Sodium fusion (for compounds containing halogen, nitrogen or sulfur)
22. Sodium iodide in acetone (for S_N2 reactions of alkyl chlorides or bromides)
23. Solubility classification (for general classification or organic compounds)
24. Tollens test (for aldehydes and reducing sugars)

Determining Solubility of Organic Compounds

1) Water Solubility

Place approximately 0.1 g or 0.2 mL (2-3 drops) of compound in a small test tube, and add about 1 mL of water in small portions. Shake test tube vigorously after the addition of each portion of solvent. Check the pH of the water to determine if your unknown is partially or completely soluble in water and whether your compound has changed the pH of the water.

- Litmus turns red: water soluble acidic compound
- Litmus turns blue: water soluble basic compound
- Litmus neutral: water soluble general compound or insoluble compound

2) 5% NaOH Solubility

Place approximately 0.1 g or 0.2 mL (2-3 drops) of compound in a small test tube, and add about 1 mL of NaOH solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If

soluble, then your unknown is behaving as an organic acid. The most common organic acids are carboxylic acids and phenols. Carboxylic acids are usually considered stronger acids than phenols, but both of these acids will react with NaOH (a strong base).

3) 5% $NaHCO_3$ Solubility

Place approximately 0.1 g or 0.2 mL (2-3 drops) of compound in a small test tube, and add about 1 mL of NaHCO3 solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If soluble, then it is a strong organic acid. If not, then it is a weak organic acid, if it dissolves in NaOH. The most common weak organic acid are phenols. Typically, only a carboxylic acid will react with NaHCO3

4) 5% HCl Solubility

Place approximately 0.1 g or 0.2 mL (2-3 drops) of compound in a small test tube, and add about 1 mL of HCl solution in small portions. Shake test tube vigorously after the addition of each portion of solvent. If HCl soluble, then it is an organic base. Amines are the most common organic base. If insoluble in all solutions, then your unknown is not an acidic or basic organic compound.

Experiment 1: Identifications of hydrocarbons

Objective:

Identification of saturated and unsaturated hydrocarbons using their properties and reactions.

Introduction

Hydrocarbons, compounds which contain only carbon and hydrogen, can be classified into several types, depending on their structure. Aliphatic hydrocarbons are divided into three classes: alkanes have only single bond. Aliphatic hydrocarbons are divided into three classes: alkanes have only single bonds, and are said to be saturated; alkenes and alkynes have carbon-carbon double or triple bonds, and are said tobe unsaturated. Aromatic hydrocarbons are cyclic compounds whose structure is related to that of benzene, with six -electrons in a six-member ring. Aliphatic Hydrocarbons such as Alkanes are relatively inert to chemical oxidizing agents such as neutral or alkaline permanganate, where alkenes are readily oxidized at room temperature. Hydrocarbons are divided into two categories. Solids and liquids some examples of solid hydrocarbons are (naphthalene, Anthracene) and liquid hydrocarbons are (toluene, benzene, n-hexane).

Procedure

All tests should be carried out in dry test tubes, and observations should be recorded on the report sheet as each experiment is performed.

(A) Solubility of hydrocarbons

In various solvents the solubility of pentene, toluene, heptane, and an unknown will be tested.

1- Put 5 ml of a polar solvent (water) into four different small test tubes.

2- Add 1 ml of each hydrocarbon to the test tubes respectively. Stopper and shake.
3- A cloudy appearance indicates the insolubility (the absence of two distinct liquid layers indicates solubility). Record your observations on the Report.
4- Add 5 ml of (non-polar solvent) to four additional test tubes.
5- Add 1 ml of each hydrocarbon to its respective test tube. Stopper and shake. Record your observations on the Report Sheet.

B) Bromine Test.

The reaction of various hydrocarbon as pentene, heptane, toluene, and an unknown with bromine will be observed. Disappearance of the color of bromine from a reaction mixture indicates a reaction is taking place (positive test).

1- Carry out this experiment in a fume hood.
2- Clean and dry four small tubes.
3- Place 1 ml of (pentene, heptane, toluene and unknown into four different small test tubes
4- Add 4 drops of 5% bromine in CCl_4 to each test tube and gently swirl the contents of the tubes.
5- If the blue changes to red, it indicates the presence of hydrogen bromide (HBr) being given off in the reaction. Report your results on the Report Sheet.
6- Observe any color changes and the speed of the changes for five minutes. Record these observations on your Report Sheet.

(C) Baeyer's Test.

The potassium permanganate (KMO_4) is a purple colorandwhen reacts with the hydrocarbon, a brown color will be observed (MnO_2).

1- Place 1 ml of each of various hydrocarbons into separate clean and dry small test tubes.
2- Add a mixture of 3 ml. of dilute potassium permanganate solution (0.5 % $KMnO_4$ solution) and 3 ml. of dilute sodium carbonate solution (10% Na_2CO_3).
3- Shake the tube for 1-2 minutes.
4- If the color of $KMnO_4$ was changed it is cyclohexene
5- If no change in color it is alkane or aromatic hydrocarbon.
6- Record your observations on the Report Sheet.

(D) Friedel-Crafts reaction.

It is the reaction of aromatic hydrocarbons with aluminum chloride ($AlCl_3$) in presence of chloroform ($CHCl_3$) to produce a brightly colored compound

1- Place 2 ml of $CHCl_3$ into four clean and dry small test tubes.
2- Add two drops various of hydrocarbon to the test tube respectively and gently stirrer the tube.
3- Add 0.5 gm. of $AlCl_3$ so that some of the solid strikes the side of the tube wall that is moisten with the unknown hydrocarbon.
4- If you observed a brightly colored compound it is aromatic hydrocarbon.
5- If no observed a brightly colored compound it is alkane (n-hexane).

Differentiate between aromatic hydrocarbons

Benzene

Physical properties:

Molecular formula (M.F) C_6H_6, colorless liquid, b.p: 80 ^oC, immiscible with water

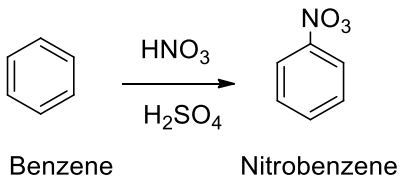
Chemical properties:

1-Freezing test:

In dry test tube, Place 1 ml of benzene then cool it in ice, it is solidified to a colorless crystalline solid, which melts to a liquid when the tube is warmed by hand.

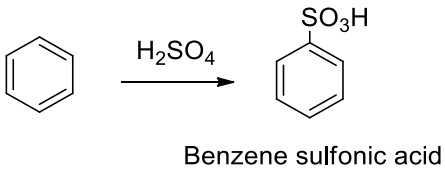
2- Nitration test:

To a mixture of concentrated nitric acid and sulphuric acid add 1 ml of benzene gradually. Shake the mixture, it becomes hot. Pour the mixture into a beaker, which contain 50 ml cold water. A yellow oil of nitrobenzene is formed.



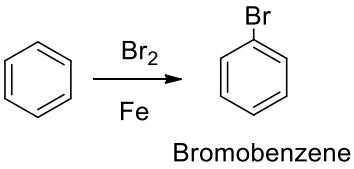
3- Sulfonation reaction:

To 5 ml of sulfuric acid add 1 ml of benzene and heat the mixture on water bath using a condenser. Observe that benzene disappear gradually. Cool then pour the liquid into cold water, a homogeneous solution of benzene sulphonic acid is obtained, which is water – soluble.

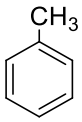


4- Bromine test:

To 1 ml of benzene add 2 ml of bromine water in test tube then add small species of iron to the mixture and notice the disappear of bromine water color. Then the mixture pours onto ice water. Oily layer from bromobenzene is formed.



Toluene



<u>Physical properties:</u>

Molecular formula (M.F) C_7H_8, b.p 110 ^oC, colorless liquid, water insoluble

<u>Chemical properties:</u>

1 - Freezing test:

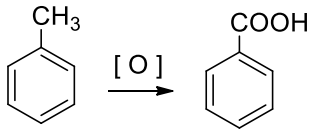
Toluene doesnot solidify readily like benzene and temperature must be reduced to – 93 ^oC to form solid.

2 –Oxidation test:

1- Add 2 ml of toluene to a solution of (potassium dichromate in conc. Sulfuric acid)
2- Heat the mixture gently under reflux for 3 hours.

3- Remove the excess of dichromate by passing sulfur dioxide gas in the solution then neutralize it with a saturated solution of sodium carbonate.

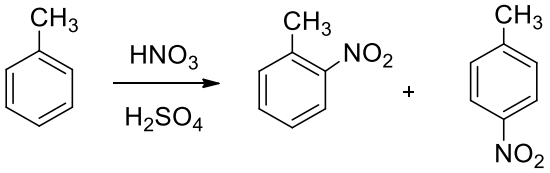
4- Concentrate the alkaline solution by evaporation then acidify it with dil.H_2SO_4. A white crystalline of benzoic acid are formed.



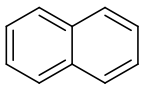
3- Nitration test:

1- Add 1 ml of toluene gradually to a mixture of concentrated nitric acid and sulfuric acid. Shake the mixture,

2- Heat gently then cool and pour the mixture into a beaker, containing 50 ml cold water. yellow heavy oil of nitro toluene is formed.



Naphthalene



<u>Physical properties:</u>

Molecular formula (M.F) $C_{10}H_8$, white crystals, b.p: 80.26 °C, Insoluble in water but soluble in acetone.

<u>Chemical properties:</u>

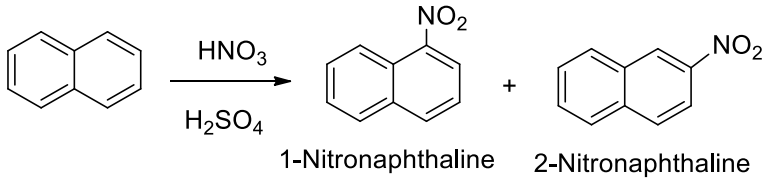
1- Nitration test:

1- Heat 0.5 g of naphthalene in 3ml. of glacial acetic acid, then cool the solution.

2- Add 1 ml of concentrated nitric acid and sulfuric acid

3- Heat the mixture gently for one minute.

4- Cool and pour the solution into baker containing cold water. Yellow solid of nitro naphthalene is formed.

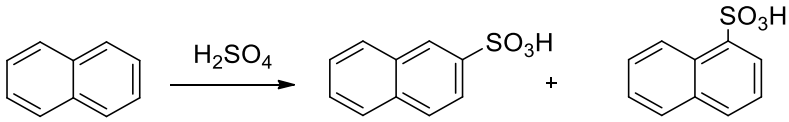


2- Sulfonation test.

1- Add conc. Sulfuric acid gradually to naphthalene at 160 ^{o}C and keep the temperature constant at this temperature for 10 minutes.

2- Cool the mixture, then pour it carefully into a beaker containing cold water, β- naphthalene sulfonic acid separated as solid hydrate

3- Recrystallize by adding half its weight of water at 70 C then adding 1/6 its weight of conc. Hydrochloric acid to give β-naphthalene sulfonic acid.



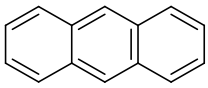
3- Picrate formation:

1- Add concentrated solution of (picric acid in acetone) to concentrated solution of naphthalene in acetone

2- Shake the mixture well then leaf it to cooled .

3- A Yellow needle from naphthalene picrate is formed.

Anthracene



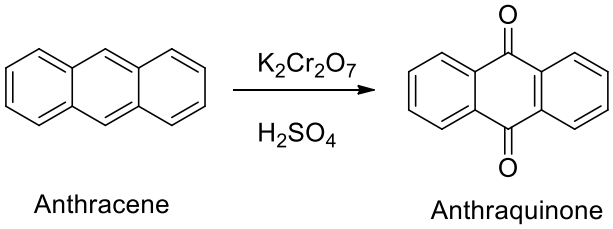
Physical properties:

Molecular formula (M.F) $C_{14}H_{10}$, colorless liquid, b.p: 218°C, insoluble in water but soluble in acetone.

Chemical properties:

1- Oxidation reaction:

1- Dissolve 1 gm of anthracene in few quantity of acetic acid and 3 ml of sulfuric acid
2- Add 4 ml of potassium dichromate solution.
3- Cool the mixture and poured onto cold water
4- A yellow precipitate of anthraquinone is formed.



2- Picrate formation:

1- To a hot concentrated solution of anthracene in acetone
2- Add concentrated solution of (picric acid in acetone).
3- Heat the red solution on the water bath for few seconds then pour it in evaporating dish and leave it for some times .
4- A red crystals of anthracene picrate is formed.

Experiment1: Identification and reactions of hydrocarbons

Name: **Sec.No.**

Date:

Physical properties

Solubility: …….

Color: ……..

Shape: ……..

Chemical properties

Exp.	Obs.	Res.

Unknown is

Experiment 2. Identifications of Alcohols and phenols.

Objective:

Chemical tests will be performed to distinguish primary, secondary and tertiary alcohols. Color tests will be performed to distinguish phenols.

Introduction:

The general formula of an alcohol is ROH in which the R is an aliphatic hydrocarbon group. Alcohols may be looked upon as derivatives of water, HOH. One hydrogen atom of water is substituted by an alkyl group (R). Like water, alcohols show hydrogen bonding. As the chain of the R group increases the hydrocarbon character of the compound overshadows the polar character of the OH group. Consequently, the solubility and boiling point of an alcohol are affected by the length of the carbon chain and the shape of the molecule. The shorter chain alcohols are water soluble, while the long chain alcohols are not soluble in water. Phenols are aromatic alcohol where the R group is aromatic ring.

To differentiate between alcohols and phenols

Ferric Chloride test:

1- Alcohols are readily differentiated from phenols using this test.
2- Addition of a drop or two of ferric chloride solution to a sample of phenol (3-4 drops) will produce a distinct violet/purple coloration.
3- Alcohols do not produce such deep coloration when treated with ferric chloride solution.

Alcohols

Alcohols contain one or more hydroxyl group. It is also classified into mono-, di- and tri hydric alcohols depending on the number of hydroxyl groups present in the molecule. Ethanol, methanol, isopropanol are examples of monohydric alcohols. Ethylene glycol is an example of dihydric alcohols. Glycerol is an example of trihydric alcohol.

Methyl alcohol

CH_3OH

Physical properties:

Molecular formula (M.F) CH_4O, colorless liquid, b.p 65 °C, miscible with water.

Chemical properties

1- Ester formation

1- In dry test tube put 1 ml of methyl alcohol then add o.5 ml of conc. H_2SO_4 and o.5 gm. of salicylic acid or its derivatives.
2- Heat the mixture for 3mintsin water bath, then cool and pour the contents in a beaker containing about 30 ml of sodium carbonate solution.
3- Note the characteristic odor of methyl salicylate.

2- Oxidation reaction

1- In dry test tube, place 0.5 ml of $K_2Cr_2O_7$ and 0.5 ml of conc. H_2SO_4,then cool.
2- Add 0.5 ml of methanol and boil gently (on water bath) notice the pungent odor of formaldehyde and change of color to green.

$$CH_3OH \xrightarrow[H_2SO_4]{K_2Cr_2O_7} HCHO + H_2O + CrSO_4 + K_2SO_4$$

Ethyl alcohol

CH_3CH_2OH

Physical properties:

Molecular formula (M.F) C_2H_6O, colorless liquid, miscible with water, b. p 78 °C.

<u>Chemical properties</u>

1- Oxidation reaction

1- Place 1 ml of $K_2Cr_2O_7$ and 0.5 ml of conc. H_2SO_4, then cool.
2- Add 0.5 ml of ethanol and boil gently (on water bath)
3- Notes the odor of acetaldehyde and change the color solution to green.

$$CH_3CH_2OH \xrightarrow{(O)} CH_3CHO$$

2- Ester formation

1- Place 1 ml of ethanol in dry test tube, then add 0.5 ml of conc. H_2SO_4 and 1 ml of acetic acid.
2- Heat the mixture gently for 3 mints in water bath, cool and pour the tube into baker containing sodium carbonate solution.
3- Not the characteristic odor of ester.

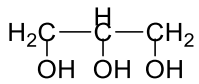
$$CH_3CH_2OH + CH_3COOH \xrightarrow{H_2SO_4} CH_3CH_2COOCH_3 + H_2O$$

3- Iodoform test

1- Add 3 ml of iodine solution to 1 ml of ethyl alcohol then add NaOH solution drop wise until the color of solution becomes straw yellow.
2- Heat the solution in water bath for 5 mints
3- Leaves it to cool gradually, a yellow ppt. of iodoform is appeared.

$$CH_3CH_2OH + 3\,I_2 \xrightarrow{NaOH} CHI_3 + HCOONa + 3HI$$

Glycerol



Physical prope

Colorless viscous liquid, odorless, has sweet taste, miscible with water, alcohol in all proportions.

Chemical properties

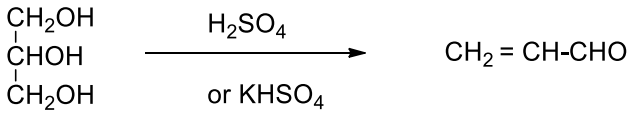
1- Oxidation reaction

Glycerol oxidized to give several products but it is ultimately transformed into CO_2 and H_2O.

1- Add 2 ml of conc. H_2SO_4 and 2 ml of $K_2Cr_2O_7$, then cool.
2- Add 0.5 ml of glycerol and boil gently (on water bath)
3- Notice the effervescence due to the evolution of CO_2.

2- Acroline test

1- Heat 0.5 gm of glycrine with 1gm of hydrogen potassium sulphate $KHSO_4$ or 2 ml of conc. Sulphuric acid in dry test tube
2- Notice odor of acroline.



3- Borax test

1- Add one drop of ph.ph to 1 ml of dil. Borax solution red color appears.
2- Add 1 ml of glycerol and note that the color disappears,
3- Heat gently and observe the appearance of the red color once more, which disappear on cooling the solution.

PHENOLS

Phenols are hydroxyl aromatic compounds which dissolved in alkali forming phenolates. Phenols are classified into mono-, di-, and tri hydric according to the number of OH groups. Monohydric: such as phenol, β and α naphthol. Dihydric: such as catechol, resorcinol and hydroquinol. Trihydric: such as pyrogallol.

General reactions of phenols

1- Ferric Chloride test

1- Take 1 ml of solution of phenol in water or alcohol
2- Add 2 drops of ferric chloride
3- Note formation of violet color which disappear by add hydrochloric acid.

2- phthalin test:

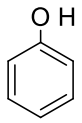
1- Mix about o.5 gm. Of phenols in dry test tube with equal amount of phthalic anhydride, 2 drop of conc. H_2SO_4.
2- Fuse the mixture on gentle flame, cool and then pour it on NaOH solution where a characteristic color appears.

3- Azo – Dye formations:

When diazotized aniline is coupled with phenols in alkaline solution red or orange dye is formed.

1- Dissolve 0.5 ml of aniline in HCl in the first test tube and then cool
2- Add cooled $NaNO_2$ solution in the second test tube and then cool.
3- Add the cold solutions (first and second test tubes) to cold solution of phenol in alkali (NaOH) in the third test tube.
4- A colored dye is formed.

Phenol



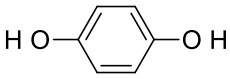
Physical properties:

Molecular formula (M.F) C_6H_6O, colorless crystalline (when pure), m p. 43°C, it is poisonous and corrosive, it turns red in air.

Chemical properties

1- With ferric chloride, it gives violet color discharged with HCl
2- With phthaline test gives a deep pink color.
3- With azo - dye it gives red ppt.
4- With bromine water awhite ppt. is formed.
5- With excess of bromine yellowish white of tri – bromo phenol is formed.

Hydroquinol



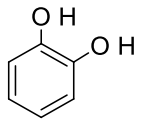
Physical properties:

Molecular formula (M.F) $C_6H_6O_2$, soluble in water, m.p. 170 °C, it is sparingly soluble in benzene.

Chemical properties

1- With ferric chloride, it gives green needles.
2- With bromine water, it gives yellowish white ppt.
3- Not gives azo dye test
4- With phthaline reaction, it gives blue violet color.
5- It reduces Fehling's solution and Tollen's reagent.

Catechol



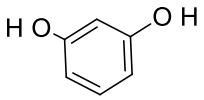
Physical properties:

Molecular formula (M.F) $C_6H_6O_2$, colorless crystals, soluble in water, alcohol, m.p. 105 °C.

Chemical properties

1- With ferric chloride, it gives green color.
2- With bromine water, it gives deep red color.
3- Not gives azo dye test
4- With phthaline reaction, it gives blue color.
5- It reduces Fehling's solution and Tollen's reagent.

Resorcinol



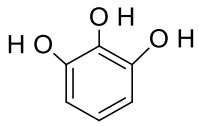
Physical properties:

Molecular formula (M.F) $C_6H_6O_2$, coluble in water, m.p. 110 °C.

Chemical properties

1- With ferric chloride, it gives deep violet color.
2- With bromine water, it gives white ppt. dissolved in excess.
3- Gives with azo dye test red ppt.
4- With phthaline reaction, it gives reddish fluorescence solution.
5- It reduces Fehling's solution and Tollen's reagent.
6- With conc. Nitric acid it gives red color.

Pyrogallol



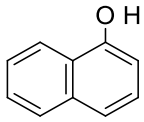
Physical properties:

Molecular formula (M.F) $C_6H_6O_3$, it is plates or needles, soluble in water and alcohol, m.p. 132 °C.

Chemical properties

1- With ferric chloride, it gives reddish color and in very dil. solution of NaOH it gives violet color.
2- Solution of pyrogallol + glycerol (solution) + conc. H_2SO_4 it gives reddish violet color.
3- Solution of pyrogallol + HCHO + conc. HCl gives white ppt. turns to pink.
4- It reduces Fehling's solution and Tollen's reagent.

α- naphthol



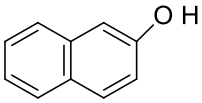
Physical properties:

Molecular formula (M.F) $C_{10}H_8O$, soluble in alcohol, ether and benzene, mp.94 °C.

Chemical properties

1- With ferric chloride, it gives greenish color at first rapidly turns violet on adding excess.
2- With bromine water, it gives de colorization occurs but no ppt.
3- Gives with azo dye test brownish red ppt.
4- With phthaline reaction, it gives green solution.

β- naphthol



Physical properties:

Molecular formula (M.F) $C_{10}H_8O$, soluble in alcohol, ether and benzene, m.p. 123°C.

Reactions of β- naphthol

1- With ferric chloride, it gives greenish color at first rapidly turns violet on adding excess.
2- With bromine water, no ppt. is formed but the color disappears.
3- Gives with azo dye test scarlet red ppt.
4- With phthaline reaction, it gives faint blue with slight fluorescence.

Experiment 2: Identification and reactions of alcohols and phenols

Data and Results

Name:

Sec.No.

Date:

Physical properties

Solubility: ……

Color: ……

Shape: ……

Chemical properties

Exp.	Obs.	Res.

Unknown is

Experiment 3: Identifications of Aldehydes and ketones.

Objective:

To identify aldehydes and ketones by using properties and reactions.

Introduction:

Aldehydes and ketones share the carbonyl functional group which features carbon doubly bonded to oxygen. In the case of In the case of aldehydes there is at least one hydrogen atom bonded to the carbonyl carbon, the other attachment maybe a carbon or hydrogen. In ketones there are two carbon atoms bonded to the carbonyl carbon and no hydrogen.In all cases the carbon(s) that are attached to the carbonyl group may be aliphatic (not part of an aromatic ring) or aromatic (part of an aromatic ring). Since they share the carbonyl group, aldehydes and ketones share much of their chemistry, but they are different enough to be considered different classes ofcompounds. This situation is similar to that of alcohols and phenols which both share the -OH group.

Procedure:

2, 4 Dinitro phenyl hydrazine test

2, 4-dinitrophenylhydrazine can be used to qualitatively detect an aldehydeor ketone functional group. A positive test is signaled by a yellow/red precipitate, known as adinitrophenylhydrazone.

$$RR'C = O + C_6H_3(NO_2)_2NHNH_2 \longrightarrow C_6H_3(NO_2)_2NHN = CRR' + H_2O$$

This reaction can be described as a condensation reaction, with two molecules joining together with loss of water. It is also called addition-elimination reaction: nucleophilic addition of $–NH_2$ group to C=O carbonyl group, followed by removal of H_2O molecule.

Method 1: *For carbonyl compounds miscible with water.*

1- Prepare a solution of 2,4-dinitro phenyl hydrazine in dilute HCl by dissolving a pinch of it in 10 ml dilute HCl by heating.
2- Cool this solution and add to it 0.5 ml of substance, and shake vigorously.
3- The colored 2,4-dinitro phenylhydrazone separates out almost immediately.
4- Filter it, wash with water, recrystallize from alcohol, dry and determine the melting point.

Method 2: *For carbonyl compounds immiscible with water*

1- To a pinch of 2, 4-dinitro phenyl hydrazine in dry test tube add 2 ml of alcohol and 1 ml conc. H_2SO_4.
2- Shake and heat on water bath to dissolve the solid.
3- To this hot solution add 0.5 ml of substance. allow it to stand until a colored precipitate is formed.
4- Wash, filter, recrystallized, and determine the melting point.

<u>To differentiate between aldehydes and ketones</u>

1-Chromic Acid Test

Aldehydes are oxidized by chromic acid, ketones are not. When an aldehyde is oxidized by orange brown chromic acid the chromic acid is reduced to Cr^{3+}, which is green. Consequently, chromic acid can distinguish between aldehydes and ketones. It is also true for other functional groups; primary and secondary alcohols for example, can be oxidized by chromic acid, causing the formation of a green color.

$$3\ RCHO \xrightarrow[3H_2SO_4]{2\ H_2CrO_4} 3\ RCOOH + 3Cr_2(SO_4)_3 + H_2O$$

2- Tollens' Test

Aldehydes are also oxidized by Tollens' reagent, a substance that contains Ag^{+1}. The silver ion is concomitantly, reduced to metallic silver. Silver ion is a weak oxidizing agent; aldehydes are very easily oxidized and are essentially unique in being able to reduce silver ion to silver metal.

Aldehydes (RCHO) , (ArCHO)

Formaldehyde

HCHO

Physical properties:

Molecular formula (M.F) is CH_2O, colorless liquid, It has characteristic pungent odor.

Chemical properties:

1- **Schiff's reagent test.** Add 2 ml of Schiff's reagent to 2 ml cold aldehyde solution shake vigorously and allow standing for two minutes – a deep – violet color which indicates the presence of aldehydic group.

2- In dry test tube put 2 ml of formaldehyde with few crystals of resorcinol then add 2 ml conc. H_2SO_4 carefully from the side of the tube. The red ring formed and white ppt. in aqueous layer turns to violet red.

3- **It is reduces Fehling's reagent**. Add 1ml of formaldehyde solution to Fehling's solution (1ml of Fehling A +1ml of Fehling B) and heating the solution notice the blue color convert to red color

$$HCHO + 2CUO \longrightarrow HCOOH + CU_2O$$

4- Add 1% phenyl hydrazine + 2 ml of formaldehyde + few drops of sodium nitroprusside solution in excess of NaOH. Blue color will appear then turns to green then red then brown.
5- Add diluted formaldehyde solution + 1% phenyl hydrazine + 5% (2) ml pot. ferricyanide +conc. HCl it gives a rose red color.
6- It gives 2, 4-dinitrophenyl hydrazine m.p 166 °C.

Acetaldehyde

CH_3CHO

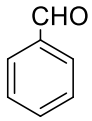
Physical properties:

Molecular formula (M.F) is C_2H_4O, colorless liquid, it has pungent, fruity odor, b.p 20 °C, miscible with water, alcohol and ether.

Chemical properties:

1- Give violet color with Schiff's reagent.
2- 2 ml aqueous sodium nitroprusside, 5 drops of NaOH and 2 ml of acetaldehyde gives a deep win red color.
3- It is Responds to iodoform test
4- Boiling 2 ml of solution with 2 ml (20%) KOH give yellow ppt.
5- It reduces Fehling's solution and Tollen's reagents.
6- Formed white crystals by reaction with sodium bisulphate.
7- It is give 2,4- dinitrophenylhydrazone, m.p 168 °C.

Benzaldehyde

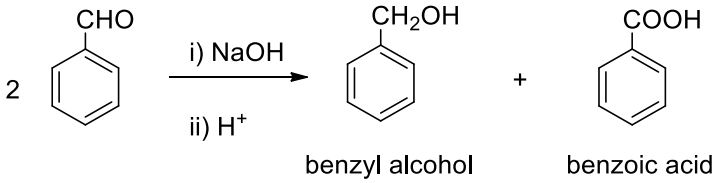


Physical properties:

Molecular formula (M.F) C_7H_6O, colorless liquid, immiscible with water.

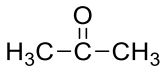
Chemical properties:

1- It gives Schiff's reagent test.
2- It reduces Fehling's solution reagents.
3- It gives violet color with $FeCl_3$.
4- It decolorize alkaline $KMnO_4$ solution by heating, then acidification with HCl, salicylic acid is formed as white ppt.
5- Cannizaro's reaction. Boiling 1 ml of benzaldehyde with 2 ml of NaOH and cooling the solution then acidification with HCl white precipitate of benzoic acid is formed.



Ketones (RCOR) , (ArCOR)

Acetone



Physical properties:

Molecular formula (M.F) is C_3H_6O, colorless liquid, it has characteristic pleasant smell, miscible with water, alcohol, and ether.

Chemical properties:

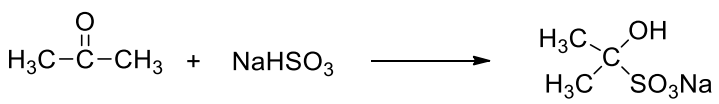
1- **Colors test**.
   - To 1 ml of sodium nitroprusside with 0.5 ml of NaOH add 1 ml of acetone and notice appearance of red color.
   - Add 1 ml of acetone to 1 ml of sodium nitroprusside with 0.5 ml of pyridine and notice appearance of blue color.
   - To 1 ml of acetone with 0.5 ml of NaOH add 0.5 gm of m-dinitrobenzene and notice appearance of red color

2- **Iodoform test.** Add 3-4 drops of iodine solution and then NaOH solution drop by drop to the sample and warm the brown color of iodine disappear and a yellow ppt. is formed.

$$H_3C-\overset{\overset{\large O}{\|}}{C}-CH_3 + 3I_2 + 4NaOH \longrightarrow CHI_3 + CH_3COONa + 3\,H_2O$$

3- Ding`s test. Addition of 2 ml of acetone and 2 ml of acidic solution of mercury sulphate then heating on water bath produce heavy white precipitate.

4- To 2 ml of standard sodium bisulphite add few drops of acetone. White crystals are formed.

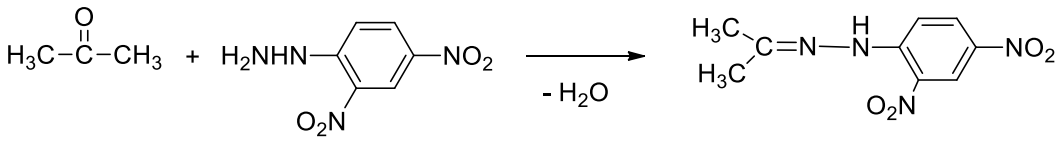


5- **Oxidation test.** Addition of 1 ml of acetone to 2 ml of acidic $KMnO_4$ solution and heating lead to disappear of violet color of permanganate.

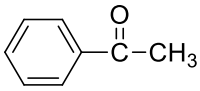
$$H_3C-\overset{\overset{\large O}{\|}}{C}-CH_3 \xrightarrow[H_2O]{KMnO_4} CH_3COOH + MnO_2$$

6- 2, 4- Dinitrophenylhydrazine test**.**

To 3 ml of alcoholic solution of 2,4-dinitrophenylhydrazine add 1 ml of acetone and heating the mixture in water bath and notice formation of yellow precipitate.



Acetophenone



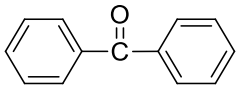
Physical properties:

Molecular formula (M.F) is C_8H_8O, colorless liquid, it has characteristic sweet smell, It is sparingly soluble in water, soluble in alcohol, ether, and chloroform.

Chemical properties

1- It responds to iodoform test.
2- Orange color is produced on dissolving in conc. H_2SO_4.
3- With 2,4- dinitrophenyl hydrazine it gives phenylhydrazone of acetophenone m.p 250 ^oC

Benzophenone



Physical properties:

Molecular formula (M.F) is $C_{13}H_{10}O$, m.p 48.5 °C, colorless solid, insoluble in water, but soluble in alcohol and ether.

Chemical properties

1- Dissolving benzophenone in conc. H_2SO_4 gives yellow solution.
2- Fusing the substance with sodium metal produces blue color.
3- It gives phenyl hydrazone with 2, 4-dinitro phenyl hydrazine.
4- Boiling the solid with NaOH gives oil drops.

Experiment2: Identifications of Aldehydes and Ketones.

Data and Results

Name:

Sec.No.

Date:

Physical properties

Solubility: ……..

Color: …….

Shape: ……

Chemical properties

Experiment	Observation	Result

Unknown is

Experiment 4: Identifications of carboxylic acids and esters.

Objective:

To study properties and reactions of carboxylic acids and esters

Introduction:

The functional group of a carboxylic acid is a carboxyl group. The general formula for an aliphatic carboxylic acid is RCOOH and for an aromatic carboxylic acid is ArCOOH. Carboxylic acids have significantly higher boiling points than other types of organic compounds of comparable molecular weight. They are polar compounds and form very strong intermolecular hydrogen bonds. Carboxylic acids are more soluble in water than alcohols, ethers, aldehydes, and ketones of comparable molecular weight. They form hydrogen bonds with water molecules through both their C=O and OH groups. They are dissolving in Na_2CO_3 with evaluation of CO_2; they are also dissolving in NaOH. Carboxylic acids are divided into two categories, aliphatic and aromatic carboxylic acids.

To use carboxylic acids neutral solution (n. solution) must be prepared first.

To the solid, add aqueous solution of ammonia (excess). Boil the solution until all ammonia odor evolved. Cool.

$$RCOOH + NH_4OH \longrightarrow RCOONH_4 + NH_3$$

Formic acid

HCOOH

Physical properties:

Molecular formula (M.F) CH_2O_2, colorless liquid, miscible with water, alcohol and ether, b.p.100 °C.

Chemical properties

1- It reduces Fehling's solution and Tollen's reagent.
2- It is decolorized $KMnO_4$
3- With $FeCl_3$: neutral solution of acid gives red color which converted to brown by boiling.
4- Ester formation: to 1 ml of acid add 1 ml of ethyl alcohol and 1 ml of conc. H_2SO_4 in attest tube, heat in water bath, and then pour to Na_2CO_3 solution. The characteristic odor of ethyl format is evolved.

$$HCOOH + C_2H_5OH \longrightarrow HCOOC_2H_5 + H_2O$$

Acetic acid

CH_3COOH

Physical properties:

Molecular formula (M.F) $C_2H_4O_2$, colorless viscous liquid, miscible with water, alcohol and ether, b.p 122 °C.

Chemical properties:

1- It does not reduce Fehling's solution and Tollen's reagent.
2- With $FeCl_3$: neutral solution of acid gives red color which converted to brown by boiling.

3- Ester formation: to 1 ml of acid add 1 ml of ethyl alcohol and 1 ml of conc. H_2SO_4 in a test tube, heat in water bath, and then pour to Na_2CO_3 solution. The characteristic odor of ethyl acetate is evolved.

$$CH_3COOH + C_2H_5OH \longrightarrow CH_3COOC_2H_5 + H_2O$$

Oxalic acid

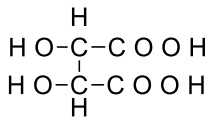
Physical properties:

Molecular formula (M.F) $C_2H_2O_4$, colorless crystalline solid, m.p. 100 °C, soluble in water and alcohol.

Chemicalproperties

1- Flaming test. When the acid or its salt is heated on a piece of porcelain it is decomposed with little or no charring.
2- When the acid is heated with conc. H_2SO_4 it is decomposed into CO and CO_2 with no charring.
3- n. solution + $CaCl_2$: A white ppt. of Ca oxalate is separated immediately on cold which is soluble in mineral acids.
4- n. solution + $AgNO_3$: gives white ppt. of Ag oxalate.
5- When heated a few drops of dil. $KMnO_4$ soln. and the acidified sol. Of oxalate the color is discharged.

Tartaric acid



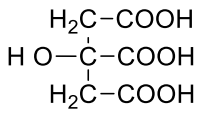
Physical properties:

Molecular formula (M.F) $C_4H_6O_6$, colorless crystalline solid, m.p. 167°C, soluble in cold water and alcohol.

Chemical properties

1- It gives Acidity test +ve.
2- With conc. H_2SO_4: when the solid is heated with conc. H_2SO_4 charring is occur with the evaluation of odor of burnet sugar.
3- Neutral solution + $CaCl_2$: it gives white ppt. after shaking from calcium tartrate, which is soluble in mineral acids.
4- n. solution + $AgNO_3$: it gives Ag mirror after heating in water bath.
5- $KMnO_4$ + neutral solution: by heating in presence of dil. H_2SO_4 a de colorization of color is occurring.

Citric acid



Physical properties:

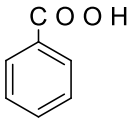
Molecular formula (M.F) $C_6H_8O_7$, colorless crystalline solid, m.p 100 °C, soluble in cold water.

Chemical properties:

1- With conc. H_2SO_4: heating the solid with conc. H_2SO_4 gives yellow color.
2- It gives Acidity test +ve.

3- with $CaCl_2$ solution: it gives white ppt. after boiling
4- Deng's test: 1 ml of $HgSO_4$solution is added to 5 ml of neutral solution. Then heat to boiling and then add 1-2 drops of 2%$KMnO_4$ where de colorization occurs and a heavy whit ppt appear.

Benzoic acid



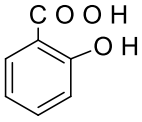
Physical properties:

Molecular formula (M.F) $C_7H_6O_2$, white crystalline solid, mp. 121°C, insoluble in cold water, but soluble by boiling and re precipitated by cooling, soluble readily in alcohol.

Chemical properties

1- It gives Acidity test +ve.
2- n. solution +neutral $FeCl_3$: gives buff ppt.
3- It gives ester test with ethyl alcohol.
4- It gives soda lime test: In dry test tube, place a layer of soda lime powder, then layer of benzoic acid and then another layer of soda lime. Then heat and note the odor of benzene.

Salicylic acid



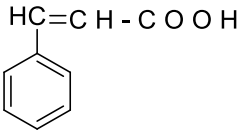
Physical properties:

Molecular formula (M.F) $C_7H_6O_3$, colorless solid, m.p. 159°C, soluble in alcohol, ether, to some extent, it is soluble in water owing to the presence of OH group.

Chemical properties

1- It gives Acidity test +ve.
2- Neutral solution + $FeCl_3$: gives violet color.
3- It gives ester test with methyl alcohol.
4- It gives soda lime test: In dry test tube, place a layer of soda lime powder, then layer of benzoic acid and then another layer of soda lime. Then heat and note the odor of phenol.

Cinnamic acid



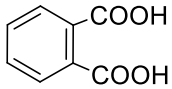
Physical properties:

Molecular formula (M.F) $C_9H_8O_2$, white monoclinic crystals, mp. 133°C, sparingly soluble in water but soluble in alcohol.

Chemical properties

1- It gives Acidity test +ve.
2- Neutral solution +n. $FeCl_3$: gives buff ppt.
3- It gives ester test with ethyl alcohol.
4- Unsaturation test: Dissolve 2 gm. Of acid in Na_2CO_3 solution (5 ml) adds 1% aqueous $KMnO_4$ solution drop wise immediate decolonization is observed.
5- Dissolve 2 gm of acid or its salt in Na_2CO_3 solution (5 ml) add Br water drop wise and note the separation of bromostyrene as color oil.

Phthalic acid



Physical properties:

Molecular formula (M.F) $C_8H_7O_4$, white solid, m.p. 191°C, it forms phthalic anhydride which dissolves in water, readily soluble in hot water and organic solvents.

Chemical properties

1- It gives Acidity test +ve.
2- Neutral solution +n. $FeCl_3$: gives buff ppt.
3- It gives fluorescein reactions with resorcinol where it gives red solution with instance green fluorescein.
4- It gives phthaline reaction with phenol where it gives bright red color.

ESTERS

RCOOR

Esters are chemical compounds consisting of a carbonyl adjacent to an ether linkage. They are derived by reaction of an acid with a hydroxyl compounds such as an alcohol or phenol. The characteristic test of esters is

Hydroxamic test

Treat few drops of the ester with cold alcoholic solution of hydroxyl amine hydrochloride. Add few drops of alcoholic KOH until the mixture is alkaline. Heat the mixture just to boiling point; acidify the mixture with dil. HCl. Add 1-2 drops of $FeCl_3$. A violet or reddish or reddish wine colorization is occurring.

$$RCOOR' + NH_2OH \longrightarrow RCONHOH + R'OH$$

Experiment 4: Identifications of carboxylic acids and esters

Data and Results

Name:

Sec.No.

Date:

Physical properties

Solubility: ……

Color: ……

Shape: ……

Chemical properties

Exp.	Obs.	Res.

Unknown is

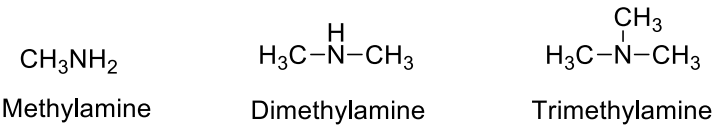
Experiment 5: Identifications of amines.

Objective:

To study properties and reactions of amines

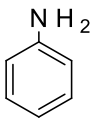
Introduction:

Amines are derivatives of ammonia, they are considered as important type of organic base found in nature. Consider as if substituted ammonia: RNH_2, R_2NH, and R_3N. R can be either aromatic or aliphatic . Amines form hydrogen bonds but not as strongly as alcohols. Nitrogen is less electronegative than oxygen. Tertiary amines cannot hydrogen bond to each other. Amines have boiling points between alkanes and alcohols. Tertiary amines boil lower, then, 10 or 20 of similar molecular weight. All amines can form hydrogen bonds with water. Amines up to 6 carbons long are water soluble due to this hydrogen bonding. Water solubility decreases as the length of the hydrocarbon portion of the molecule increases. Amines are classified by the number of carbons directly bonded to the nitrogen atom: A primary amine has one (RNH_2= 1°); A secondary amine has two (R_2NH = 2°); A tertiary amine has three (R_3N = 3°)



Primary aromatic amines

Aniline

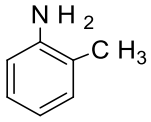


Physical properties: Molecular formula (M.F) C_6H_7N, colorless to yellow liquid, b.p.184° C, sparingly soluble in water, When aniline is exposed to air it is darkness in color and become black.

Chemical properties:

1- Azo dye test: Dissolve aniline with 2.5 ml of conc. HCl, cool and dilute with about 3 ml H_2O, cool in ice bath and add with shaking 2 ml of diluted $NaNO_2$ solution. Then cool and add this diazotized solution to cooled solution of β-naphthol dissolved in 10 % NaOH, a scarlet ppt. is appeared.
2- Add 2drops of aniline to dil. H_2SO_4, then add $K_2Cr_2O_7$solution, a green blue or black ppt. is formed due to oxidation.
3- With $FeCl_3$, solution of aniline gives pale green color.
4- Shake 2 drops of aniline with 5 ml of water; add few drops of NaOCl solution, a purple color is formed which soon turns brown.

_o_-toludine



Physical properties:

Molecular formula (M.F) C_7H_9N, liquid, b.p. 199° C, sparinglysoluble in water but soluble in mineralacids.

Chemical properties:

1- In azo dye test: it gives orange or red ppt.
2- With $FeCl_3$, gives greenish color.
3- Shake 2 drops of o-toludine with 5 ml of alcohol; add few drops of NaOCl solution, a brown color is formed.

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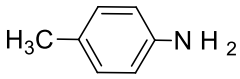
p-toludine



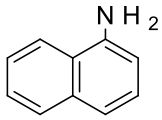
Physical properties:

Molecular formula (M.F) C_7H_9N, solid, mp.45° C, sparingly soluble in water but soluble in mineral acids.

Chemical properties:

1- In azo dye test: it gives orange or red ppt.
2- With $FeCl_3$, gives brown color.
3- Shake 2 drops of p-toludine with 5 ml of alcohol
4- Add few drops of NaOCl solution, a yellow color is formed.

α – Naphthyl amine



Physical properties:

Molecular formula (M.F) $C_{10}H_9N$, colorless solid, When exposed to air it becomes violet, mp.47 - 50 ° C, sparingly soluble in water but soluble in mineral acids.

Chemical properties:

1- In azo dye test: it gives orange or red ppt.
2- With $FeCl_3$, gives blue color.
3- conc. HCl +H_2O + $FeSO_4$ it gives green ppt.

Aliphatic amino compounds

Glycine

$NH_2 – CH_2$ - COOH

Physical properties:

Molecular formula (M.F) $C_2H_5NO_2$, colorless crystalline solid, soluble in water, it exhibited acidic and basic characters.

Reactions of glycine

1- It is gives nitration test –ve
2- Gives weak acidity.
3- Solution + cupper acetate gives blue color
4- Solution + $FeCl_3$gives red color.

Experiment No. 5: Identifications and reactions of amines.

Data and Results

Name:

Sec.No.

Date:

Physical properties

Solubility: ……

Color: ……

Shape: ……

Chemical properties

Exp.	Obs.	Res.

Unknown is

Simple Scheme for differentiation between solid compounds

Exp.	Obs.	Res.
Acidity test	+ve	It is an acid
Nitration test	+ ve	It is aromatic acid
n-solution +$FeCl_3$	Violet color	It is salicylic acid
	Buff ppt.	May be benzoic acid or cinnamic acid or phthalic acid
Nitration test	-ve	It is aliphatic acid May be oxalic acid, tartaric acid or citric acid **(differentiate between them)**
If Acidity test - ve it is not acid		
Solution +$FeCl_3$	Dark color	It is phenolic compound
Solubility in water	Soluble in water	May be catechol, pyrogalol, hydroquinol, resorcinol **(differentiate between them)**
	In soluble in water	May be α- naphthol or β-naphthol **(differentiate between them)**
If solution +$FeCl_3$ gives - ve it is not phenol		

Test of ketones 2,4- di nitro phenyl hydrazine test	Orange or red crystals	It is benzophenone
Test of hydrocarbon (picrate test)	Yellow crystals	It is naphthalene
	Red crystals	It is anthracene

Simple Scheme for differentiation between miscible liquids

Exp.	**Obs.**	**Res.**
Acidity test	+ ve	It is acid May be acetic or formic acid **(differentiate between them)**
If acidity test - ve it is not an acid		
Schiff's test	Pink color	It is aldehyde may be formaldehyde or acetaldehyde **(differentiate between them)**
If Schiff's test – ve it is not aldehyde		
Iodoform test	Yellow crystals on cold	It is acetone
	Yellow crystals on hot	It is ethyl alcohol

If iodoform test – ve		
Esterification with salicylic acid	Ester odor	It is methyl alcohol
If esterification with salicylic acid - ve		
Borax test	Red color	glycerol

Simple Scheme for differentiation between immiscible liquids

Exp.	**Obs.**	**Res.**
Schiff's test	Pink color	It is Salicyaldehyde
If Schiff's test – ve not aldehyde		
Test of ketones 2,4- di nitro phenyl hydrazine test	Orange or red crystals	It is acetophenone
Solution + $FeCl_3$	Dark color	It is phenol
If Solution + $FeCl_3$	**- Ve it is not phenol**	

Freezing test	Gives solid	It is benzene
	Ve-	It is toluene

Simple Scheme for differentiation between solid compounds

Exp.	**Obs.**	**Res.**
Nitration test	- ve	Aliphatic amine (glycine)
	+ ve	Aromatic amine
Azo dye test	+ve	Primary aromatic amine May be Anthranilic acid or *p*-amino benzoic acid or α-naphthyl amine or *p*-toludine
Acidity test	+ve	It is Anthranilic acid or *p*-amino benzoic acid **(differentiate between them)**
Acidity test	-ve	It is not Anthranilic acid or *p*-amino benzoic acid
Solution + $FeCl_3$	Blue color Yellow color	α- naphthyl amine *p*-toludine
If Azo dye test – ve not Primary aromatic amine		

Reaction with nitrous acid	Yellow color	It is di phenyl amine

Simple Scheme for differentiation between liquids.

Exp.	**Obs.**	**Res.**
Azo dye test	**Ve +**	Primary aromatic amine May be Aniline or *o*-toluidine
Solution +$FeCl_3$	brown color bale green color	*O*-toluidine Aniline
If Azo dye test – ve it is not Primary aromatic amine		
liquid +HNO_3	Deep green color	(N,N) Di methyl aniline

After identification of each compound you must perform confirmation tests for each compound.