

King Abdul Aziz University Faculty of Science Department of Chemistry

Fundamental Organic Chemistry

Chem. 230 (Lab Manual)



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9. A lab coat, gloves should be worn during lab experiments.

10. If a chemical should splash in your eyes or on your skin,

quickly wash with water for at least 20 minutes.

8. Long hair must be tied back, and clothing must be secured

7. Contact lenses may be not being worn in the lab.

and shoes must completely cover the foot.

- 6. Keep hands from face, eyes, mouth, and body while using chemicals or lab equipment and wash your hands with soap and water after performing all experiments.
- 5. Read carefully instructions of equipment before use .

4. Always work in a well-ventilated area.

- 3. Do not drink or eat food and gum in the lab.
- 2. In the lab area do not touch any equipment, chemicals, or other

materials.

1. Never work alone in the lab.







- 11. All chemicals in the lab are dangerous because of that do not taste, or smell any chemicals.
- 12. Never return unused chemicals to their original container.
- 13. Never remove chemicals or other materials from the lab area.
- 14. Examine glassware before each use and never use cracked or dirty glassware.
- 15. Do not inter hot glassware in cold water.

16. Do not operate a hot plate by yourself and take care that hair, clothing, and hands are a safe distance from the hot plate at all times.

17. Never look into a container that is being heated.







Flammable substances



Explosive substances



harmful substances



oxidizing substances



Corrosive substances





First aid in lab

Chemical or causes	neutralizing wash or treatment	
Acids	2M ammonium carbonate	
Alkalis	M acetic acid	
Bromine	2M ammonia	
Hydro fluoric acid	As for acids, then hospital treatment	
Phenol	Ethanol, then hospital treatment	
Phosphorus	0.1 M copper sulphate	
Sodium	Ethanol on a cotton wool pad	
Poison swallowed	Plenty of water at once if the patient is conscious	
Corrosive poisons	Plenty calcium hydroxide(lime water) as soon as	
possible		
Gassing	In case of gassing acknowledge of artificial respiration	
method		
	May be required ,and the correct person(s)to summon	
should	be listed in the emergency procedure	
Cuts	The wound is washed well with cold water, inspected,	
	And any foreign bodies removed. Then an intersected	
	cream of certrimide is applied and the affected part	
	protected with a suitable dressing	
Severe bleeding	Firm pressure round the wound, a cover with a pad	
	And firm bandaging	
Electrical mishaps	These are usually avoidable by the correct installation	
	of equipment	















SEPARATION TECHNIQUES

Introduction:

Apure substance contains only one kind of molecule; an impure substance is a mixture of molecules. When the different molecules of a mixture each behave in a different way under the conditions of some procedures, the procedure can result in a separation of the different molecules. The theory of each of the separation procedures described in this section is presented from the point of view of the different behavior that can be expected from different molecules under the experimental conditions.

In this part we will discuss the different types of techniques which used for separation of organic compounds.

Technique 1

Filtration:

Filtration is a technique used for two main purposes. The first is to remove solid impurities from a liquid or a solution. The second is to collect a solid product from the solution from which it was precipitated or crystallized. Two different kinds of filtration are in general use: gravity filtration and vacuum (or suction) filtration.



🖶 Gravity filtration:

Apiece of filter paper and conical glass funnel to support it are all that are required for gravity filtration see **fig.** (1). In order to maximize the rate at which the liquid flows through the filter paper, the paper should be folded as indicated in the folded paper is then dropped into the funnel.



Fig. (1): Gravity filtration

Steps of folded filter paper:

<u>Step 1</u>: Fold the paper in half ,then in quarters ,creasing the folds as you proceed. However, do not crease the very center of paper (the point),which might become weakened.



<u>Step 2</u>: Open the quarters to a half- sized piece, and then fold the edges in to the centerfold







<u>Step 3</u>: Open the paper again to a half-sized piece, then accordion-pleat, using the existing fold lines as guides. (again, do not crease the center of the paper.)



<u>Step 4</u>: crease the folds (except at the point), then open the filter paper and place it in an appropriately sized funnel.



4 Vacuum or suction filtration:

Vacuum, or suction, filtration is more rapid than gravity filtration. In vacuum or suction filtration, a partial vacuum is created below the filter, causing the air pressure on the surface of the liquid to increase the rate of flow through the filter paper. A typical apparatus is



Fig. (2): Suction filtration

Illustrated in **fig.** (2) .A circle of filter paper just large enough to cover the holes in the bottom of the Hirsch or Buchner funnel should be used.





Crystallization:

When a solid organic compound is prepared in the laboratory or isolated from some natural source, such as leaves, it is almost always impure. A simple technique for the purification of such a solid compound is **crystallization**. The compound is first dissolved in a minimum amount of hot solvent. If insoluble impurities are present, the hot solution is filtered. If the solution is contaminated with colored impurities, it may be treated with decolorizing charcoal and filtered.

Crystallization is not the same as precipitation. Precipitation is the *rapid* formation of solid material, while crystallization is the *slow* formation of a crystalline solid. If a hot saturated solution is cooled too quickly, then the compound may precipitate instead of crystallizing. A precipitated solute may contain many impurities trapped in the rapidly formed crystal structure. On the other hand, when a solution is allowed to crystallize slowly, impurities tend to be excluded from the crystal structure because the molecules in the crystal lattice are in equilibrium with the molecules in solution.

Solvents for Crystallization:

The ideal solvent for the crystallization of a particular compound is one that:

- (1) Does not react with the compound .
- (2) Boils at a temperature that is below the compound's melting point .
- (3) Dissolves a moderately large amount of the compound on hot .



- (4) Dissolves only a small amount of compound on cold.
- (5) Is moderately volatile so that the final crystals can be dried readily .
- (6) Is nontoxic, nonflammable, and inexpensive.

In addition, impurities should be either highly insoluble in the solvent else highly soluble, so that they remain in solution during the crystallization.

General guidelines for predicting solubilities based upon the structures of organic compounds do exist. For example, an *alcohol*, a compound containing the hydroxyl (-OH) group as its functional group, may be soluble in water because it can form hydrogen bonds with water molecules. If an alcohol's molecules are largely hydrocarbon, the alcohol may be insoluble in water, but will probably be soluble in other alcohols, such as ethanol (CH₃CH₂OH). *Carboxylic acids* (compounds containing – CO₂H groups) and *amines* (compounds containing NH₂, N H, or N groups) also can form hydrogen bonds an are also generally soluble in polar solvents such as alcohols.

Compounds that are largely hydrocarbon in structure are not soluble in polar solvents because C - C and C - H bonds are not polar. For these compounds, we would choose a non polar solvent – for example, low-boiling petroleum ether, which is a mixture of alkanes such as pentane, $CH_3(CH_2)_3CH_3$, and hexane, $CH_3(CH_2)_4CH_3$. Thus, in choosing crystallization solvents, chemists generally follow the rule of thumb: like dissolves like.





solvent	Bp(°C)	Useful for	
water	100	Salts amides some	
		carboxylic acids	
methanol	65	General use	
ethanol	78	General use	
acetone	56	General use	
ethylacetate	77	General use	
dichloromethane	40	General use	
Diethyl ether	35	General use	
Chloroform	61	General use	
toluene	111	Aromatic compound	
benzene	80	Aromatic compound	
Hexane(petroleum	69	hydrocarbons	
ether)			

 Table 1: Some common crystallization solvents

Ideally, a compound to be crystallized should be soluble in the hot solvent, but insoluble in the cold solvent. When a proper solvent cannot be found, a chemist may use a **solvent pair**.





Steps in Crystallization:

1) Dissolving the compound. The first step in crystallization is dissolving the compound in a *minimum amount* of the appropriate hot solvent in an Erlenmeyer flask. An Erlenmeyer flask is used instead of a beaker or other container for several reasons. The solution is less likely to splash out and dust is less likely to get in.

Safety Note:) Toxic solvents should be heated only in a hood.

2) *Filtering insoluble impurities*. Filtering a hot, saturated solution inevitably results in cooling and in evaporation of some of the solvent. Therefore, a premature crystallization of the compound on the filter paper and in the funnel may be observed. A few precautions can minimize this premature crystallization.

3) Crystallizing the compound. Cover the flask containing the hot, saturated solution with a watch glass or inverted beaker to prevent solvent evaporation and dust contamination.

4) Isolation the crystals. Crystals are separated from their mother liquor by filtration.

Suggested experiments:

Crystallize one of the following compounds from the indicated solvents:

1) benzoic acid from water.

2) Acetanilide from water . 3) Naphthalene from 95% ethanol .







Sublimation:

Sublimation is a process whereby a solid is purified by vaporizing and condensing it without its going through an intermediate liquid state. Solid compounds that evaporate (that is, pass directly from the solid phase to the gaseous phase) are rather rare; solid CO_2 (dry ice) is a familiar example of such a compound. Even though both solids and liquids have vapor pressures at any given temperature, most solids have very low vapor pressures.

Sublimation can be used to purify some solids just as distillation can be used to purify liquids. In sublimation, nonvolatile solid impurities remain behind when the sample evaporates, and condensation of the vapor yields the pure solid compound. Sublimation has the advantages of being fast and clean because no solvent is used. Unfortunately, most solid compounds have vapor pressures too low for purification in this fashion. Also, sublimation is successful only if the impurities have much lower vapor pressures than that of the substance being purified.

🖶 Atmospheric Sublimation:

The atmospheric sublimation apparatus shown can be used only for a solid with a relatively high vapor pressure. Place the sample in a filter flask equipped with a water-cooled cold finger or a test tube filled with ice. Warm the flask on a hot plate or in a water bath, taking care not to melt the solid. Crystal growth on the test tube (and on the cooler flask sides) soon occurs. Periodically, cool the



apparatus, remove the cold finger or test tube carefully, and scrape the sublimed crystals into a tared container or onto a watch glass. Determine the melting point of the sample with a sealed capillary **fig. (3)**.



Fig.(3): atmospheric sublimation

4 Vacuum Sublimation:

For a vacuum sublimation, choose a side-arm test tube to hold the sample, and then insert a smaller test tube fitted into a neoprene adapter or large-holed stopper. Place ice in the inner tube, connect the side arm with heavy-walled rubber tubing to a trap (preferably chilled in an ice bath) and then to the aspirator. For an illustration of the trap and a discussion of using the aspirator.

Turn on the aspirator and press the test tubes together to obtain a seal. Then, warm or heat the outer test tube. When the sublimation is complete, allow the apparatus to cool before breaking the vacuum. Take care in breaking the



vacuum and removing the tube so that the sublimed crystals remain on the tube until they are scraped off. Fig. (4).



Fig.(4): Vacuum sublimation



- 1) Perform an atmospheric sublimation of hexachloroethane .
- 2) Vacuum sublime naphthalene or caffeine.

Problems:

Which of the following compounds could be subjected to sublimation at

atmospheric pressure?

- (a) Compound A:vapor pressure at its melting point=770 mmHg
- (b) Compound B:vapor pressure at its melting point=400 mmHg
- (c) Compound C:vapor pressure at its melting point=10 mmHg



Extraction Using a Separatory Funnel:

Extraction is the separation of a substance from one phase by another phase. The term is usually used to describe removal of a desired compound from a solid or liquid mixture by a solvent. In a coffee pot, caffeine and other compounds are extracted from the ground coffee beans by hot water. Vanilla extract is made by extracting the compound vanillin from vanilla beans.

In the laboratory, several types of extraction techniques have been developed. The most common of these is *liquid-liquid extraction*, or simply "extraction." Extraction is often used as one of the steps in isolation a product of an organic reaction. After an organic reaction has been carried out, the reaction mixture usually consists of the reaction solvent and inorganic compounds, as well as organic products and by-products. In most cases, water is added to the reaction mixture to dissolve the inorganic compounds. The organic compounds are then separated from the aqueous mixture by extraction with an organic solvent that is immiscible with water. The organic compounds dissolve in the extraction solvent while the inorganic impurities remain dissolved in the water.

The most commonly used device to separate the two immiscible solutions in an extraction procedure is the **separatory funnel.** Typically the aqueous mixture to be extracted is poured into the funnel first, and then the appropriate extraction solvent is added. The mixture is shaken to mix the extraction solvent and the aqueous mixture, and then is set aside for a minute or two until the aqueous and organic layers have separated. The stopcock at the bottom of the



separatory funnel allows the bottom layer to be drained into a flask and makes possible the separation of the two layers **fig.(5)**. The result (ideally) is two separate solutions: an organic solution (organic compounds dissolved in the organic extraction solvent), and an inorganic solution (inorganic compounds dissolved in water). Unfortunately, often the water layer still contains some dissolved organic material. For this reason, the water layer is usually extracted one or two more times with fresh solvent to remove more of the organic compound.

After one or more extractions and separations, the combined organic solutions are usually extracted with small amounts of fresh water to remove traces of inorganic acids, bases, or salts; treated with a solid drying agent to remove traces of water; and then filtered to remove the hydrated drying agent. Finally, the solvent is evaporated or distilled. The organic product can then be purified by a technique such as crystallization or distillation.





Distribution Coefficients:

When a compound is shaken in a separatory funnel with two immiscible solvents, such as water and diethyl ether $(CH_3CH_2OCH_2CH_3)$, the compound distributes itself between the two solvents. Some dissolves in the water and some in the ether. How much solute dissolves in each phase depends on the solubility of the solute in each solvent. The ratio of the concentrations of the solute in each solvent at a particular temperature is a constant called the **distribution**

coefficient or partition coefficient (K).

Concentration in solvent₂ K = ------

Concentration in solvent₁

Where solvent₁ and solvent₂ are immiscible liquids.

Steps in extraction:

- 1) Preparation of the separatory funnel.
- 2) Adding the liquids. Be sure the stopcock is closed.

Safety Note:

Never add a volatile solvent to a warm solution. If you are using a flammable solvent, make sure there are no flames in the vicinity!

3) Mixing the layers.

Insert the stopper and, *holding the stopper in place with one hand*, pick up the separatory funnel and invert it. Immediately open the stopcock with your other hand to vent solvent fumes or carbon dioxide. Swirl the separatory funnel gently to further drive off solvent vapors or gases.



After venting, close the stopcock, gently shake or swirl the mixture in the inverted funnel, then re-vent the fumes. If excessive pressure build-up is not observed, the separatory funnel and its contents may be shaken up and down vigorously in a somewhat circular motion for 2-3 minutes so that the layers are thoroughly mixed. Vent the stopcock several times during the shaking period. After completing the shaking, vent the stopcock one last time. Place a large Erlenmeyer flask under the stem of the separatory funnel in case the stopcock should develop a leak. Allow the separatory funnel to sit until the layers have separated.



4) *Separation the layers.* Before proceeding, make sure the stopper has been removed. (It is difficult to drain the lower layer from a stoppered funnel. Because a vacuum is created in the top portion of the funnel.)





5) Cleaning the separatory funnel.

Suggested experiments:

1) Separate a mixture of 2-naphthol and benzoic acid by an ether-bicarbonate extraction.

2) Separate a mixture of benzoic acid, p-chloroaniline, and naphthalene using dichloromethane as the organic solvent.



Chromatograph:

Chromatography is a general term that refers to a number of related techniques used for analyzing, identifying, or separating mixtures of compounds.

All chromatographic techniques have one principle in common liquid or gaseous solution of the sample, called the **mobile phase**, is passed (moved) through an adsorbent, called the **stationary phase**. The different compounds in the sample move through the adsorbent at different rates because of physical differences (such as vapor pressure)and because of different interactions (adsorptivities, solubilities. etc.)with the stationary phase.



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1 - Column Chromatography

Column chromatography is used to separate mixtures of compounds. In this technique, a vertical glass column is packed with a polar adsorbent along with a solvent. The sample is added to the top of the column; then, additional solvent is passed through the column to wash the components of the sample, one by one (ideally), down through the adsorbent to the outlet.

The sample on the column is subjected to two opposing forces: the solvent dissolving it and the adsorbent adsorbing it. The dissolving and the adsorption constitute an equilibrium process, with some sample molecules being adsorbed and others leaving the adsorbent to be moved along with the solvent, only to be re-adsorbed farther down the column. A compound (usually a non polar one) that is highly soluble in the solvent, but not adsorbed very strongly, moves through the column relatively rapidly. On the other hand, a compound (usually a more polar compound) that is more highly attracted to the adsorbent moves through the column more slowly.

Because of the differences in the rates at which compounds move through the column of adsorbent, a mixture of compounds is separated into *bands*.





(1) The sample has just started to move into column of adsorbent .

(2) and (3) as more solvent is passed through the column, the sample moves down and begins to separate into its components because of differences in attraction to the adsorbent and solvent.

(4) The faster-moving compound is eluted into a flask.



- 1- Separatec a mixture of permanganate and dichromate on silica gel column.
- 2- Isolate and separate plant pigments from extracts of carrots, tomatoes ,etc.
- 3- Separate *o*-and *p*-nitro anilines.





2- Thin-Layer Chromatography(TLC)

Thin-layer chromatography is a variation of column chromatography. Instead of a column, a strip of glass or plastic is coated on one side with a thin layer of alumina or silica gel.

In a TLC analysis, about 10 μ L of a solution of the substance to be tested is placed ("spotted") in a single spot near one end of the plate, using a micro capillary. The plate is "developed" by placing it in a jar with a small amount of solvent. Shows a TLC plate in a developing jar. The solvent rises up the plate by capillary action, carrying the components of the sample with it. Different compounds in the sample are carried different distances up the plate because of variations in their adsorption on the adsorbent coating. If several components are present in a sample, a column of spots is seen on the developed plate, with the more polar compounds toward the bottom of the plate and the less polar compounds toward the top.

As an analytical tool, TLC has a number of advantages: it is simple, quick, inexpensive, and requires only small amounts of sample. Tlc is generally used as a qualitative analytical technique, such as checking the purity of a compound or determining the number of components in a mixture. We can use TLC to follow the course of a reaction by checking the disappearance of starting material.





The R_f Value:

The distance that the spot of a particular compound moves up the plate relative to the distance moved by the solvent front is called the **retention factor**,

distance traveled by the compound

Equipment for TLC:

TLC sheets and plates. Commercial TLC sheets are coated with silica gel (SiO_2) or alumina (Al_2O_3) . Choose the type that gives the best separation for your particular mixture.

If commercial TLC sheets are unavailable, plates can be made from microscope slides and a slurry of 1g aluminum oxide G or silica gel G and 2mL



chloroform (CAUTION : toxic). A 2 : 1 mixture by volume of dichloromethane and methanol (also toxic) may be substituted for the chloroform.

Dip two slides, back-to-back, in the slurry. Allow the excess slurry to drain. Separate the slides and allow them to dry in a fume hood. Then wipe excess adsorbent from the backs and edges of the slides. Making satisfactory plates requires practice; therefore, prepare a number of plates and select the most evenly coated ones. Microscope slide plates are shorter than commercial sheets; consequently, the separation of components is not as clean.

Pipets. Commercial 10- μ L disposable pipets are best for spotting. If commercial pipets are not available, draw out some soft glass tubing or melting-point capillary tubes in a flame. The diameter of the pipet should be about 1/4 of the diameter of a melting-point capillary.

Developing jars. Developing chambers with the proper solvent system may be prepared in advance and kept in the fume hood. Any tall jar, such as an instant coffee jar or mason jar, with a lid or screw top, may be used for developing a TLC plate. The jar should be narrow enough to hold the plate upright inside, without the danger of its falling over. The lid of the jar should be impervious to solvent fumes.



- 1- Analyze a mixture of amino acids.
- 2- Analyze a mixture of ink.







Problems:

- **1-** Calculate the R_f values for the following compounds:
- (a) Spot,5 cm; solvent front, 20cm
- (b) Spot,3 cm; solvent front,12 cm
- (c) Spot,9.8cm;solvent front,12cm



Technique 6

Distillation:

Distillation is a general technique used for removing a solvent, purifying a liquid or separation the components of a liquid mixture. In distillation, liquid is vaporized by boiling, then condensed back to a liquid, called the distillate or condensate, and collected in a separate flask.

Types of distillation:

1-Fractional distillation

Miscible liquids are much more difficult to separate. Mixtures of miscible liquids can be separated by fractional distillation. It will provide the boiling points of the liquids are not too close.

If we want to separate a mixture of ethanol and water. The diagram below is suitable for this process. The fractionating column is packed with glass



bead. It provides a large surface area for vaporization and condensation of the liquid mixture.

Ethanol is more volatile than water, since it has a lower boiling point (78°C). The vapor rises up the fractionating column when the mixture is heated. Because ethanol is more volatile, the vapor contains more ethanol. The hot vapor condenses upon touching the cold glass beads. There is a continues rise of hot vapor up the fractionating column at the same time. Hot vapor will make the condensed vapor boils again. It will contain more and more ethanol as the vapor rises up to the fractionating column.



Fig.(8): Fractional distillation



The above process is to be repeated many times before the vapor consists only pure ethanol. During the process the escaping vapor is measured by a thermometer of the fractionating column. The temperature will remain steady for some time and will then rise quickly and become pure ethanol.

When the ethanol has boiled off completely, the escaping vapor will consist of pure water only.

Generally, for fractional distillation to work best, the difference in boiling points of liquids in the mixture should be greater than 10C. The separation will not be complete if it is not.

Fractional distillation is used in industry to separate oxygen and nitrogen from liquid air. In whisky production it is used to increase ethanol.

2-Vacuum distillation

Vacuum distillation is a method of distillation whereby the pressure above the liquid mixture to be distilled is reduced to less than its vapor pressure (usually less than atmospheric pressure) causing evaporation of the most volatile liquid(s) (those with the lowest boiling points). This distillation method works on the principle that boiling occurs when the vapor pressure of a liquid exceeds the ambient pressure. Vacuum distillation is used with or without heating the solution



3-Steam distillation

Steam distillation is a special type of distillation (a separation process) for temperature sensitive materials like natural aromatic compounds. Many organic compounds tend to decompose at high sustained temperatures. Separation by normal distillation would then not be an option, so water or steam is introduced into the distillation apparatus. By adding water or steam, the boiling points of the compounds are depressed, allowing them to evaporate at lower temperatures, preferably below the temperatures at which the deterioration of the material becomes appreciable. If the substances to be distilled are very sensitive to heat, steam distillation can also be combined with vacuum distillation. After distillation the vapors are condensed as usual, usually yielding a two-phase system of water and the organic compounds, allowing for simple separation.





When a mixture of two practically immiscible liquids is heated while being agitated to expose the surfaces of both the liquids to the vapor phase, each constituent independently exerts its own vapor pressure as a function of temperature as if the other constituent were not present. Consequently, the vapor pressure of the whole system increases. Boiling begins when the sum of the partial pressures of the two immiscible liquids just exceeds the atmospheric pressure (approximately 101 kPa at sea level). In this way, many organic compounds insoluble in water can be purified at a temperature well below the point at which decomposition occurs. For example, the boiling point of bromobenzene is 156 °C and the boiling point of water is 100 °C, but a mixture of the two boils at 95 °C. Thus, bromobenzene can be easily distilled at a temperature 61 C° below its normal boiling point.

Steam distillation is employed in the manufacture of essential oils, for instance, perfumes. In this method, steam is passed through the plant material containing the desired oils. It is also employed in the synthetic procedures of complex organic compounds. Eucalyptus oil and orange oil are obtained by this method on the industrial scale.





HYDROCARBONS

Compound containing only Carbon and Hydrogen and classified into:

Alkanes R-H

- Alkanes are saturated hydrocarbon compounds.
- Chemically, alkanes are unreactive compounds because they are saturated compounds , all the bonds are strong sigma bonds.
- The general formula of alkanes is C_nH_{2n+2}
- Example: Cyclohexane





2- Potassium Permanganate Test:		
In a test tube put 1ml of	The violet color of the	It is saturated
cyclohexane + few drops of dilute	KMnO ₄ dose not	compound.
acidic KMnO ₄ with shaking.	changes.	

Alkenes R-CH=CH-R

- Alkenes are unsaturated hydrocarbon compounds containing double bond .
- They are reactive compounds because they contains weak π bond.
- The general formula of alkenes is C_nH_{2n} .
- Example: Cyclohexene





Put 3ml of cyclohexanol in dry test tube + 0.5 ml of conc. $H_2SO_4(2)$ and then perform the following tests:





Alkynes (Acetylenes)

- Alkynes are unsaturated hydrocarbon compounds containing triple bond .
- The alkynes are also reactive compounds because they contains weak π bonds .
- The general formula of alkynes is C_nH_{2n-2}
- Example: Acetylenes HC == CH



Acetylene gas prepared by adding water to calcium carbide $CaC_2(5)$



Put 0.25 gm from calcium carbide in dry test tube add 1ml of water and note the evolution of acetylene gas, perform the following experiments on it:
Test	Observation	Result
1-Bromination Test : Pass the acetylene gas on the test tube contains bromine.	The orange color of the bromine disappears Immediately.	It is unsaturated compound (6).
HC≡CH	HC==CH _Br ₂	Br Br HC CH Br Br (6)
2- Potassium Permanganate		
Test :		
Pass the acetylene gas on the test	The violet color of the	It is unsaturated
tube contains dilute acidic	KMnO ₄ disappears	compound.
KMnO ₄ with shaking.	immediately.	
3- Pass the acetylene gas on the	White precipitate	The white precipitate
test tube contains ammonical	forms.	is Silver acetylide (7).
silver nitrate.		
HC≡CH + 2 Ag +	→ Ag—C=C—Ag	+ 2 H⁺
	Silver acetylide	(7)



AROMATIC HYDROCARBONS

Examples on Aromatic hydrocarbons:





Benzene

Toluene

The most characteristic reactions for aromatic hydrocarbons are:

1) Nitration reaction

2) Sulphonation reaction





Benzene is a colorless liquid, immiscible in water but miscible in all organic solvent, flammable and burned by heavy smoked yellow flame which indicate that it rich with carbon.

Test	Observation	Result
1-Nitration Test :		
In a test tube put 1ml of		
$conc.HNO_3$ acid + 1ml of conc.	Yellow oily layer	The yellow oily layer
H_2SO_4 acid + 0.5ml of benzene	will be separated	is Nitro benzene (8).
then pour into beaker contains cold		
water.		





2-Toluene



Toluene is a color less liquid , the boiling point is 110°C immiscible in water but miscible in organic solvent behave like benzene on burning .

Test	Observation	Result
1-Nitration Test :		
In a test tube put 1ml of conc.HNO ₃ acid + 1ml of conc.	Yellow oily layer will be separated	The yellow oily layer is <i>o</i> -Nitrotoluene
H_2SO_4 acid + 0.5ml of Toluene then pour into beaker contains cold water .		& <i>p</i> -Nitrotoluene (9).
$\begin{array}{c} \begin{array}{c} CH_{3} \\ HNO_{3} \text{ Conc.} \\ H_{2}SO_{4} \text{ Conc.} \end{array} \end{array} \xrightarrow{\begin{array}{c} CH_{3} \\ H_{2}SO_{4} \text{ Conc.} \end{array}} + \begin{array}{c} CH_{3} \\ H_{2}SO_{4} \text{ Conc.} \end{array} $		
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ALCOHOLS COMPOUNDS

R-OH

- Alcohols are compounds contains one or more hydroxyl groups (OH)
- Alcohols divided according to the type of carbon atom attached to the hydroxyl group in the molecule to Primary, Secondary& Tertiary alcohols.

Oxidation test:

It was found that Primary alcohols oxidizing to aldehyde the Secondary alcohols oxidizing to ketone, but tertiary alcohols not oxidized according to the following equations.





1-Methanol CH₃OH

Methyl alcohol is colorless liquid , have characteristic odor , miscible in water , toxic , burned by blue flame .

Test	Observation	Result
1-Oxidation Reaction:		
In a test tube put 1 ml of methanol	The orange color	The green color is chromium
+1ml of potassium dichromate	of the solution	sulphate $Cr_2(SO_4)_3$ which
$K_2Cr_2O_7$ + drops of conc. sulphuric	changes to green.	formed due to the oxidation of
acid .		methanol to Formaldehyde (10).
$K_2Cr_2O_7 + 4H_2SO_4 + 3CH_3OH \longrightarrow$	0 - 3H $-C$ H + K ₂ SO ₄	+ $Cr_2(SO_4)_3$ + 7 H ₂ O (10)
2-Esterification :		
In a test tube put 1ml of methanol	Vix (oil of winter	The product is
+ 0.5 gm of salicylic acid + drops	green) odor	methyl salicylate (11).
of conc. H_2SO_4 & heat in water	appear.	
bath for 3 min. then cool and pour		
the content of the test tube into		
beaker contains Na ₂ CO ₃ .		
$\begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} $		
	meth	yl salicylate (11)
	41	



2-Ethanol CH₃CH₂OH

Ethyl alcohol is colorless liquid , have characteristic odor , miscible in water , toxic , burned by colorless flame .

Test	Observation	Result
1-Oxidation Reaction:		
In a test tube put 1ml of ethanol	The orange color	The green color is chromium
+1ml of potassium dichromate	of the solution	sulphate $Cr_2(SO_4)_3$ which
$K_2Cr_2O_7$ + drops of conc. sulphuric	changes to green.	formed due to the oxidation of
acid .		ethanol to acetaldehyde (12)
K ₂ Cr ₂ O ₇ + 4 H ₂ SO ₄ + 3 CH ₃ CH ₂ OH ——	→ 3 CH ₃ — ^U — н	+ K_2SO_4 + $Cr_2(SO_4)_3$ + 7 H_2O chromium sulphate (12)
 2-Esterification : In a test tube put 1ml of ethanol + 0.5 gm of sodium acetate + drops of conc. H₂SO₄ & heat in water bath for 3 min. then cool and pour the content of the test tube into beaker contains Na₂CO₃ solution . 	Fruity odor appears (apple odor) .	The product is Ethyl acetate (13) .







Glycerol is a colorless viscous liquid, melted at 10 °C, miscible in water and alcohol and has a sweet taste.

The most characteristic test for glycerol is the Borax test.

Test	Observation	Result
1-Borax Test :		
In a test tube add 2 drops of	The color	The disappearance of (ph.ph)
phenolphthalein (ph.ph) to	disappears and is	color on cold due to the
dilute borax solution then a pink	restored on heating.	formation of strong acid and
color is produced Add few		by heating this acid
drops of glycerol & shake.		dissociated into boric acid
		& glycerol, the pink color
		returned (15).

 $Na_2B_4O_7 + 7H_2O \longrightarrow 2NaOH + 4H_3BO_3$







1-Formaldehyde H = C H

Formaldehyde is a gas and is available as aqueous solution which has a characteristic pungent odor.

Test	Observation	Result
2,4-dinitrophenyl hydrazine test:		
In a test tube put 1 ml of HCHO	Yellow precipitate is	The ppt. is hydrazone
+1ml of 2,4- dinitro phenyl	formed.	derivative this prove that
hydrazine .		formaldehyde contain
		carbonyl group (16).
$O_2 N \longrightarrow NHNH_2 + O = C \xrightarrow{H} \xrightarrow{-H_2O} O_2 N \longrightarrow NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 N \longrightarrow NO_2 NHN = C \xrightarrow{H} + H_2O$ $NO_2 N \longrightarrow NO_2 N$		
2-Schiff's Reagent:		
In a test tube put 1 ml of HCHO	Magenta color	It's Formaldehyde
+ drops of Schiff's reagent.	appears rapidly.	
3-Reduction Reaction		
A)- Ammonical AgNO ₃	A silver mirror is	Due to Oxidation of
In a test tube put 1ml of HCHO	deposited on the inner	HCHO to formic acid
+3 ml of amm. AgNO ₃ & heat in	wall of the test tube.	& reduction for Ag ₂ O to
water bath for 3 min.		Ag metal (17).







2-Acetaldehyde $CH_3 - C - H$

It is a liquid b.p 21° generally used in aqueous solution which has also characteristic odor, miscible in water ,alcohol and ether.

Test	Observation	Result
1-2,4-dinitrophenyl hydrazine test : In a test tube put 1ml of CH ₃ CHO +1ml of 2,4-dinitro phenyl hydrazine	Yellow precipitate is formed.	The ppt. is hydrazone derivative this prove that acetaldehyde contain carbonyl group
 2-Schiff's Reagent: In a test tube put 1 ml of CH₃CHO + drops of Schiff's reagent. 	Magenta Violet color appears rapidly.	It's Acetaldeyde
 3-Reduction Reaction A)- Ammonical AgNO₃ In a test tube put 1ml of CH₃CHO + 3 ml of amm. AgNO₃ & heat in water bath for 3 min. 	A silver mirror is deposited on the inner wall of the test tube.	Due to Oxidation of CH ₃ CHO to acetic acid & reduction for Ag ₂ O to Ag metal .
 B)- Fehling's solution In a test tube put 1ml of CH₃CHO +1ml of Fehling solution A +1ml of Fehling solution B & then heat on water bath for 3 min . 	Solution turns green and after some time a red ppt.	Due to Oxidation of CH ₃ CHO to acetic acid & reduction for CuO to Cu ₂ O (red ppt.)







Benzaldehyde is colorless liquid if pure with the odor of bitter almonds immiscible with water & sinks in it but miscible with alcohol .

Test	Observation	Result
1-2,4-dinitrophenyl hydrazine test:	Dark yellow	The ppt. is hydrazone
In a test tube put 1ml of benzaldehyde	precipitate is	derivative this prove
+1ml of 2,4-dinitro phenyl hydrazine .	formed.	that benzaldehyde
		contain carbonyl group
2-Schiff's Reagent:	magenta color	
In a test tube put 1 ml of benzaldehyde	appears after some	It's benzaldehyde
+ drops of Schiff's reagent.	time and vigorous	
	shaking.	
3-Reduction Reaction	A silver mirror is	Due to Oxidation of
A) Ammonical AgNO ₃	deposited on the	benzaldehyde to
In a test tube put 1ml of benzaldehyde	inner wall of the	benzoic acid
+ 3 ml of amm. AgNO ₃ & heat in water	test tube by gentle	& reduction for
bath for 3 min.	heating.	Ag ₂ O to Ag metal.



$\begin{array}{c} O\\ \textbf{4-Acetone} \qquad CH_3 - \overset{||}{C} - CH_3 \end{array}$

Acetone is colorless liquid with a characteristic odor, inflammable, miscible with water, alcohol & ether .

Test	Observation	Result
1-2,4-dinitrophenyl hydrazine test: In a test tube put 1ml of acetone +1ml of 2,4-dinitrophenylhydrazine	Yellow precipitate is formed.	The ppt. is hydrazone derivative this prove that acetone contain carbonyl group .
 2-Schiff's Reagent: In a test tube put 1 ml of acetone + drops of Schiff's reagent. 3-Iodoform Test : In the test tube put 1ml of acetone +3ml of Iodine then add drops of NaOH immediately. 	magenta color appears slowly, need long time Yellow ppt. is formed on cold.	Acetone is ketone not aldehyde . The Yellow ppt. is Iodoform CHI ₃ . (22) .
$CH_{3} - C - CH_{3} \xrightarrow{3 _{2}} CH_{3}CO$ $Iodal$ $Iodal$	H ₃ ↓ + CH ₃ COONa m (22)	
In the test tube put 1ml of acetone + 0.5 ml of Sodium nitroprusside then put drops of NaOH with shaking.	Deep Red Solution Formed.	It's Acetone characteristic test for (- COCH ₃).



CARBOXYLIC ACIDS

Líquíd organic acids

- All acids contains carboxyl group COOH.
- Acids divided into two major classes.

Aliphatic acid and aromatic acid. Also can divide into different types according to number of carboxylic group in the molecules or according to type or number of another group attached to acid molecules.





To Make Neutral Solution N.S.:

Add to the acid aqueous NH_3 (NH_4OH) till the solution is just alkaline to litmus. Boil the solution until the evolution of NH_3 gas ceases (this can be ensured by exposing a red litmus paper to vapor).

> General properties for acids :

- 1. Soluble in sodium hydroxide.
- 2. Soluble in sodium carbonate or sodium bicarbonate with eff. of CO₂ gas.
- 3. When heated the acids with sodalime CO_2 evolved.



1. Acids react with alcohol in present of conc. H_2SO_4 produce ester with specific odor .

$$RCOOH + ROH \longrightarrow RCOOR + H_2O$$

- 2. N.S of acid with ferric chloride solution. Gives specific color for each acid.
- 3. Acids Solution turns litmus paper to red.



1- Formic acids HCOOH

Formic acid is a colorless liquid, immiscible in water, alcohol and ether.

It has a pungent irritating odor. All formate as soluble except those of Ag, Hg, and some basic salt.

Test	Observation	Result
1-Acidity test: 1ml of acid +2ml of NaHCO ₃	CO_2 evolves with effervescence.	Compound is carboxylic acid (23).
HCOOH + NaHCO	B HCOONa + H	$I_2O + CO_2$
2-Ferric chloride test	Red color turned with	Red color is ferric format
1ml of N.S. of acid + few drops	heating into Brown ppt.	$(HCOO)_3$ Fe and brown ppt.
of FeCl ₃ .		is basic ferric formate
		(HCOO) Fe (OH) $_2$ (24).
$3 \text{ HCOONH}_4 + \text{FeCl}_3 \longrightarrow (\text{HCOO})_3 \text{Fe} + 3 \text{ NH}_4 \text{Cl}$		
$(\text{HCOO})_3\text{Fe} \xrightarrow{2 \text{H}_2\text{O}} (\text{HCOO})\text{Fe}(\text{OH})_2$		
	basic ferric format	e (24)
3-Mercury chloride test:	White ppt. immediately	White ppt. is Hg ₂ Cl ₂
1ml of N.S. of acids +1ml	appear &change to gray	& the gray ppt. is
HgCl ₂ & added excess then boil	ppt. with boiling.	metallic Hg (25).





2- Acetic acid CH₃COOH

Acetic acid is a colorless liquid, with a penetrating odor of vinegar, miscible with H₂O, alcohol and ether . Most of its salts soluble with water except those of Ag, mercurous and basic salts.

Test	Observation	Result	
1-Acidity test: 1ml of acid + 2ml of NaHCO ₃ .	CO_2 evolves with effervescence.	Acetic acid is carboxylic acid (28).	
CH ₃ COOH + NaHCO ₃ -	$\longrightarrow CH_3COONa + H_2O + CO_2$ Sodium acetate(28)		
2-Ferric chloride test : 1ml of N.S. of acid + few drops of FeCl ₃ .	Red color turn with heating to brown ppt.	Red color is Ferric acetate $(CH_3COO)_3$ Fe and brown ppt. is basic ferric acetate (CH_3COO) Fe $(OH)_2$ (29)	
$3 \text{ CH}_3\text{COONH}_4 + \text{FeCl}_3 \longrightarrow (\text{CH}_3\text{COO})_3\text{Fe} + 3\text{NH}_4\text{Cl}$ (CH ₃ COO) ₃ Fe $\xrightarrow{2\text{H}_2\text{O}}$ (CH ₃ COO) Fe(OH ₂) Basic ferric acetate			
		(29)	
	57		



3-Mercury chloride test 1ml of N.S. of acids $+1$ ml of HgCl ₂ & added excess then boil.	White ppt. Change to yellow ppt. with boiling.	Confirmatory test for acetic acid.
4-Permanganate test:- 1ml of warm acid + drops of KMnO ₄ .	The color will not disappear .	No oxidation for acetic acid occur.
5-Esterification test: In dry test tube put 1ml of acids +1ml of ethanol +1ml of conc. H ₂ SO ₄ & heat in water bath then pour into beaker contain Na ₂ CO ₃ solution .	apple odor	This odor characteristic for Ethyl acetate CH ₃ COOC ₂ H ₅ (30) .
CH ₃ COOH + CH ₃ CH ₂ OH	H_2SO_4 CH_3COOO Ethyl ac	$DCH_2CH_3 + H_2O$ cetate(30)

3-Lactic acid CH₃CH(OH)COOH

Is light yellow color liquid, frozen at 110°C miscible with water and alcohols.

Test	Observation	Result	
1-Acidity test: 1ml of acid +2ml of NaHCO ₃ .	CO_2 evolves with effervescence.	Lactic acid is from carboxylic acid .	
CH ₃ -CH-COOH + NaHCO ₃ —	$\rightarrow CH_3-CH-COONa + CO_2 + H_2O \\OH \\Sodium lactate (31)$		
2-Ferric chloride test:			
1ml of N.S. of acid+ few drops	No change for FeCl ₃	The acid is lactic acid	
of FeCl ₃ .	color.	specific test for lactic acid	
3-Iodoform test:			
1ml of acid + 3ml of iodine then	Yellow ppt. is	Yellow ppt. is Iodoform	
add NaOH with shaking	formed on cold .	CHI ₃ (32).	
ОН О [0] Сн₃снсоон —→ сн₃ссоон	$\begin{array}{cccc} OH & O & O \\ I & [O] & II & 3I_2 & II \\ CH_3CHCOOH & \longrightarrow & CH_3CCOOH & 3I_2 & CI_3CCOOH & 2NaOH & CHI_3 + COONa \\ & & & & & & & \\ Iodoform & & & & \\ & & & & & \\ Iodoform & & & \\ & & & & & \\ & & & & & \\ \end{array}$		
4-Permanganate test:-			
1ml of warm acids + drops of	The color of	oxidation for hydroxyl	
KMnO ₄ .	KMnO ₄ disappear.	group in lactic acid .	
	59		











Sixth:

In dry test tube put 1ml of unknown + 0.5gm of salicylic acid + 2drop of conc. H_2SO_4 and heated in water bath for 3 min. then pour the content into beaker containing Na₂CO₃ solution.

Vix smell appear (Methyl salicylate) unknown is methanol.

Neutral Solution N.S.:

Add to the acid aqueous \mathbf{NH}_3 ($\mathbf{NH}_4\mathbf{OH}$) till the solution is just alkaline to litmus Boil the solution until the evolution of \mathbf{NH}_3 gas ceases (this can be ensured by exposing a red litmus paper to vapor).

Experiment to differentiate between formic and acetic acid :

Test	Formic acid	Acetic acid
1-Mercury chloride test: 1ml of N.S. of acid +1ml of HgCl ₂ & added excess then boil .	White ppt. immediately appear & change to gray ppt. with boiling .	White ppt. change to yellow ppt. with boiling .
2-Permanganate test: 1ml of warm acid + drops of $KMnO_4$.	The color disappear.	The color will not disappear.

Experiment to differentiate between formaldehyde and Acetaldehyde :

Test	Formaldehyde	Acetaldehyde
1-Resorcinol test: In a test tube put 1ml of HCHO + drops of resorcinol +1ml of H_2SO_4 conc. added down the side of the test tube.	Red violet ring is formed at the interface (A white ppt. appears over the ring) .	- Ve
2- nitroprusside sodium test: In the test tube put 1ml of aldehyde + 0.5 ml of Sodium nitroprusside then put drops of NaOH with shaking .	- Ve	Dark red color .



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AROMATIC ORGANIC ACIDS

1-Benzoic acid C_6H_5COOH

Benzoic acid is a white crystalline solid undergo sublimation by heating partially soluble in cold water, freely soluble in hot water and ethanol m.p. is $121 \, ^{\circ}C$.

Test	Observation	Result
1-Acidity test :		
0.25gm of acid +1ml of conc.	CO ₂ evolves with	The compound from
NaHCO ₃ solution.	effervescence	carboxylic acid.
2-Ferric chloride test :		The ppt. is basic ferric
1ml of N.S. + drops of $FeCl_3$.	buff ppt.	benzoate (33).
COONH ₄	COO)Fe(OH) ₂	
+ FeCl ₃	+ NH	4₄Cl(33)
3-Estrification test :		
0.25gm of acid +1ml of ethanol	Specific odor	The odor due to Ethyl
+1ml of conc. H_2SO_4 & heat in water		benzoate (34).
bath 3 min. then pour into beaker		
contain NaHCO ₃ solution .		
$\begin{array}{c} \text{COOH} \\ + C_2H_5\text{OH} \end{array} \begin{array}{c} H_2\text{SO} \\ \hline \end{array}$	P_4 COOC ₂ H ₅ +	H ₂ O
	Ethyl benzoate	(34)
	64	



2-Salicylic acid $C_6H_5(OH)COOH$

Salicylic acid is a white crystalline solid undergo sublimation by heating, partially soluble in cold water, freely soluble in hot water and ethanol m.p. is 159 $^{\circ}$ C.

Test	Observation	Result	
1-Acidty test :			
0.25gm of acid +1ml of conc.	CO ₂ evolves with	The compound from	
NaHCO ₃ solution.	effervescence	carboxylic acid	
2-Ferric chloride test :		Specific test of	
1ml of N.S. + drops of $FeCl_3$.	Violet color	salicylic acid	
3-Estrification test :			
0.25gm of acid +1ml of methanol			
+1ml of conc. H ₂ SO ₄ & heat in	Vix odor	The odor due to Methyl	
water bath 3 min. then pour into		salicylate ester (35).	
beaker contain Na ₂ CO ₃ solution			
$\begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ $			
	Methyl salicy	·lat(35)	



DI CARBOXYLIC ACID

1-Oxalic acid

Oxalic acid is a colorless crystalline solid freely soluble in water and alcohol, it undergo pyrolysis without charring up on heating .

Test	Observation	Result
1-Acidity test :		
0.25gm of acid +1ml of NaHCO ₃	CO ₂ evolves with	The compound from
solution .	effervescence	carboxylic acid.
2-Ferric chloride test :		
1ml of N.S. + drops of $FeCl_{3}$.	No change for FeCl ₃	Oxalic or tartaric or citric
	color	acid.
3-Calcium chloride test :		
1ml of N.S. of acid +1ml of $CaCl_2$	White ppt. on cold	The ppt. is calcium oxalate
		specific test for oxalic acid
4-Silver nitrate test:		
1ml of N.S. of acids +2ml of silver	White ppt. dissolve	The ppt. is silver oxalate.
nitrate AgNO ₃ .	in ammonia and	
	HNO ₃ acid	
5- potassium Permanganate test:		
1ml of warm acid + drops of	The color of	Oxidation for oxalic acid.
KMnO ₄ .	KMnO ₄ disappear	



2-Tartaric acid

НО----СН----СООН | НО----СН---СООН

Colorless crystalline solid, freely soluble in water and alcohols. Heating of acid or its salts, they undergo charring with evolution of burned sugar odor.

Test	Observation	Result
1-Acidity test:	CO ₂ evolves with	The compound from
0.25gm of acid +1ml of NaHCO ₃	effervescence	carboxylic acid
2-Ferric chloride test:	No change for FeCl ₃	Oxalic or tartaric or
1ml of N.S. of acid + drops of FeCl ₃	color	citric acid
3-Calcium chloride test:	White ppt. appear after	The ppt. is calcium
1ml of N.S. of acid +1ml of $CaCl_2$	scratching the inner wall	tartarate specific test
	of the tube	for tartaric acid
4-Silver nitrate test:		
1ml of N.S. of acids + 2ml of silver	White ppt. soluble in	The ppt. is silver
nitrate AgNO ₃ .	excess of N.S.	tartarate
5- potassium Permanganate test :	The color of KMnO ₄	Oxidation of
1ml of warm acid + drops of KMnO ₄	disappear on heating	Tartaric acid
6-Fenton's test:	Green color turns into	
1ml of acid + 1drop of dilute solution	intense violet color on	specific test for
of FeSO ₄ then 1drop of 10% H_2O_2 .	adding excess NaOH	tartaric acid
	solution.	
7-Potassium dichromate test :	The color change to	specific test for
1ml of acid +1ml $K_2Cr_2O_7$ & heat	green .	tartaric acid



$$\begin{array}{c} \mathsf{CH}_2 - \mathsf{COOH} \\ \textbf{3-Citric acid} & \mathsf{HO} - \mathsf{C} - \mathsf{COOH} \\ | \\ \mathsf{CH}_2 - \mathsf{COOH} \end{array}$$

Citric acid is Colorless crystalline solid, freely soluble in water and alcohols. By heating a heavy vapor with irritating smell evolved and finally undergo charring

Test	Observation	Result	
1-Acidity test :			
0.25gm of acid +1ml of NaHCO ₃	CO ₂ evolves with	The compound from	
solution.	effervescence	carboxylic acid	
2-Ferric chloride test:			
1ml of N.S. + drops of FeCl ₃ .	No change for FeCl ₃	Oxalic or tartaric or	
	color	citric acid	
3-Calcium chloride test :			
1ml of N.S. of acid +1ml of	White ppt. appear	The ppt. is calcium citrate	
CaCl ₂ .	after boiling	specific test of citric acid	
4-Silver nitrate test :			
1ml of N.S. of acids + 2ml of	White ppt. dissolve	The ppt. is silver citrate	
silver nitrate AgNO ₃ .	in ammonia		
5-Denig's test :	Color of		
2ml of N.S. + 2ml of denig's A	permanganate will	specific test of citric acid	
& heating until boiling then add	disappear and heavy		
2 drops of deng's B .	white ppt. formed		



PHENOL

Phenol is aromatic compound contains one or more hydroxyl group (OH) connected directly to aromatic nucleus.

Phenol classified according to the number of hydroxyl contained group into :

📥 Monohydric phenol Dihydric phenol ∔ Trihydric phenol

> General properties of phenol:

1-Solubility:

Phenol soluble in sodium hydroxide solution (NaOH) or potassium hydroxide (KOH), phenol differ from acids it doesn't dissolve in basic carbonate like sodium carbonate Na₂CO₃ and potassium carbonate k_2CO_3 .

2-Ferric chloride test:

This test used to identify phenols it gives complex with red, blue, Purple or green color.

3-Azodye formation test:

Diazonum salt react with phenols in basic medium via substitution forming compounds, known as AZODYE, most of them used in cloth dying. Azodyes are colored and they color depend on the kind of phenol and the aromatic amine which used to form the azo compound.

4-Bromination test:

Phenols react with bromine forming the substituted product which are mainly solids.



ОН

1-Phenol

Phenol is a colorless crystals in pure state, which turned to light red when exposed to air it has characteristic odor. Soluble in water, alcohols, ethers, and sodium hydroxide. Highly toxic .

Test	Observation	Result
1-Ferric chloride test:Diluted solution of phenol in alcohol then add2 drop of FeCl₃.	Violet color appear	The compound from phenols
2-Azo dye formation test: Take 3 test tube Tube (1): 1ml of aniline +3.5ml of conc. HCl Tube (2): 1ml of sodium nitrite NaNO ₂ Tube (3): phenol dissolved in excess NaOH. Keep the 3 test tubes in ice bath& Put tube (2)- \rightarrow (1) slowly, then add mixture to tube (3).	Red dye	Dye is Benzene azo phenol (36)











Resorcinol is solid ,dissolve in water and alcohol but in soluble in benzene.

Test	Observation	Result
1-Ferric chloride test:		
Diluted solution of resorcinol in alcohol then	Deep violet color	The compound
add 2 drop of FeCl ₃ .	appear	from phenols
2-Azo dye formation test:		
Take 3 test tube		
Tube (1): 1ml of aniline + 3.5ml of conc. HCl	Red dye	The dye is
Tube (2): 1ml of sodium nitrite NaNO ₂		Benzene azo
Tube (3): resorcinol dissolved in excess NaOH.		resorcinol
Keep the 3 test tube in ice bath & Put tube		
(2) \rightarrow (1) slowly, then add mixture to tube (3).		
3-Chloroform test :		
1ml of resorcinol dissolved in excess 20% NaOH	Red fluorescent	Compound is
+1ml of chloroform CHCl ₃ then heat in water bath		resorcinol
4-Phethalen test:		
In dry test tube add 0.5gm of resorcinol	Red fluorescent	Compound is
+ 0.5 gm of phthalic anhydride +2drop of	in the inner &	resorcinol
conc. H_2SO_4 , and gently fuse together allow to	Green fluorescent	
cool, and then add 30% NaOH solution in excess	in the wall.	


Solid, insoluble in water, alcohol, ether and sod.hydroxide solution.

Test	Observation	Result
1-Ferric chloride test:		
Diluted solution of α -naphthol in alcohol then add	Greenish color	The compound
2 drop of FeCl ₃ .	turns to violet	from phenols
2-Azodye formation test:		
Take 3 test tube		
Tube (1): 1ml of aniline +3.5ml of conc. HCl	Brownish red	Dye is
Tube (2): 1ml of sodium nitrite NaNO ₂	ppt.	Benzene azo
Tube (3): α -naphthol dissolved in excess NaOH.		α -naphthol
Keep the 3 test tube in ice bath Put tube (2) \rightarrow (1)		
slowly, then add mixture to tube (3).		
3-Chloroform test :		
1ml of α -naphthol dissolved in excess 20% NaOH	Blue color turns	Compound is
+1ml of chloroform CHCl ₃ then heat in water bath	to green	α -naphthol
4-Phethalein test:		
In dry test tube add 0.5gm of α -naphthol		Compound is
+ 0.5 gm of phthalic anhydride +2drop of	Green Color	α -naphthol
conc. H_2SO_4 , and gently fuse together allow to cool ,		
and then add 30% NaOH solution in excess.		



4-B-Naphthol



Test	Observation	Result
1-Ferric chloride test:		
Diluted solution of β -naphthol in alcohol then	Greenish color	The compound
add 2 drop of FeCl ₃ .	turns to violet	from phenols
2-Azo dye formation test:		
Take 3 test tube		
Tube (1): 1ml of aniline +3.5ml of conc. HCl		Dye is Benzene
Tube (2): 1ml of sodium nitrite NaNO ₂	orange dye	azo β -naphthol
Tube (3): β -naphthol dissolved in excess NaOH.		
Keep the 3 test tube in ice bath & Put tube		
(2) \rightarrow (1) slowly, then add mixture to tube (3).		
3-Chloroform test :		
1ml of β -naphthol dissolved in excess 20%	Blue color turns	Compound is
NaOH +1ml of chloroform CHCl ₃ then heat in	to green.	β -naphthol
water bath .		
4-Phethalen test:		
In dry test tube add 0.5gm of β -naphthol		Compound is
+ 0.5 gm of phthalic anhydride +2drop of	Green Color	β -naphthol
$conc.H_2SO_4$, and gently fuse together allow to		
cool, and then add 30% NaOH solution in exces		



Scheme for solid sample

Organic compound

Solid compound:

1- Acids:

Benzoic acid - Salicylic acid - Oxalic acid - Tartaric acid - Citric acid.

2- Sodium salt for acid:

Sodium acetate - Sodium format - Sodium Benzoate - Sodium Salicylate Sodium Oxalate -Sodium Tartarate - Sodium Citrate.

3- Ammonium salt for acid:

Ammonium acetate - Ammonium format - Ammonium Citrate Ammonium Salicylate - Ammonium Oxalate - Ammonium Tartarate.

4- Aniline salts:

1-Aniline Hydrochloride



2- Aniline Sulfides



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Confirmatory test:

Test	Formic acid or its salt	Acetic acid or its
		salt
1-Mercuric chloride test:	White ppt. immediately	White ppt. change to yellow by
1ml of N.S. of unknown	change to gray with	boiling
+1ml of $HgCl_2$ and boil.	boiling	
2-Permanganate test:		
1ml of warm acid	The color will disappear	The color will not disappear
+ drops of $KMnO_4$.		

Test	Hydrochloride Aniline	Sulfides Aniline
Unknown +AgNO ₃ .	white ppt. from silver chloride	-ve
Unknown+BaCl ₂ .	-ve	white ppt. from silver sulphate



Notes:

Preparation of N.S. for ammonium and sodium salt :

By dissolve the salt in water only .