



جامعة الزهراء
Al-Zahraa University for Women



FACULTY OF PHARMACY

Pharmaceutical Chemistry Department

First Year

Organic Chemistry

Laboratory Course

Semester 1

Course Organisers

Name : Dr. Nafeesa Querban

Name : Asst. Dhuha Mahdi Al-Bahrani

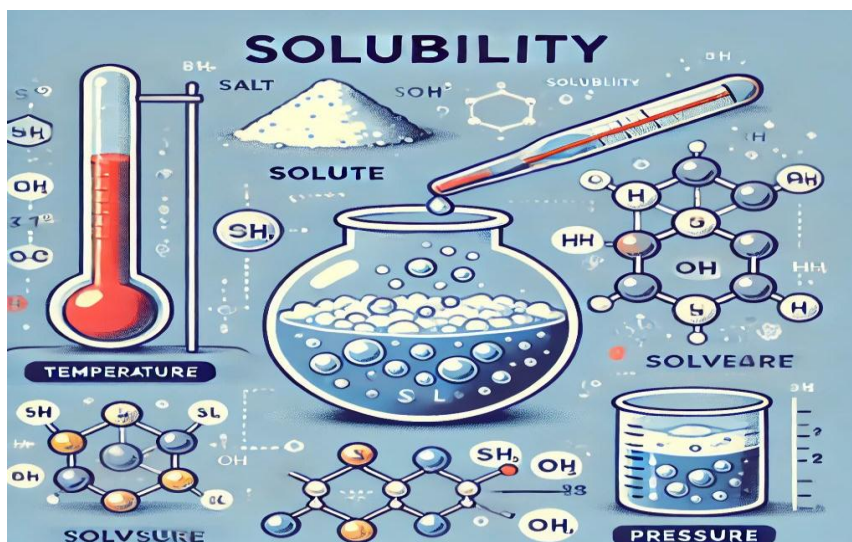
Head of Department

Dr. Rafid A. Taj Aldeen

PhD Drug Chemistry

Introduction to Solubility:

Solubility is a physical property that describes the ability of a substance (solute) to dissolve in another substance (solvent) to form a homogeneous solution. Solubility is influenced by several factors, including the nature of the solute and solvent, temperature, and pressure. Substances can be fully soluble, partially soluble, or insoluble, depending on their characteristics.



Definition of Solubility:

Solubility is the amount of solute that can be dissolved in a specific amount of solvent at a given temperature and pressure to form a saturated solution. Solubility is usually measured in units such as grams per liter or moles per liter.

Solubility classification of organic cpd. gives an idea about:

- 1- The type of the functional group present in the cpd.
2. The polarity & the molecular weight of the cpd., (Hydrocarbons are insoluble in water because of their non-polar nature, and if an unknown cpd. is partially soluble in water this indicates that a polar functional group is present).

★ As the M.wt increases, the water solubility decreases ($C \leq 5$ water soluble).

3. The nature of the compound (acidic, basic, neutral).

Solubility Classification of organic cpd.s is accomplished by testing the solubility of the compounds in the following solvents:

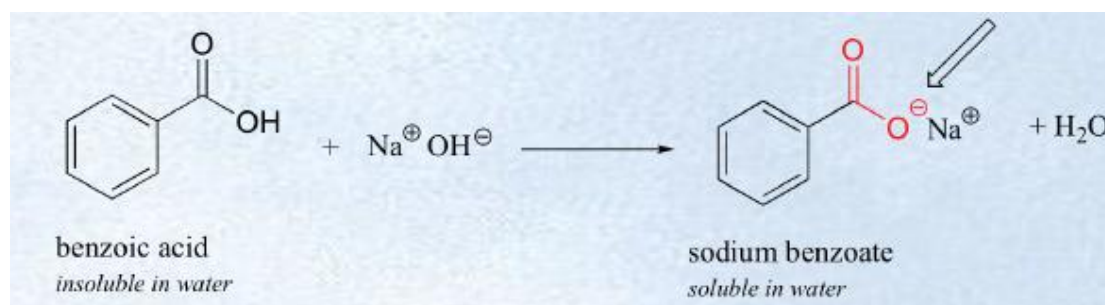
- Distilled water.
- Ether.
- 5% sodium hydroxide solution.
- 5% sodium bicarbonate solution.
- 5% hydrochloric acid solution.
- Cold concentrated sulfuric acid.

The solubility in certain solvents often leads to more specific information about the functional group.

For example:

Benzoic acid is insoluble in water, but it is converted by 5% NaOH solution to sodium benzoate salt which is readily soluble in water.

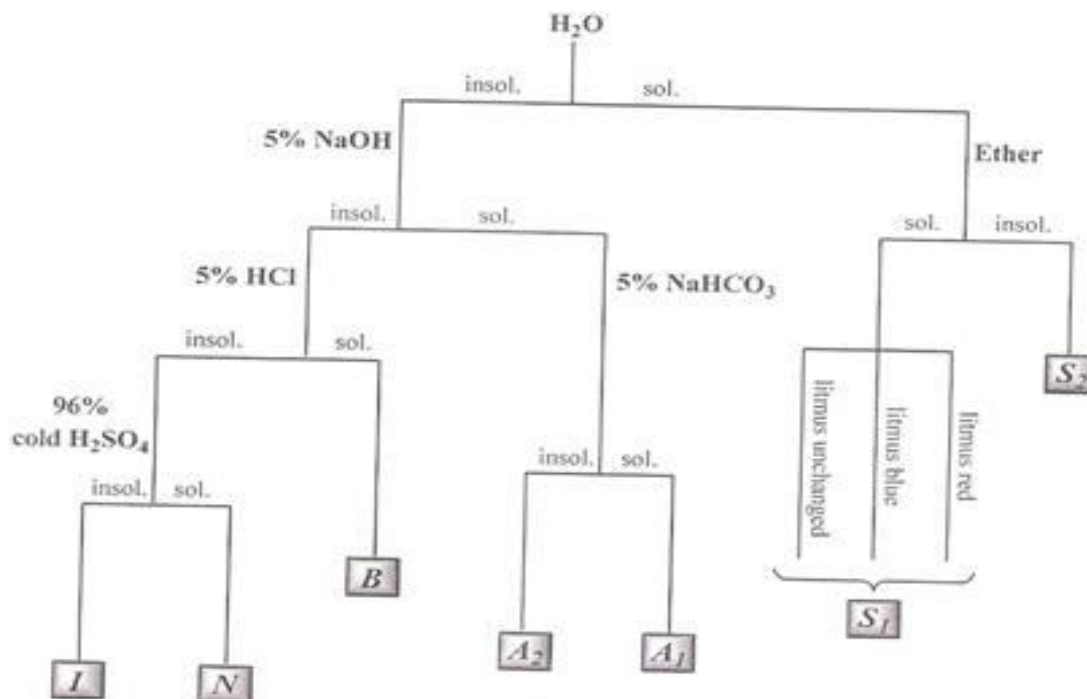
Ionic – very



★ When an unknown cpd. is insoluble in water and soluble in 5% NaOH sol. this indicates the presence of an acidic functional group.

- ♦ Generally, and for solubility classification purposes, the cpd. is said to be soluble in any solvent if it dissolves to the extent of about 3% (0.1gm/3 ml or 0.2 ml/3 ml).
- ♦ This is achieved by dissolving about 0.1 gm of solid or 3-4 drops of liquid organic cpd. in gradually increasing volumes of the solvent up to 3 ml (maximum allowed volume) with shaking.

- ◆ When the cpd. is more soluble in aqueous acid or aqueous base than in water, such increased solubility is the desired positive test for acidic or basic functional groups.



Determination of Solubility Class Flow Chart

1. Water

- Water is a polar solvent with a dielectric constant equals to 80.
- It has the ability to form hydrogen bonding.
- It is amphoteric; it can act either as an acid or a base.

Therefore, it can dissolve:

1. Salts of ammonium ion (RNH_4^+) or organic acids salts with alkali metal cations (RCOO^-).
2. Ionic compounds.
3. Polar compounds “ like dissolves like ”.
4. Organic cpd.s with low m.wt (carbon < 5) such as alcohols, carboxylic acids , aldehydes & ketones .

2. Ether

- Ether is a non-polar solvent having a dielectric constant of 4.3.
- It cannot form H-bond (unassociated liquid).

It differs from water in that:

- **It cannot dissolve ionic cpd.s such as salts.**
- It is an organic solvent for non-polar cpd.s (like dissolves like).
- It dissolves most water insoluble cpd.s

Therefore, in the determination of solubility class, the importance of ether is for water soluble cpd.s only & no further solubility tests using the remaining solvents are to be done.

Accordingly, 2 probqabilities are there :

1- Cpd.s soluble in both water & ether.

These cpd.s are :

- Nonionic.
- Contain five or less carbon atoms.
- Contain an active group that is polar & can form H - bond.
- Contain only one strong polar group. This division of cpd.s is given **S1** class , it includes, **e.g.**, aldehydes, ketones & aliphatic acids.

2- Cpd.s soluble in water only (not is ether)

These cpd.s are:

- Ionic.
- Contain two or more polar groups with no more than four carbon atoms per each polar group.

This group is classified as **S2** class, it includes ionic salts such as salts of carboxylic acids & amines & cpd.s with more than one active group such as poly hydroxylated cpd.s & carbohydrates.

Note: The solubility in ether is tested only for water soluble cpd.s. for water insoluble cpd.s use the left side of the scheme, i.e., test solubility in sodium hydroxide solution rather than ether.

5% NaOH and 5% NaHCO₃

- Water insoluble cpd.s must be tested first in 5% NaOH solution which is a basic solvent. It reacts with water insoluble cpd.s that are capable of donating protons such as strong & weak acids.

❖ The stronger the acid, the weaker the base it can react.

Water insoluble cpd.s that dissolve in 5% NaOH sol. must also be tested for solubility in 5% NaHCO₃ sol. **Therefore**, for water insoluble acidic cpd.s NaOH sol. considered as a **detecting solvent** where as NaHCO₃ sol. is called as a **sub classifying solvent** since it can react with strong acids **only**.

That is, these two solvents give an idea about the acidity degree of the compound.

- Note that testing solubility in 5% NaHCO₃ sol. is not needed if the cpd. is insoluble in 5% NaOH sol., but rather, 5% HCl sol. should be used.

There are two probabilities

1. *Cpd.s soluble in both bases.*

This group is given class **A1** which includes, strong acids “which *can react with weak bases*”, carboxylic acids & phenols with electron withdrawing groups, e.g. –NO₂ “protons are weakly attached & can be given easily”.

2. *Cpd.s soluble in 5% NaOH sol. only.*

This group is given **class A2**, it includes, phenols, amides & amino acids (weak acids).

5% HCl sol.

If the cpd. is insoluble in water & NaOH sol. , this mean it is not an acidic compound but rather it may be a basic, neutral or inert cpd.. 5% HCl sol. can dissolve basic cpd.s such as amines (**RNH₂**).

If the cpd. is soluble in this solvent, then it is given **class B**, it includes primary, secondary, & tertiary amines.

Cold concentrated H₂SO₄

If the cpd. is insoluble in water, 5% NaOH sol. & 5% HCl sol., solubility in cold conc. H₂SO₄ should be tested.

If the cpd. is soluble in this acid it belongs to **class N** which includes, neutral cpd.s such as high m.wt. alcohols, aldehydes, ketones, esters & ethers (carbon atoms > 4) & unsaturated hydrocarbons.

On the other hand, cpd.s that are insoluble in cold conc. H₂SO₄ belong to **class I** , it includes inert aliphatic (saturated) hydrocarbons , aromatic hydrocarbons , haloalkanes & aryl halides.

Questions and exercises

- 1-** Water solubility test is the 1st test to run, explain why?
- 2-** Show by chemical equation, how can cold conc. H₂SO₄ dissolves oxygen containing neutral cpd.?
- 3-** Determine the solubility class & the nature of the following unknowns:
 - (a)** Unknown “ X ” is insoluble in water & gives bubbles with 5% NaHCO₃.
 - (b)** Unknown “ Y ” is insoluble in water , insoluble in 5% NaOH, but soluble in 5% HCl .



IDENTIFICATION OF ALCOHOLS

Alcohols: are organic compounds of the general formula **$R-OH$** , where R is any alkyl group. The alkyl group may be primary, secondary or tertiary and it may be open chain or cyclic, aliphatic or aromatic. Examples of alcohols include methanol, ethanol and cyclohexanol.

Physical properties

- ✓ Alcohols are colorless liquids with a special odor.
- ✓ Alcohols are miscible with water except benzyl alcohol, cyclohexanol, and secondary butanol (which is very slightly soluble in water).

Ignition: Aliphatic alcohol burn with blue flame (without smoke) while alcohols that contain aromatic ring burn with yellow flame (with smoke).



Chemical properties

- ♦ Alcohols are neutral compounds that don't change the color of the litmus paper.
- ♦ All reactions of alcohols are related to its active hydroxyl and are of two types:
 - a) Removal of the hydroxyl itself as in the reaction with hydrogen halides. To form alkyl halides or in the dehydration reaction to form double bond.
 - b) Removal of the proton only of the hydroxyl as in the formation of esters or in the reaction with active metals such as sodium.

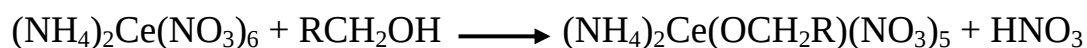


Chemical Reactions

General test (Ceric ammonium nitrate reagent)

Ceric ammonium nitrate (yellow solution) is an oxidizing agent that reacts with alcohols to give a red complex and with phenol to give a brown to greenish brown precipitate.

This test gives positive result with primary, secondary and tertiary alcohols up to 10 carbons.



Yellow

Red complex

a) Water soluble (miscible) alcohols: Mix two drops of alcohol with one drop with ceric ammonium nitrate solution. A red complex indicates a positive test.

b) Water insoluble (immiscible) alcohols: Mix two drops of alcohol with 0.5 ml of dioxane, shake well and add one drop of the reagent to get a positive red complex.

Lucas test

The Lucas test is a chemical test used to detect the type of alcohol (primary, secondary, or tertiary) based on the alcohol's reaction with Lucas reagent. Lucas reagent is a mixture of concentrated hydrochloric acid (HCl) and anhydrous zinc chloride (ZnCl₂).

Principle of the test:

The test is based on the alcohol's ability to form alkyl chloride when reacted with Lucas reagent. This reaction occurs at different rates depending on the type of alcohol.

Test results:

- **Tertiary alcohols:** React very quickly with Lucas reagent, forming a white suspension (alkyl chloride precipitate) immediately or within a few seconds.
- **Secondary alcohols:** React more slowly, with the white suspension appearing after several minutes.
- **Primary alcohols:** Do not react or react very slowly with Lucas reagent, with no white suspension forming or only forming after several hours.

Interpretation of results:

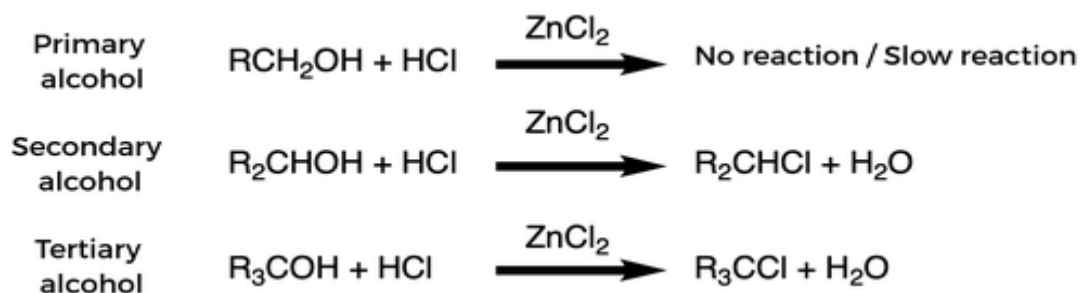
- The rapid reaction of tertiary alcohols is due to the stability of the carbocation formed during the reaction, as the tertiary carbocation is more stable than secondary and primary ones.

Uses:

This test is used in quick chemical analyses to classify alcohols and determine their type based on the speed of their reaction with the reagent.

The mechanism of this reaction involves a carbocation intermediate: first an OH⁻ ion is removed from the alcohol, leaving the carbon atom in the molecule positively charged. Then a Cl⁻ ion adds to the positively charged carbon atom. The result is the

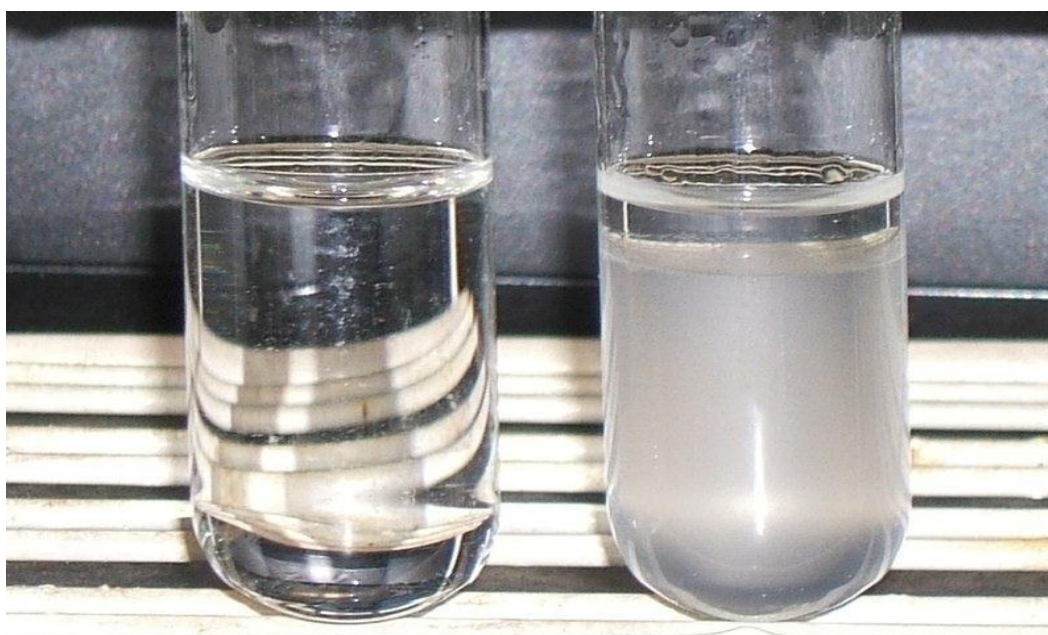
alcohol is converted to a chlorinated alkane. Since tertiary carbocations are much more stable than primary or secondary carbocations, tertiary carbocations would be faster in performing the reaction.



Procedure

Mix 1 ml of alcohol with 2 ml of Lucas reagent, shake and heat gently then observe the results:

- ❖ Tertiary alcohols give two phases that separate within 2-3 minutes.
- ❖ Secondary alcohols give two phases that separate after 15- 20 minutes (give a cloudy solution).
- ❖ In primary alcohols one layer appears.



- ◆ negative test (left) with ethanol, and positive test (right) with t-butanol

The Iodoform Test

Is a qualitative test used to detect compounds containing a methylcarbinol group ($\text{-CH}_3\text{CH(OH)}$), which include some alcohols and ketones? This test is particularly useful for detecting secondary alcohols that have a methyl group attached to the carbon atom bearing the hydroxyl group, such as ethanol and acetone.

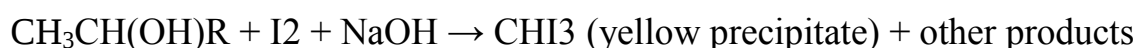
Substances that can be detected

- Secondary alcohols that have a methyl group, such as 2-propanol
- Ketones such as acetone.
- Ethanol (although it is a primary alcohol)

Steps of the Iodoform Test:

1. A small amount of the sample (alcohol or ketone) is added to a solution containing iodine (I_2) and sodium hydroxide (NaOH).
2. If the compound contains a methylcarbinol group, an oxidation reaction occurs that produces a yellow precipitate of iodoform (CHI_3).
3. The precipitate appears as a yellow precipitate with a characteristic odor similar to that of medical disinfectants.

Chemical formula for the reaction:



Compounds that give a positive result:

- a. Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$)
- b. 2-Propanol ($\text{CH}_3\text{CH(OH)CH}_3$)
- c. Acetone (CH_3COCH_3)

Compounds that do not give a positive result:

1. Unmethylated primary alcohols such as methanol (CH_3OH).
2. Secondary alcohols or ketones that do not contain a methyl group next to the carbonyl group.

Importance of the iodoform test:

- It is mainly used in analytical organic chemistry to detect secondary alcohols and ketones.
- It helps in determining the presence of compounds that have the appropriate chemical structure for the iodoform reaction.

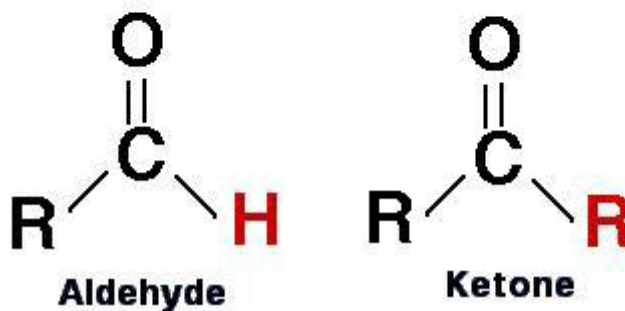
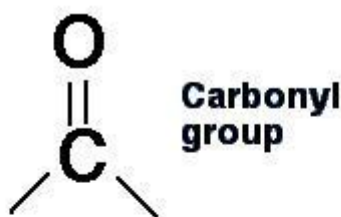
Questions

1. Write an equation to show the reaction between tert-butyl alcohol and Lucas reagent.
2. How to distinguish between primary, secondary and tertiary alcohol?

Aldehydes and Ketones

ALDEHYDES: STRUCTURE AND NOMENCLATURE

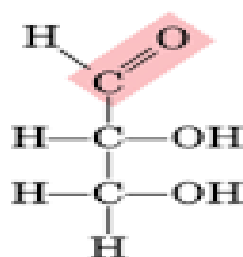
Aldehydes and ketones are organic compounds which incorporate a carbonyl functional group, **C=O**. The carbon atom of this group has two remaining bonds that may be occupied by hydrogen or alkyl or aryl substituents. If at least one of these substituents is hydrogen, the compound is an **aldehyde** RCHO or RCH=O . If neither is hydrogen, the compound is a **ketone** RCOR' (R and R' =alkyl or aryl).



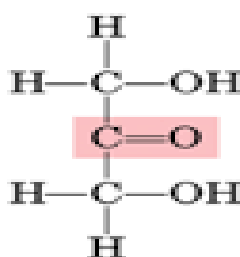
Some Common Classes Carbonyl Compounds

Class	General Formula	Class	General Formula
ketones	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{R}'$	aldehydes	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{H}$
carboxylic acids	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{OH}$	acid chlorides	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{Cl}$
esters	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{O}-\text{R}'$	amides	$\text{R}-\overset{\text{O}}{\underset{\parallel}{\text{C}}}-\text{NH}_2$

Aldehyde - ethanal $\begin{array}{c} \text{H} \\ \\ \text{H}-\text{C}-\text{C}=\text{O} \\ \quad \diagdown \\ \text{H} \quad \text{H} \end{array}$ • Carbonyl group at the end of the carbon chain • Suffix -al • Functional group is -CHO	Ketone - propanone $\begin{array}{c} \text{H} \quad \text{O} \quad \text{H} \\ \quad \parallel \quad \\ \text{H}-\text{C}-\text{C}-\text{C}-\text{H} \\ \quad \quad \\ \text{H} \quad \quad \text{H} \end{array}$ • Carbonyl group is not at the end of the carbon chain • Suffix -one • Functional group is -CO
--	--



Aldose



Ketose

Difference Between Aldehyde and Ketone

♦ Chemical Structure

- Aldehydes have the form of R-CHO.
- ketones have the form of R-CO-R'.

♦ Reactivity

- Aldehydes are more reactive than ketones. Aldehydes undergo oxidation forming carboxylic acids.
- Ketones cannot be oxidised without breaking the carbon chain.

♦ IUPAC Nomenclature

- Aldehydes end with the suffix 'al'
- Ketones end with the suffix 'one.'

♦ Location of Carbonyl Group

- Aldehydes always occur at the end of a carbon chain.
- Ketones always occur at the middle of the chain.

♦ Natural Occurrence

- Aldehydes are usually found in volatile compounds such as fragrance compounds.
- Ketones are commonly found in sugars and are referred to as ketoses in general. However, there are aldehyde sugars which are called aldoses

Difference Between Aldehyde and Ketone

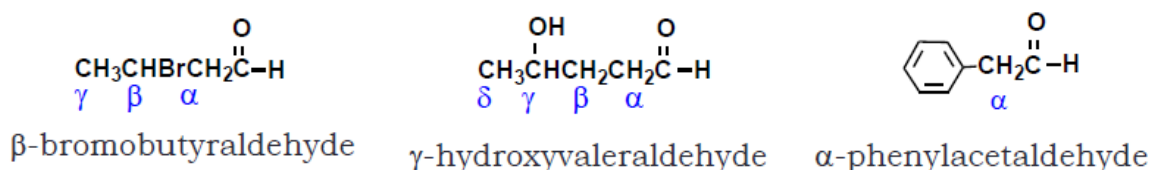
Aldehydes have the form of $R-CHO$	Ketones have the form of $R-CO-R'$
More reactive than ketones; Aldehydes undergo oxidation forming carboxylic acids	Cannot be oxidised without breaking the carbon chain
Always occur at the end of a carbon chain	Always occur in the middle of the chain
Usually found in volatile compounds	Usually found in sugars

Common Names of Aldehydes

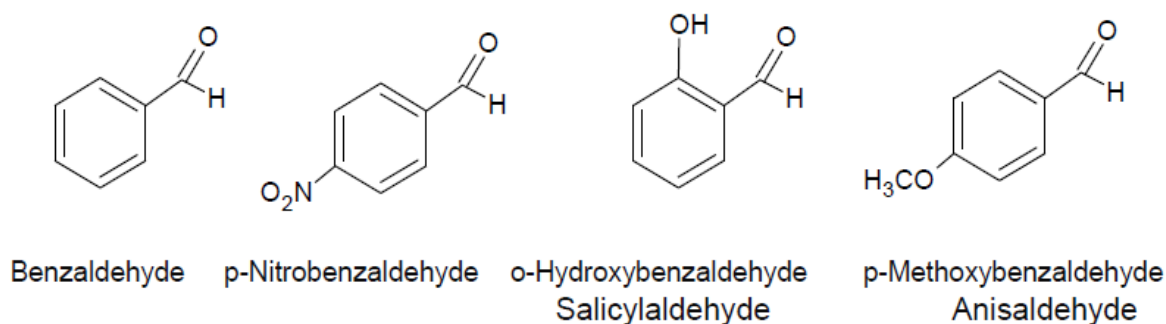
- In the common system, [aldehydes](#) are named from the common names of the corresponding carboxylic acid.
- The „*ic acid*’ ending is replaced with „*aldehyde*”.
- The aldehyde group is always [at the end](#) of a chain (terminal).

Structure	IUPAC name	Common name	Structure	IUPAC	Common name
HCO_2H	methanoic acid	formic acid	HCHO	methanal	formaldehyde
$\text{CH}_3\text{CO}_2\text{H}$	ethanoic acid	acetic acid	CH_3CHO	ethanal	acetaldehyde
$\text{CH}_3\text{CH}_2\text{CO}_2\text{H}$	propanoic acid	propionic acid	$\text{CH}_3\text{CH}_2\text{CHO}$	propanal	propionaldehyde
$\text{CH}_3(\text{CH}_2)_2\text{CO}_2\text{H}$	butanoic acid	butyric acid	$\text{CH}_3(\text{CH}_2)_2\text{CHO}$	butanal	butyraldehyde
$\text{CH}_3(\text{CH}_2)_3\text{CO}_2\text{H}$	pentanoic acid	valeric acid	$\text{CH}_3(\text{CH}_2)_3\text{CHO}$	pentanal	valeraldehyde
$\text{CH}_3(\text{CH}_2)_4\text{CO}_2\text{H}$	hexanoic acid	caproic acid	$\text{CH}_3(\text{CH}_2)_4\text{CHO}$	hexanal	caproaldehyde

- Substituents locations are given using Greek letters (α , β , γ , δ , ϵ , ω .) beginning with the carbon *next to* the carbonyl carbon, the α -carbon.

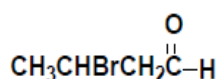


- Aromatic aldehydes are usually designated as derivatives of the simplest aromatic aldehyde, **Benzaldehyde**

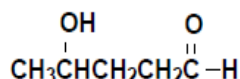


IUPAC Nomenclature of Aldehydes:

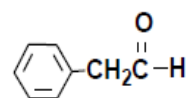
- Select the longest continuous carbon chain that contains the $\text{C}=\text{O}$ group and replace the ending -e by the suffix **-al**
- The **CHO** group is assigned the number "1" position and takes precedence over other functional groups that may be present such as $-\text{OH}$, $\text{C}=\text{C}$
- If the CHO group is bonded to a ring, name the **ring** and add the suffix **-carbaldehyde**.



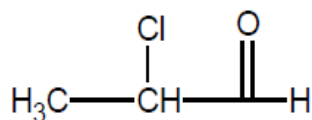
3-bromobutanal



4-hydroxypentanal



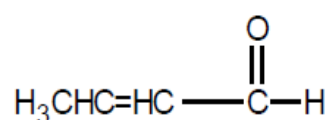
2-phenylethanal



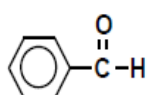
2-Chloropropanal



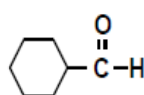
3-Hydroxypropanal



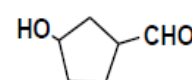
2-Butenal



benzenecarbaldehyde



cyclohexanecarbaldehyde



3-hydroxycyclopentanecarbaldehyde

Nomenclature of Ketones

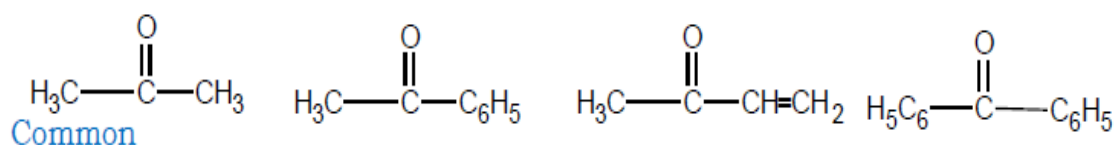
Common name:

listing the alkyl substituents attached to the carbonyl group alphabetically, followed by the word ketone. As with aldehydes, substituents locations are given in common names using Greek letters

(α , β , γ , δ , ϵ , ω .) beginning with the α -carbon.

❖ IUPAC system:

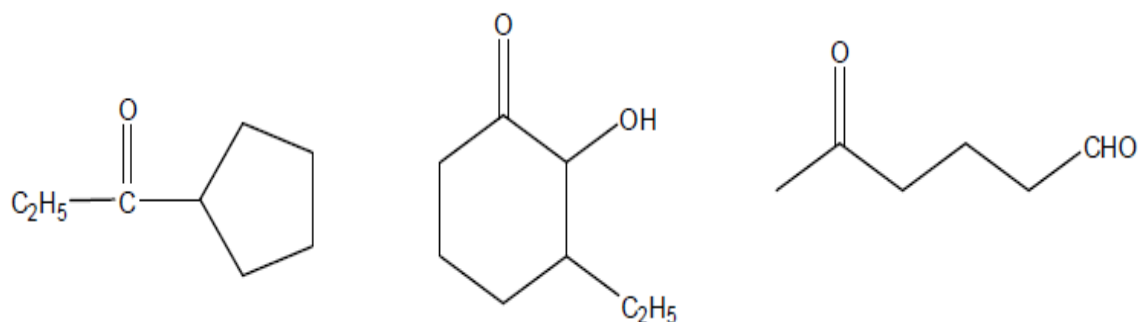
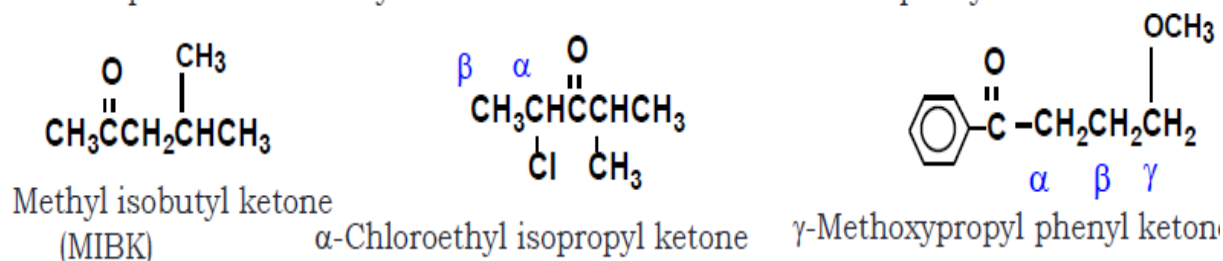
- Find the longest chain containing the carbonyl group, and change the *-e* ending of the parent alkane to the suffix *-one*.
- Number the carbon chain to give the carbonyl carbon the lower number. Apply all of the other usual rules of nomenclature.
- Ketones are just below aldehydes in nomenclature priority.
- A ketone group is named as an „oxo“ substituent in an aldehyde.



Dimethyl ketone Methyl phenyl ketone Methyl vinyl ketone Diphenyl ketone
Acetone Acetophenone Benzophenone

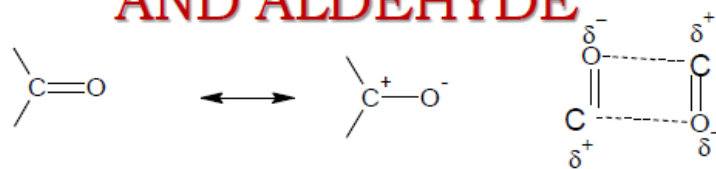
IUPAC

Propanone Phenyl ethanone 3-Buten-2-one Diphenylmethanone

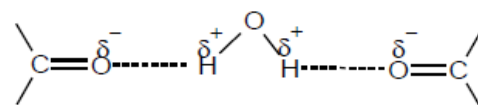
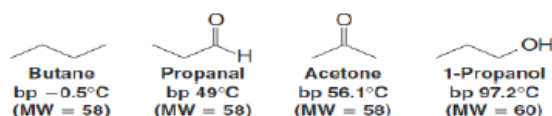


Cyclopentylpropanone 3-Ethyl-2-hydroxycyclohexanone 5-Oxohexanal

PHYSICAL PROPERTIES OF KETONES AND ALDEHYDE

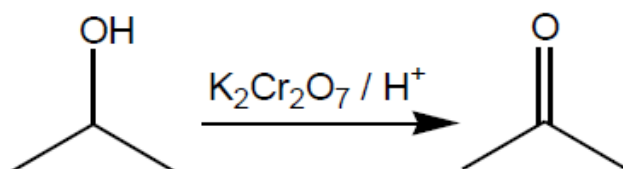
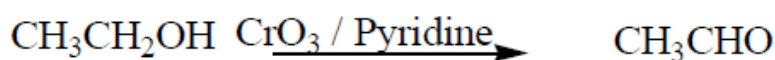


- Aldehydes and ketones are **polar** compounds, Because the polarity of the carbonyl group.
- Polarization of CO group creates **Dipole-dipole attractions** between the molecules of aldehydes and ketones, resulting in **higher boiling points** than **nonpolar alkanes and ether**.
- aldehydes and ketones **lower** than **alcohols** Because Dipole-dipole attractions, are not as strong as interactions due to hydrogen bonding.
- The lower aldehydes and ketones are soluble. Acetone, formaldehyde and acetaldehyde are miscible in water.

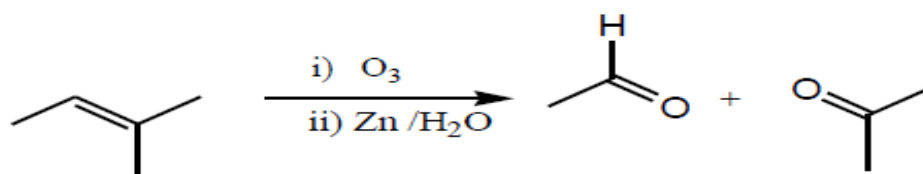


Preparation of Aldehydes and Ketones

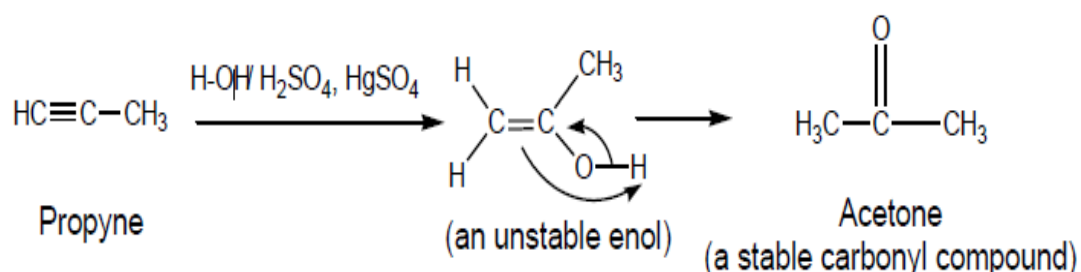
1. Oxidation of alcohols



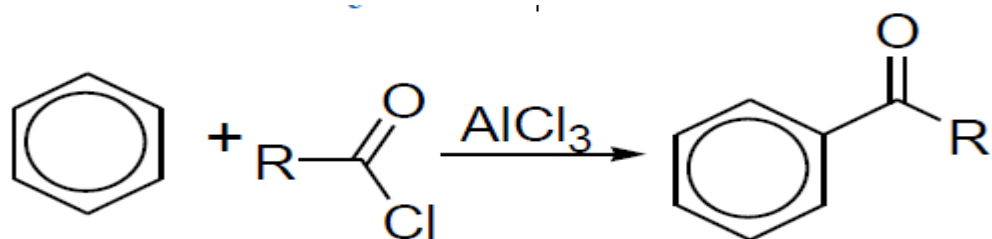
2. Ozonolysis of alkenes



3. Hydration of alkynes

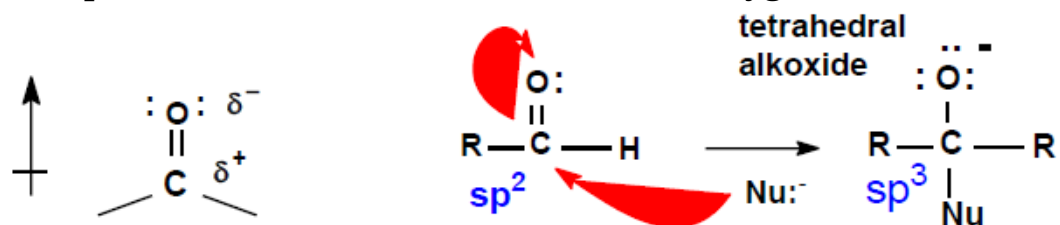


4. Friedel Crafts acylation



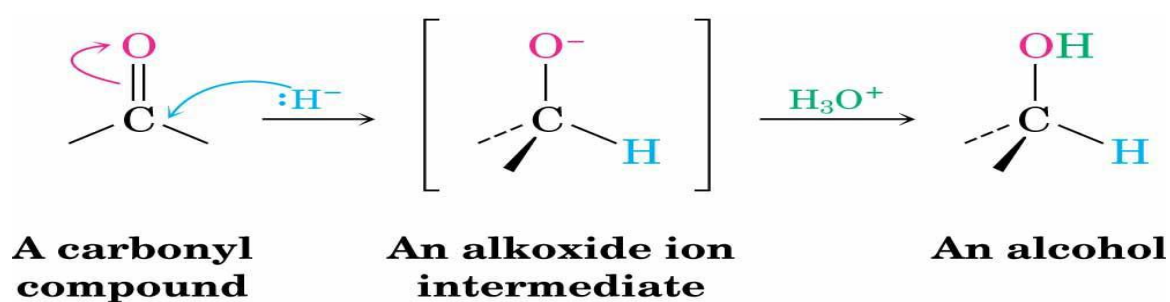
REACTIONS OF ALDEHYDES AND KETONES

Nucleophilic Addition Reaction to the carbon-oxygen double bond.

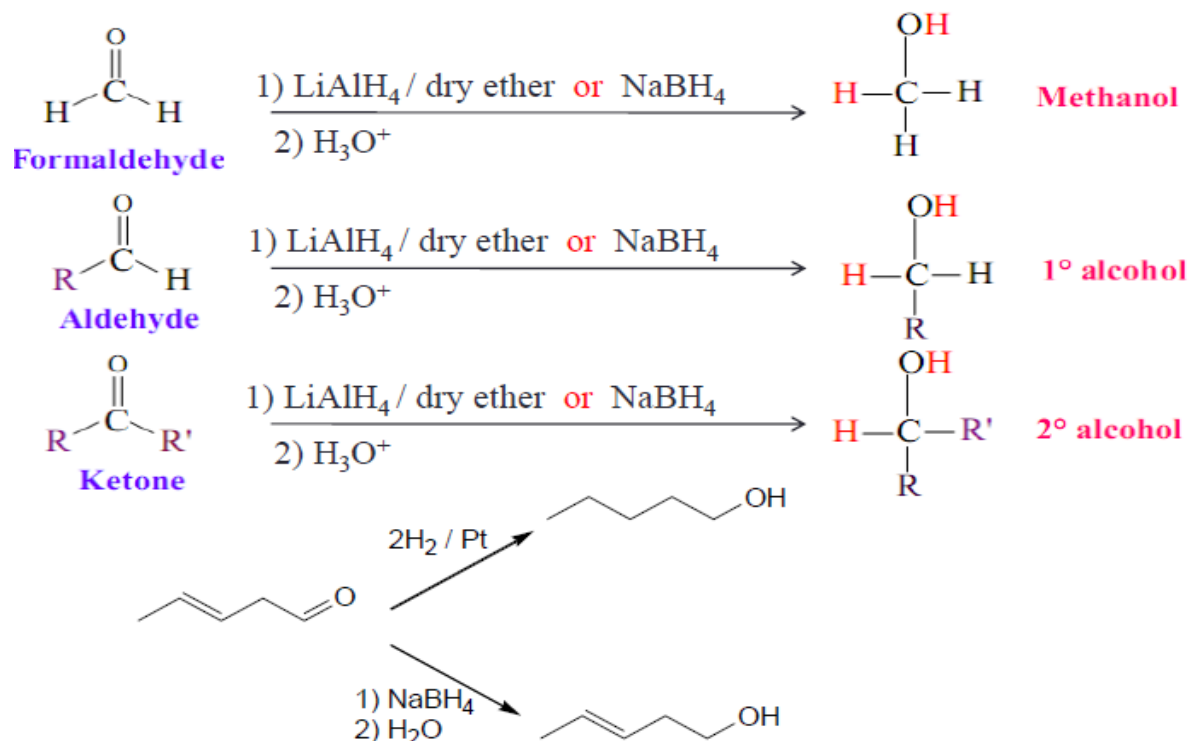


1. Reduction of carbonyl group

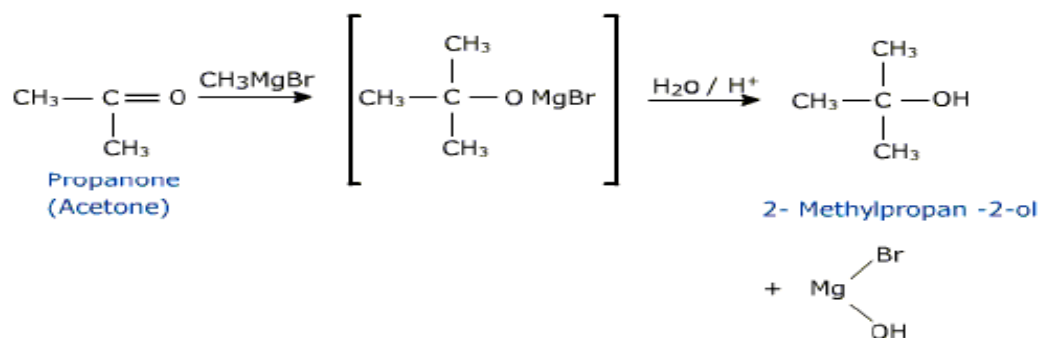
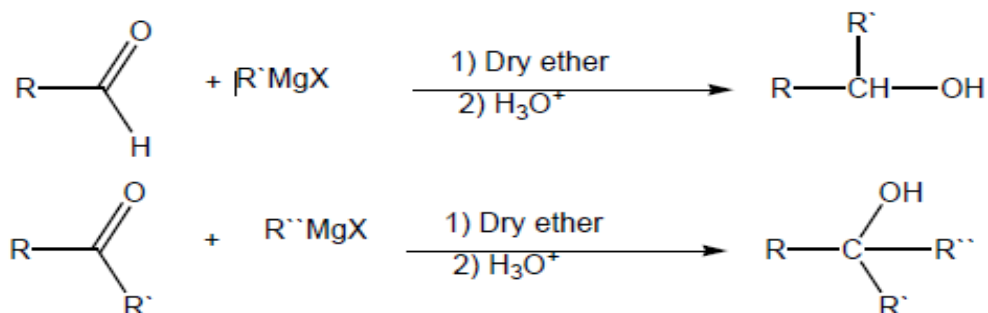
Addition of metal hydrides: Formation of alcohols.



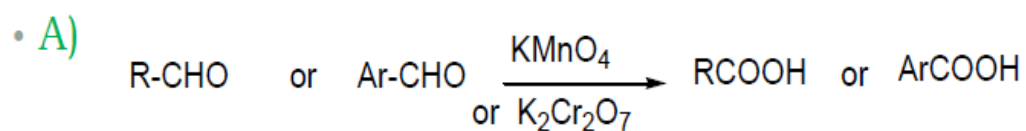
Reduction by hydride reagents, Lithium aluminium hydride LiAlH₄ or Sodium boron hydride NaBH₄.



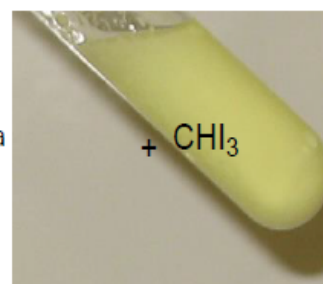
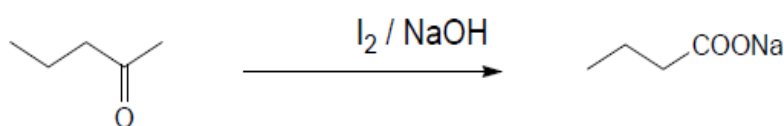
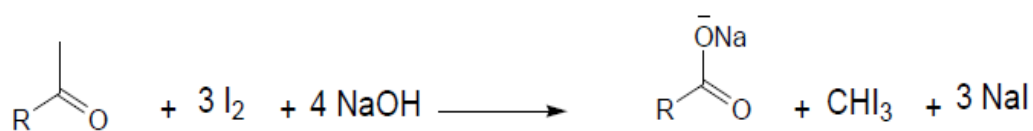
2. Addition of Grignard Reagents: Formation of alcohols



3. Oxidation reaction

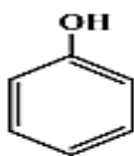


- B) **Iodoform reaction:** The reaction occurs in any aldehyde or ketone containing CH_3CO .

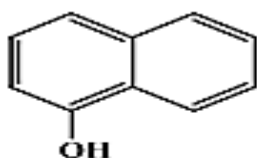
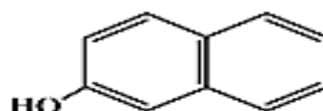
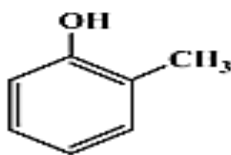


Identification of Phenols

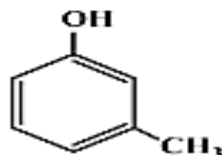
Phenols are organic compounds with direct benzene or substituted benzene bound to a hydroxyl ring. They have the general formula Ar-OH. Phenols, according to the number of OH groups, are categorized into mono-, di-, and trihydric; Monohydric: such as phenol, o-cresol, m-cresol, p-cresol, beta- and alpha-naphthol. Dihydric: such as hydroquinone and resorcinol. Trihydric: Pyrogallol, for instance.



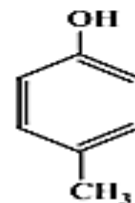
phenol

 α -naphthol β -naphthol

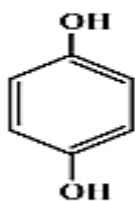
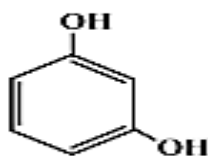
o-cresol



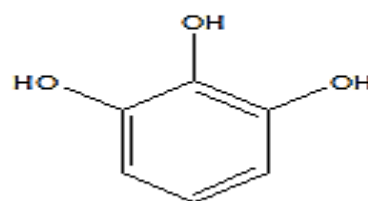
m-cresol



p-cresol

hydroquinone
(quinol, hydroquinol)

resorcinol



Pyrogallol

Physical properties

1. Because of intermolecular hydrogen bonding, they have high boiling points.
2. Owing to air oxidation, they are colored and have a special odor. Pure substances are colorless.
3. Liquids (e. g., o- and m-cresol) or stable crystalline compounds are phenols (e. g., phenol and resorcinol).

4. Due to its capacity to create hydrogen bonding with water, phenol is itself soluble in water. For other phenols, by increasing the molecular weight, the solubility in water decreases. Owing to the presence of the aromatic ring, phenols burn with a yellow smoky flame.

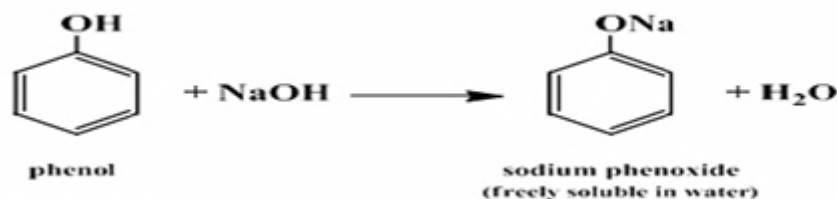
Chemical properties

Phenols are weak acid compounds, but they are soluble only in solid alkaline solutions (e. g., sodium hydroxide solution). For this reason, they fall into solubility class A2. The existence of the withdrawing group of phenyl ring electrons strengthens the acidity of the phenol, allowing it solubility class A1(e. g, nitrophenol). Phenol itself however, since it is soluble in water, is of solubility class S1.

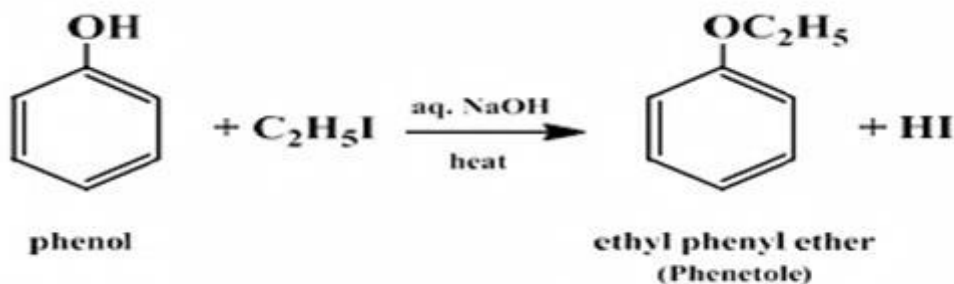
Types of phenols reactions

1. Reactions at the phenolic hydroxyl group (-OH); e.g., ether formation and salt formation:

- phenol reacts with sodium hydroxide to form sodium phenoxide.

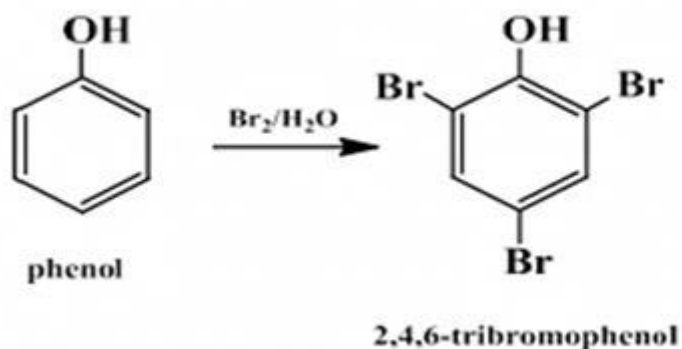


phenol reacts with ethyl iodide; Ethyl phenyl ether is formed.



2. Aromatic ring replacement, e.g., bromination and nitration reactions:

- Reaction with bromine water.



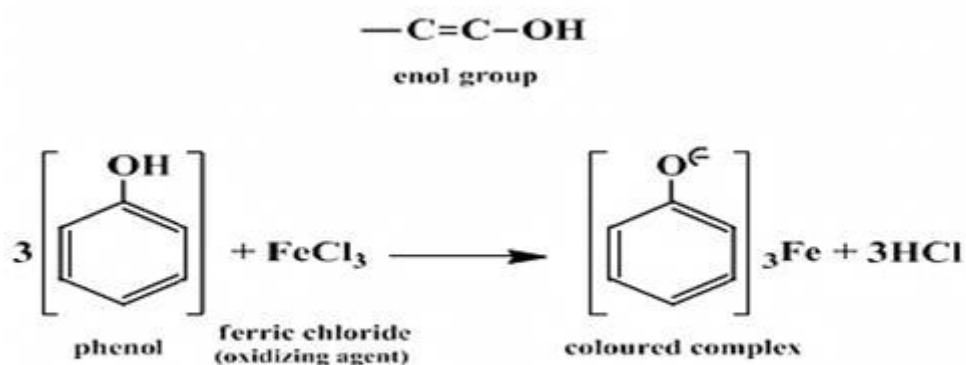
- Reaction with dilute nitric acid.



Chemical Reactions

1. Reaction with ferric chloride

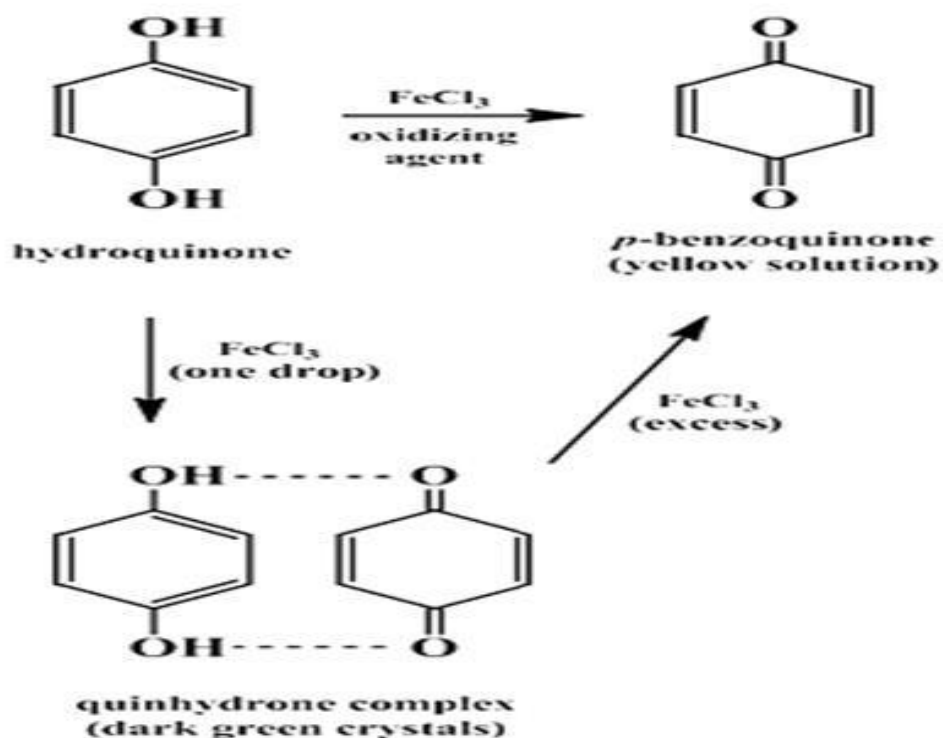
Due to the presence of the enol group, phenols react with ferric chloride to give colored compounds. This reaction is actually known to be a test for any compound with the enol group.



Procedure

- 1) To a very dilute phenol aqueous solution (30-50 mg in 1-2 mL of water) or to a few stable phenol crystals (50-100 mg) dissolved in water.
- 2) Apply 1 drop of a solution of ferric chloride and observe the resulting colour:

Compound	Colour
Phenol, <i>m</i> -cresol, Resorcinol	Violet or blue
α - and β -naphthol	No special colour
Hydroquinone	Deep green
<i>o</i> - and <i>p</i> -cresol	Greenish blue



2. Reaction with bromine water

This reaction is an example of the phenyl ring replacement reaction (mentioned earlier).

Procedure:

1) Add the bromine water progressively to a concentrated aqueous solution of the phenol or to the phenol itself. The bromine is first decolorized and then a white or yellowish-white precipitate of a poly-bromo-derivative is formed with all phenols except hydroquinone and alpha- and beta-naphthol when applied to an excess.

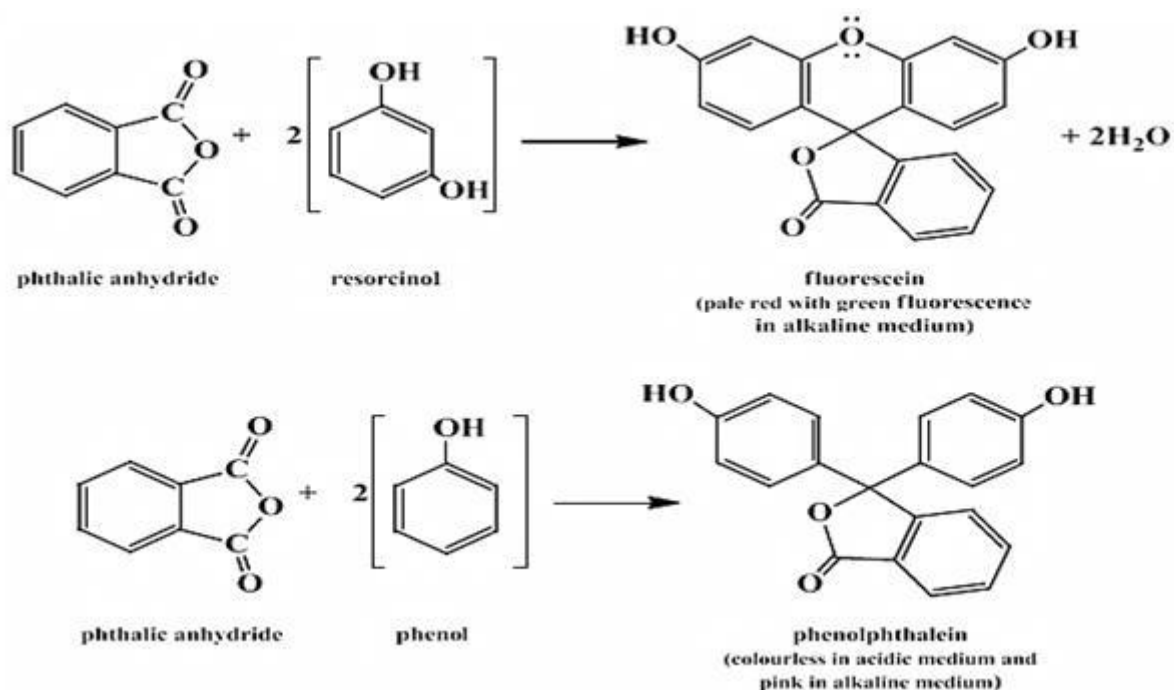
2) A deep red coloration is created when bromine water is gradually added to a hydroquinone solution followed by the separation of deep green crystals that then dissolve, giving a yellow solution.

3) Naphthols decolorize bromine water, but typically no bromine compound precipitate can be obtained.

Due to the difficulties of separating the bromo compound from the initial phenol, this test is not quite satisfactory for certain phenols that are insoluble in water.

3. Phthalein test

Yield of Phenols When reacted with phthalic anhydride and a small amount of concentrated sulphuric acid, some phenols yield phthaleins in alkaline solutions that offer unique colors (sometimes with fluorescence). The following are examples of Phenol and Resorcinol:



Resorcinol's fluorescence is due to the presence of an oxygen linkage Between the phenolic nuclei of the two (in basic medium).

Procedure

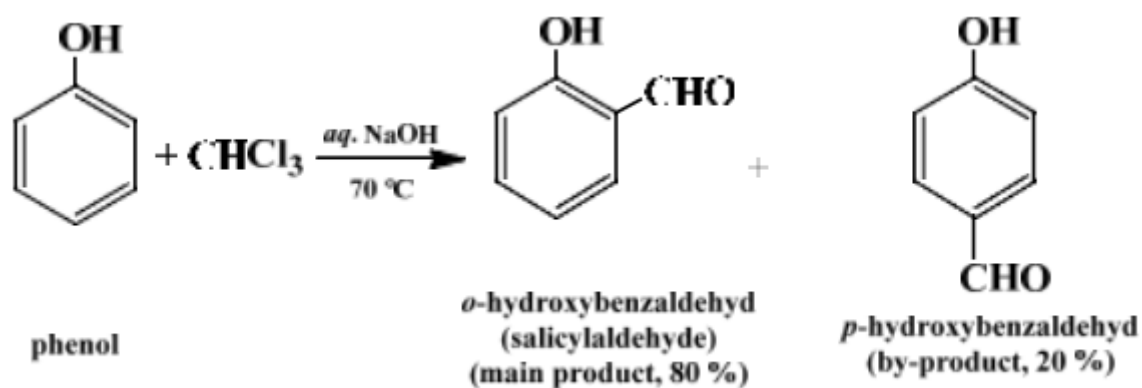
- 1) Place about 0.1 g of phenol and an equivalent quantity of phthalic anhydride or phthalic acid in a dry test tube, mix well and add 1-2 drops of concentrated sulphuric acid.
- 2) Heat gently for 1 minute on a direct flame before the mixture's crystals melt and fuse. Cool the test tube and apply more than 10% of the sodium hydroxide solution. The results should be:

Compound	Colour
α -naphthol	Green colour
m-cresol	Blue to pink
Phenol	Red to pink
<i>o</i> -cresol	Red-violet
β -naphthol	Very pale green with slight fluorescence
<i>p</i> -cresol	No change
Resorcinol	Pale red colour with green fluorescence
Hydroquinone	Violet colour

If the resultant colour is not so clear you can dilute with water.

4. Reimer-Tiemann Reaction

The treatment of phenol with a solution of chloroform and aqueous sodium hydroxide introduces an aldehyde group (-CHO) into the **ortho-** or **para-**position aromatic ring:



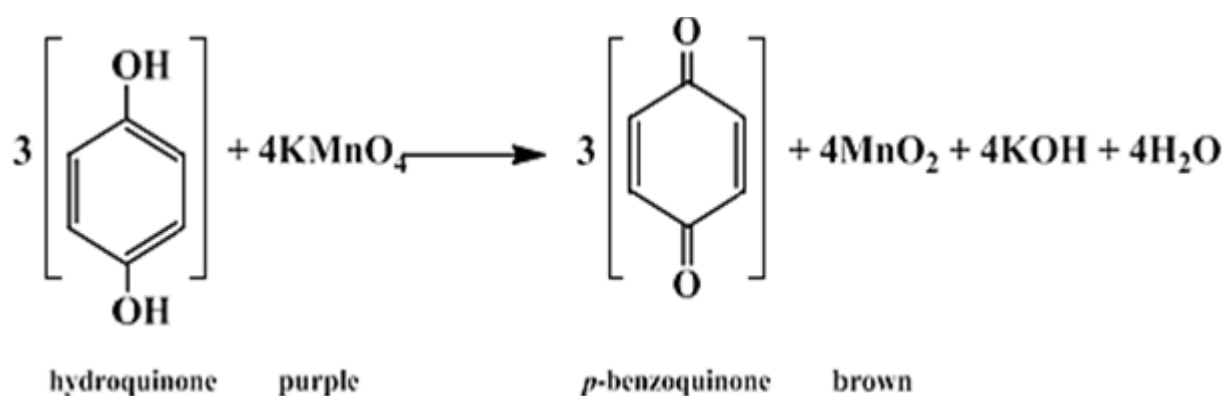
Procedure

- 1) Add 1mL of 30 percent sodium hydroxide solution and 1 mL of chloroform to around 0,2 g of phenol.
- 2) Heat and note the colour of the aqueous layer in the water bath.:

Compound	Colour
Phenol	Yellow or no colour
Hydroquinone	Deep brown
α -naphthol	Dark green
m-cresol	Yellow
o-cresol	Deep orange
β -naphthol	Pale orange
p-cresol	Deep blue that turns to green
Resorcinol	Red colour with weak fluorescence

5. Reduction of potassium permanganate

Phenols reduce solutions of potassium permanganate and undergo quinone oxidation. The manganese is reduced to +4, which is brown, from +7, which gives a purple solution. For dihydroxylated phenols, this test is extremely effective compared to phenol itself.



Procedure

- 1)** Add **2%** aqueous potassium permanganate solution drop by drop with shaking until the purple colour of the permanganate persists.
- 2)** For 0.1 g or 0.2 mL (3-4 drops) of the compound, add 2 mL of water or ethanol.
- 3)** If the color of the permanganate is not changed within **0.5-1** minutes, allow the mixture to stand for **5** minutes with vigorous shaking periodically. A good outcome for the presence of phenols is the absence of the purple color and the formation of a brown suspension, which is manganese (**II**) **oxide**, at the bottom of the test tube.