

KEVA INTERNSHIP

By Samika Jain

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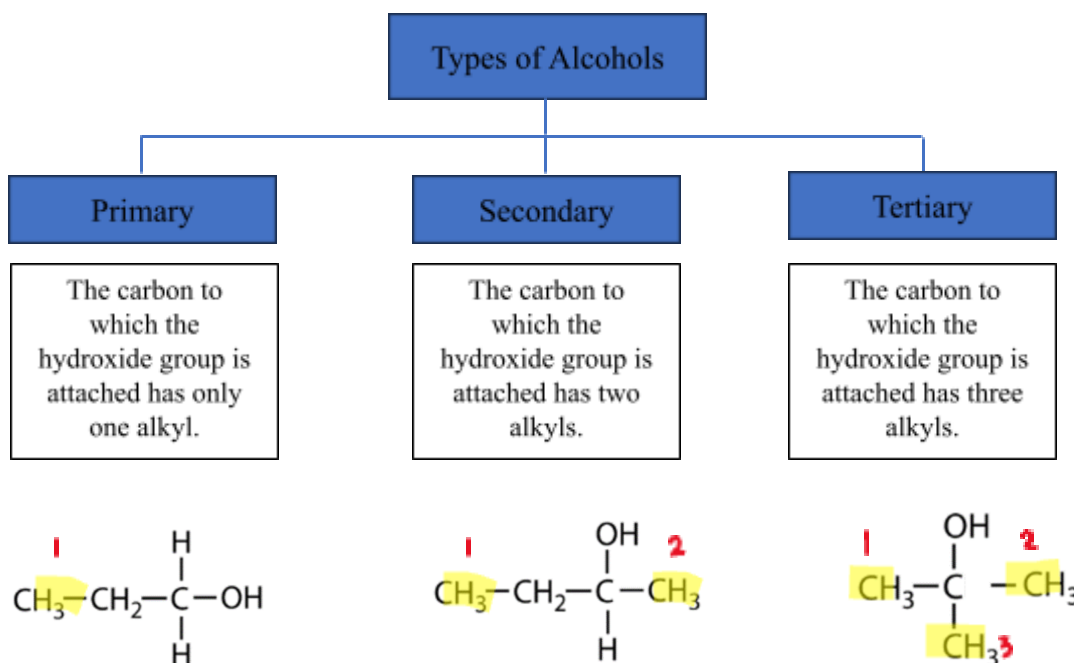
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ALCOHOLS

The Three types of Alcohols

There are primarily 3 main types of alcohols. These include:

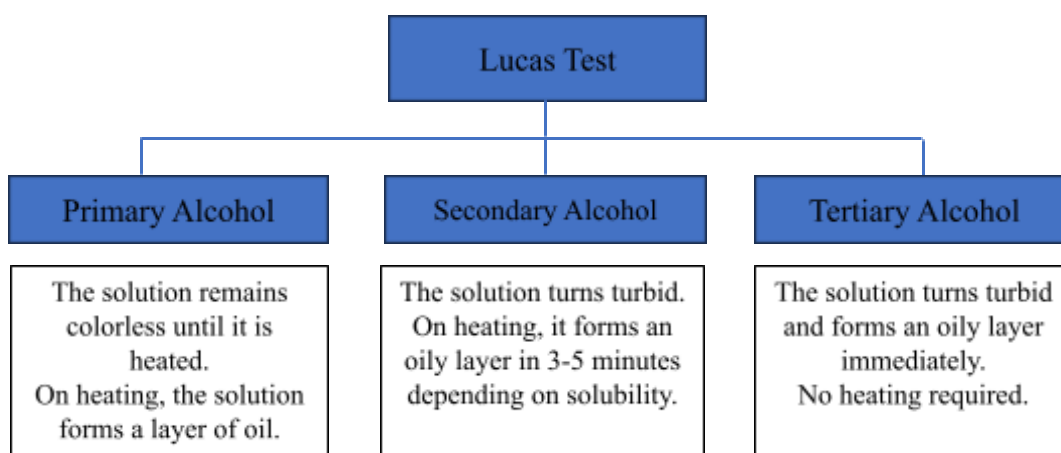
- (i) Primary Alcohol
- (ii) Secondary Alcohol
- (iii) Tertiary Alcohol

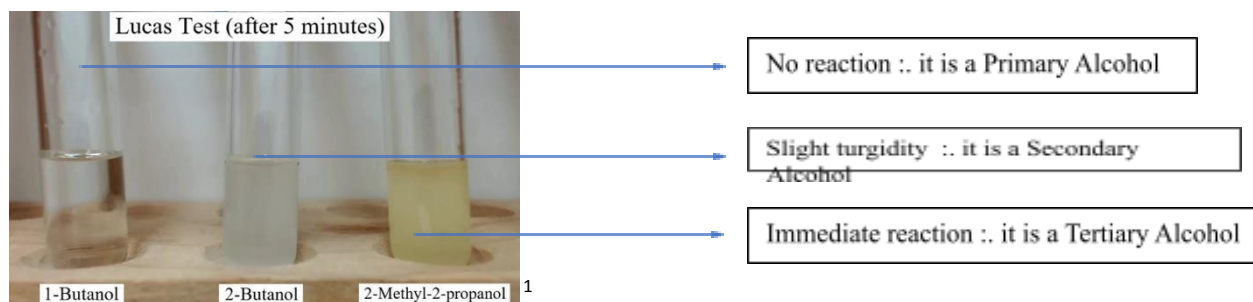


Tests for the 3 types of Alcohols

There are primarily 3 main types of alcohols. These include:

- (i) **Lucas Test**
 - a solution of anhydrous zinc chloride in concentrated hydrochloric acid called the Lucas reagent.
 - Based on the presence of turbidity or oil formation, one can differentiate the 3 types of alcohols.





The Main Difference between Primary and Secondary Alcohols

PRIMARY ALCOHOLS	SECONDARY ALCOHOLS
Less reactive	More reactive
High rate of esterification	Low rate of esterification
Difficult to dehydrate	Easy to dehydrate
When oxidized results in aldehydes	When oxidized results in ketones
More acidic in nature	Less acidic in nature
No oily layer in the Lucas Test	Forms oily layer in the Lucas Test
Red color in Myer's Test	Blue color in Myer's Test

OXIDISING ALCOHOLS

Oxidizing the Three types of Alcohols

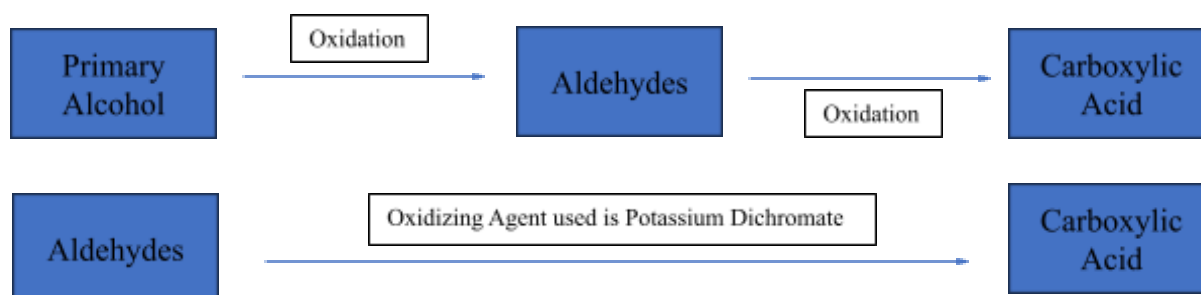
There are primarily 3 main types of alcohols. When oxidized, they form these reactions:

- (i) Primary Alcohol - Aldehydes
- (ii) Secondary Alcohol - Ketones
- (iii) Tertiary Alcohol - None

The Functional Groups

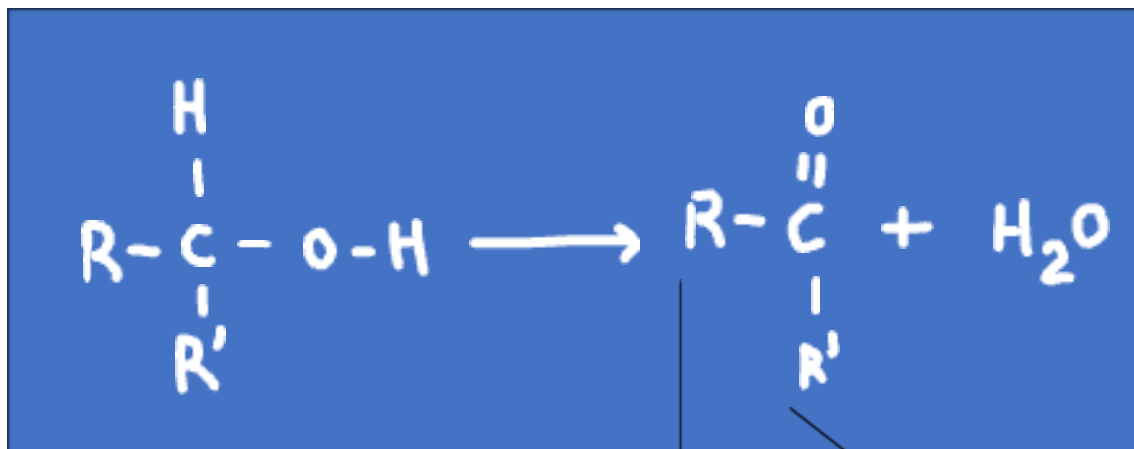
Both Aldehydes and Ketones have a carbonyl functional group.

- (i) Aldehydes – (RCHO)
- (ii) Ketones – (RCO)



¹ "Google Image Result for <https://i.ytimg.com/vi/ZuAKxcxdDF8/Maxresdefault.jpg>," www.google.com, accessed June 26, 2023

In Summary:



If either (or both) of these is Hydrogen, then it's an aldehyde.

If both R and R' are alkyl groups, then it's a ketone.

COLUMN CHROMATOGRAPHY

It is a technique which is used to separate a single chemical compound from a mixture dissolved in a fluid.



Like the name suggests, column chromatography involves the use of an apparatus called the column. The dark upper regions are the impurities that have been collected towards the upside of the column.

The picture shows the process in which a musk scent is trying to be made.

These conical flasks are where the solvent, or liquid needed, is collected in several batches.

The process in which Keva creates new solvents is carried out in two simple steps:

- (i) Researching already available scholarly articles
- (ii) Replicating the compounds
- (iii) Modifying parts of the compound

In the picture, the column's size has been appropriately chosen by the mass of the solution needed to be used i.e., 40 grams.

Allow the solvent front to reach $3/4^{\text{th}}$ of the level of the paper.

Silica Gel separation

Column chromatography is the ideal method of chromatography for purification and separation. It is a technique in which the stationary phase is solid adsorbents like silica gel and activated alumina powder and the mobile phase is a liquid.

The principle of active compound separation depends on the activity of adsorbents and polarity of the solvent. If the polarity of the solvent is very low and the activity of the adsorbent is strong and high, then result of separation of compound is good. On the other hand, if the polarity of the solvent is very high and the activity of adsorbents is high then it gives poor results of compound separation. It means purification and isolation of compounds are not 100% pure.

Silica Gel is seen in the lab in the form of sheets: One side is aluminum, and the other is coated in a thin layer of silica, which will help see the separation.



The Silica acts like the stationary phase while the solvent is the mobile phase.

Using the general formula for a chemical reaction: $A + B \rightarrow C$, we can see that C's chromatogram has both parts of B and A.

The dot seen here shows the start solution. Some of it can be seen because as the time of the reaction goes on, the reactants get used up \therefore Showing a small, faded dot.

Polarity:

Polarity is the temporary separation of charges in a compound. Column chromatography depends on the polarity of the solvent too. The more polar the compound, the more the interaction with the solvent.

In the set-up above, 99% hexane was used to ensure that polarity was low. Hexane is highly non-polar which is helpful because if the compound is polar, then a non-polar solvent will be needed to ensure that it is not flushed out.



2



3

Here are some Locating Agents:

² "Silica Gel Column Chromatography," Sorbead India, n.d.

³ "MilliporeSigma TLC Silica Gel 60 Aluminum Sheets - Partition Chromatography, Thin Layer Chromatography," www.fishersci.com, accessed June 26, 2023



Some examples include:

- (i) KMnO_4
 - Oxidizing agent
 - Pink in color
 - Double bond tests

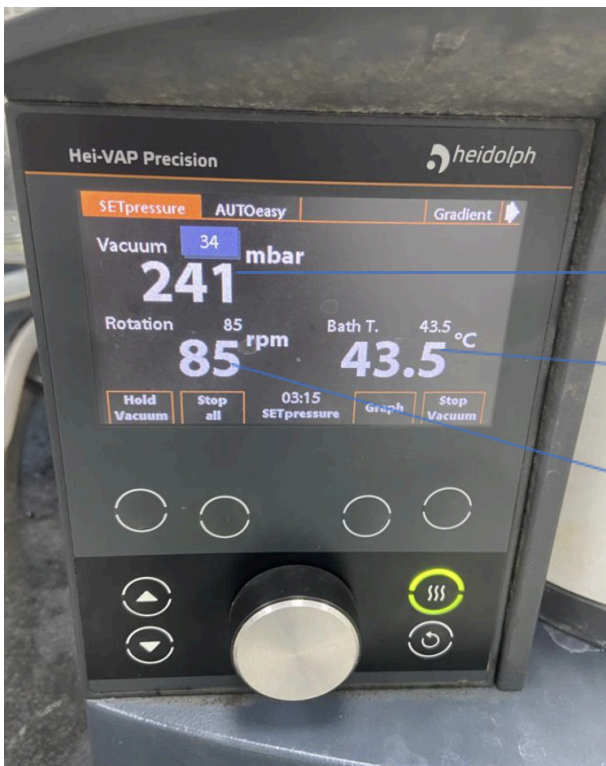
- (ii) PMA
 - Usually only used for locating aldehydes
 - Always used as a stain

- (iii) Iodine
 - Brown gas
 - Can be used in general as a stain for anything

ROTARY EVAPORATOR

It is a piece of equipment primarily used to remove solvent from a sample through "evaporation under reduced pressure".

The presence of reduced pressure in the apparatus causes the solvent (in the round bottom flask) to boil at a lower temperature than normal.

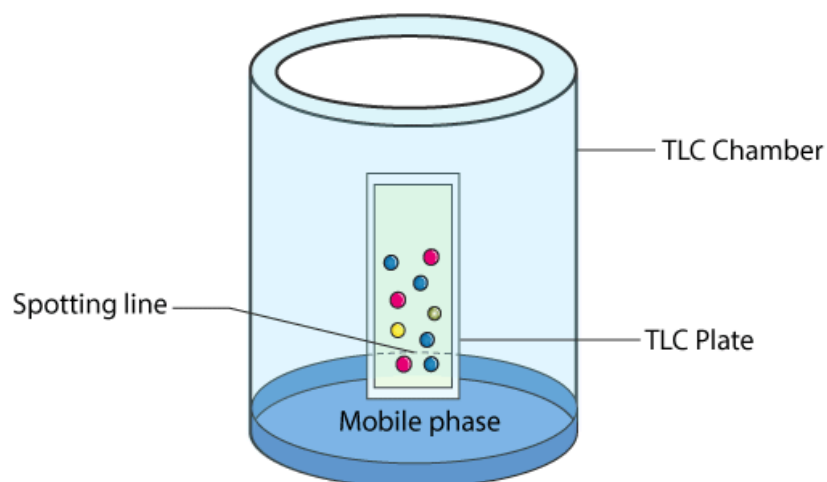


Pressure in millibar

Temperature in degree C

Rotations per minute

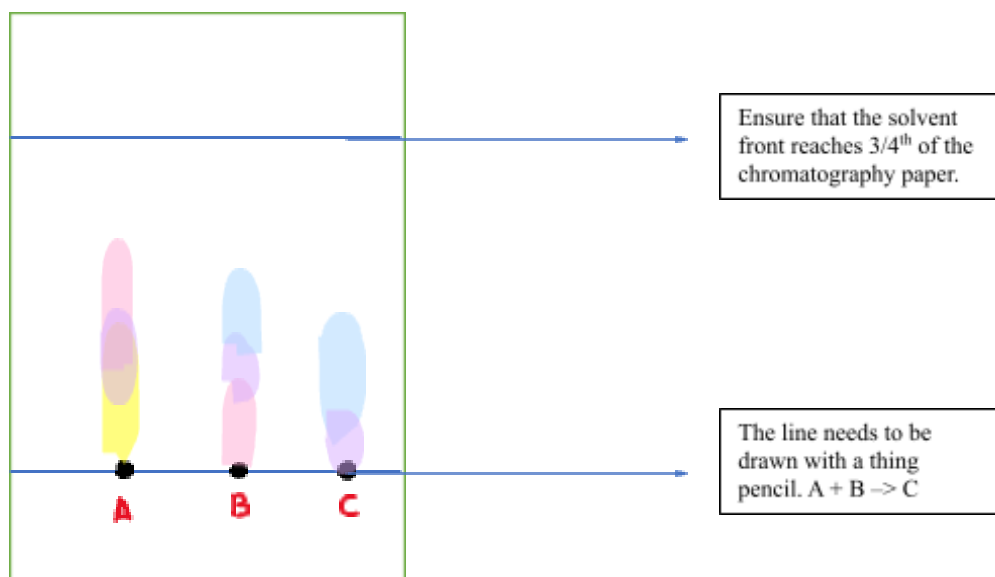
THIN LAYER CHROMATOGRAPHY



4

Apparatus Needed for TLC

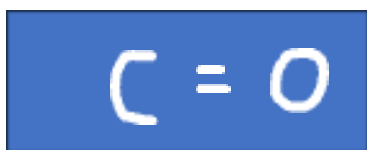
1. **Plates** – ready-made plates are used which are chemically inert and stable. The stationary phase is applied on its surface in the form of a thin layer. The stationary phase on the plate has a fine particle size and has a uniform thickness.
2. **Chamber** – Chamber is used to develop plates. It is responsible to keep a steady environment inside which will help in developing spots. Also, it prevents the solvent evaporation and keeps the entire process dust-free.
3. **Mobile phase** – Mobile phase is the one that moves and consists of a solvent mixture or a solvent. This phase should be particulate-free. The higher the quality of purity the development of spots is better.



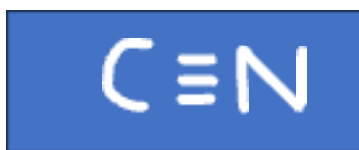
Procedure:

- The chromatogram was placed under UV light
- The colored compounds revealed themselves in the presence of the UV rays.

⁴ BYJU'S, "Thin Layer Chromatography," BYJUS (Byju's, February 21, 2019)



AND



These compounds give a color when placed under ultra-violet light.

Factors affecting TLC:

If you know that one component of a mixture is insoluble in each solvent, but another component is freely soluble in it, it often gives good separations. How fast the compounds travel up the plate depends on two things:

- If the compound is soluble in the solvent, it will travel further up the TLC plate
- If the compound likes the stationary phase, it will stick to it, which will cause it to not move very far on the chromatogram.

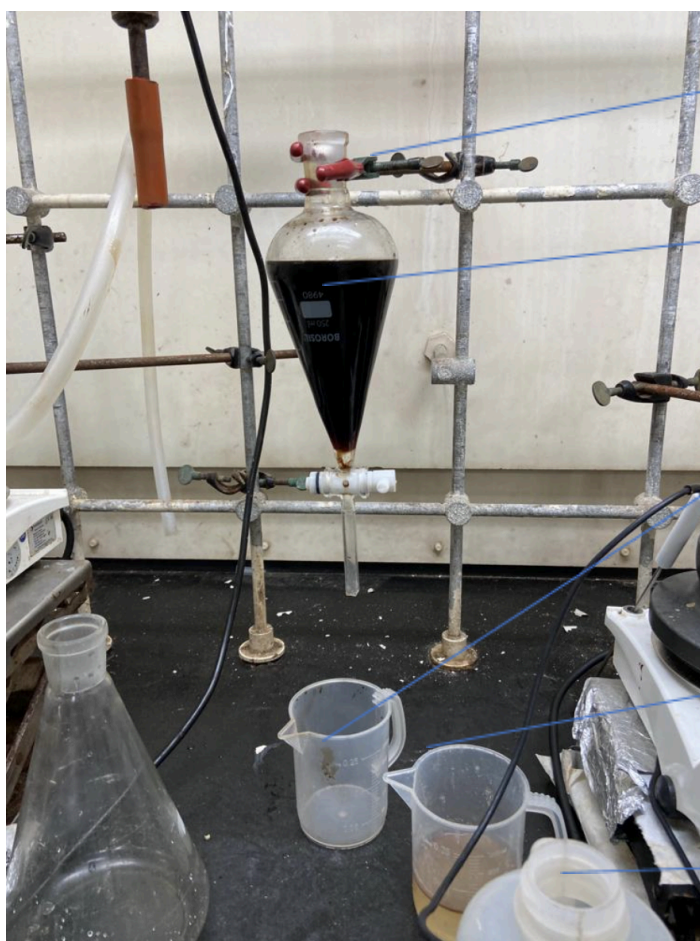
You should be able to determine which by looking at the R_f value.

SEPARATING FUNNEL

“Work-up Method” is useful because:

- (i) Reduces wastage
- (ii) Ensure proper separation
- (iii) Quenches the reagent

Some reagents added to the mixture need to be quenched. We started with the mixture and poured it inside the separating funnel with a filter funnel for controlled pouring.



A clamp holds the apparatus in place.

The mixture in the funnel separates into organic layer on top and the aqueous layer seen at the bottom.

The solution is then flushed out several times to ensure that any remaining compound particles are not wasted.

The flushed-out solution is collected in a beaker. The process is repeated several times.

A solvent called ethyl acetate is used which is highly polar.

The black solution (as seen above) is acidic in nature i.e., with a pH of lesser than 7. A neutralization reaction has occurred due to the adding of Sodium Bicarbonate. The pH paper turned green after neutralisation.



Due to the reaction being a neutralization reaction, there is effervescence in the form of carbon dioxide given out.

Choosing a Solvent

It is very important to choose the correct solvent because:

- (i) A solvent can control the temperature of a reaction
- (ii) The compound should be soluble or insoluble in it depending on how the compound must be extracted

ETHYL ACETATE is the perfect solvent because it is a methyl ester. It can be rinsed several times to ensure all the compound is reused and not wasted. This will also ensure the getting rid of impurities.



The process is repeated several times

Traces of a precipitate can be seen in the solution in the filter funnel. Upon adding excess the precipitate will dissolve.



Organic Solvent: The wanted compound.

Aqueous Solvent: The unwanted watery part.

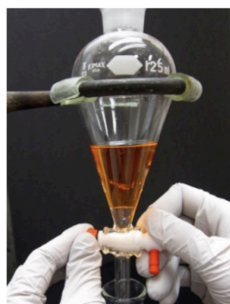
BRINE WATER WORKUP

What is a Brine solution?

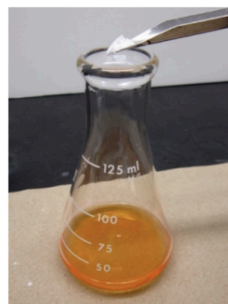
Brine, also called saltwater, specifically a highly concentrated water solution of the common salt. Natural brines take place underground, in seawater, or as salt lakes and are commercially essential sources of the common salt and other salts, such as sulphates and chlorides of potassium and magnesium.



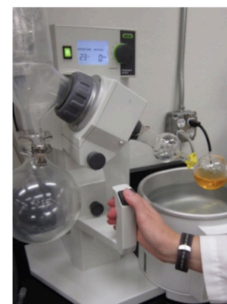
a) Conduct the reaction.



b) Perform multiple extractions and/or washes to partially purify the desired product.

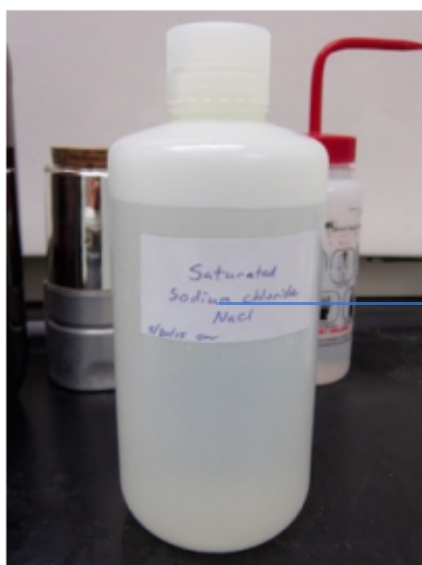


c) Remove trace water with a drying agent.
d) Filter or decant the drying agent.



e) Remove the solvent with a rotary evaporator
f) Further purify if necessary.

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The organic solvents that require a brine wash before exposure to a solid drying agent are diethyl ether and ethyl acetate.

The purpose of this wash is to remove large amounts of water than may be dissolved in the organic layer. Although the organic layer should always be later exposed to a drying agent (e.g., anhydrous sodium sulphate, magnesium sulphate, or calcium chloride), these reagents at best remove only small amounts of water.

Brine works to remove water from an organic layer because it is highly concentrated (since NaCl is so highly water soluble). A saturated NaCl(aq) solution is highly ordered, causing a large motivation for water to draw into the solution from the organic layer, therefore making it more dilute.

Solvent	Grams of water dissolved in 100mL solvent
Ethyl Acetate	2.92g

⁵ Lisa Nichols, "4.7: Work-Ups," Chemistry LibreTexts, March 7, 2018

Dichloromethane	0.32g
Hexanes	0.007g

Hexane is extremely non-polar.

After this the use of Sodium Sulphate was noted. Sodium sulphate is a drying agent. It is useful because it can remove the excess water from the aqueous layer left behind during a work-up reaction. Here are the steps:

- (i) Remove the water at a moderate rate
- (ii) The solution should be allowed to sit with the drying agent for some time
- (iii) Solution with can usually be decanted



This is the organic layer. It is nonpolar. Any impurities in it are highly polar.

This is the aqueous layer. It is a brine water solution and therefore is HIGHLY polar. Any polar impurities in the organic layer will be attracted and pulled down to the aqueous layer by the brine.

6

VACUUM DISTILLATION

⁶ Roth Carl, "Separating Funnel JPG," Roth carl, n.d.



This is the control for the vacuum. If there is too much pressure it could cause spillage.

These are the knobs that must be secured tightly to ensure no entry of air in vacuum-like condition.

THE BASICS OF VACUUM DISTILLATION:

- (i) It uses the principle of vacuum to lower the BP
- (ii) Useful for substances that have high boiling points

Vacuum Distillation

Vacuum distillation is the perfect solution when you have elements that decompose when heated at atmospheric pressure – or – that have high boiling points and would have high system heating requirements.

Because of the Vacuum, a suction was created, ensuring that all the moisture and remaining water left behind, (since the sodium sulphate still left a little bit of moisture,) is all removed.

The boiling point of the organic compound was reduced, therefore requiring less energy, lower temperatures, and less electricity so overall wastage of heat and energy was less.



Pressure of the vacuum in mbar (millibar)

RPM – rotations per minute

Vacuum control

The next step is column packing, which is done after confirming the results using infrared radiation.

CONDENSOR

The lightest molecules reaching the top of the column (a.k.a. distillate or "tops") leave the column and flow to a condenser. The condenser cools the steam, turning it back into a liquid of highly concentrated distillate

Your condenser determines the column pressure. Vapor condenses at specific pressure/temperature combinations. System pressure must be precise to avoid over cooling the liquid. Vacuum distillation helps lower the required pressure for separation in the column. Condensers are usually either:

- Total vacuum
- Partial vacuum



COLUMN PACKING

Feed, or the product being distilled, enters the column and falls towards the bottom. Inside the column, liquid falls and steam from the bottom rises. This separates heavier molecules from lighter ones. Separation is facilitated by the material in the column, which is either:

- Trays – common for taller columns, more robust applications
- Loose Packing – popular in smaller columns/applications

INFRARED SPECTROSCOPY

⁷ Taken from a scholarly paper

DEFINITION: Infrared Spectroscopy is the analysis of infrared light interacting with a molecule. This can be analyzed in three ways by measuring absorption, emission, and reflection.

The main use of this technique is in organic and inorganic chemistry.

- (i) It is used by chemists to determine functional groups in molecules.
- (ii) IR Spectroscopy measures the vibrations of atoms, and based on this it is possible to determine the functional groups.
- (iii) Generally, stronger bonds and light atoms will vibrate at a high stretching frequency (wavenumber).

Approximate Frequency (cm ⁻¹)	Description	Bond Vibration	Notes
3500 - 3200	broad, round	O-H	much broader, lower frequency (3200-2500) if next to C=O
3400-3300	weak, triangular	N-H	stronger if next to C=O
3300	medium-strong	=C-H (sp C-H)	
3100-3000	weak-medium	=C-H (sp ² C-H)	can get bigger if lots of bonds present
3000-2900	weak-medium	-C-H (sp ³ C-H)	can get bigger if lots of bonds present
2800 and 2700	medium	C-H in O=C-H	two peaks; "alligator jaws"
2250	medium	C=N	
2250-2100	weak-medium	C=C	stronger if near electronegative atoms
1800-1600	strong	C=O	lower frequency (1650-1550) if attached to O or N middle frequency if attached to C, H higher frequency (1800) if attached to Cl
1650-1450	weak-medium	C=C	lower frequency (1600-1450) if conjugated (i.e. C=C-C=C) often several if benzene present
1450	weak-medium	H-C-H bend	
1300 - 1000	medium-strong	C-O	higher frequency (1200-1300) if conjugated (i.e. O=C-O or C=C-O)
1250-1000	medium	C-N	
1000-650	strong	C=C-H bend	often several if benzene present

⁸ The table shows a preview of what different bonds appear like using IR Spectroscopy.

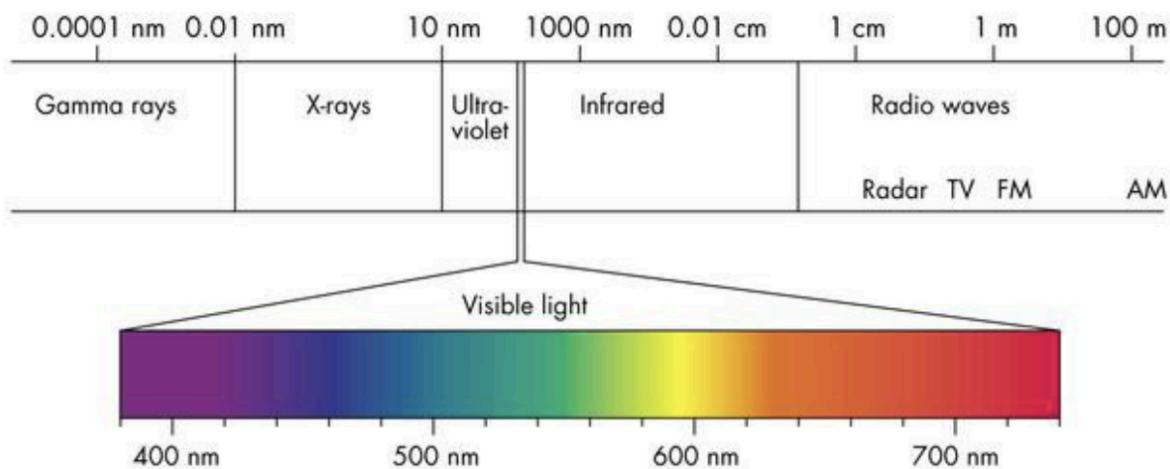
IR spectroscopy (which is short for infrared spectroscopy) deals with the infrared region of the electromagnetic spectrum depending on 2 things:

1. *light having a longer wavelength*
2. *and a lower frequency than visible light.*

Infrared Spectroscopy generally refers to the analysis of the interaction of a molecule with infrared light.

Infrared Light

⁸ Chemistry LibreTexts. "Interpreting Infrared Spectra," October 3, 2013.



TLC STAINS

1. KmnO_4
 - (i) Oxidizing agent
 - (ii) Purple in color
 - (iii) Creates a yellow stain

2. PMA
 - (i) Green in color
 - (ii) Basic stain
 - (iii) Blue-grey spots created

3. 4DNP
 - (i) Tests for aldehydes
 - (ii) Tests for ketones

4. Iodine
 - (i) Sublime stain
 - (ii) Used for all
 - (iii) Completely binds to the compound
 - (iv) Creates a yellow-brown spot

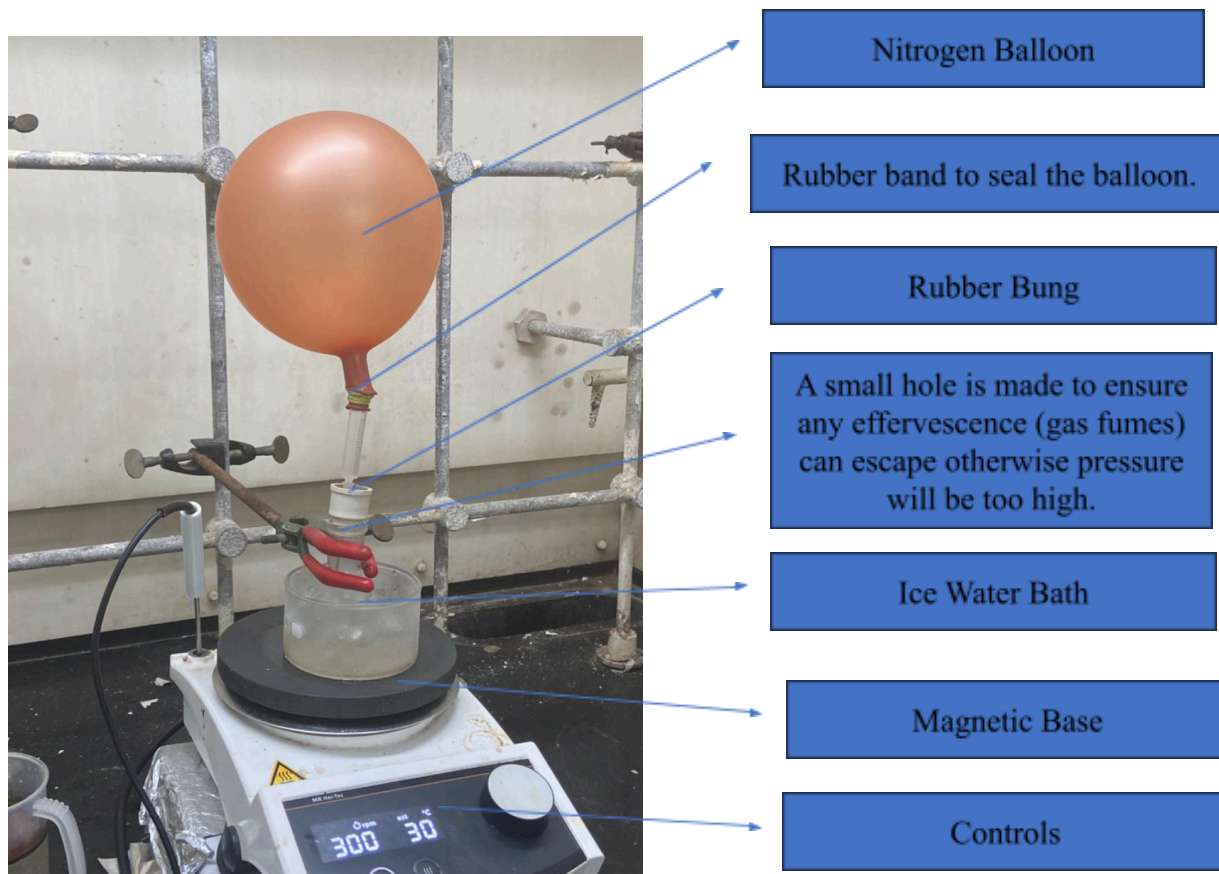
5. Bromocresol
 - (i) Green in color
 - (ii) Used to test for acidic compounds

INCLUDEPICTURE
 "https://encrypted-tbn0.gstatic.com/images?q=tbn:ANd9GcRjtdZ6oWe_1221TaQwbcd1zxGYB0P
 BI2mnmtg&usqp=CAU" *
 MERGEFORMATINET

Any compounds which are **UV-active** can also be visualized. Spot(s) must be lightly traced with a pencil while visible, since when the UV light is removed, the spots disappear.

NITROGEN ATMOSPHERE

⁹ Mini Physics, "The Electromagnetic Spectrum," Mini Physics, May 22, 2016



First the compound is placed inside a tiny round bottom flask. An H-O bond is in the center. The reaction will take place around the O-H bond only.

Next, 500mg of starting material was added. It was in the form of a thick viscous liquid, and we placed inside using a pipette. The solvent needed for this reaction is dichloromethane. Based on the equation $\text{Density} = \text{Mass}/\text{Volume}$, we can weigh the mixture on a balance, find the density and then use it to calculate the volume needed instead of directly adding it in.

A syringe was used to add the solvent in instead of a pipette because a syringe has a gradient scale, where as a pipette does not, so there is more accuracy when using a syringe.

The small round bottom flask is placed inside an ice bath because low temperatures will be needed since the mixture has to remain cold (at a temperature lesser than rtp). The water bath also controls the temperature and ensures uniform heating.

Now the base + solvent + starting material is in one mixture. The reagent is added next, which is ethyldisopropylamine. It is part of the amine group and therefore has a CONH bond.

After adding in the reagent, a balloon with nitrogen gas is used to cover the round bottom flask. The balloon is added to make sure that the solution is in a nitrogen atmosphere because the solution cannot contact any moisture. If not taken care of, this moisture would quench the reagent and render the experiment useless. The solid reagent is then added in.

Effervescence of HCl gas is seen, and since HCl is acidic in nature, the fumes have a pH of less than 7. When a pH paper is placed in the fumes it turns slightly red. However, if a damp litmus paper was used then the red color would be brighter.

REDUCING FUNCTIONAL GROUPS

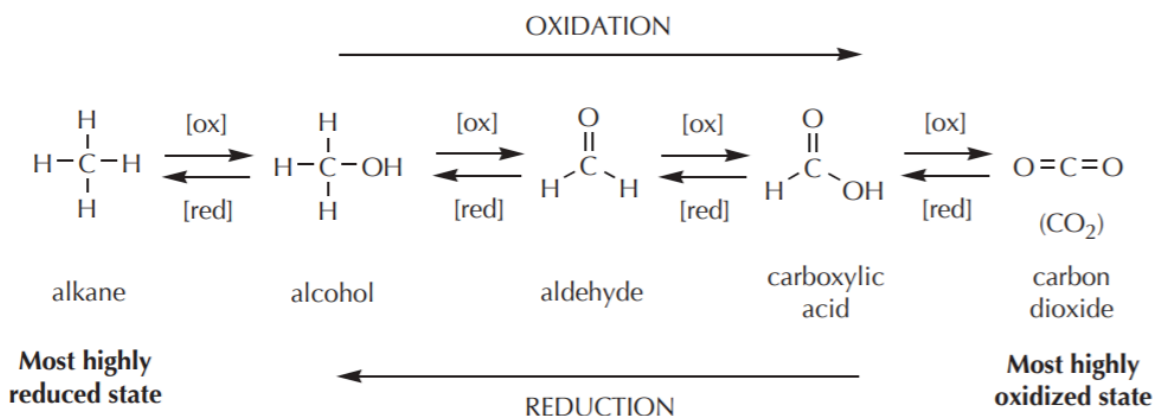
In ionic reactions, the process of oxidation and reduction is based on the removal/addition of oxygen and electrons.

Definition

With reference to organic molecules, oxidation is a process by which a carbon atom gains bonds to more electronegative elements, most commonly oxygen. Reduction is a process by which a carbon atom gains bonds to less electronegative elements, most commonly hydrogen.

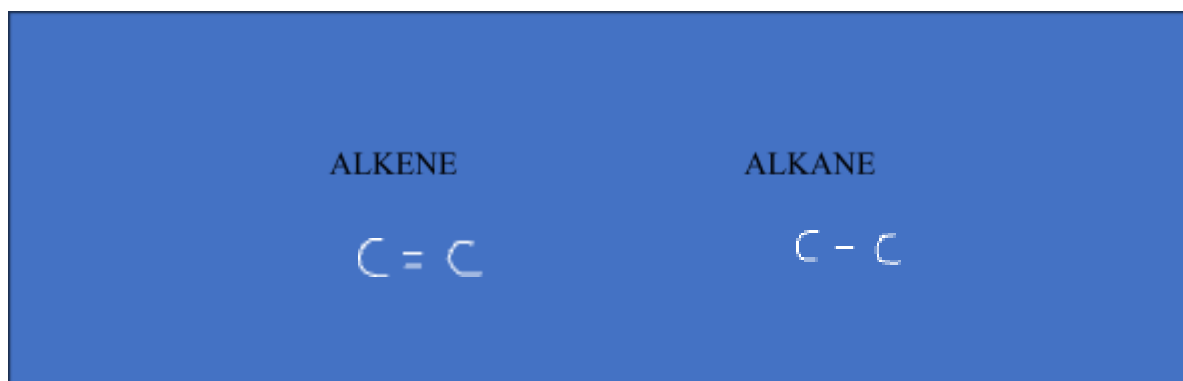
For example, during the previous reaction, the base change resulted in the formation of a liquid, not a solid compound. Therefore, a conclusion arose that the problem wasn't the base, it was the presence of an additional functional group in the starting material.

The starting material was an alkene, which meant that it has a double covalent bond.



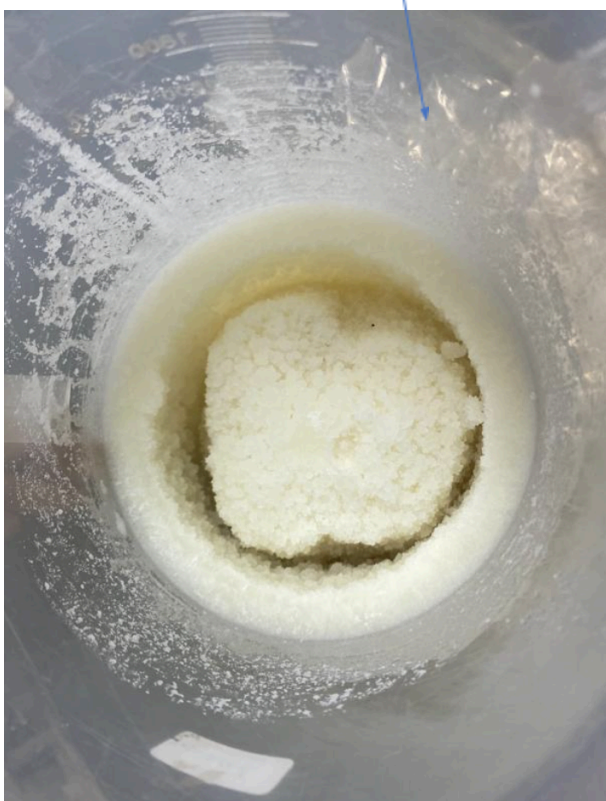
10

Therefore, it was established that the double bond in the starting material was interfering with the reaction.



¹⁰ "19.1: Oxidation and Reduction in Organic Chemistry," Chemistry LibreTexts, May 22, 2020

By changing the material from a double to single bond, the hydrogen had to be added in order to reduce the alkene starting material to an alkane.



START MATERIAL

END MATERIAL

Yellow liquid which has been decanted from the white solid.

The white solid remains which have been obtained by removing top liquid layer.

The process in which you pour out a distinctive liquid layer over a solid layer. When there is a need to separate a solid-liquid mixture, on occasion it is possible to pour off the liquid while leaving the solid behind. This process is called decanting, and is the simplest separation method.

CO-DISTILLATION

According to acclaimed Agricultural chemist Barry Tickes from the University of Arizona and Arizona Agricultural Experiment Station, the definition of co-distillation is when a herbicide evaporates or changes from a liquid to a vapor with water. This can occur from soil, water or plant surfaces and can be responsible for substantial loss of some herbicides.

In general, co-distillation is greatest when:

- Temperature is high
- moisture is high
- pH is high
- organic matter is low.

By adding in the solvent toluene, all the water and moisture is absorbed and separated from the main compound.

Here are toluene's properties:

- similar BP as water
- pulls out any water from mixture
- helps in separation of water

Since the boiling point of toluene is 110 degrees Celsius, in the Rotary Evaporator, the boiling point had to be set to 50 degrees Celsius. An observation was made that in the rotating round-bottom flask, the water and toluene along with the yellow compound, were separated into layers.

KUGEL RORH DISTILLATION

Separation of small amounts of compounds with close boiling points is difficult in a fractional distillation setup and can alternatively be performed via Kugel Rohr Distillation. Typically high boiling compounds are used and vacuum is applied.

Here are the steps to carry out this kind of distillation:

1. Place compound mixture in small round bottom flask. The amount of compound should be small. We took 5 grams.
2. Attach Kugel Rohr flasks - typically two additional flasks are used.
3. Induce vacuum, if desired. You can cool the external Kugel Rohr flasks with an ice bath.
4. Heat the Kugel Rohr and start rotating motor until distillation occurs.

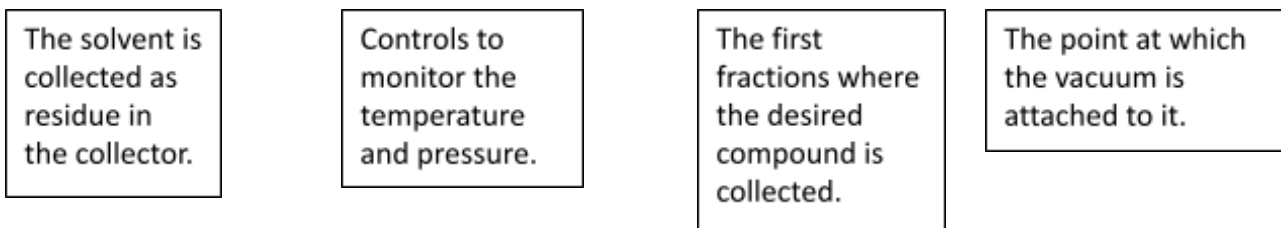
In this microscale, short-path technique, a small quantity of material is redistilled using several glass bulbs connected in line.

DEFINITION

A method of separating mixtures based on differences in their volatilities in a boiling liquid mixture. As the sample mixture to be purified is heated, its vapours rise a short distance into a vertical condenser (called a 'Kugelrohr') where they are cooled by water. This technique is used for small amounts of samples with high boiling points (usually >300 deg. C) under greatly reduced pressure.¹¹



¹¹ Official definition according to the Royal society of Chemists, last updated, 2021

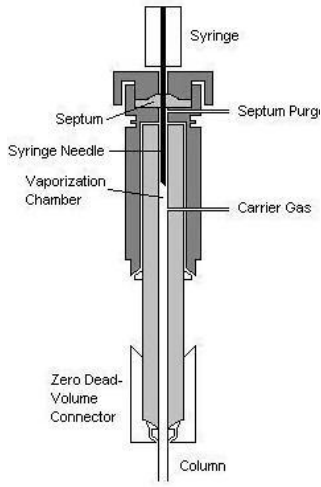


The mechanism works in such a way that The other bulbs can be used to collect the distillates sequentially, when the desired fraction is being collected the bulb is cooled with water or ice to aid condensation. A motor drive is often used to rotate the string of bulbs to reduce bumping, give even heating, and increase the surface area for evaporation.

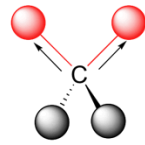
ANALYTICAL MACHINES

<i>Gas Chromatography</i>	<i>Infrared Spectroscopy</i>	<i>High Performance Liquid Chromatography</i>	<i>Mass Spectroscopy</i>
<p>In gas chromatography, the components of a sample are dissolved in a solvent and vaporized in order to separate the mixture.</p> <p>This is done by distributing the sample between two phases: a stationary phase and a mobile phase.</p> <p>Mobile Phase = chemically inert gas that serves to carry the molecules of the analyte through the heated column.</p>	<p>Depends on the vibrational energy between different functional groups.</p> <p>Different types of functional groups have different vibrations.</p>	<p>It was first used as a method of separating coloured compounds.</p> <p>In analytical chemistry, a quantitative metric of every experimental parameter is desired, and so separation efficiency is measured through HPLS.</p>	<p>In order to measure the characteristics of individual molecules, a mass spectrometer converts them to ions so that they can be affected by electro-magnetic fields.</p> <p>The ions are sorted and separated according to their mass and charge. The Mass Analyzer</p> <p>The separated ions are then measured, and the results displayed on a chart. The Detector</p>

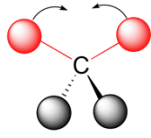
The stationary phase is either a solid adsorbent, or an inert liquid.



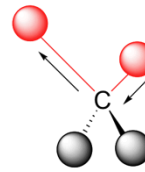
Carrier gas must be chemically inert mobile-phase employed in gas chromatography. Helium is most commonly used.



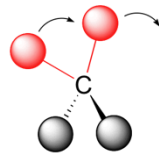
symmetric stretching



scissoring



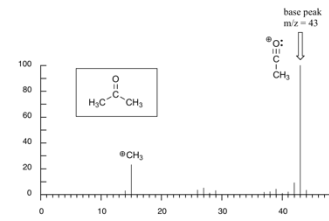
asymmetric stretching



rocking

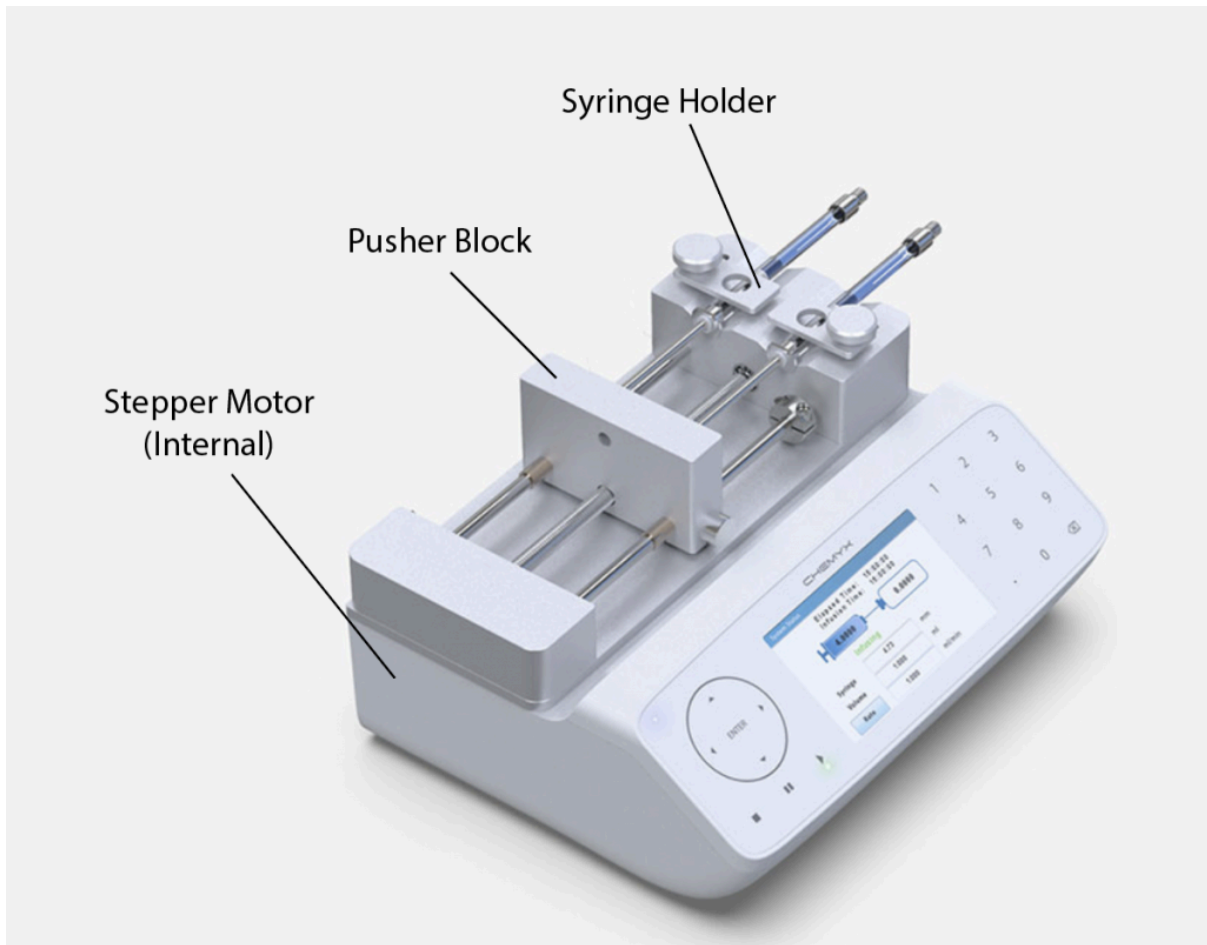


It too uses, a mobile and stationary phase, but unlike GC, the mobile phase is in liquid form, and not a gas.



SYRINGE PUMP

A syringe pump is a motor-driven precision pump that uses one or more syringes to deliver precise and accurate amounts of fluid in high-impact research.



Uses of Syringe Pumps:

- At the research lab, syringe pumps can be used in almost any application that involves precise metering, especially at the micro and nanoscale.
- They are used in many research fields as precise dosing systems, or to accurately deliver small quantities of reagents, mix minuscule volumes, and add traces of specific chemicals over the course of the experiment.
- Syringe pumps compatible with microfluidic applications are available in the market, which facilitates research in fields such as micro-environmental control.
- Microfluidic applications of syringe pumps have increased dramatically in the vaccine research area.

COW HEAD DISTILLATION

This receiver can be used with short path style distilling heads. This type of adapter is also called an "udder". Both this and "cow style" refer to the multiple outlets that are reminiscent of a cow udder.



A place where the inert gas tube can be attached.

3 joints where multiple outlets to collect different substances are attached.

These traditional "cow" type distillation receivers have three receivers 45° apart. Condensers with lower drip tip inner joints will protrude into the spherical section, permitting the distillate to fall directly into the receiving flasks without running down the inner wall of the receiver. The distribution adapter has a serrated connection for attachment to a vacuum or inert gas source.

TYPES OF SCENTS

TAKEAWAY 1:

Rose oil is one of the most widely used essential oils for perfumes and cosmetics. The oil has been reported to possess a wide range of physiological activities, including analgesic, hypnotic, and anti-inflammatory properties.

Based on the Journal Club meeting, there are 3 types of rosy alcohols:

- (i) Citronellol
- (ii) Geraniol
- (iii) Nerol

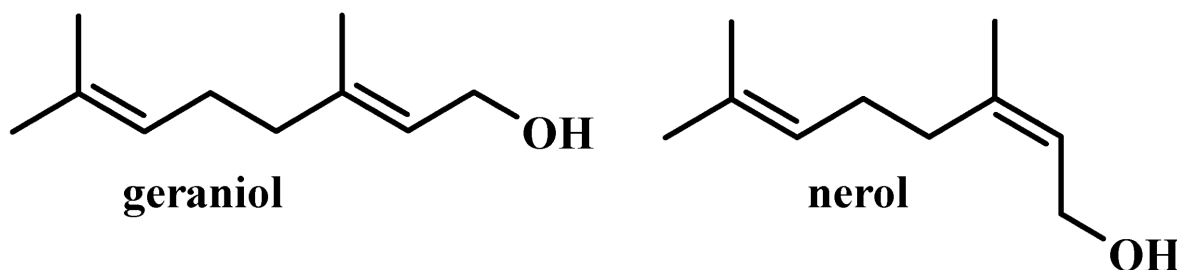
All these produce monoterpenoid products. The meaning of monoterpenoid products is that they are:

- (i) C₁₀ isoprenoids and constitute a large family of natural products
- (ii) They have been used as ingredients in food, cosmetics, and therapeutic products.
- (iii) represent a set of chemicals widely diffused in plants whose basic structure consists of two linked isoprene units
- (iv) They have been used as ingredients in food, cosmetics, and therapeutic products.

TAKEAWAY 2:

I also learned that mercury is a common catalyst in the Thomas Process which is used to prepare rosy alcohol based-perfumes.

However, geraniol and nerol are not widely used because they are sometimes skin irritants and is banned from being used in the rose perfumery process.



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TAKEAWAY 3:

The saturated or unsaturated compound are those which contains C=C double bond(s) or C≡C triple bonds.

If aldehyde, ketones, alcohols etc contain such double or triple C-C bonds, they can be said as unsaturated otherwise they are saturated.

This means that when put in a bromine water solution, these unsaturated aldehydes will decolourise the bromine water.

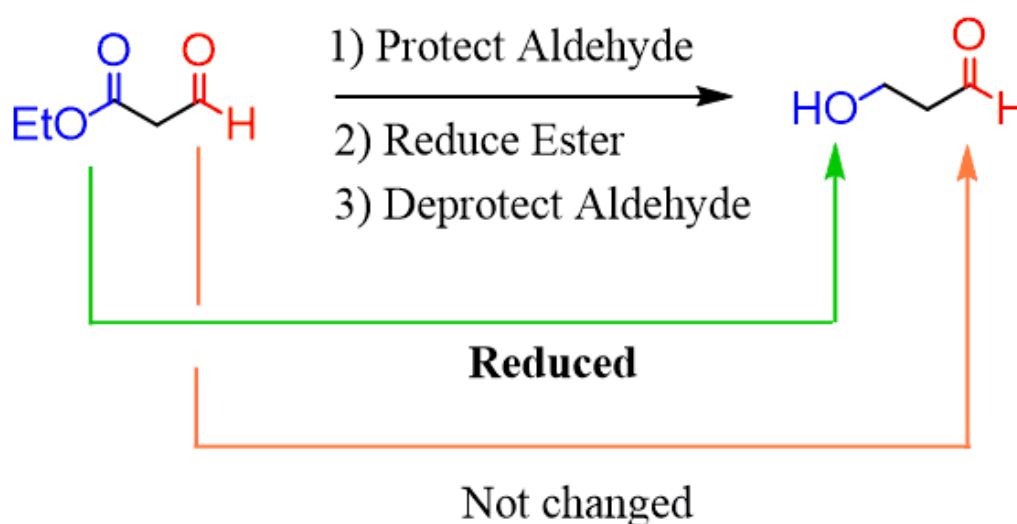
TAKEAWAY 4: THE PROTECTION OF ALDEHYDES

¹² Wanda Mączka, Katarzyna Wińska, and Małgorzata Grabarczyk, "One Hundred Faces of Geraniol," *Molecules* 25, no. 14 (July 21, 2020): 3303

What is an aldehyde?

It is an organic compound containing the group —CHO , formed by the oxidation of alcohols. In organic chemistry, an aldehyde is an organic compound containing a functional group with the structure R—CH=O . Aldehydes have a boiling point of between 130-140 degree Celsius.

Aldehydes are also more reactive than esters and when mixed with, for example LiAlH_4 , they will react first before the ester is reduced and that is when the aldehyde is mostly gone. So, we need to temporarily protect the aldehyde, reduce the ester, and then remove the protecting group to bring the aldehyde back in the normal form in order to unprotect/remove protection.



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Acetal Properties:

- They are stable and show a lack of reactivity in neutral to strongly basic environments
- As long as they are not treated by acids, especially aqueous acid, acetals exhibit all the lack of reactivity associated with ethers in general.

DROP WISE REACTIONS

Why add a liquid reagent drop wise?

A reaction was conducted in which the reagent(ethyl acetate) had to be added into the flask

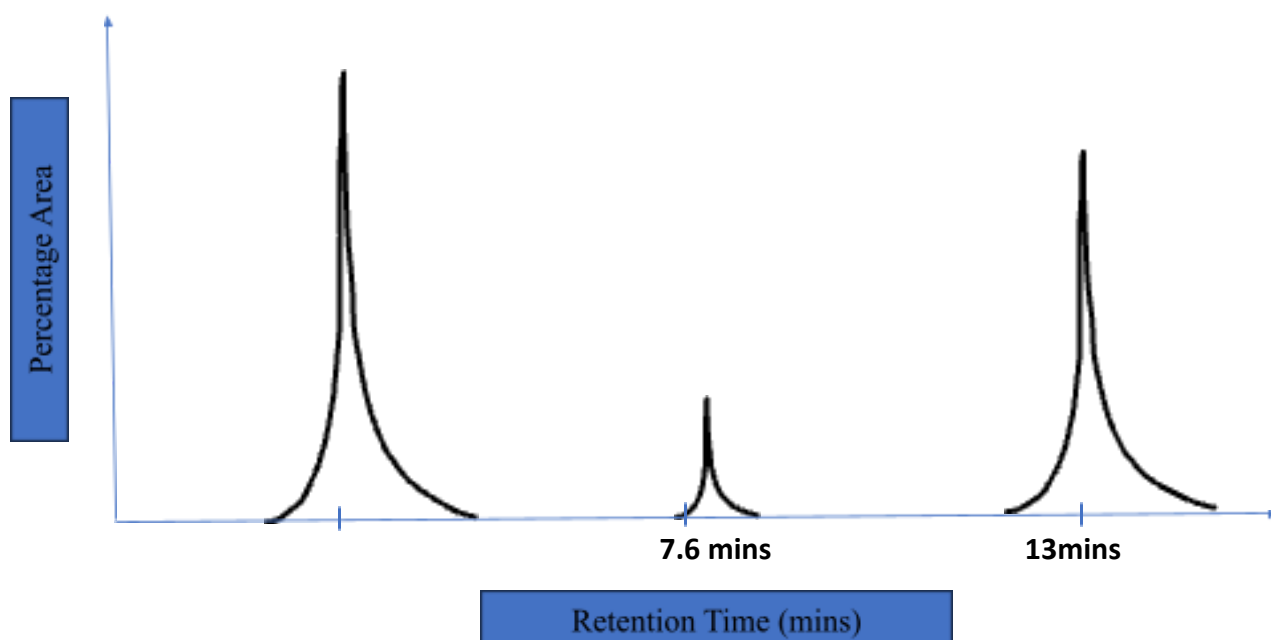
¹³ "Acetals as Protecting Groups for Aldehydes and Ketones," Chemistry Steps, May 31, 2018

one drop at a time. Dropwise addition is simply adding a liquid to a reaction slowly with a pipet. Only a few drops at a time.

The reason for this is because:

1. To prevent the risk of effervescence
2. Adding something drop-wise keeps things nicely under control, allows immediate reaction
3. the reaction is addition controlled and will stop if the addition is halted
4. prevents side reactions
5. stops accumulation of dangerous amounts of reactants/intermediates

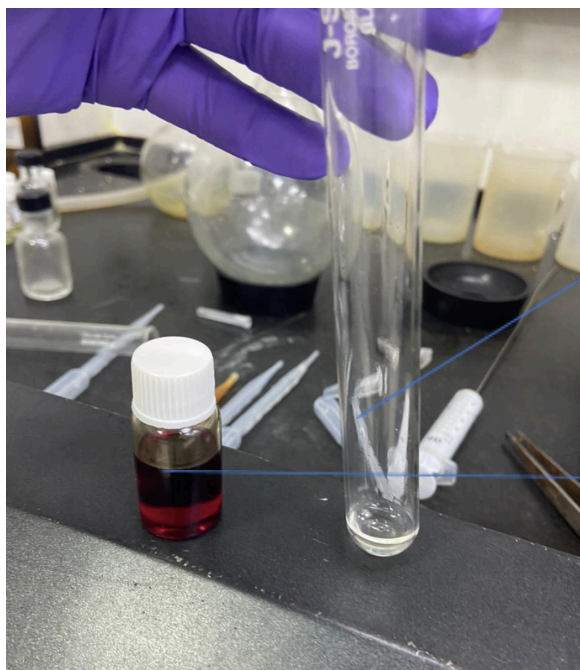
GC GRAPH READINGS



Additional Notes:

- The gas passed through the GC is always a noble gas
- This is done, in order to ensure that the gas doesn't react with the vial
- The noble gases most commonly used are He and N
- He and/or N is the mobile phase
- The vial's liquid sample injected into the column is the stationary phase
- Sometimes H and O are used for ignition in order to ensure that the vial's liquid gets heated to form a gaseous vapour form.

BROMINE WATER TEST

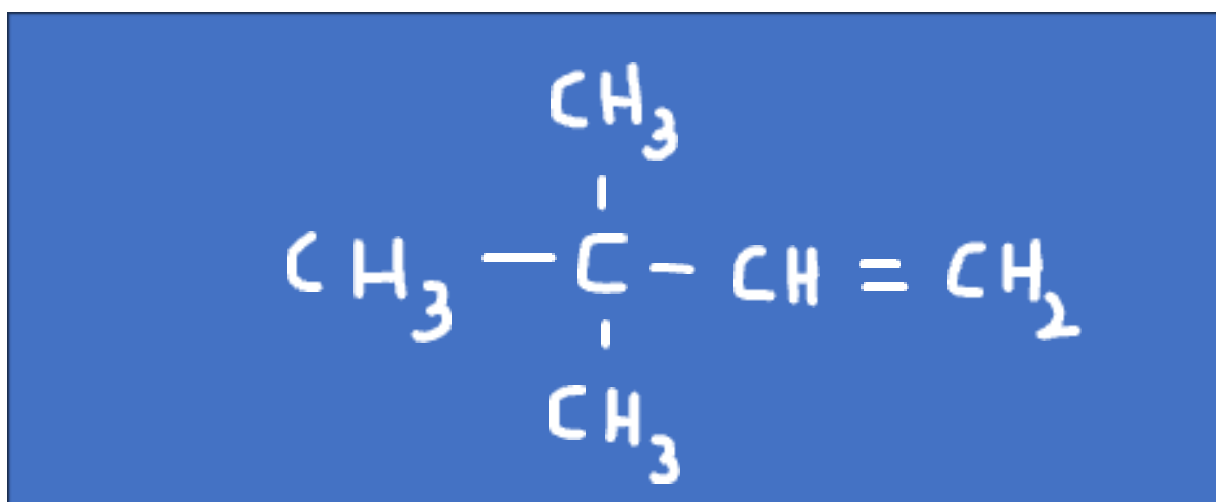


This is the solution after adding in the alkene. It has turned from orange, brown to colorless, meaning the alkene was unsaturated.

This is the bromine water. Originally, it is orange, brown in color.

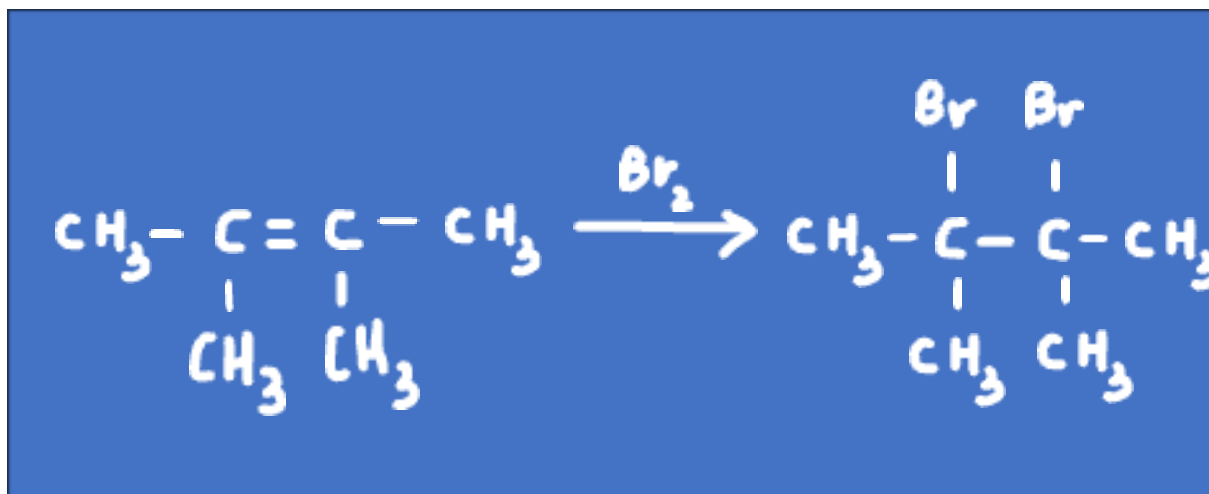
This is also known as the saturation test, because it helps identify if a hydrocarbon is saturated or unsaturated. It is a qualitative test (since the observation is a colour change,) used to identify the alkene or alkane functional groups present in the compound. Alkene groups react with bromine water in the dark condition and undergo an addition reaction to give a decolourised solution.

Name of alkene added: Dimethylbutene



The presence of the C = C double covalent bond ensures that the addition reaction will take place.

This is the entire reaction with bromine water: It is known as an addition reaction because the double covalent bond can break in order to ensure that more molecules can add on.



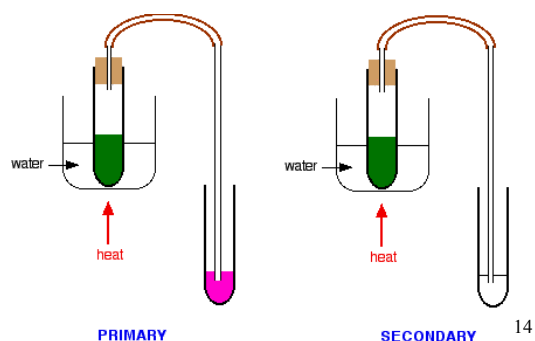
The double bond breaks, and therefore is allowed to add on more bromine. Since there is no more bromine present in the liquid solution, it loses its orange-brown colour and turns colourless.

OXIDISING ALCOHOLS

Alcohols are compounds in which one or more hydrogen atoms in an alkane have been replaced by an -OH group.

Primary alcohols can be oxidized to form aldehydes and carboxylic acids; secondary alcohols can be oxidized to give ketones. Tertiary alcohols, in contrast, cannot be oxidized without breaking the molecule's C-C bonds.

The oxidising agent used in these reactions is normally a solution of sodium or potassium dichromate(VI) acidified with dilute sulphuric acid. If oxidation occurs, the orange solution containing the dichromate(VI) ions is reduced to a green solution containing chromium(III) ions.



QUENCHING

¹⁴ Jim Clark, "Oxidation of Alcohols," Chemguide, 2015

Quenching is the soaking of a metal at a high temperature, followed by a rapid cooling process to obtain certain desirable material properties. It also refers to the deactivation any unreacted reagents.

How can a quenching reaction be carried out?

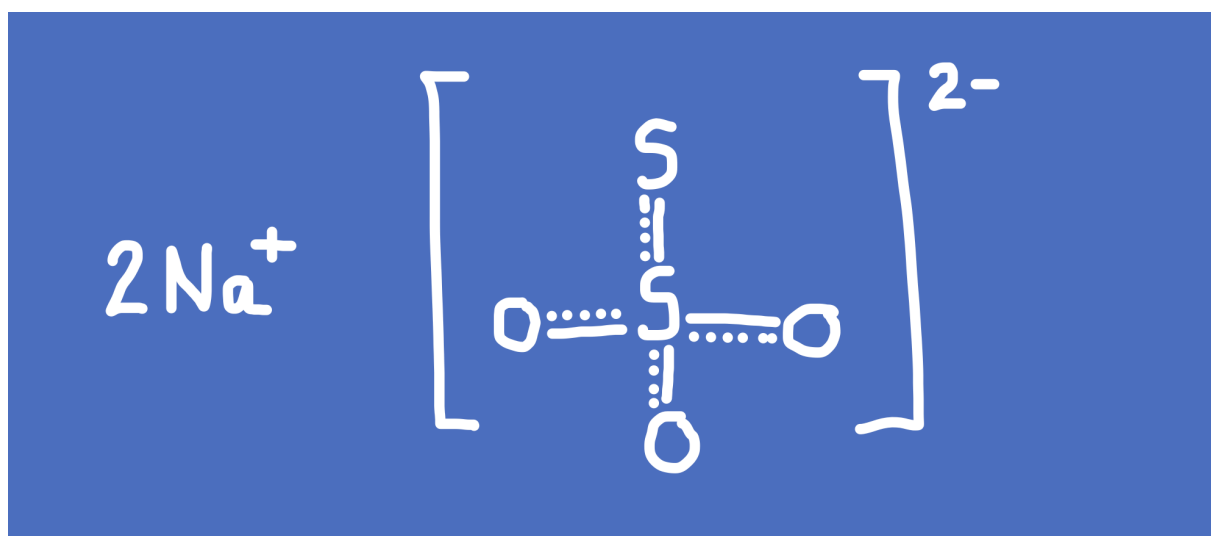
- The quenching of a reaction can be made by diluting the reaction mixture.
- The reaction is supposed to be completed if it is kept for long time or strongly heated.

It is also done by adding an antisolvent to induce precipitation, and collecting or removing the solids. Overall, Quenching is a term used to describe the introduction of a material that combines with any unused reactants and effectively stops a reaction.

SODIUM THIOSULPHATE

Sodium thiosulfate readily dissolves in water giving thiosulfate ions, which is one of the useful reducing agents. It appears as a bright white colourless crystal or even in powder form. The substance is known to possess alkaline nature when decomposed to sulphide and sulphate in the air.

- Sodium thiosulfate appears as a white translucent, colourless crystal and is an inorganic compound.
- It is a water-soluble substance and is also soluble in the oil of turpentine but not in alcohol.
- The substance has a melting point of about 48 to 52 C.
- This chemical substance is highly stable in nature and is said to be incompatible with some strong oxidising agents and strong acids.
- Thiosulfate anion readily reacts with the dilute acids producing sulphur, sulphur dioxide and also water.
- The chemical has a density measuring about 1.667 g/mL



DEFINITION: Nuclear magnetic resonance (NMR) spectroscopy is the study of molecules by recording the interaction of radiofrequency (Rf) electromagnetic radiations with the nuclei of molecules placed in a strong magnetic field.

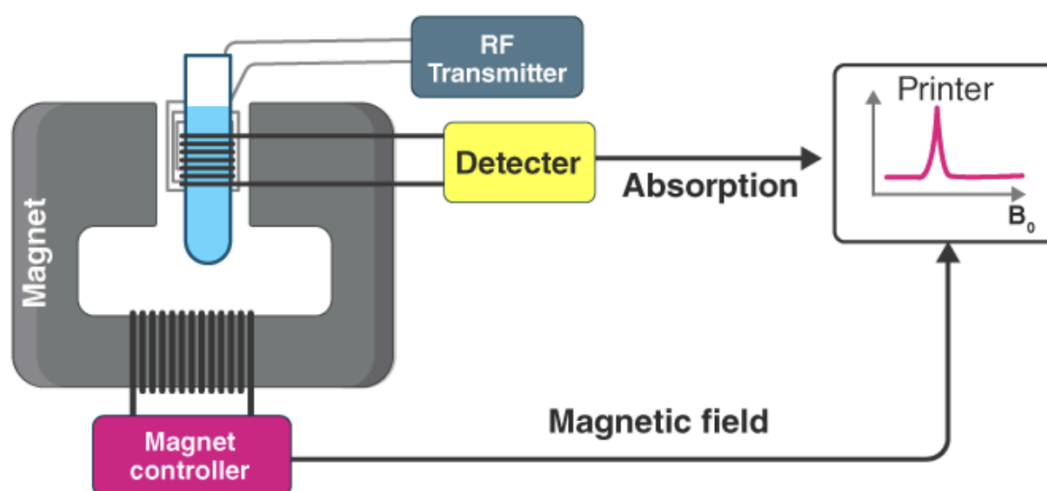
NMR helps identify the:

- (iv) Properties of atoms based on magnetic abilities
- (v) Determines the physical and chemical properties

NMR helps in giving detailed information about:

- *Structure*
- *Dynamics*
- *reaction state*
- *chemical environment*

of molecules.

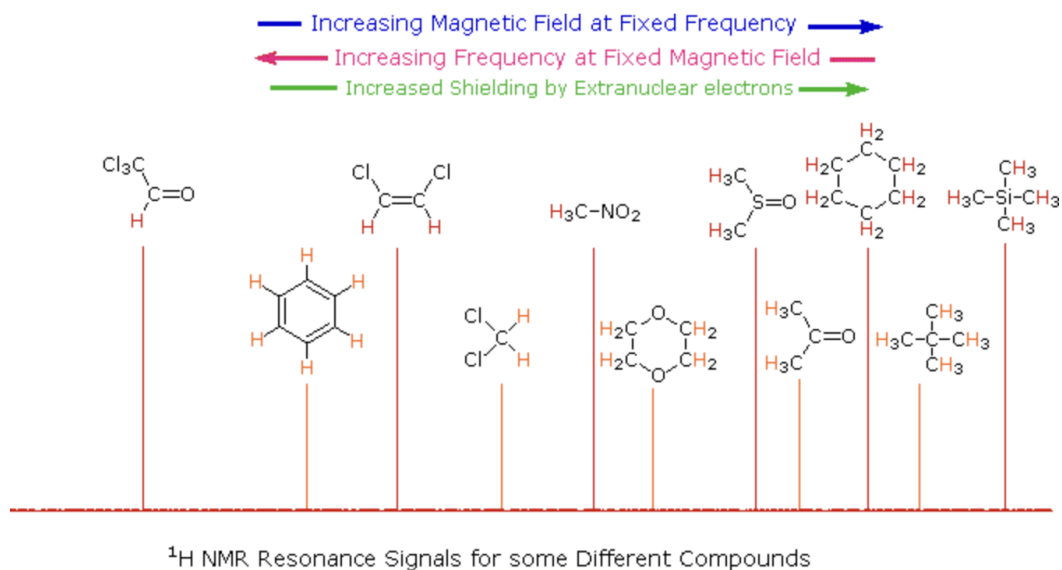


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A Brief History of NMR

Over the past fifty years nuclear magnetic resonance spectroscopy, commonly referred to as NMR, has become the preeminent technique for determining the structure of organic compounds. Of all the spectroscopic methods, it is the only one for which a complete analysis and interpretation of the entire spectrum is normally expected. Although larger amounts of sample are needed than for mass spectroscopy, nmr is non-destructive, and with modern instruments good data may be obtained from samples weighing less than a milligram.

¹⁵ "NMR Spectroscopy (Nuclear Magnetic Resonance) - Principle, Working, Chemical Shift, Instrumentation & Applications of NMR Spectroscopy," BYJUS, accessed July 20, 2023



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The nucleus consists of elementary particles called neutrons and protons, which contain an intrinsic property called spin. Like electrons, the spin of a nucleus can be described using quantum numbers of l for the spin and m for the spin in a magnetic field.

The NMR uses the spin of the molecules in order to derive information about the molecules structure, movements and chemical environment.

NMR Working Principle:

- All nuclei are electrically charged and many have spin.
- Transfer of energy is possible from base energy to higher energy levels when an external magnetic field is applied.
- The transfer of energy occurs at a wavelength that coincides with the radio frequency.
- Also, energy is emitted at the same frequency when the spin comes back to its base level.
- Therefore, by measuring the signal which matches this transfer the processing of the NMR spectrum for the concerned nucleus is yield.

NMR is used for a number of scientific reasons:

- (i) perfumery compound discovery
- (ii) Nuclear magnetic resonance imaging to study human body
- (iii) determine structures of important chemical and biological compounds
- (iv) environmental testing
- (v) petroleum industry
- (vi) earth's field NMR and magnetometers

¹⁶ William Reusch, "NMR Spectroscopy," Msu.edu, 2013

