Instrumental technique presentation

Gas Chromatography –Mass Spectrometry (GC-MS)

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Chromatography is the separation of a mixture of compounds (solutes) into separate components. Gas Chromatography (GC) is one of these techniques, derived from the fact that the mobile phase is gaseous. Hyphenated or hybrid techniques are common in chemistry. For example,

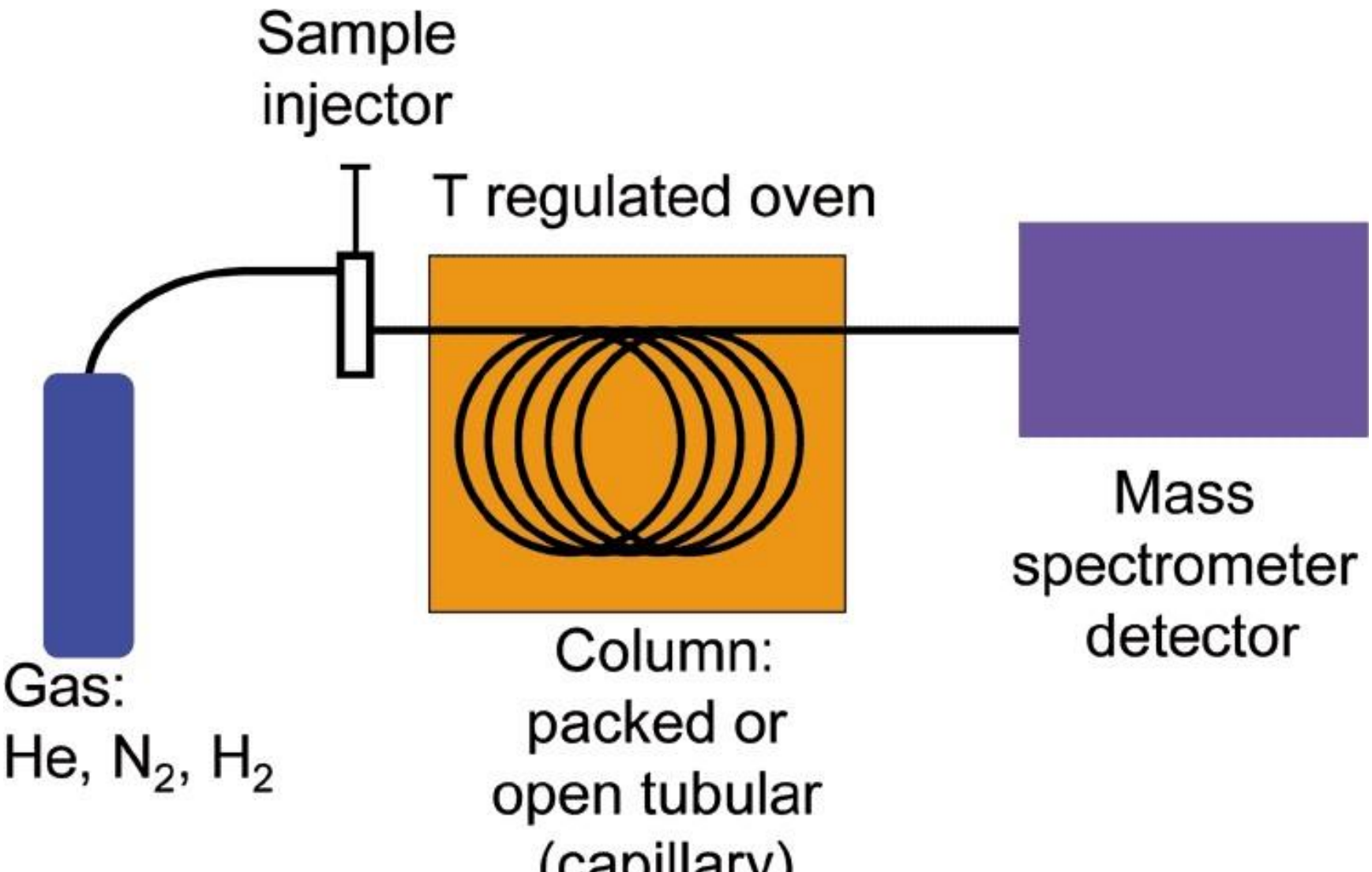
1. Gas chromatography-mass spectrometry
2. Gas chromatography-infrared spectroscopy
3. Liquid chromatography-mass spectrometry
4. Liquid chromatography-NMR spectroscopy
5. Liquid chromatography-infrared spectroscopy
6. Capillary electrophoresis-mass spectrometry, etc.

Out of all these techniques GC-MS is popular as it has several applications compare to other techniques.

Common Applications

- Quantitation of pollutants in drinking and wastewater using official U.S. Environmental Protection Agency (EPA) methods.
- Quantitation of drugs and their metabolites in blood and urine for both pharmacological and forensic applications.
- Identification of unknown organic compounds in hazardous waste dumps.
- Identification of reaction products by synthetic organic chemists.
- Analysis of industrial products for quality control.

Basic block diagram of GC-MS



Basic GC-MS theory

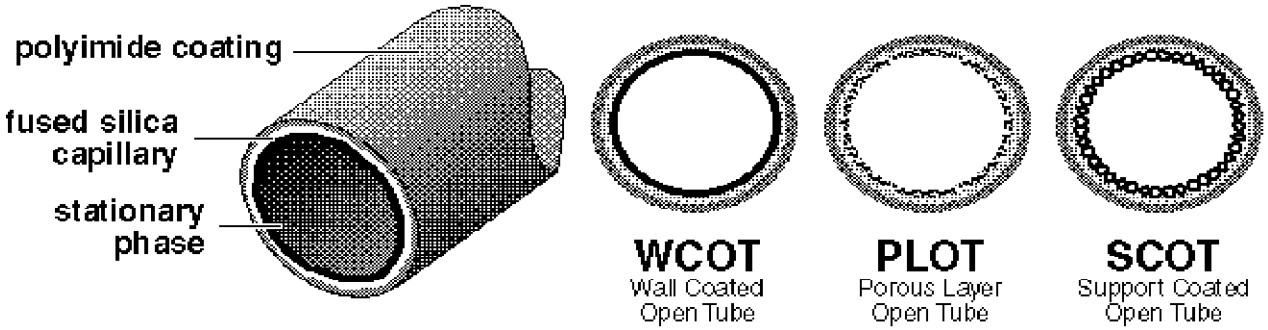
- Sample injected onto column via injector
- GC then separates sample molecules
- Effluent from GC passes through transfer line into the Ion Trap/Ion source
- Molecules then undergo electron /chemical ionisation
- Ions are then analysed according to their mass to charge ratio
- Ions are detected by electron multiplier which produces a signal proportional to ions detected

Column Types

Capillary Columns

Length: 10m to 100m

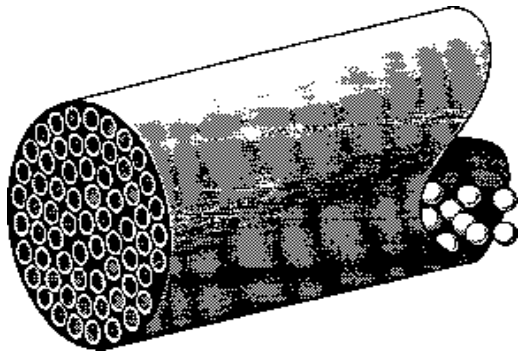
Diameter: 180um, 250um, 320um & 530um I.d



Packed Columns

Length: <2m

Diameter: 1/8" & ¼" OD



Examples for stationary phases, cyanopropylphenyl dimethyl polysiloxane, carbowax polyethyleneglycol

Open Tubular (capillary) Columns:

When the stationary phase is uniformly distributed on the interior surface of column it is called an open tubular (capillary) column.

Open tubular columns are longer, smaller in diameter, and more efficient than packed columns.

The most common stationary phases used for open tubular columns are polysiloxanes.

Polysiloxanes are silicon atoms which have attached oxygen and R groups.

The R groups can vary, which makes polysiloxanes very versatile.

WCOT :

The wall coated open tubular column consists of a capillary tube with its interior surface coated in a tiny layer of stationary phase.

The most common type of wall coated open tubular column used is fused-silica, because it is stronger, inert, reliable, easy to use, and flexible.

The fused silica column also has a layer of polyimide on the outside of the column, which makes the column flexible and extends the life of the column.

SCOT :

A support-coated open tubular column has a thin layer (approximately 30 μm) of liquid support matter.

This type of open tubular column has a greater amount of stationary phase than the wall coated column, so it can handle a larger quantity of sample.

PLOT :

A porous-layer open tubular (PLOT) column is very similar to a support-coated open tubular column.

The only difference between the two types of columns is that a PLOT does not have a liquid stationary phase. PLOT columns are used for gas solid chromatography.

PLOT columns have a solid layer of carbon, molecular sieves, cyclodextrins, inorganic oxides, or porous polymers, coating the inner wall of the column. PLOT columns can be up to 100 meters long. The inner diameter of a PLOT column is between 0.25 and 0.53 mm. The stationary phase coating is between 5 and 50 micrometers thick.

Packed column :

Unlike GC capillary columns, which are referred to as WCOT (Wall Coated Open Tubular), or PLOT (Porous Layered Open Tubular), packed columns are what their name implies, they are packed full of fine particles and not “Open”. Because they are packed, they have a much higher pressure drop across the column. This is why they tend to be much shorter in length than a capillary column.

Samples

State:

Organic compounds must be in solution for injection into the gas chromatograph.

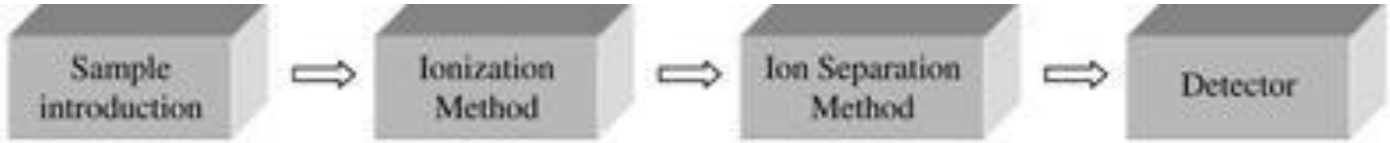
The solvent must be volatile and organic (for example, hexane or dichloromethane).

Nano gram sample requirement.

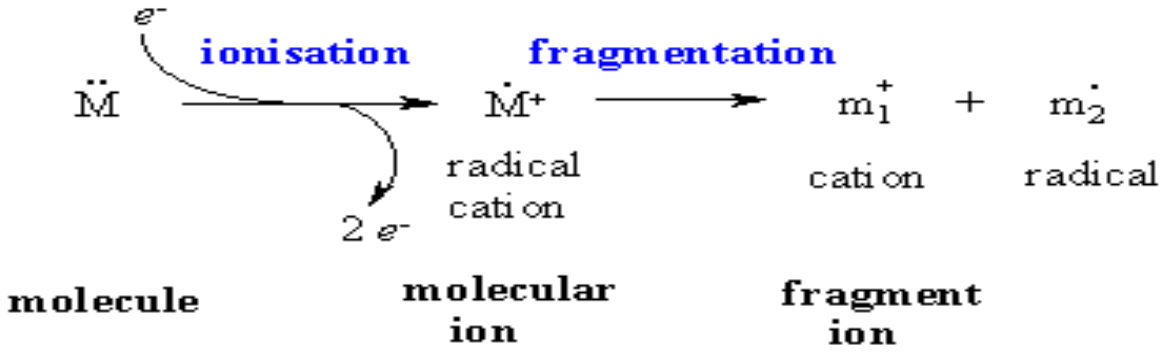
General Limitations:

1. Vapour pressure >10^{-10} torr
2. Otherwise chemically derivatized (e.g. trimethylsilyl ethers)
3. Determining positional substitution on aromatic rings is often difficult
4. Certain isomeric compounds are not possible to differentiate with MS

MS Process



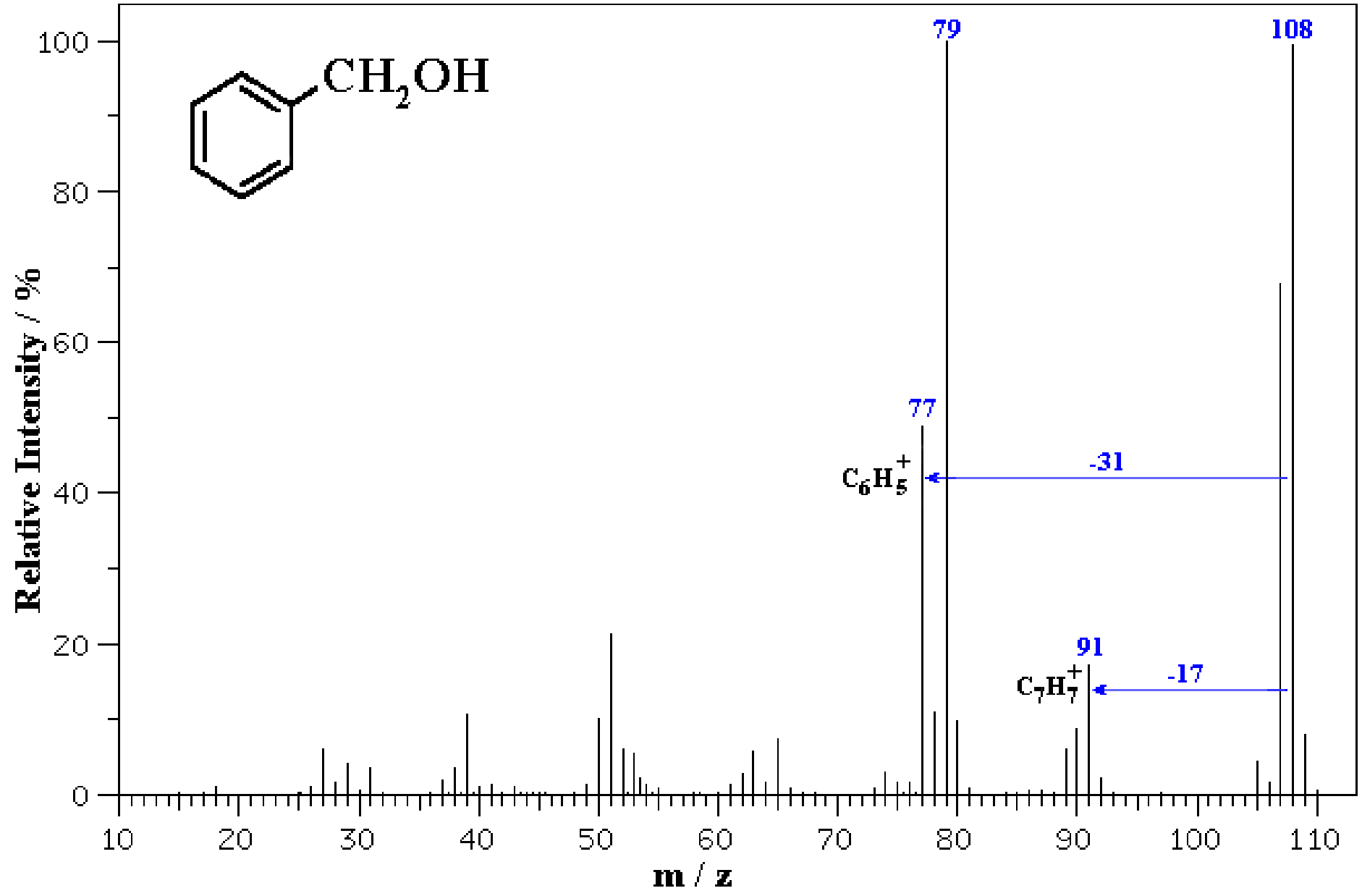
Electron Ionisation



Quadrupole :

It is basically 4 rods that have a combination of Radio-frequency (RF) and direct current (DC) voltages applied to them. The combination is chosen to allow only ions with a specific m/z or a range of m/z to transmit through the analyzer.

Because of the simplicity to use, sensitivity, and quick scan speeds these are useful for applications as GC-MS and LC-MS.


CH2OH
79
108
77
C6H5+
-31
91
C7H7+
-17
Relative Intensity / %
m / z
0
20
40
60
80
100
10
20
30
40
50
60
70
80
90
100
110

Limitations :

Pressure range is 10^{-5} to 10^{-6}

Background interference

Regular removal of column should not be done

Minimum use of water based sample for detection through MS as it can damage the filament.

Thank u