

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,

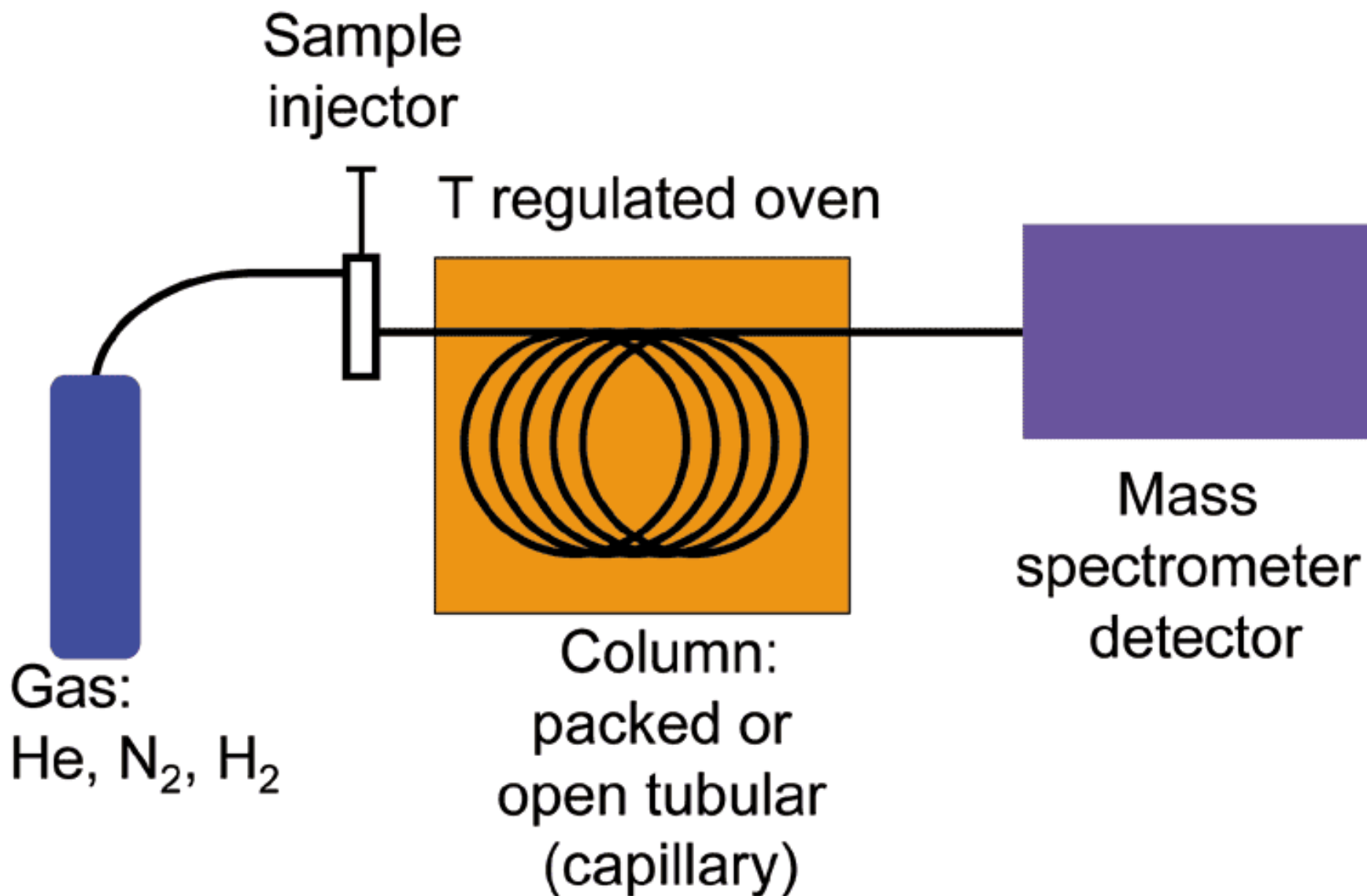
1. Gas chromatography-mass spectrometry
2. Gas chromatography-infrared spectroscopy
3. Liquid chromatography-mass spectrometry
4. Liquid chromatography-NMR spectroscopy
5. Liquid chromatography-infrared spectroscopy
6. Capillary electrophoresis-mass spectrometry, etc.

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

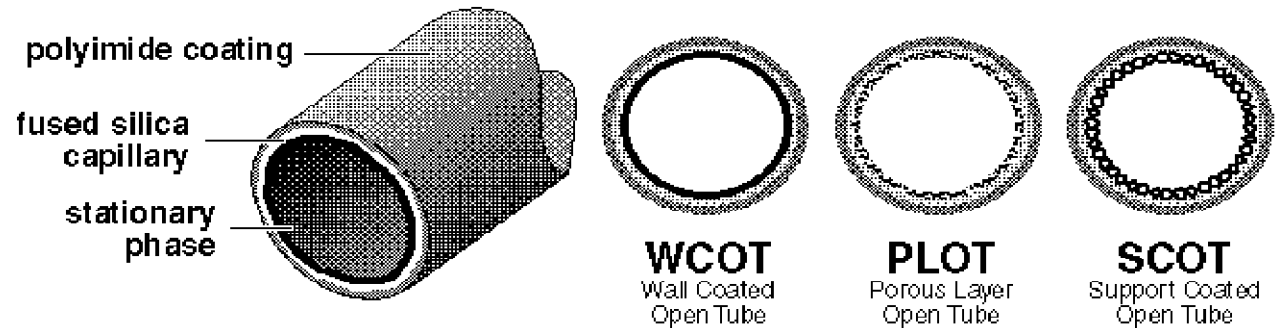
- Electron multiplier passes the ion current signal to system electronics
- Signal is amplified
- Result is digitised
- Results can be further processed and displayed

Column Types

Capillary Columns

Length: 10m to 100m

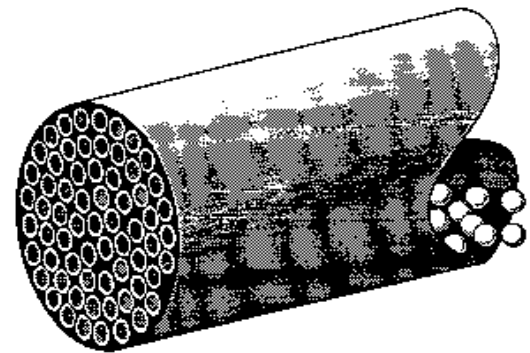
Diameter: 180µm,
250µm, 320µm &
530µm I.d



Packed Columns

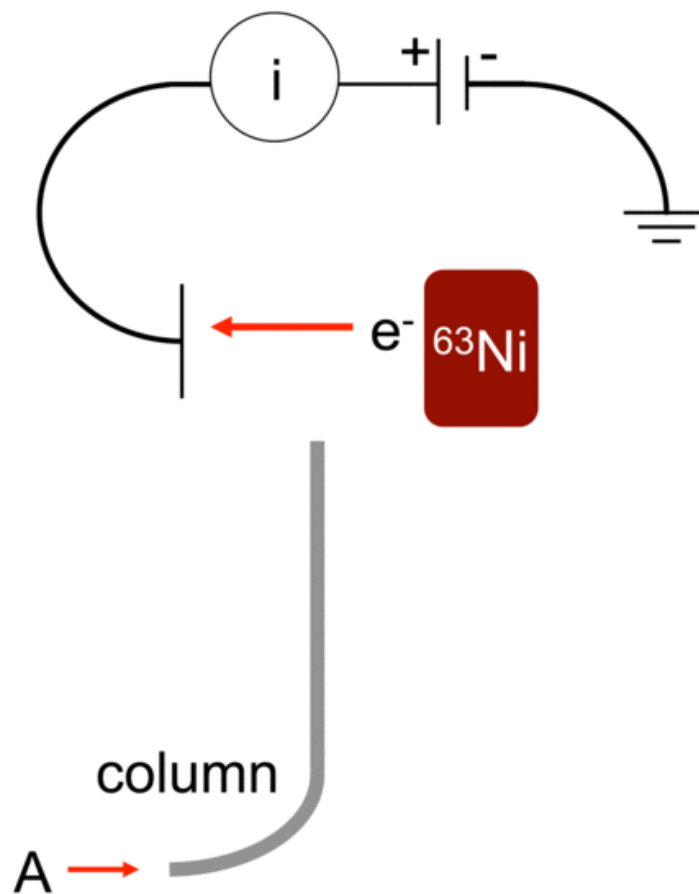
Length: <2m

Diameter: 1/8" & 1/4" OD



Examples for stationary phases, cyanopropylphenyl dimethyl polysiloxane, carbowax polyethyleneglycol

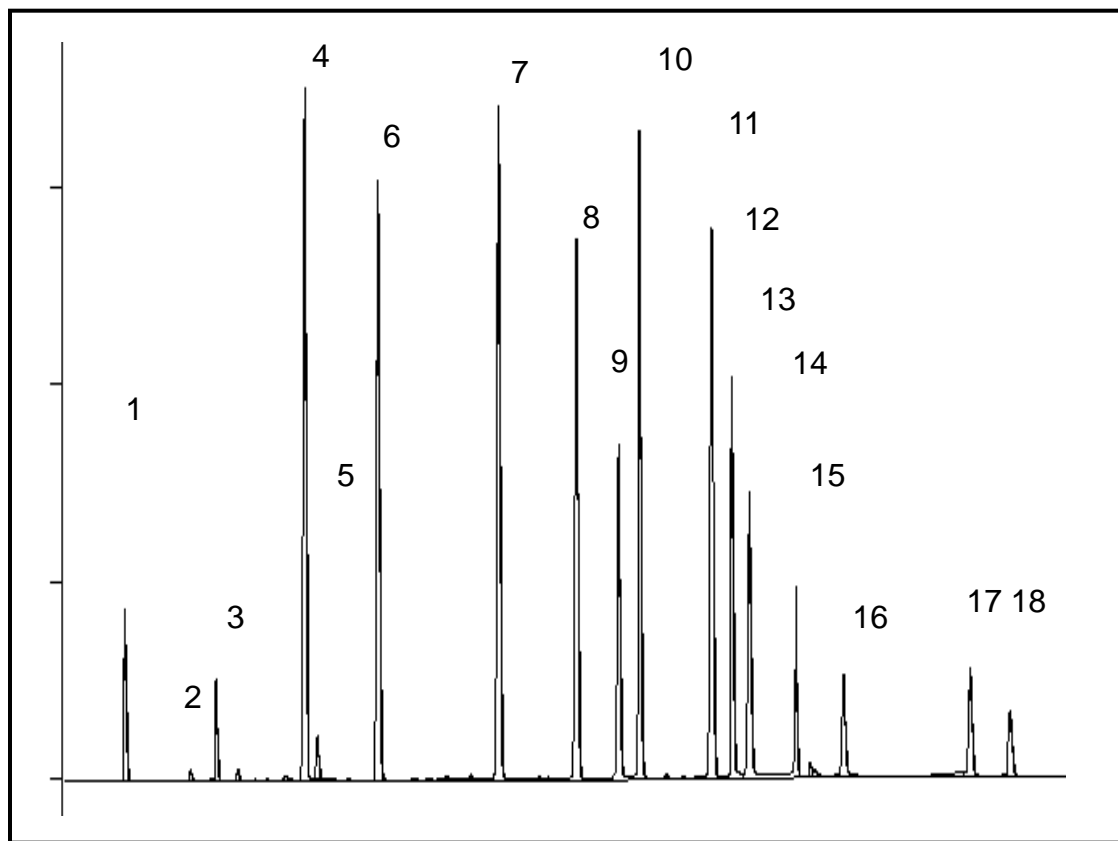
Electron capture detector



halogens, organometallic
compounds, nitriles, or nitro
compounds.

Analysis of Halogenated Pesticides

1. α -HCH
2. γ -HCH
3. β -HCH
4. Heptachlor
5. δ -HCH
6. Aldrin
7. Heptachlor epoxide
8. Endosulfan I
9. 4,4'-DDE
10. Dieldrin
11. Endrin
12. 4,4'-DDD
13. Endosulfan II
14. 4,4'-DDT
15. Endrin aldehyde
16. Endosulfan sulfate
17. Methoxychlor
18. Endrin ketone



2ppb in Water

Samples

State:

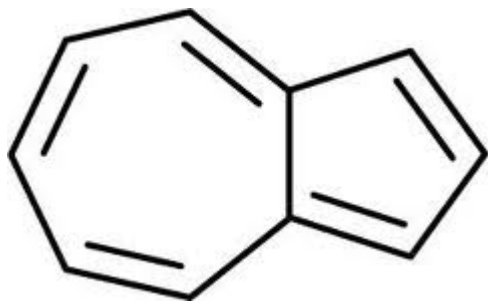
Organic compounds must be in solution for injection into the gas chromatograph. The solvent must be volatile and organic (for example, hexane or dichloromethane).

Amount:

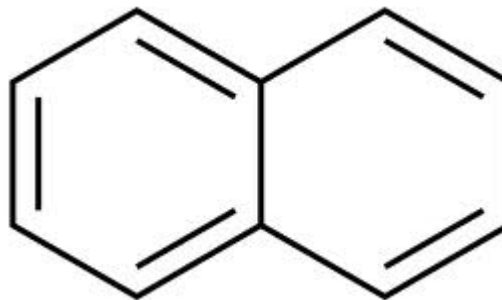
1 to 100 pg per component are routine

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



Azulene



Naphthalene

General precautions:

1. High-quality (and expensive) solvents
2. Glassware to heat at 450 °C after solvent and acid washing

Highly compatible:

1. Two vapor phase sample
2. Low quantity of sample (less than 1 ng)

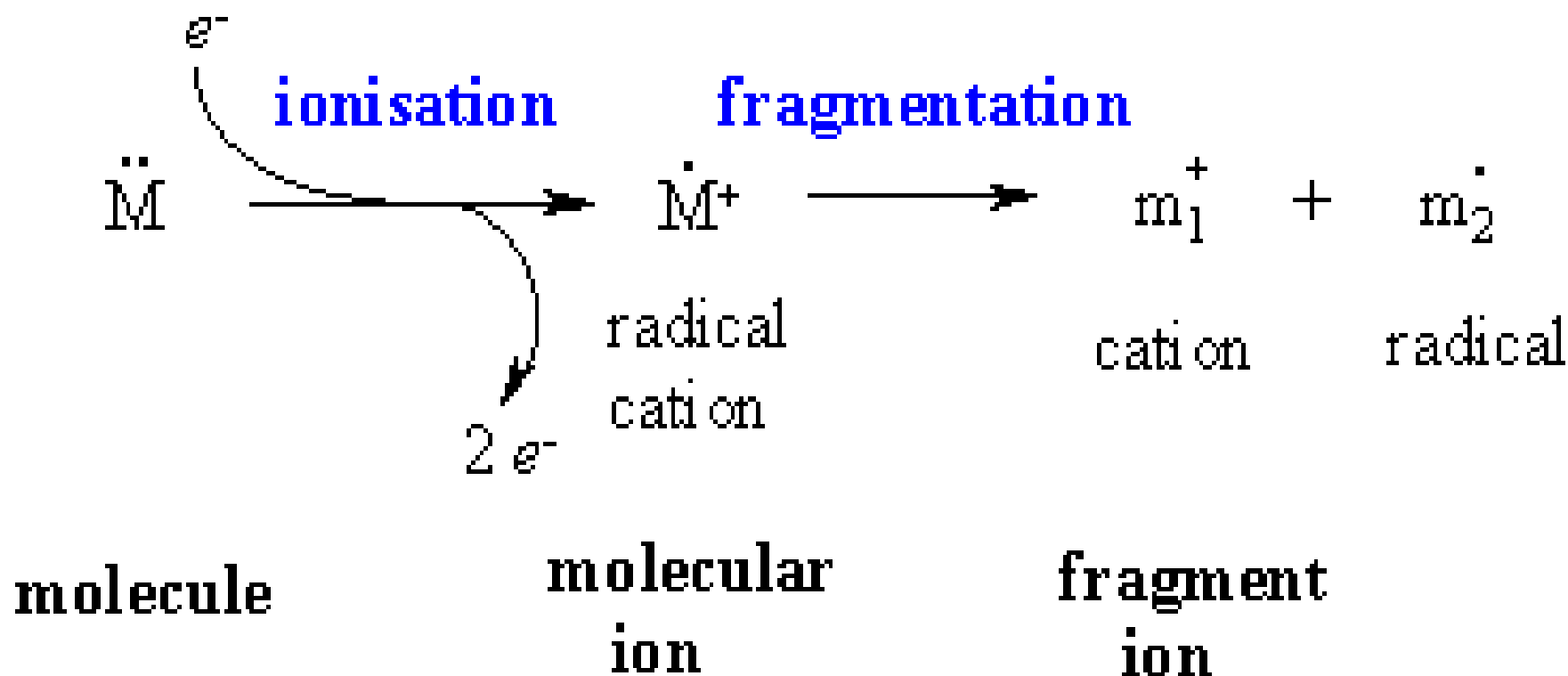
Highly incompatible:

1. GC exiting at nearly 760 torr
2. MS works at 10^{-6} - 10^{-5} torr

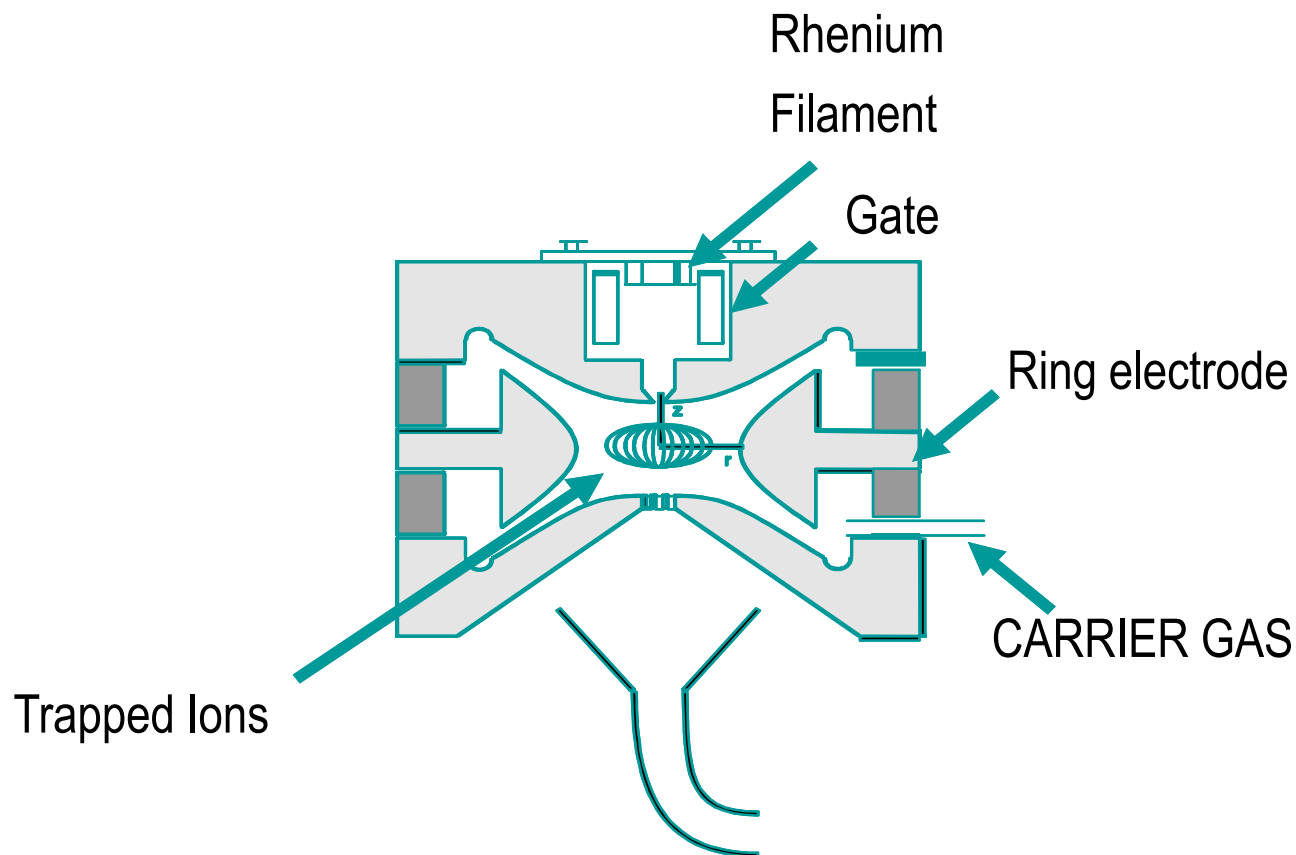
MS overview

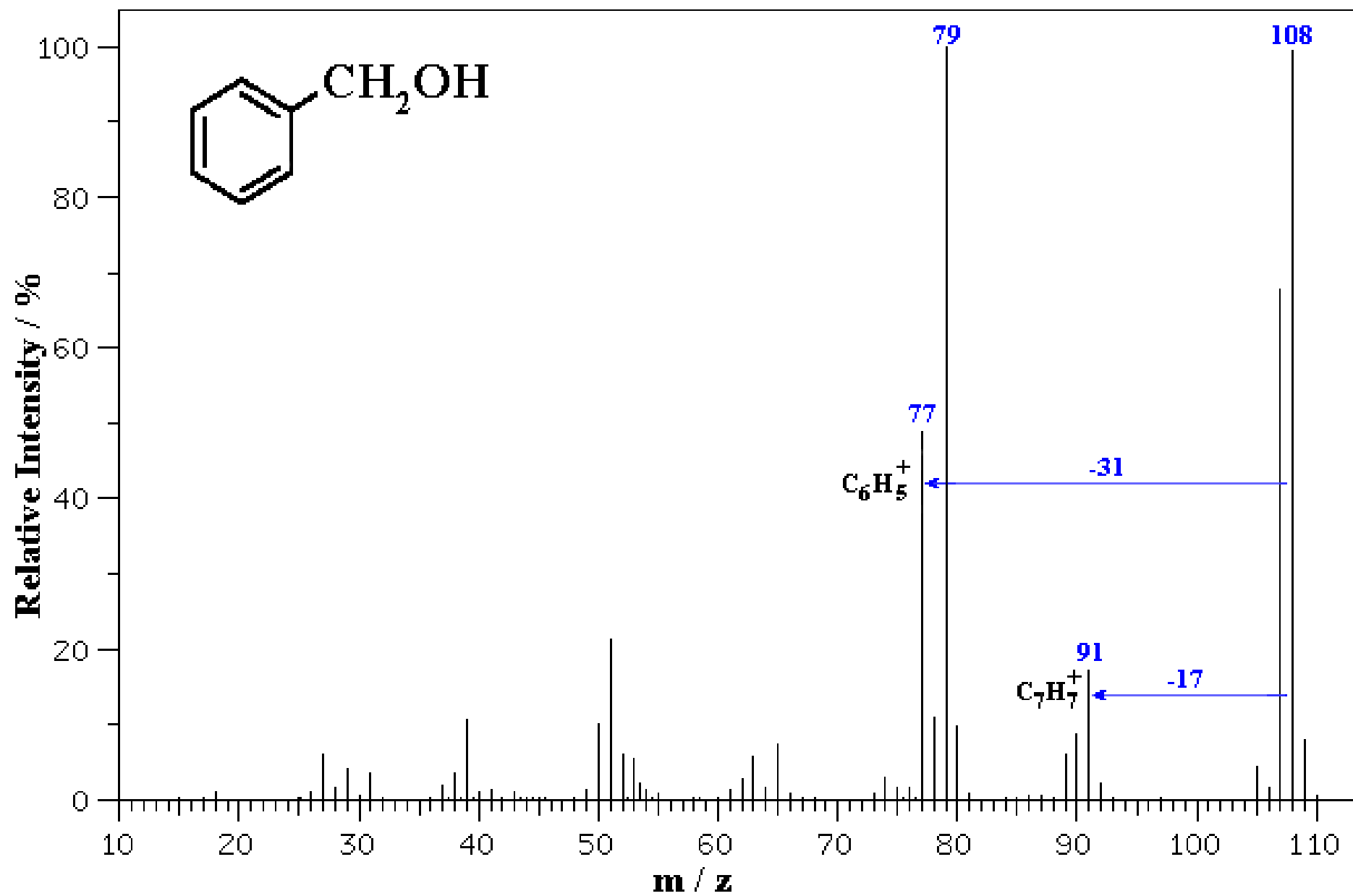
- Basic Mass Spectrometry Theory
- Types of Ionisation
 - Electronic Ionisation
 - Chemical Ionisation
- Interpretation of Mass Spectra
- Ion Trap Theory
- Components of the Ion Trap

Electron Ionisation



Electron Ionization Happens Inside the Ion Trap





Thanks