

GC

GAS CHROMATOGRAPHY

BASICS OF CHEMISTRY



Part - 2 of 2

GC DETECTOR



1. As Solutes Elute From The Column, They Interact With The Detector. The Detector Converts This Interaction Into An Electronic Signal That Is Sent To The Data System.
2. The Magnitude Of The Signal Is Plotted Versus Time (From The Time Of Injection) And A Chromatogram Is Generated.

TYPES OF GC DETECTORS

FLAME IONIZATION DETECTOR (FID)

NITROGEN PHOSPHORUS DETECTOR (NPD)

ELECTRON CAPTURE DETECTOR (ECD)

THERMAL CONDUCTIVITY DETECTOR (TCD)

FLAME PHOTOMETRIC DETECTOR (FPD)

PHOTOIONIZATION DETECTOR (PID)

ELECTROLYTIC CONDUCTIVITY DETECTOR (ELCD)

MASS SPECTROMETER (MS)

GC DETECTOR



3. Some Detectors Respond To Any Solute Eluting From The Column While Others Respond Only To Solutes With Specific Structures, Functional Groups Or Atoms Detectors That Exhibit Enhanced Response To Specific Types Of Solutes Are Called Selective Detectors.

TYPES OF GC DETECTORS

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GC DETECTOR



4. Some Detectors Respond To Any Solute Eluting From The Column While Others Respond Only To Solutes With Specific Structures, Functional Groups Or Atoms Detectors That Exhibit Enhanced Response To Specific Types Of Solutes Are Called Selective Detectors.
5. Most Detectors Require One Or More Gases To Function Properly.

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GC DETECTOR



6. There Are Combustion, Reagent, Auxiliary And Makeup Gases. In Some Cases, One Gas May Serve Multiple Purposes.
7. The Type Of Detector Gas Is Dependent On The Specific Detector And Is Fairly Universal Between GC Manufacturers.
8. The Flow Rates For Each Type Of Detector Varies Between GC Manufacturers. It Is Important To Follow The Recommended Flow Rates To Obtain The Optimal Sensitivity, Selectivity And Linear Range For A Detector.

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IDEAL DETECTOR HAS

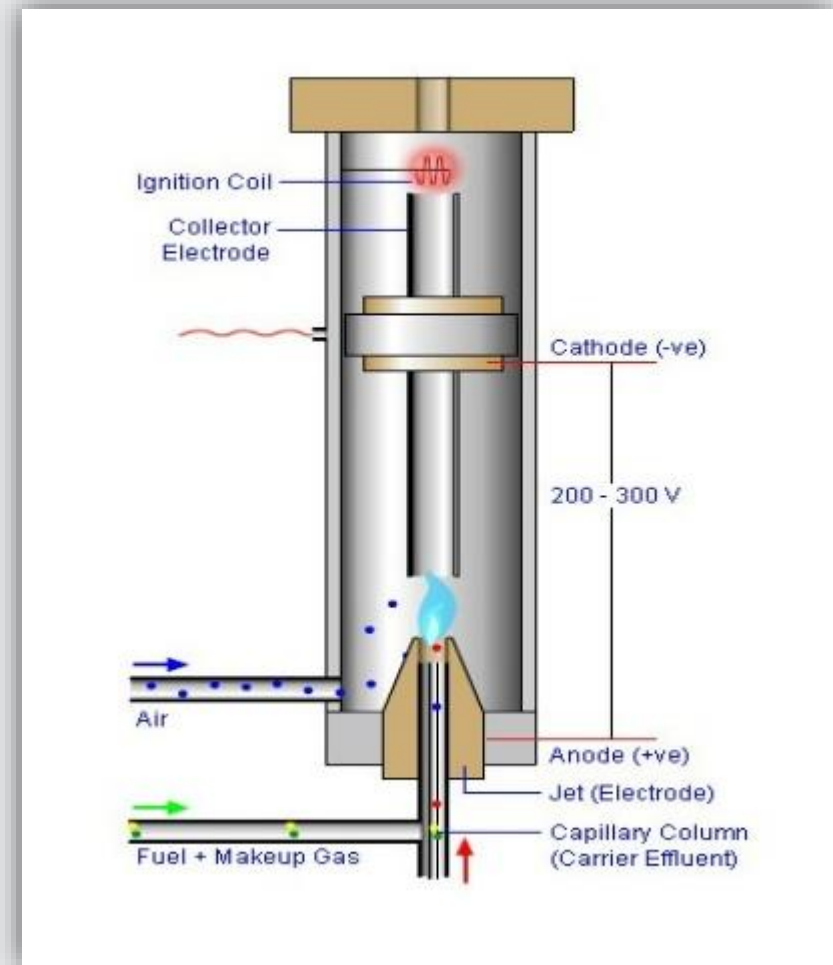


1. High Sensitivity.
2. Good Stability & Reproducibility.
3. A Linear Response To Solute.
4. Negligible Base Line Noise.
5. Should Be Inexpensive.
6. Capable Of Providing Data Necessary For Identification Of Solute.
7. A Short Response Time Independent Of Flow Rate.
8. High Reliability & Ease Of Operation
9. Non-Destructive Mode Of Action.
10. Responses Independent Of Mobile Phase Composition.
11. A Temperature Range From Room Temperature To At Least 500°C.

FLAME IONIZATION DETECTOR (FID)



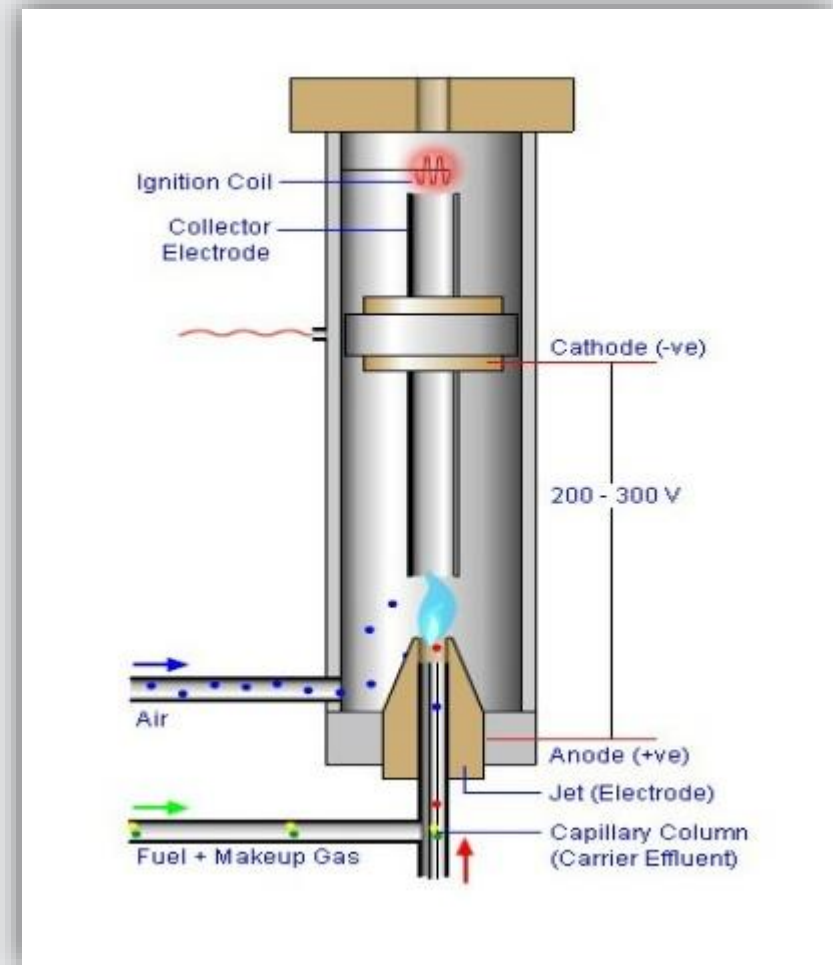
- 1. Mechanism:** Compounds Are Burned In A Hydrogen-air Flame. Carbon Containing Compounds Produce Ions That Are Attracted To The Collector. The Number Of Ions Hitting The Collector Is Measured And A Signal Is Generated.
- 2. Selectivity:** Compounds With C-H Bonds. A Poor Response For Some Non-hydrogen Containing Organics (e.g., Hexachlorobenzene).



FLAME IONIZATION DETECTOR (FID)



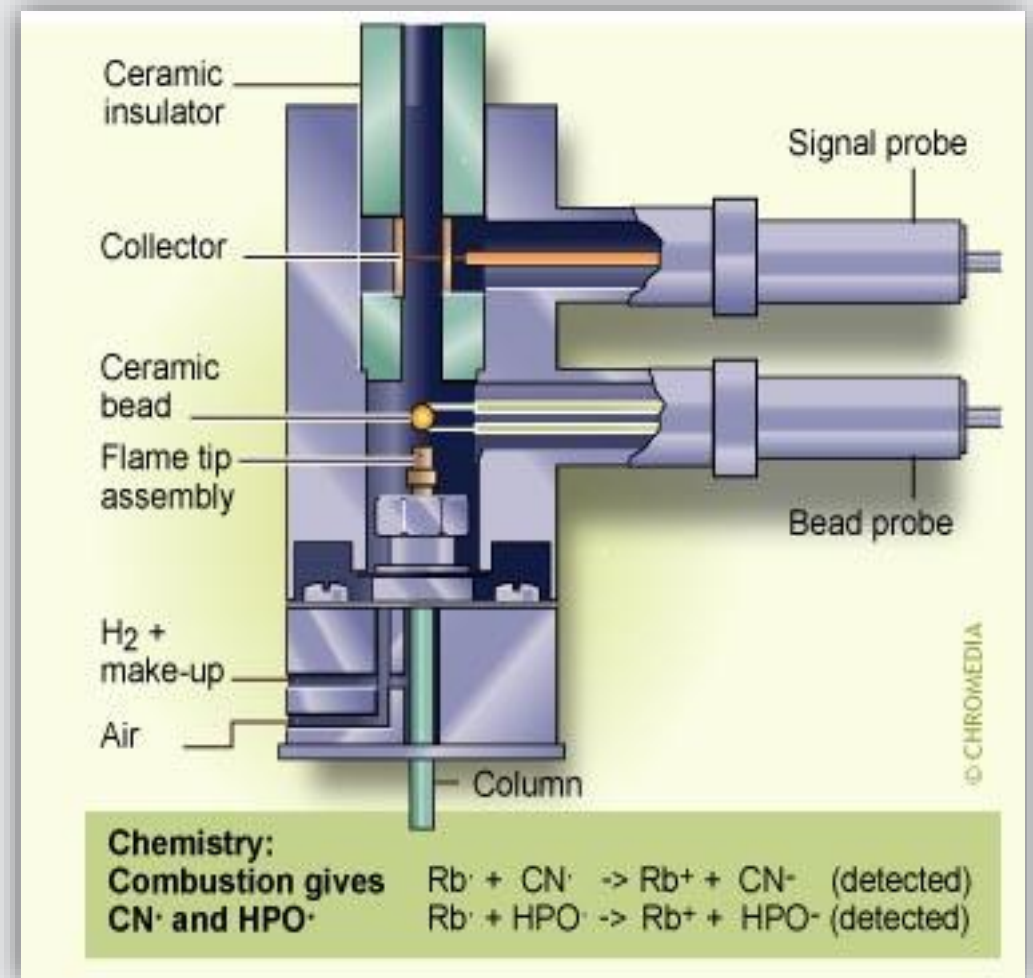
- 3. Sensitivity:** 0.1-10 ng
- 4. Linear Range:** 10^5 - 10^7
- 5. Gases:** Combustion - Hydrogen And Air; Makeup - Helium Or Nitrogen
- 6. Temperature:** 250-300°C, and 400-450°C For High Temperature Analyses.



NITROGEN PHOSPHORUS DETECTOR (NPD)



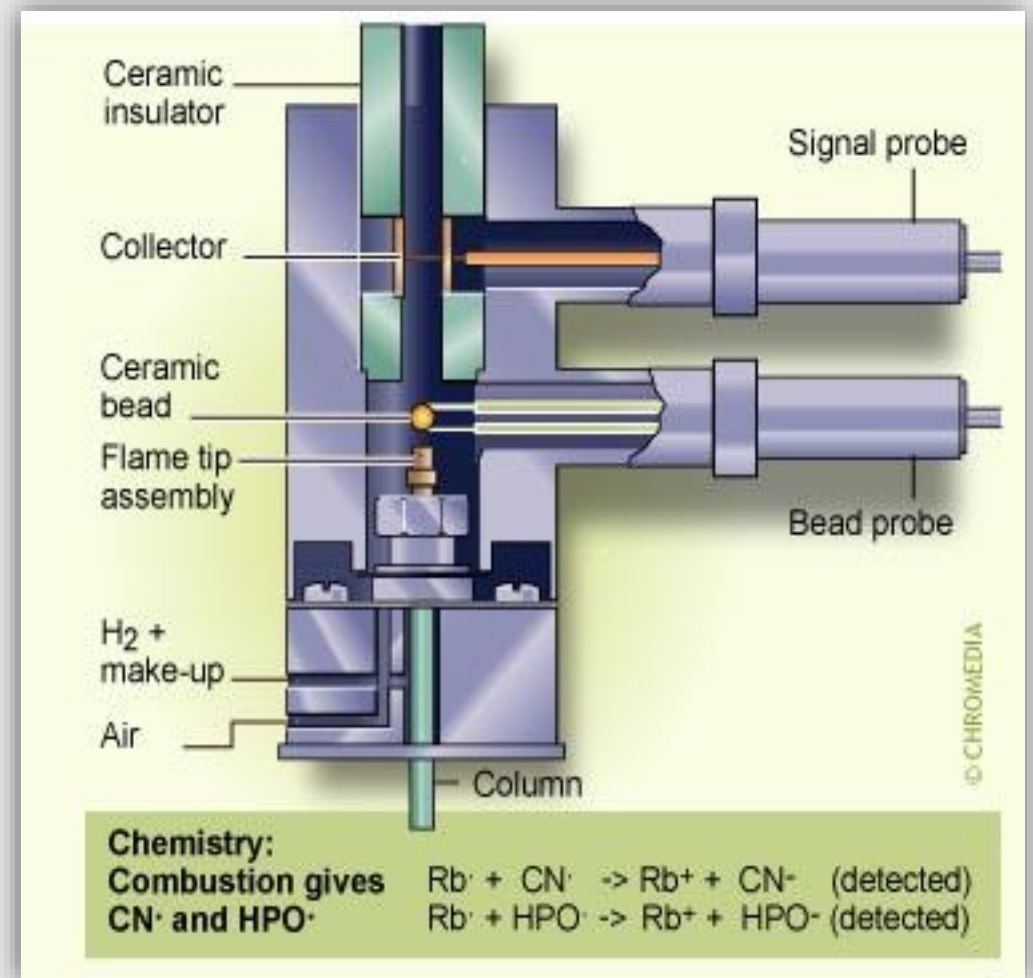
1. Mechanism: Compounds Are Burned In A Plasma Surrounding A Rubidium Bead Supplied With Hydrogen And Air. Nitrogen And Phosphorous Containing Compounds Produce Ions That Are Attracted To The Collector. The Number Of Ions Hitting The Collector Is Measured And A Signal Is Generated.



NITROGEN PHOSPHORUS DETECTOR (NPD)



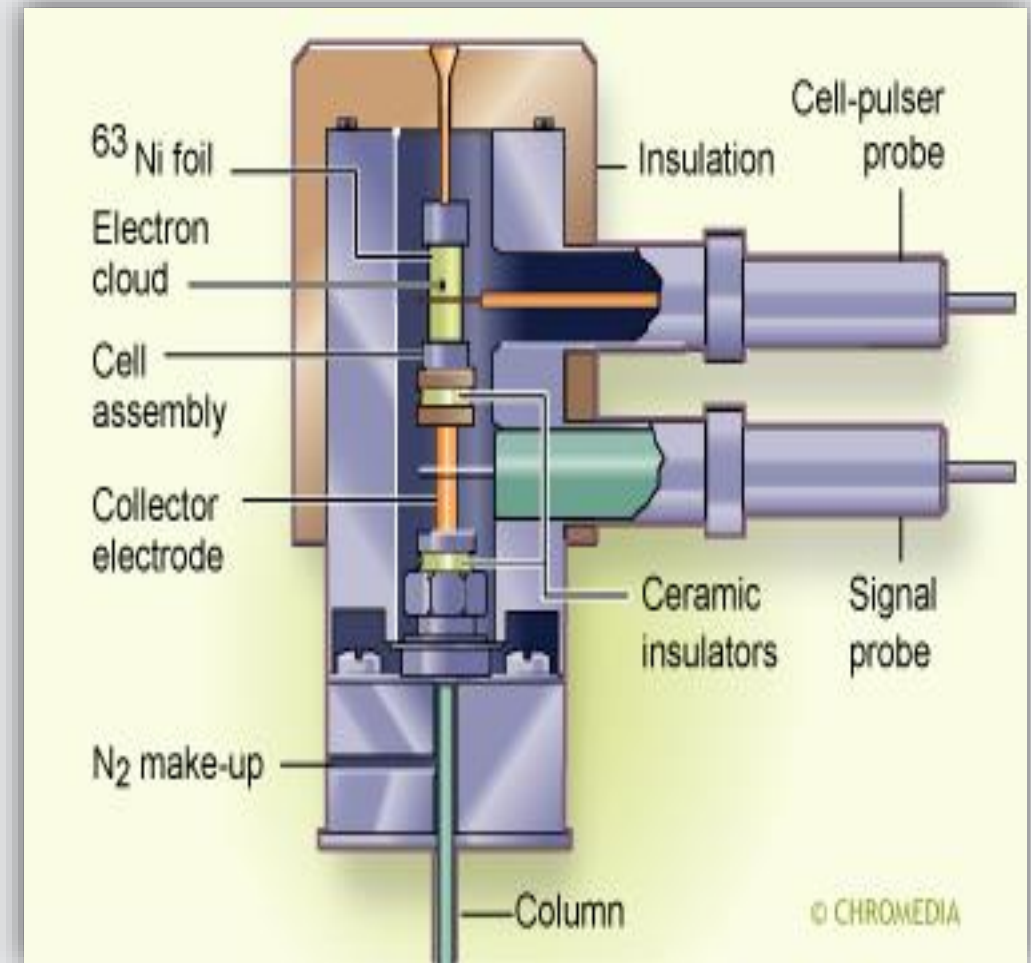
- 2. Selectivity:** Nitrogen And Phosphorous Containing Compounds
- 3. Sensitivity:** 1-10 pg
- 4. Linear Range:** 10^4 - 10^6
- 5. Gases:** Combustion - Hydrogen And Air; Makeup - Helium
- 6. Temperature:** 250-300°C



ELECTRON CAPTURE ELECTRODE (ECD)



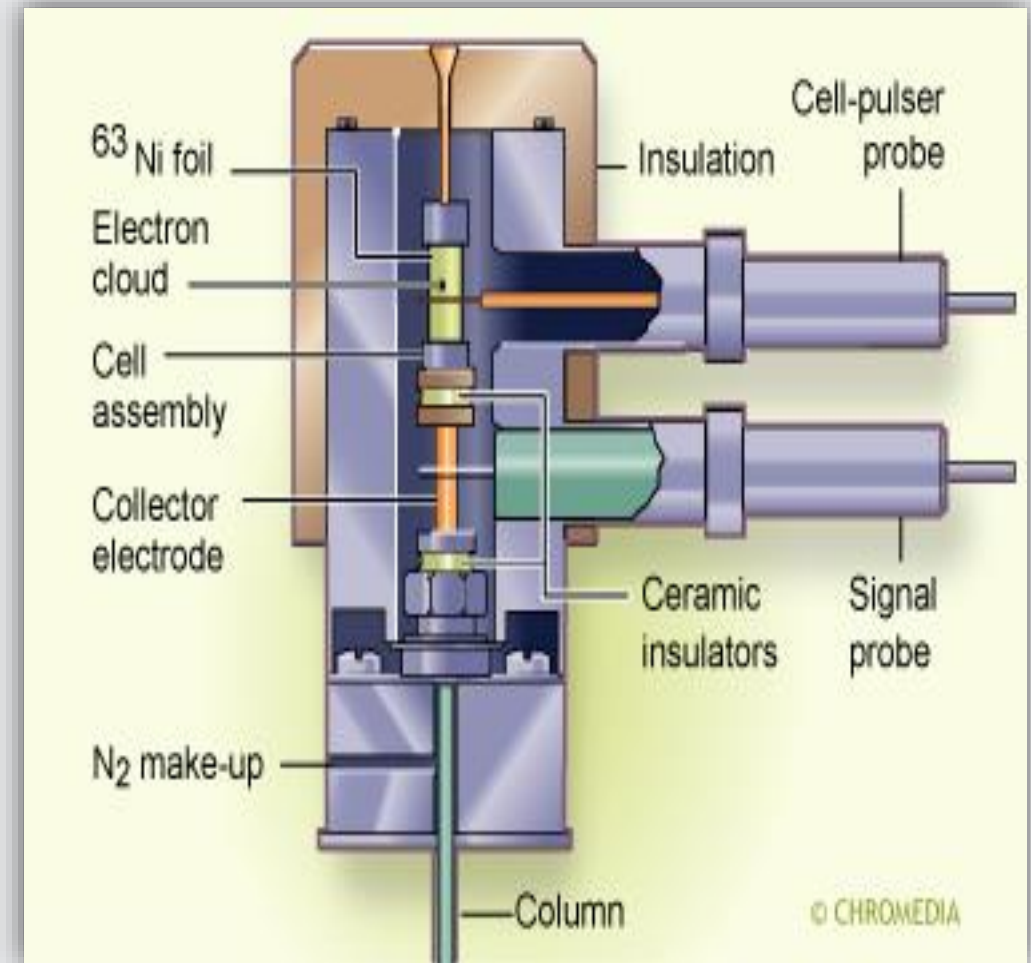
- Mechanism:** Electrons Are Supplied From A ^{63}Ni Foil Lining The Detector Cell. A Current Is Generated In The Cell. Electronegative Compounds Capture Electrons Resulting In A Reduction In The Current. The Amount Of Current Loss Is Indirectly Measured And A Signal Is Generated.



ELECTRON CAPTURE ELECTRODE (ECD)



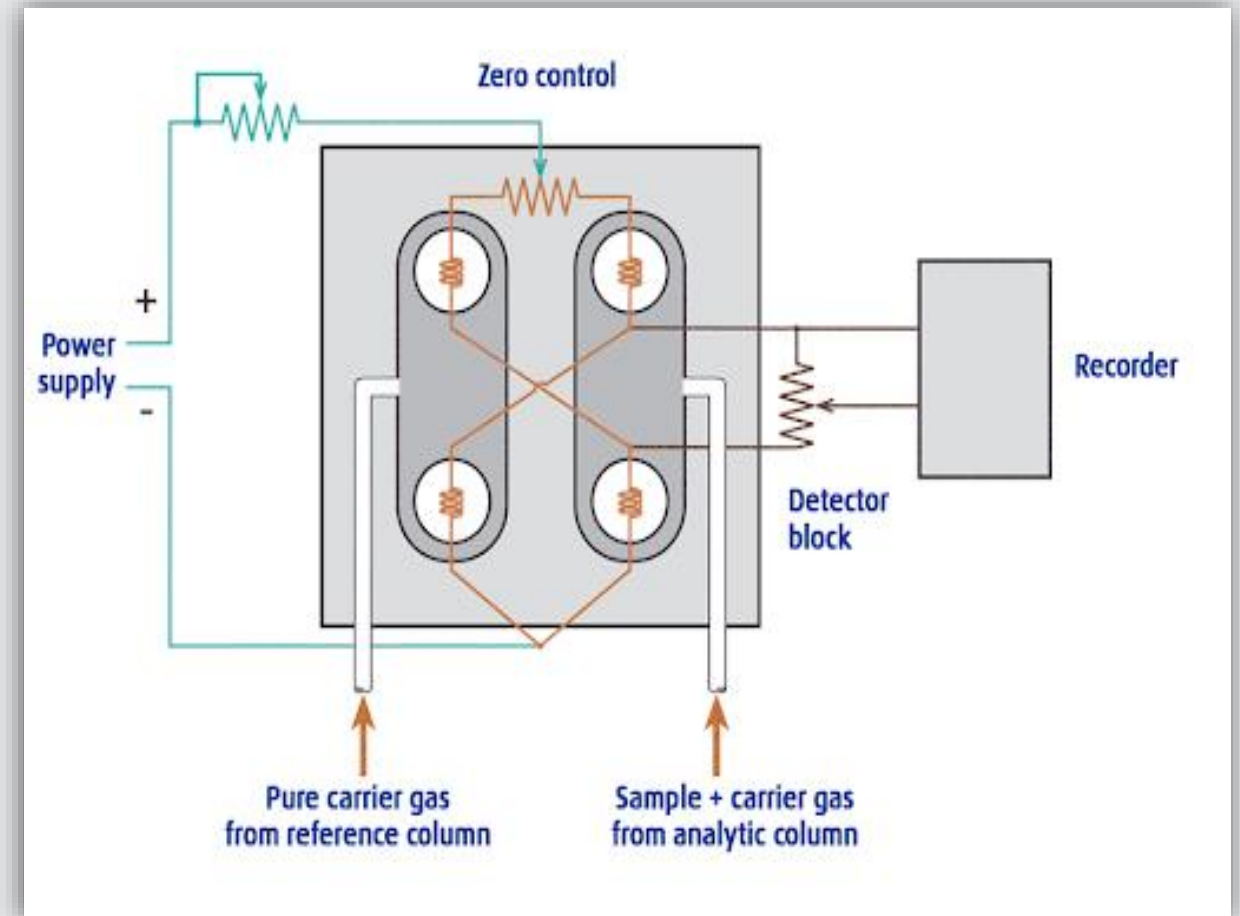
- 2. Selectivity:** Halogens, Nitrates And Conjugated Carbonyls
- 3. Sensitivity:** 0.1-10 pg (Halogenated Compounds); 1-100 pg (Nitrates); 0.1-1 ng (Carbonyls)
- 4. Linear Range:** 10^3 - 10^4
Gases: Nitrogen Or Argon/Methane
- 5. Temperature:** 300-400°C



THERMAL CONDUCTIVITY DETECTOR (TCD)



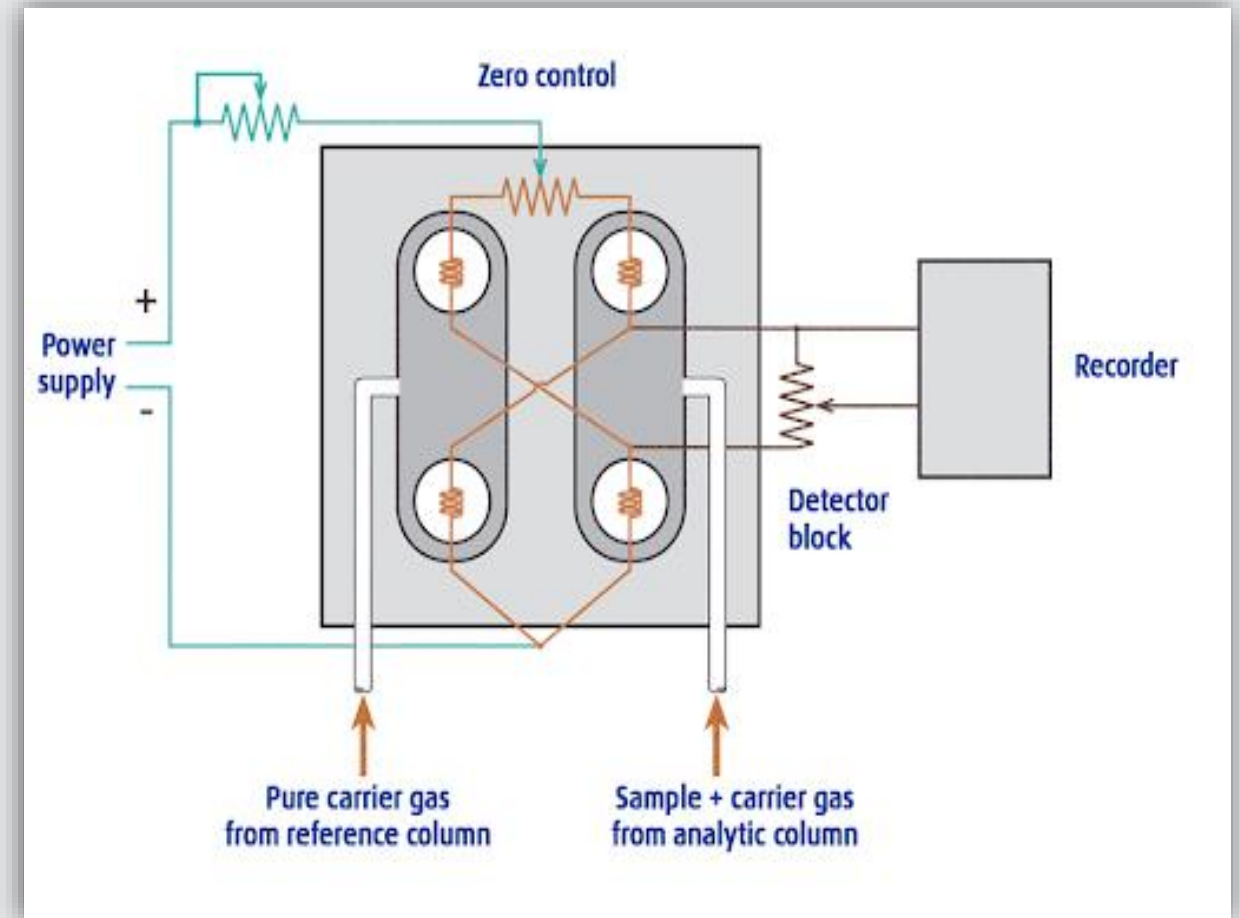
1. **Mechanism:** A Detector Cell Contains A Heated Filament With An Applied Current.
2. As Carrier Gas Containing Solutes Passes Through The Cell, A Change In The Filament Current Occurs. The Current Change Is Compared Against The Current In A Reference Cell. The Difference Is Measured And A Signal Is Generated.



THERMAL CONDUCTIVITY DETECTOR (TCD)



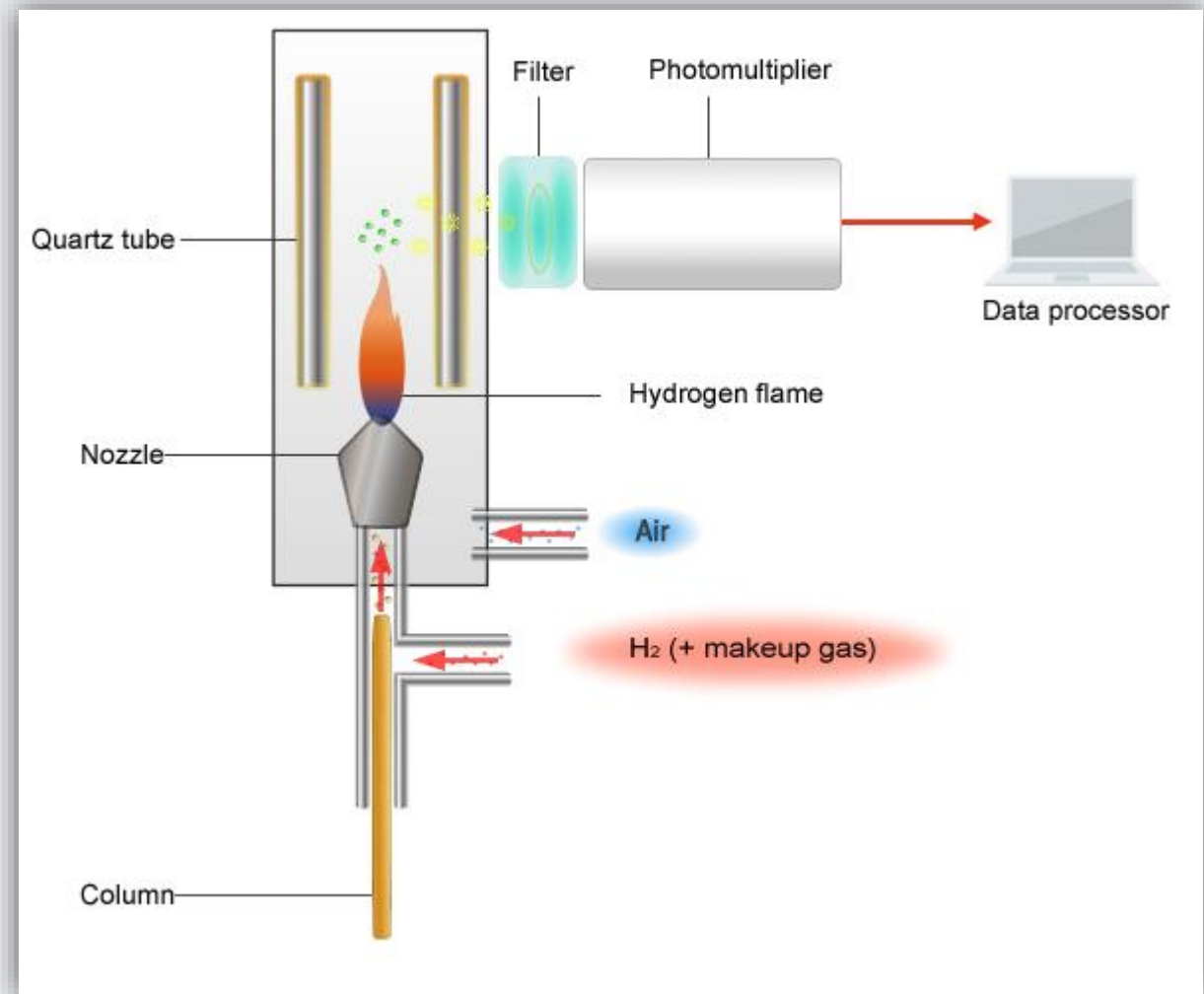
- 3. Selectivity:** All Compounds Except For The Carrier Gas
- 4. Sensitivity:** 5-20 ng
- 5. Linear Range:** 10^5 - 10^6
- 6. Gases:** Makeup - Same As The Carrier Gas
- 7. Temperature:** 150°C - 250°C



FLAME PHOTOMETRIC DETECTOR (FPD)



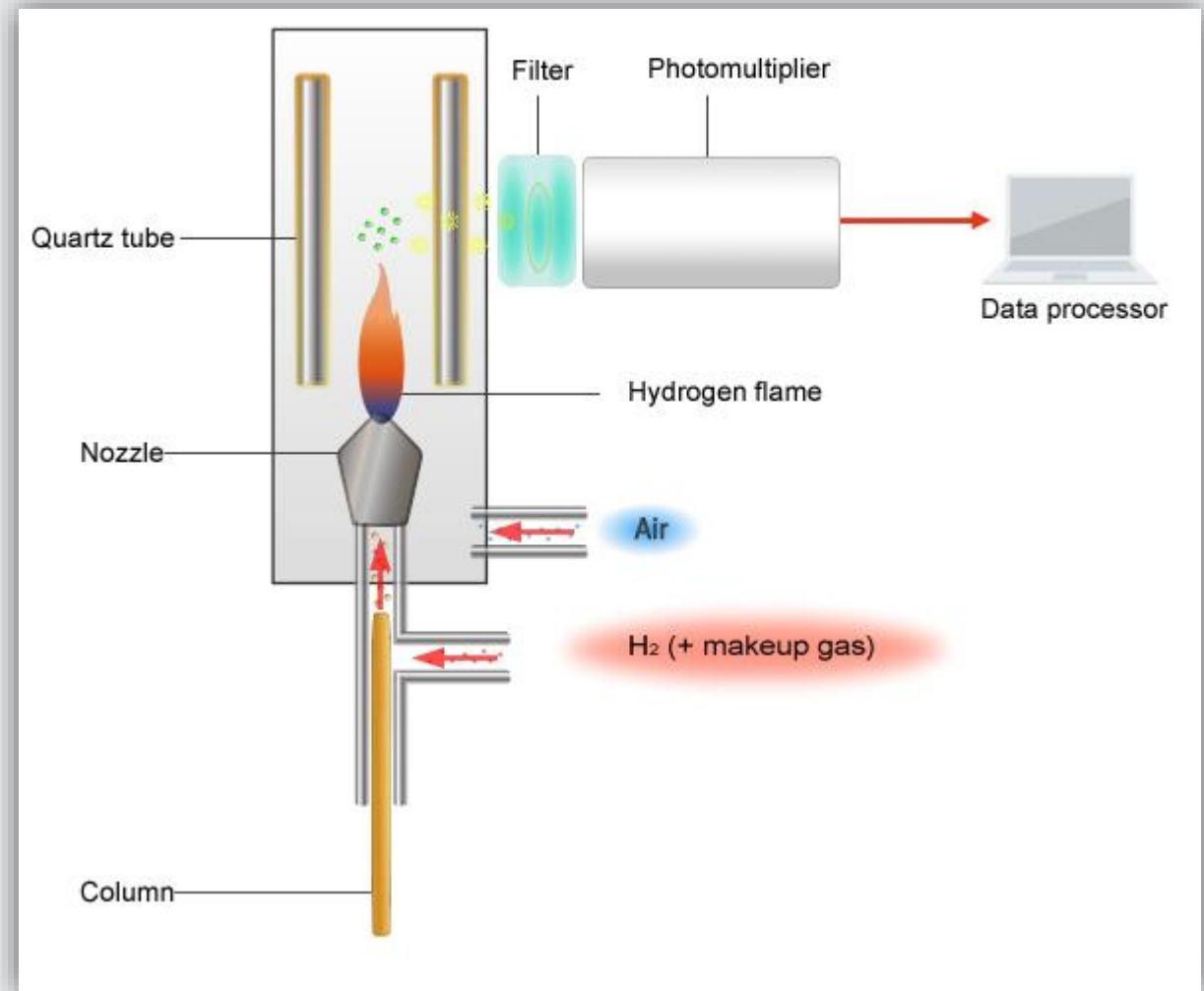
- Mechanism:** Compounds Are Burned In A Hydrogen-Air Flame. Sulfur And Phosphorous Containing Compounds Produce Light Emitting Species (Sulfur At 394 nm And Phosphorous At 526 nm).
- A Monochromatic Filter Allows Only One Of The Wavelengths To Pass. A Photomultiplier Tube Is Used To Measure The Amount Of Light And A Signal Is Generated. A Different Filter Is Required For Each Detection Mode.



FLAME PHOTOMETRIC DETECTOR (FPD)



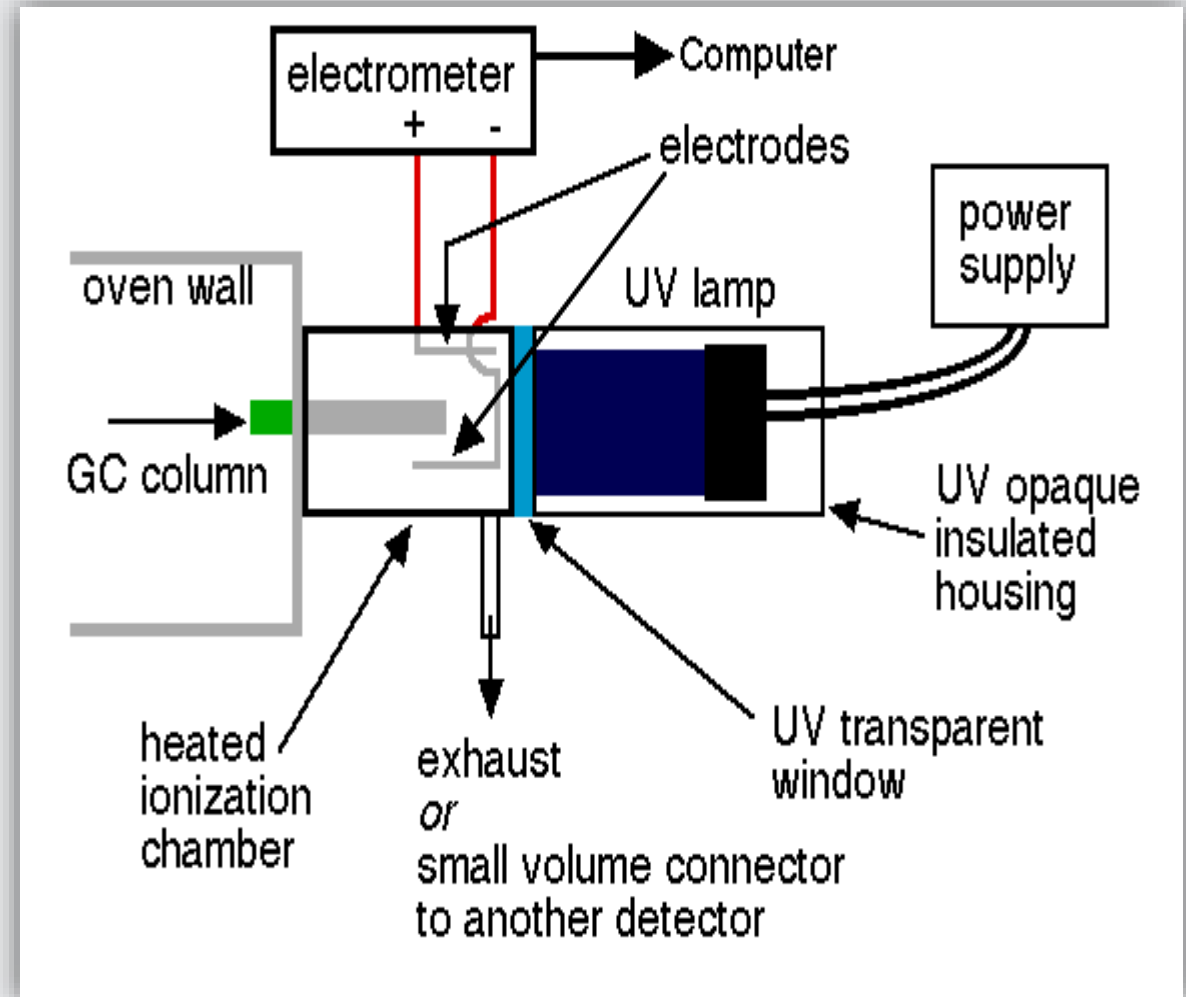
- 3. Selectivity:** Sulphur Or Phosphorous Containing Compounds. Only One At A Time.
- 4. Sensitivity:** 10-100 pg (sulfur); 1-10 pg (Phosphorous)
- 5. Linear Range:** Non-Linear (Sulfur); 10^3 - 10^5 (Phosphorous)
- 6. Gases:** Combustion - Hydrogen And Air; Makeup - Nitrogen
Temperature: 250°C -300°C



PHOTOIONIZATION DETECTOR (PID)



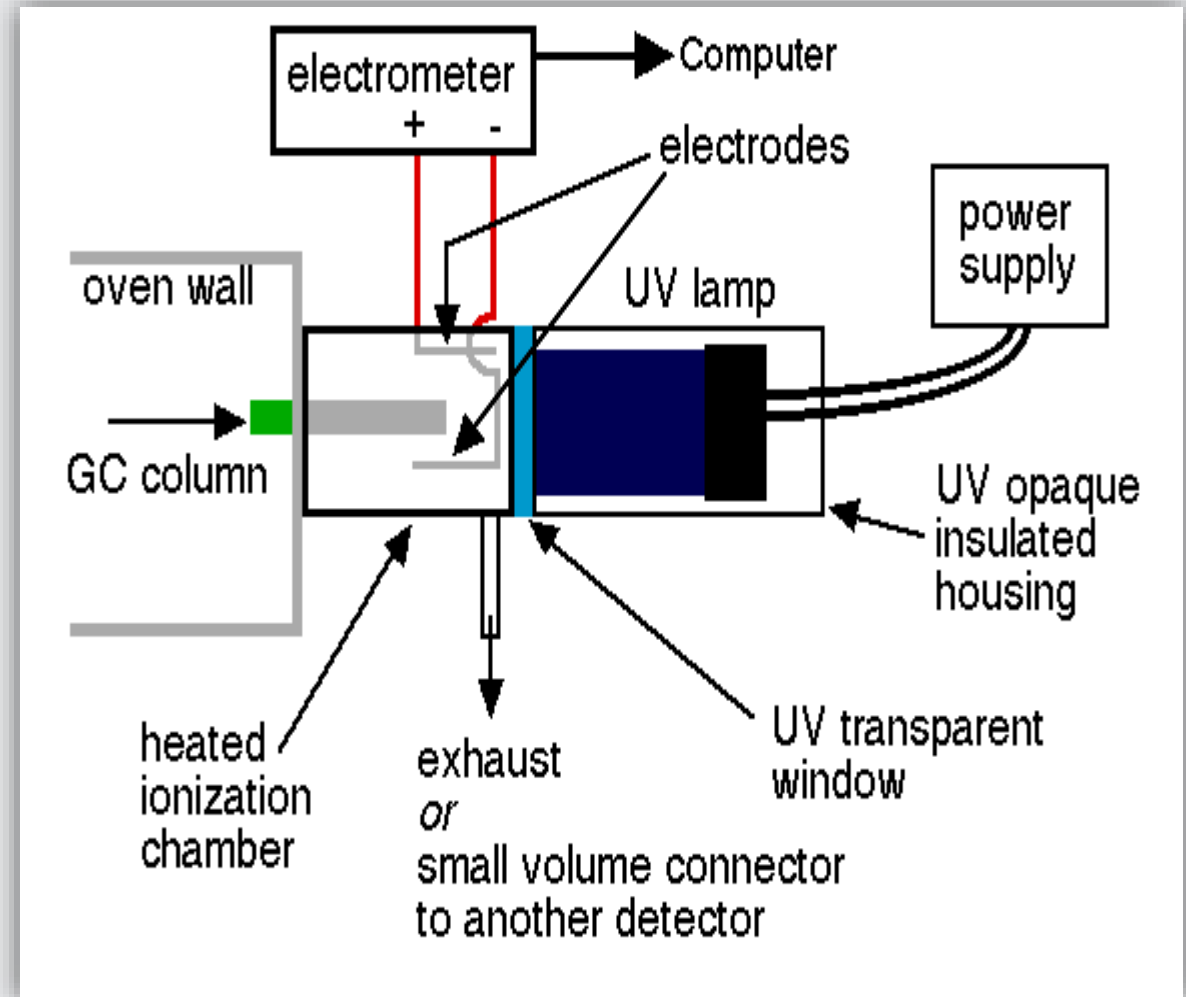
- Mechanism:** Compounds Eluting Into A Cell Are Bombarded With High Energy Photons Emitted From A Lamp.
- Compounds With Ionization Potentials Below The Photon Energy Are Ionized. The Resulting Ions Are Attracted To An Electrode, Measured, And A Signal Is Generated.



PHOTOIONIZATION DETECTOR (PID)



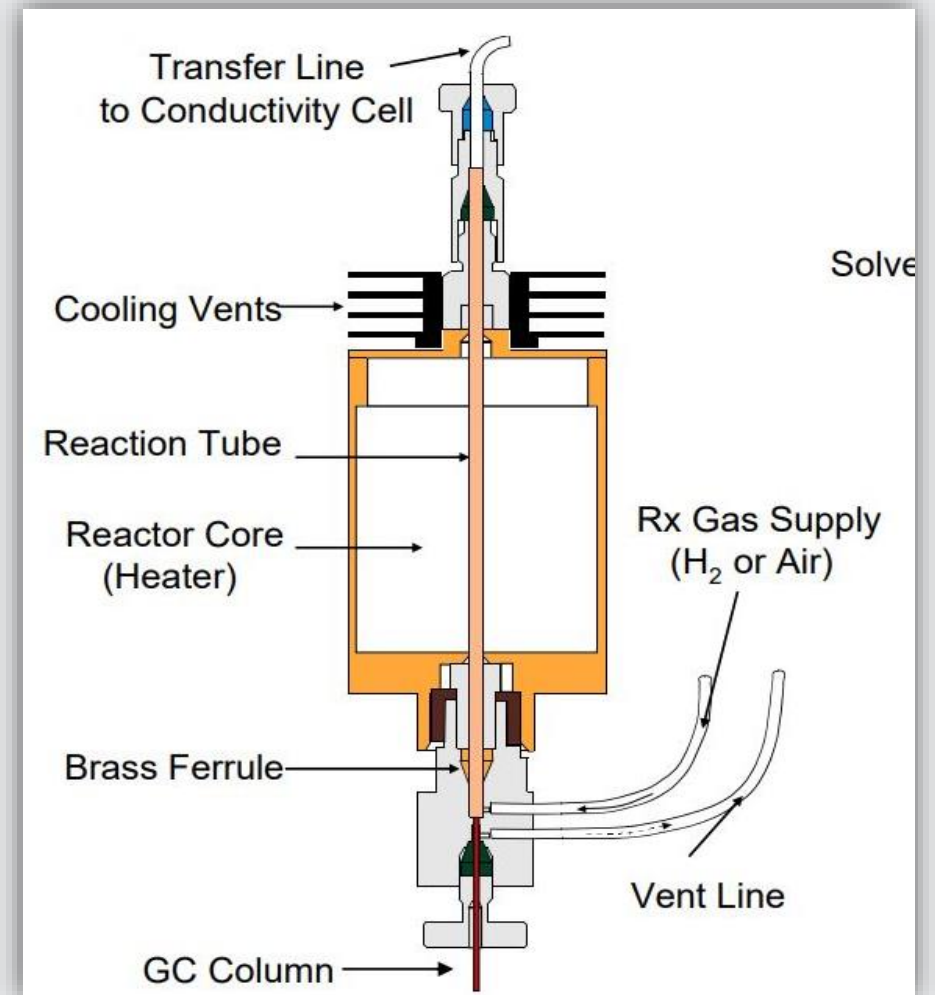
- 3. Selectivity:** Depends On Lamp Energy. Usually Used For Aromatics And Olefins (10 eV lamp).
- 4. Sensitivity:**
25-50 pg (Aromatics);
50-200 pg (Olefins)
- 5. Linear Range:** 10^5 - 10^6
- 6. Gases:** Makeup - Same As The Carrier Gas
- 7. Temperature:** 200°C



ELECTROLYTIC CONDUCTIVITY DETECTOR (ELCD)



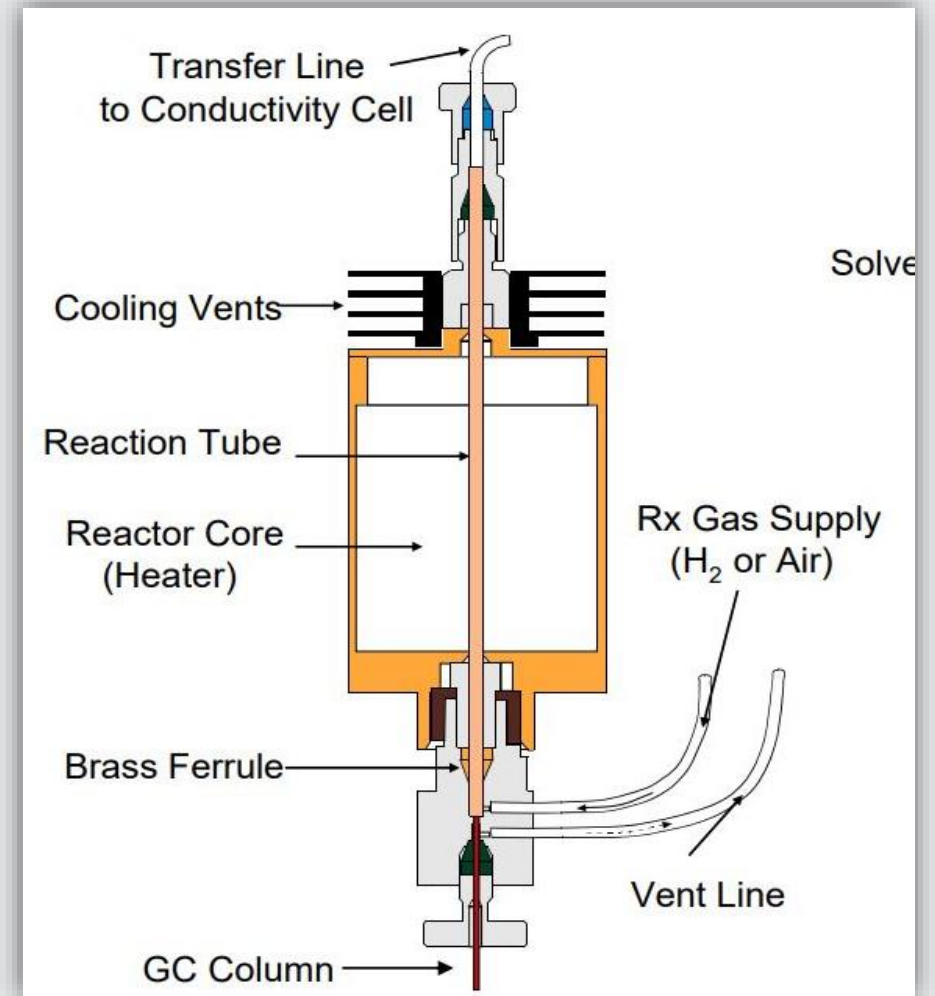
- Mechanism:** Compounds Are Mixed With A Reaction Gas And Passed Through A High Temperature Reaction Tube. Specific Reaction Products Are Created Which Mix With A Solvent And Pass Through An Electrolytic Conductivity Cell.
- The Change In The Electrolytic Conductivity Of The Solvent Is Measured And A Signal Is Generated. Reaction Tube Temperature And Solvent Determine Which Types Of Compounds Are Detected.



ELECTROLYTIC CONDUCTIVITY DETECTOR (ELCD)



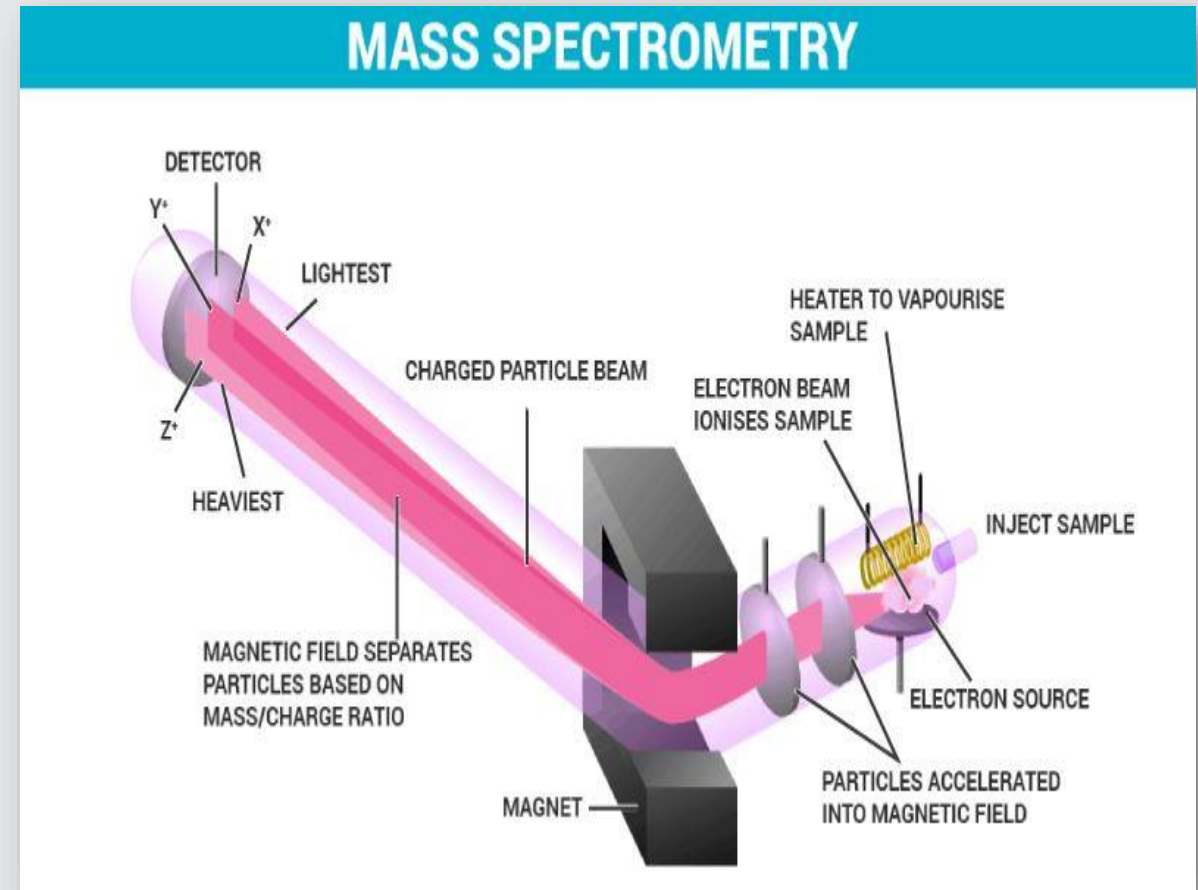
- 3. Selectivity:** Halogens, Sulfur Or Nitrogen Containing Compounds. Only One At A Time.
- 4. Sensitivity:** 5-10 pg (Halogens); 10-20 Pg (S); 10-20 Pg (N)
- 5. Linear Range:** 10^5 - 10^6 (Halogens); 10^4 - 10^5 (N); $10^{3.5}$ - 10^4 (S)
- 6. Gases:** Hydrogen (Halogens And Nitrogen); Air (Sulfur)
- 7. Temperature:** 800-1000°C (Halogens), 850-925°C (N), 750-825°C (S)



MASS SPECTROMETER DETECTOR (MSD)



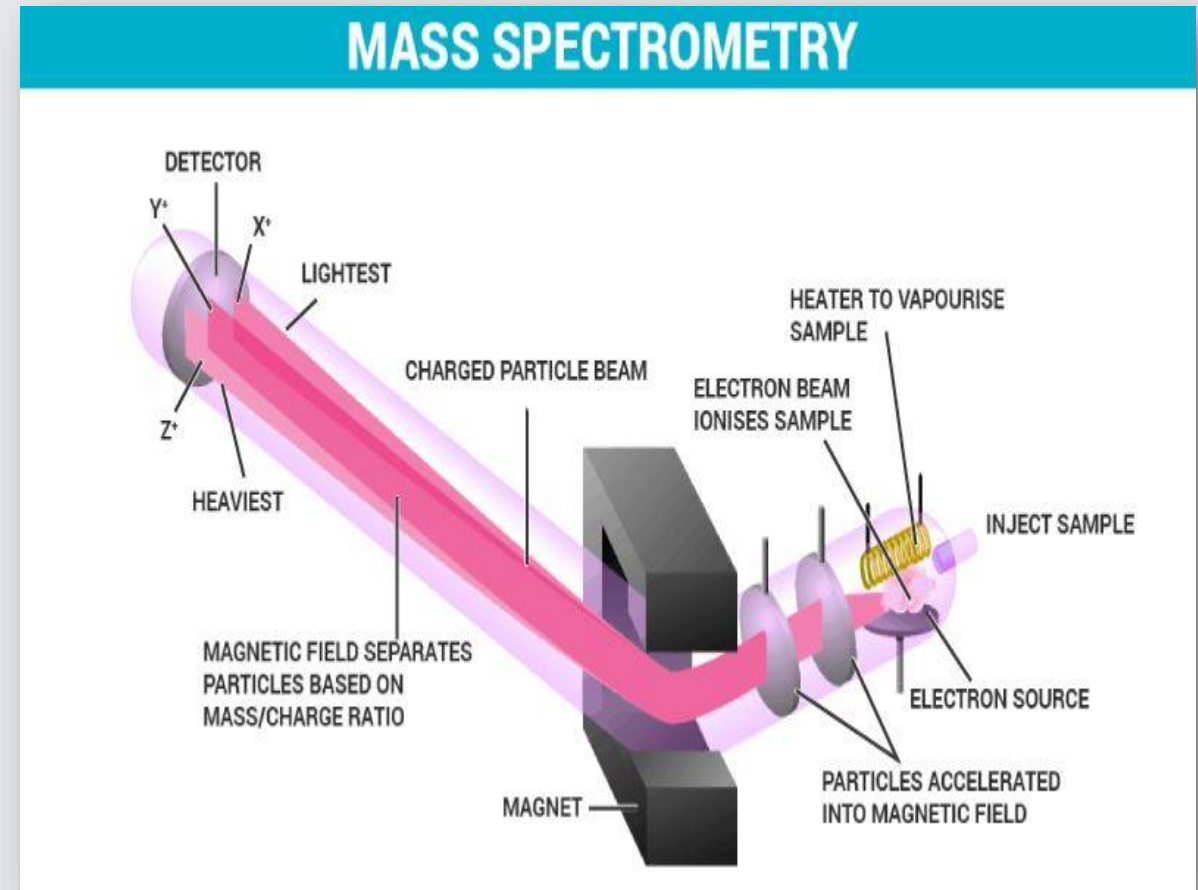
1. **Mechanism:** The Detector Is Maintained Under Vacuum. Compounds Are BombarDED With Electrons (EI) Or Gas Molecules (CI). Compounds Fragment Into Characteristic Charged Ions Or Fragments. The Resulting Ions Are Focused And Accelerated Into A Mass Filter.
2. The Mass Filter Selectively Allows All Ions Of A Specific Mass To Pass Through To The Electron Multiplier. All Of The Ions Of The Specific Mass Are Detected.



MASS SPECTROMETER DETECTOR (MSD)



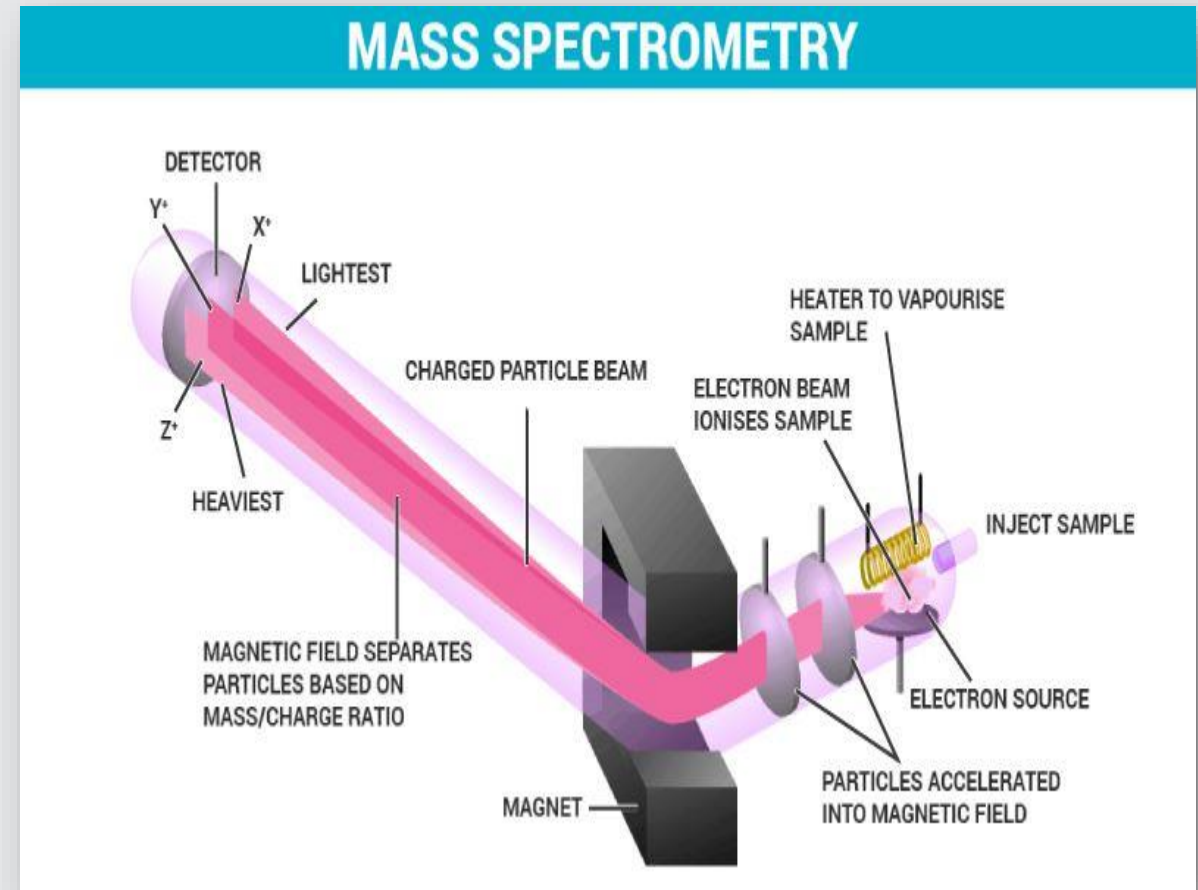
3. The Mass Filter Then Allows The Next Mass To Pass Through While Excluding All Others. The Mass Filter Scans Stepwise Through The Designated Range Of Masses Several Times Per Second. The Total Number Of Ions Are Counted For Each Scan.
4. The Abundance Or Number Of Ions Per Scan Is Plotted Versus Time To Obtain The Chromatogram (Called The TIC). A Mass Spectrum Is Obtained For Each Scan Which Plots The Various Ion Masses Versus Their Abundance Or Number.



MASS SPECTROMETER DETECTOR (MSD)



- 5. Selectivity:** Any Compound That Produces Fragments Within The Selected Mass Range. May Be An Inclusive Range Of Masses (Full Scan) Or Only Select Ions (SIM).
- 6. Sensitivity:** 1-10 ng (Full Scan); 1-10 pg (SIM)
- 7. Linear Range:** 10^5 - 10^6
- 8. Gases:** None
- 9. Temperature:** 250-300°C (Transfer Line), 150-250°C (Source)



MASS SPECTROMETER DETECTOR (MSD)



TYPES OF MASS SPECTROMETERS/ ANALYZERS

MAGNETIC-DEFLECTION MASS ANALYZER

DOUBLE-FOCUSING SECTOR SPECTROMETERS

QUADRUPOLE MASS ANALYZER

TIME-OF-FLIGHT SPECTROMETER

ION TRAP MASS SPECTROMETER

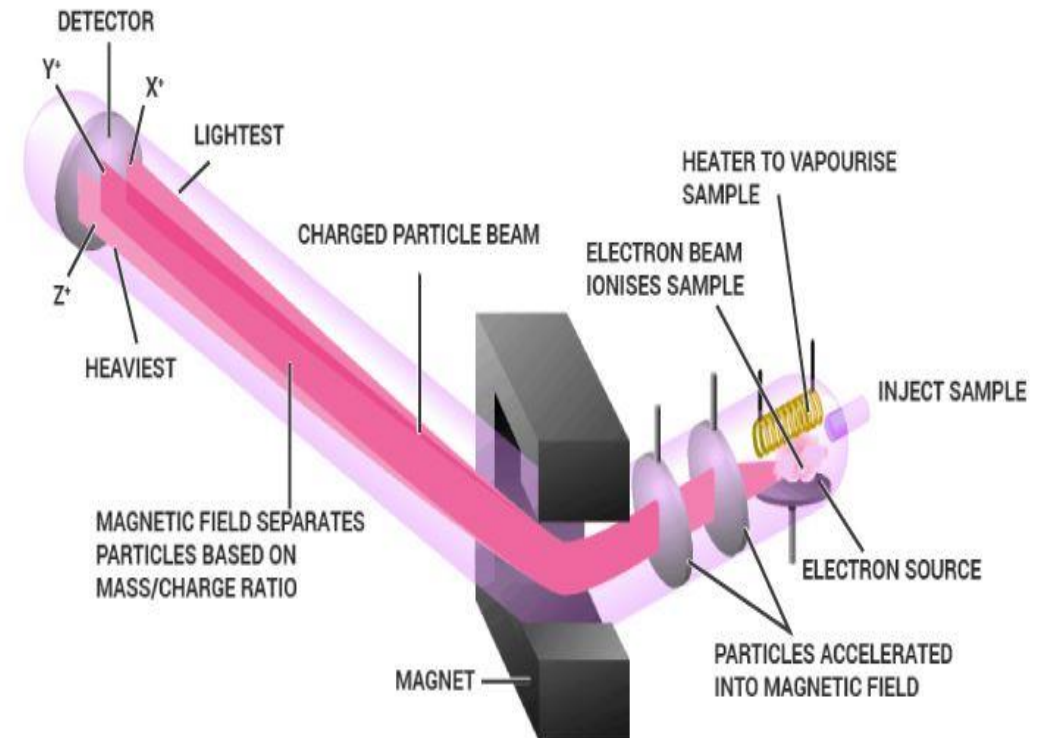
FOURIER-TRANSFORM MASS SPECTROMETER

TANDEM MASS SPECTROMETERS

TRIPLE QUADRUPOLE MASS SPECTROMETERS

INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETER

MASS SPECTROMETRY



PC, INTEGRATOR OR RECORDER



1. A Supervisory Control & Data Acquisition (SCADA) System Installed A PC, Integrator Or Recorder Should Be Connected To The Detector To Plot The Gas Chromatogram To Be Evaluated By The Analyst.
2. It Should Also Have The Ability To Back Up All The Necessary Data For Future Reference & Possibly Compliant With 21CFR Requirements.

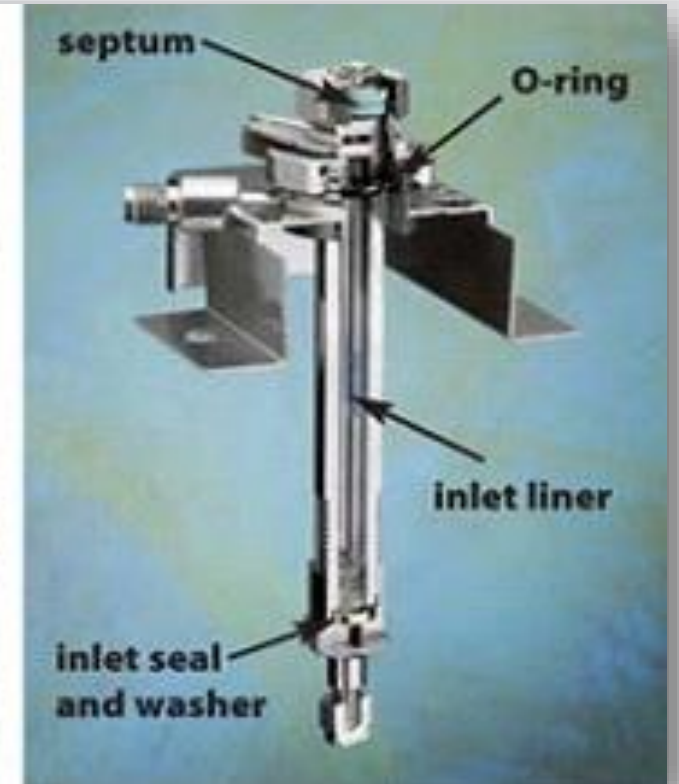


GC METHODOLOGY / PROCEDURE



SAMPLE INJECTION & VAPORIZATION

1. A Small Amount Of Liquid Sample To Be Analyzed Is Drawn Up Into A Syringe.
2. The Syringe Needle Is Positioned In The Hot Injection Port Of The Gas Chromatograph And The Sample Is Injected Quickly.

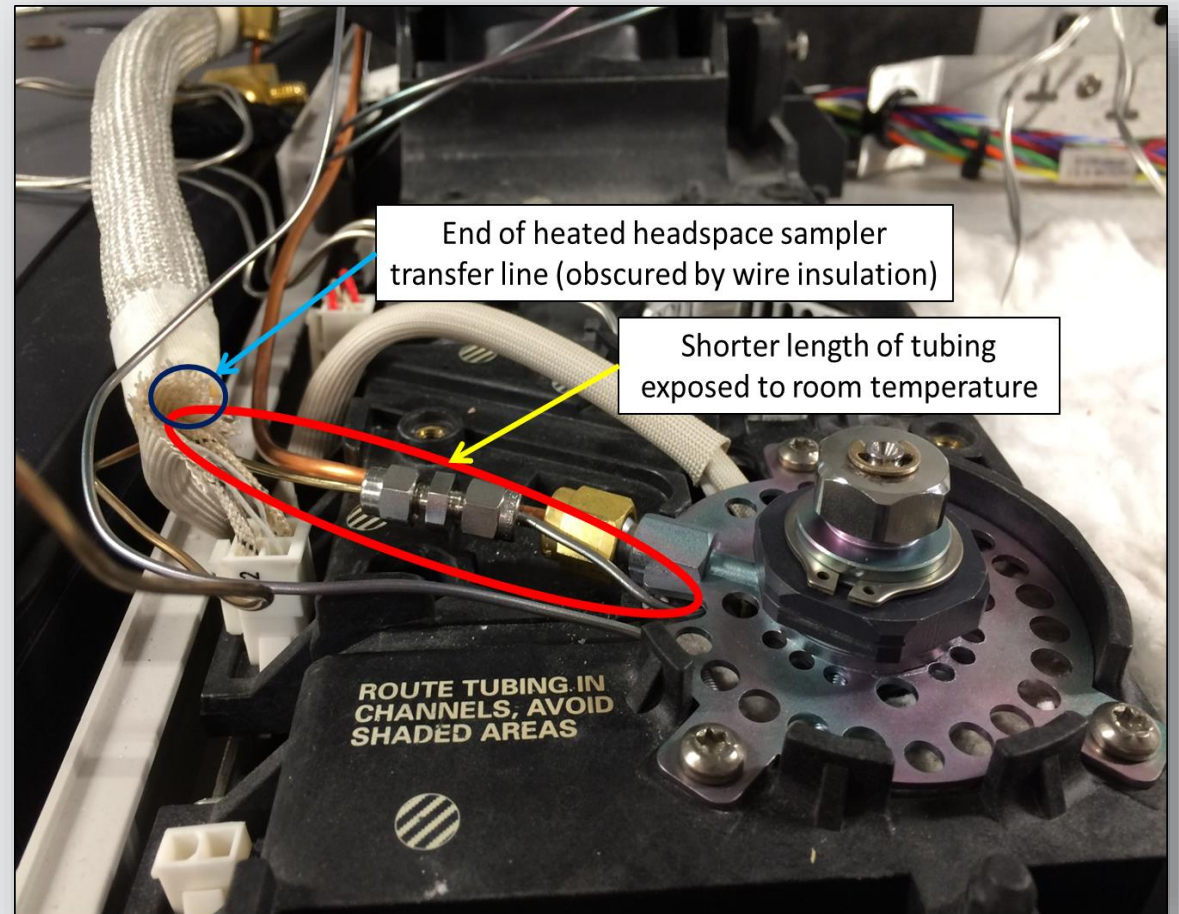


GC METHODOLOGY / PROCEDURE



SAMPLE INJECTION & VAPORIZATION

3. The Injection Of The Sample Is Considered To Be A “Point” In Time, That Is, It Is Assumed That The Entire Sample Enters The Gas Chromatograph At The Same Time, So The Sample Must Be Injected Quickly.



GC METHODOLOGY / PROCEDURE



SAMPLE INJECTION & VAPORIZATION

4. The Temperature Is Set To Be Higher Than The Boiling Points Of The Components Of The Mixture So That The Components Will Vaporize.
5. The Vaporized Components Then Mix With The Inert Gas Mobile Phase To Be Carried To The Gas Chromatography Column To Be Separated.

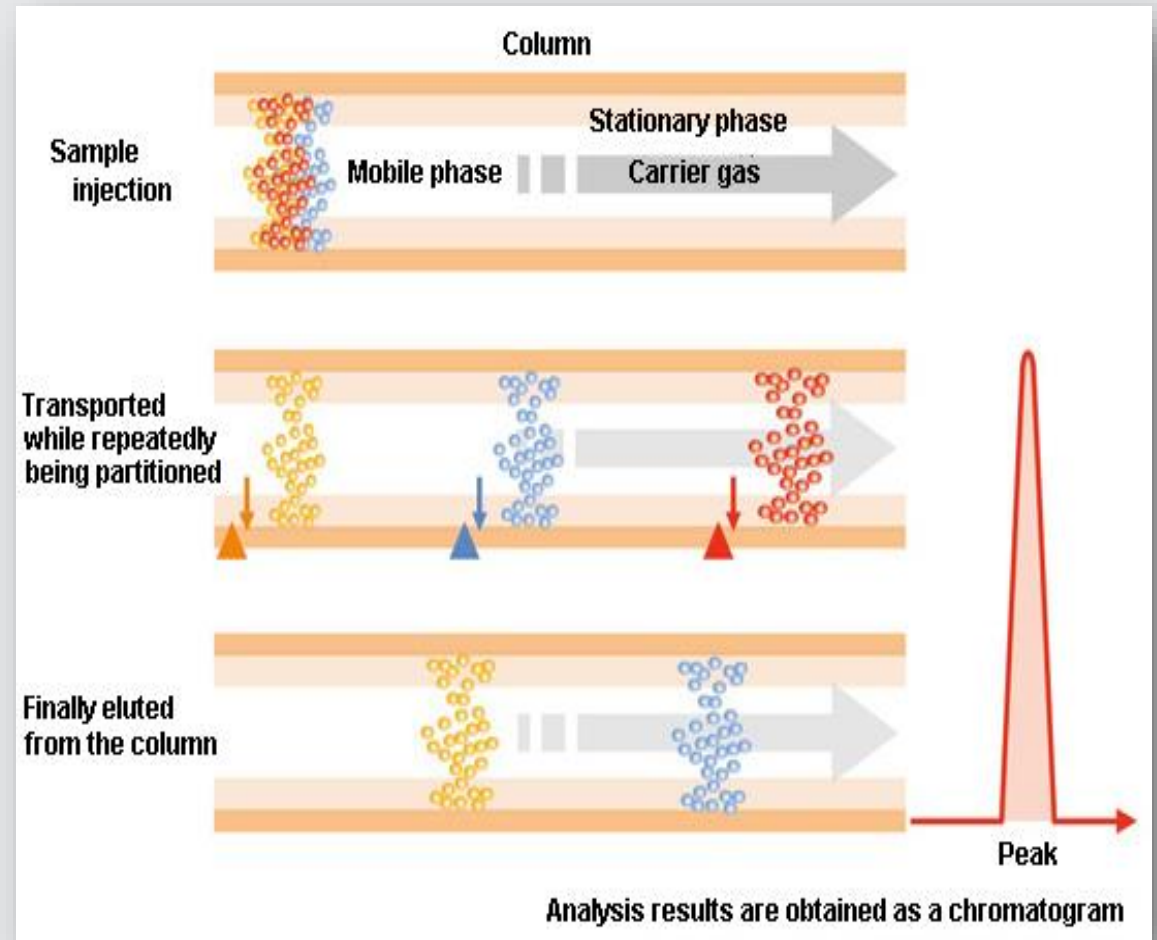


GC METHODOLOGY / PROCEDURE



SEPARATION IN THE COLUMN

1. Components In The Mixture Are Separated Based On Their Abilities To Adsorb On Or Bind To, The Stationary Phase.
2. A Component That Adsorbs Most Strongly To The Stationary Phase Will Spend The Most Time In The Column (Will Be Retained In The Column For The Longest Time) And Will, Therefore, Have The Longest Retention Time (R_t). It Will Emerge From The Gas Chromatograph Last.



GC METHODOLOGY / PROCEDURE



SEPARATION IN THE COLUMN

3. Components In The Mixture Are Separated Based On Their Abilities To Adsorb On Or Bind To, The Stationary Phase.
4. A Component That Adsorbs Most Strongly To The Stationary Phase Will Spend The Most Time In The Column (Will Be Retained In The Column For The Longest Time) And Will, Therefore, Have The Longest Retention Time (R_t). It Will Emerge From The Gas Chromatograph Last.

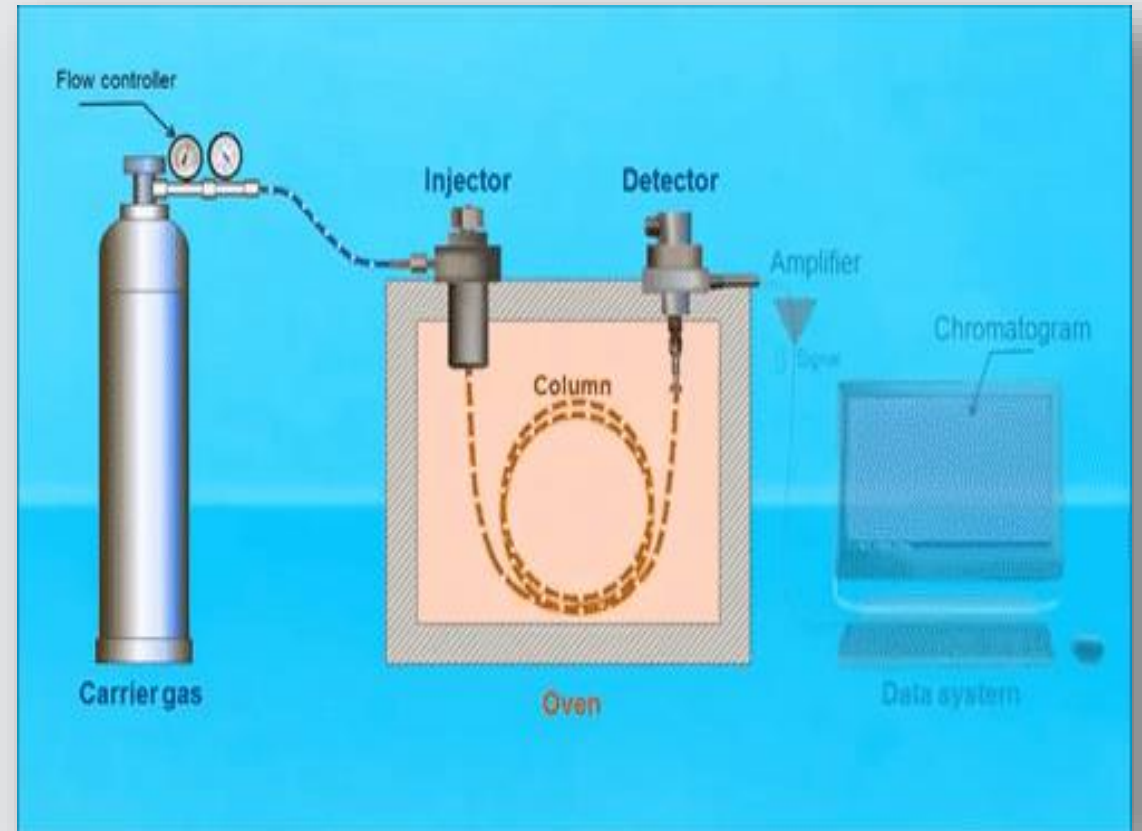


GC METHODOLOGY / PROCEDURE



SEPARATION IN THE COLUMN

5. A Component That Adsorbs The Least Strongly To The Stationary Phase Will Spend The Least Time In The Column (Will Be Retained In The Column For The Shortest Time) And Will, Therefore, Have The Shortest Retention Time (R_t). It Will Emerge From The Gas Chromatograph First.

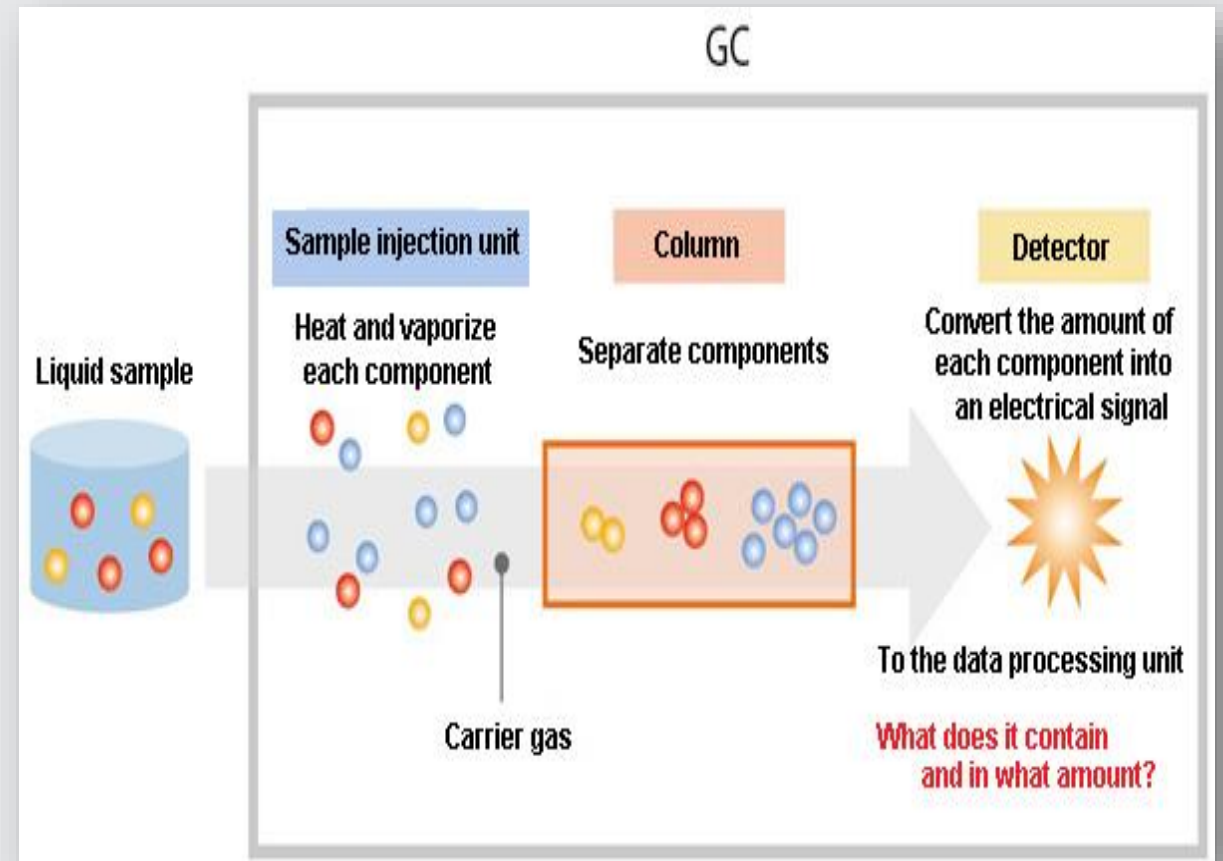


GC METHODOLOGY / PROCEDURE



SEPARATION IN THE COLUMN

6. If We Consider A 2 Component Mixture In Which Component 'A' Is More Polar Than Component 'B' Then:
7. Component 'A' Will Have A Longer Retention Time In A Polar Column Than Component 'B'.
8. Component 'A' Will Have A Shorter Retention Time In A Non-polar Column Than Component 'B'.

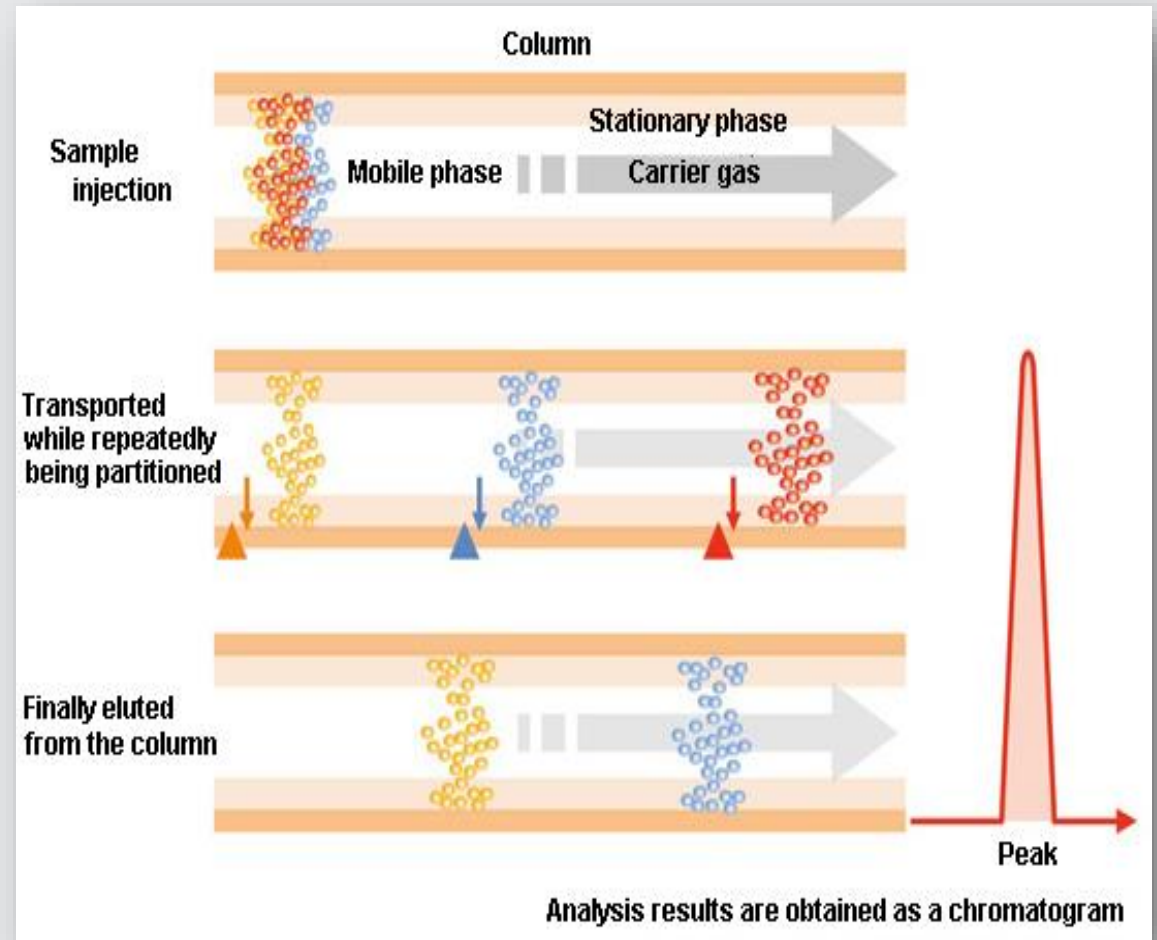


GC METHODOLOGY / PROCEDURE



DETECTING & RECORDING RESULTS

1. The Components Of The Mixture Reach The Detector At Different Times Due To Differences In The Time They Are Retained In The Column.
2. The Component That Is Retained The Shortest Time In The Column Is Detected First. The Component That Is Retained The Longest Time In The Column Is Detected Last.

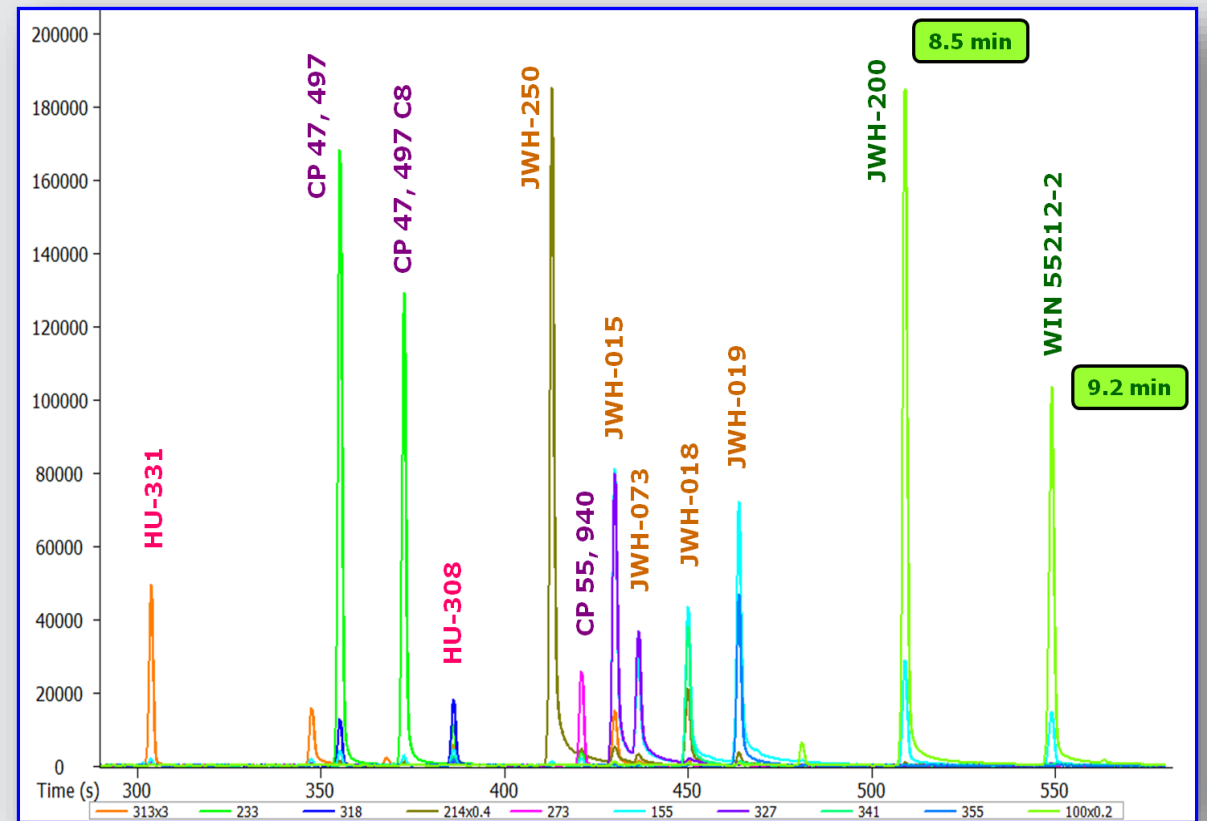


GC METHODOLOGY / PROCEDURE



DETECTING & RECORDING RESULTS

- The Detector Sends A Signal To The Chart Recorder Which Results In A Peak On The Chart Paper. The Component That Is Detected First Is Recorded First. The Component That Is Detected Last Is Recorded Last.



ADVANTAGES OF GC



1. The Use Of Longer Columns And Higher Velocity Of Carrier Gas Permits The Fast Separation In A Matter Of A Few Minutes.
2. Higher Working Temperatures Up To 500°C And The Possibility Of Converting Any Material Into A Volatile Component Make Gas Chromatography One Of The Most Versatile Techniques.



ADVANTAGES OF GC



3. GC Is Popular For Environmental Monitoring And Industrial Applications Because It Is Very Reliable And Can Be Run Nearly Continuously.
4. GC Is Typically Used In Applications Where Small, Volatile Molecules Are Detected And With Non-aqueous Solutions.
5. GC Is Favored For Non-polar Molecules.



LIMITATIONS OF GC



1. Compound To Be Analyzed Should Be Stable Under GC Operation Conditions..
2. They Should Have A Vapor Pressure Significantly Greater Than Zero.
3. Typically, The Compounds Analyzed Are Less Than 1,000 Da, Because It Is Difficult To Vaporize Larger Compounds.



LIMITATIONS OF GC



4. The Samples Are Also Required To Be Salt-free; They Should Not Contain Ions.
5. Very Minute Amounts Of A Substance Can Be Measured, But It Is Often Required That The Sample Must Be Measured In Comparison To A Sample Containing The Pure, Suspected Substance Known As A Reference Standard.



CONCLUSION

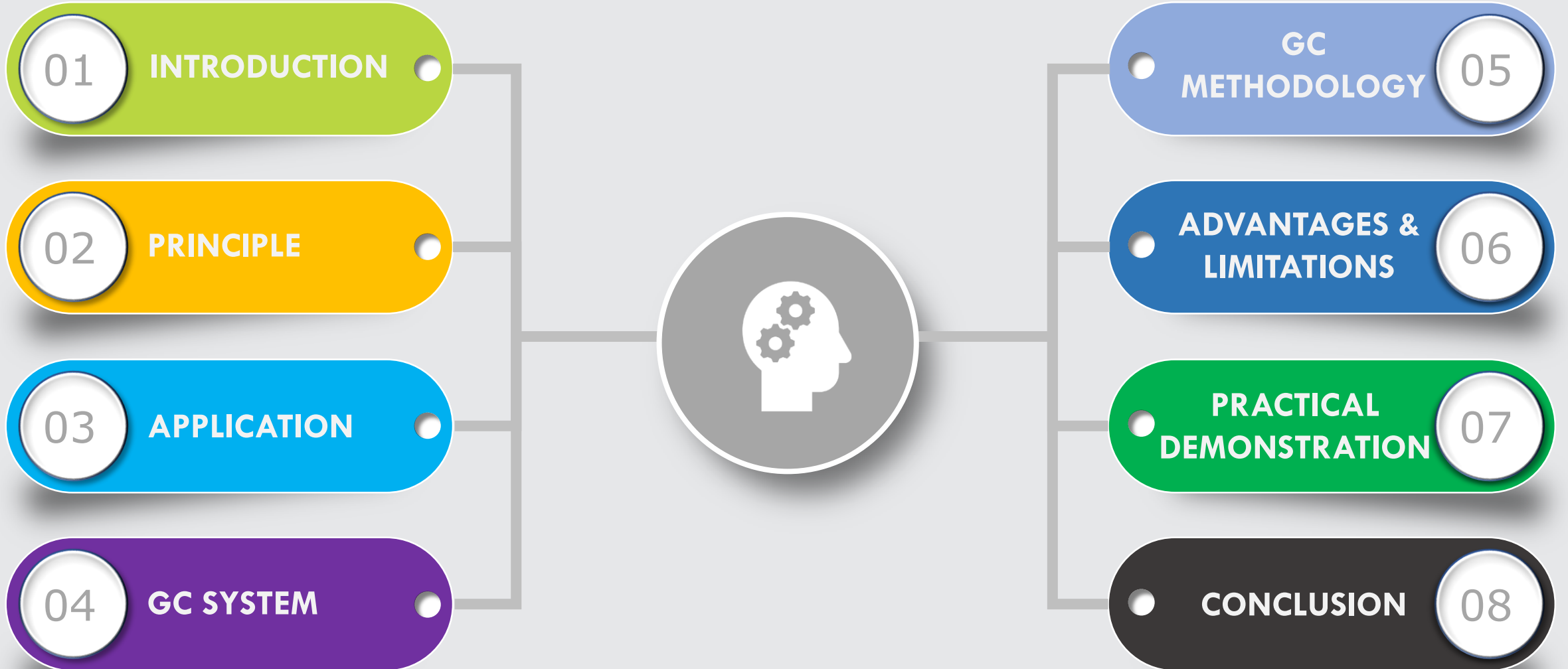


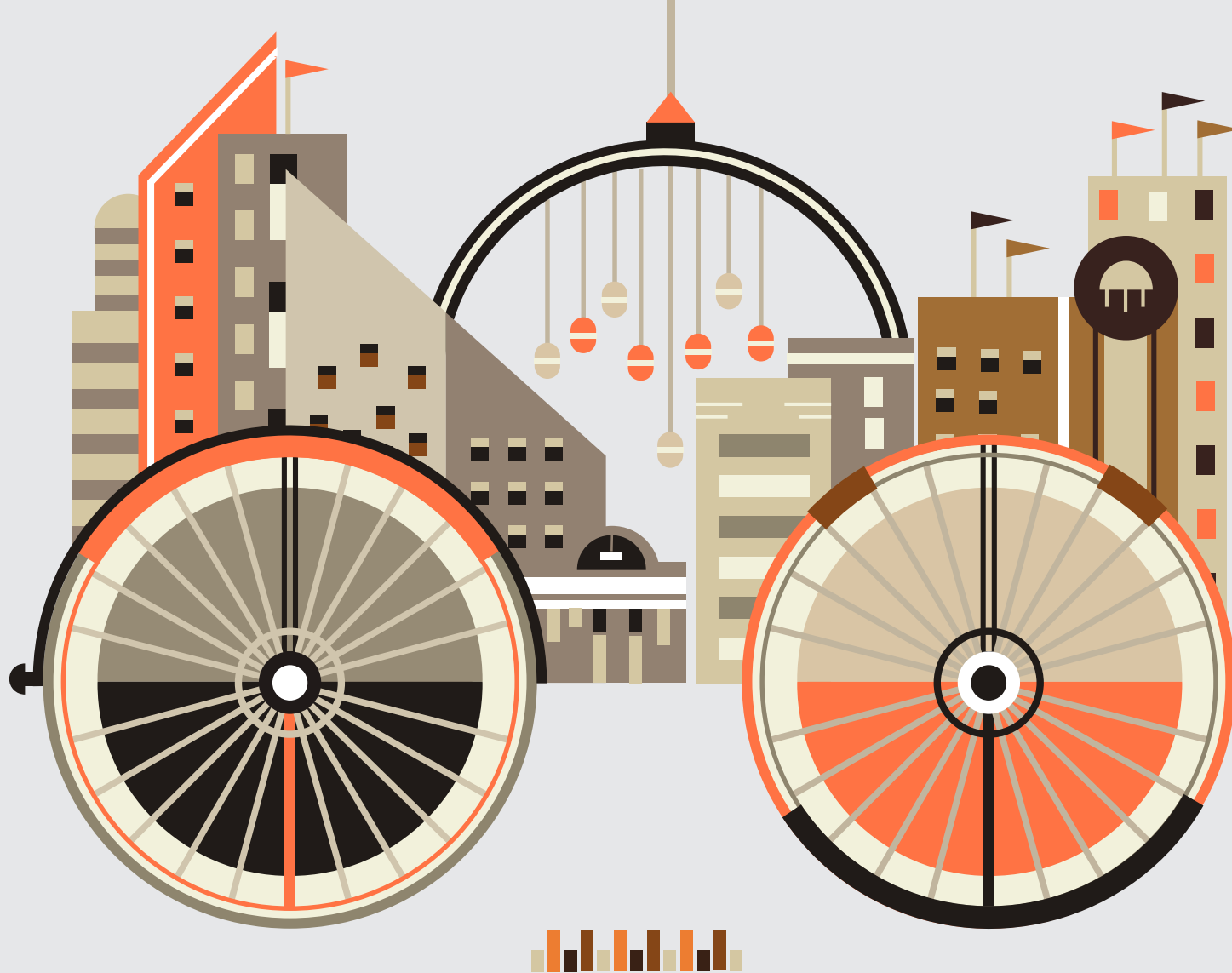
Gas Chromatography Is One Of The Most Important Tools In Chemistry Because Of Its Simplicity. Sensitivity And Effectiveness In Separating Components Of Mixtures. It Is Widely Used For Quantitative & Qualitative Analysis Of Mixtures & For The Purification Of Compounds.

END OF PART -2



SUMMARY





THANK YOU VERY MUCH