Chromatography (from Greek χρώμα:chroma, colour and γραφειν:“grafein” to write) is the collective term for a family of laboratory techniques for the separation of mixtures. It involves passing a mixture dissolved in a "mobile phase" through a stationary phase, which separates the analyte to be measured from other molecules in the mixture and allows it to be isolated.

Gas Chromatography (GC), is a type of chromatography in which the mobile phase is a carrier gas, usually an inert gas such as helium or an unreactive gas such as nitrogen, and the stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside glass or metal tubing, called a column.

The instrument used to perform gas chromatographic separations is called a gas chromatograph. The wikipedia
Gas Chromatography
Basics

Introduction

• Spectacular development since its beginnings in the fifties

• It has been used to solve many problems in industry, medicine, biology and environmental analysis.

• It is now used as a routine technique and control in a variety of areas

• Offers a better resolution for VOC

• Limitation: Thermal Lability. The compounds must be stable at the required temperature for volatilization

• The mobile phase is a gas: carrier gas

• The stationary phase could be
  Standard adsorbent solid
  a microscopic layer of liquid or polymer on an inert solid support, inside glass or metal tubing, called a column
Gas Chromatography Basics

Basic components

Gas Supply Unit
  Flow controller
  Flow Programmer

Sampling Unit
  Injector
    (Manual or Automatic)
  Injector oven

Injection Unit
  Injector and Injector Oven Controller

Column Unit
  Column
  Column oven

Detector Unit
  Detector
  Detector oven

Microprocessor for Flow Controller and Programme

Column Oven Controller and Programmer

Detector Electronics and Computer Data Acquisition and Processing System

Detector Oven Controller

Chromatogram

Some Definitions…..
Gas Chromatography

Chromatogram

**The injection point** is that point in time/position time when the sample is placed on the column.

**Position of the peak-maximum of an unretained solute**

**Base Line**

Any part of the chromatogram where only mobile phase is emerging from the column.

**Volume (V)_0**

**Volume of mobile phase passed through the column between the injection point and the dead point**

Thus, \( V_0 = F \cdot t_0 \) where \( F \) is the flow rate in mL/min.

**Corrected Retention Volume (V')_r**

**Volume of mobile phase passed through the column between the injection point and the peak maximum**

\( V_r = F \cdot t_r \)

where \( F \) is the flow rate in mL/min.

**Retention Time (t)_r**

**Time elapsed between the injection point and the peak maximum.**

Each solute has a characteristic retention time.

**Retention Volume (V)_r**

**Corrected Retention Volume (V')_r**

**Volume of mobile phase passed through the column between the dead point and the peak maximum**

or

retention volume minus the dead volume

\( V'_r = V_r - V_0 = F \cdot (t_r - t_0) \)

where \( F \) is the flow rate in mL/min.

**Time elapsed between the dead point and the peak maximum.**

**ELUTION Point of Unretained Solute**

**Peak Width at Base**

**Dead Point**

**Dead Time t_o**

**Volume of mobile phase passed through the column between the injection point and the dead point.**

Each solute has a characteristic retention time.
**Gas Chromatography**

**Basics**

**Carrier gas**

Mission: to transport the components mixture from the injector to the detector, going through the column where they are separated

It must be chemically inert to solutes and to stationary phase.

Selection and velocity influence efficiency and retention time.

Typical carrier gases include helium, nitrogen, argon, and hydrogen.

Which gas to use is usually determined by:

Sample type
Sample's Matrix
Detector to be used
Safety and Availability
Purity and Price

---

Mechanisms which contribute to band broadening: Van Deemter equation for plate height.

\[ \text{HETP} = A + \frac{B}{u} + C \cdot u \]

**HETP**: Height Equivalent to a Theoretical Plate

where \( u \) is the average velocity of the mobile phase. \( A \), \( B \), and \( C \) are factors which contribute to band broadening.

**A - Eddy diffusion**

The mobile phase moves through the column which is packed with stationary phase. Solute molecules will take different paths through the stationary phase at random. This will cause broadening of the solute band, because different paths are of different lengths.

**B - Longitudinal diffusion**

The concentration of analyte is less at the edges of the band than at the centre. Analyte diffuses out from the center to the edges. This causes band broadening. If the velocity of the mobile phase is high then the analyte spends less time on the column, which decreases the effects of longitudinal diffusion.

**C - Resistance to mass transfer**

The analyte takes a certain amount of time to equilibrate between the stationary and mobile phase. If the velocity of the mobile phase is high, and the analyte has a strong affinity for the stationary phase, then the analyte in the mobile phase will move ahead of the analyte in the stationary phase. The band of analyte is broadened. The higher the velocity of mobile phase, the worse the broadening becomes.
The plate model supposes that the chromatographic column is contains a large number of separate layers, called theoretical plates. Separate equilibrations of the sample between the stationary and mobile phase occur in these "plates". The analyte moves down the column by transfer of equilibrated mobile phase from one plate to the next.

Plates serve as a way of measuring column efficiency, either by stating the number of theoretical plates in a column, \( N \) (the more plates the better), or by stating the plate height; the Height Equivalent to a Theoretical Plate (the smaller the better).

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### Carrier gas

<table>
<thead>
<tr>
<th>Carrier gas</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrogen</td>
<td>Cheap</td>
<td>Long run times</td>
</tr>
<tr>
<td></td>
<td>Safe</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Easy purification</td>
<td></td>
</tr>
<tr>
<td>Hydrogen</td>
<td>Cheap</td>
<td>Can react with several compounds: For instance double bond addition. Explosive (although not under GC conditions)</td>
</tr>
<tr>
<td></td>
<td>Low pressure drop in the column</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Shorter run time</td>
<td></td>
</tr>
<tr>
<td>Helium</td>
<td>Safe</td>
<td>Expensive</td>
</tr>
<tr>
<td></td>
<td>Good compromise between Nitrogen and hydrogen</td>
<td></td>
</tr>
<tr>
<td>Argon</td>
<td>Cheap</td>
<td>Type of detector used</td>
</tr>
<tr>
<td></td>
<td>Safe</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Easy purification</td>
<td></td>
</tr>
</tbody>
</table>
Gas Chromatography
Basics
Carrier gas

With GCs made before the 1990s, carrier flow rate was controlled indirectly by controlling the carrier inlet pressure, or “column head pressure.”

The actual flow rate was measured at the outlet of the column or the detector with an electronic flow meter, or a bubble flow meter, and could be an involved, time-consuming, and frustrating process.

The pressure setting was not able to be varied during the run, and thus the flow was essentially constant during the analysis.

Many modern GCs, however, electronically measure the flow rate, and electronically control the carrier gas pressure to set the flow rate. Consequently, carrier pressures and flow rates can be adjusted during the run, creating pressure/flow programs similar to temperature programs.

Gas Chromatography
Basics
Sample Injection systems

The GC can be used for all kind of samples- solid, liquid or gas if they are stable at the working temperature

The amount of sample introduced should not cause an overload or saturation in the column or the detector

The maximum amount of sample depend mainly on the type of column

The injection technique depend on:

- Physical state
- Concentration range
- Chromatographic conditions: column, detector, flow rate

For liquid and solid samples the limiting factor is the necessary time for the sample vaporization
Gas Chromatography
Basics

Sample Injection systems

**INJECTOR TEMPERATURE** High enough to volatilize the sample without decomposition

*Generic rule:* $T_{\text{inj}} = 50^\circ\text{C}$ above the Boiling Point of the less volatile compound

**Injection Volume** will depend of the column type and the physical state of the sample

<table>
<thead>
<tr>
<th>COLUMN</th>
<th>Liquid samples</th>
<th>Gas samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Packed $\varnothing = 3.2\ \text{mm (1/4”)}$</td>
<td>$0.2\ \mu\text{L} \ldots 20\ \mu\text{L}$</td>
<td>$0.1\ \text{ml} \ldots 50\ \text{ml}$</td>
</tr>
<tr>
<td>capillar $\varnothing = 0.25\ \text{mm}$</td>
<td>$0.01\ \mu\text{L} \ldots 3\ \mu\text{L}$</td>
<td>$0.001\ \text{ml} \ldots 0.1\ \text{ml}$</td>
</tr>
</tbody>
</table>

Solids: In General they are solved in a solvent.

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Gas Chromatography
Basics

Sample Injection systems

- GC column efficiency requires that the sample be of suitable size (to prevent column overloading) and be introduced as a plug of vapour.
- Two common approaches include for introduction of $0.01 \ldots 50\ \mu\text{l}$ include: Microsyringe and valve loop.
- The syringe technique is most common and can be used with both gas and low viscosity liquid samples by inserting the needle through a rubber septum to the column inlet port.
- The region into which the needle projects must be heated in order to flash vaporise the sample.
- However, overheating of the rubber septum must be avoided to prevent out gassing.
- The most popular inlet for capillary GC is the split/splitless injector.
- If this injector is operated in split mode, the amount of sample reaching the column is reduced (to prevent column overloading) and very narrow initial peak widths can be obtained.
- For maximum sensitivity, the injector can be used in so-called splitless mode, then all of the injected sample will reach the column.
- Injection may be manual or automated.
**Sample Injection systems**

Sample Valve Injection

Sample valves are convenient for on-line gas stream analysis.

In position A the stream to be sampled flows through a loop of calibrated volume while the carrier gas alone passes through the column.

In position B the loop is placed in the carrier gas stream and the entrapped sample is swept along to the column.

Sample valves are becoming more prevalent for quantitative work employing both liquids and gases to introduce a reproducible volume of sample onto a column.
**Gas Chromatography**

**Basics**

**Sample Injection systems**

- **liquid samples**

1. Injector nut
2. Septum
3. Septum purge
4. Carrier gas inlet
5. Split vent
6. Glass insert
7. Column

**Split-splitless**

1. Septum
2. Carrier gas inlet
3. Carrier gas inlet
4. Split vent
5. Column

**On-column**

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**Headspace Analysis**

- Head space analysis is a technique where the vapours in the gas above, and in equilibrium with, a solid or liquid is sampled.

- The advantage of this approach is that GC can be used instead of HPLC, thus providing four to five orders of magnitude greater sensitivity.

- Procedure involves the extraction of a volume of the equilibrium gas over the sample (usually about 10 ml) by a syringe through either a vial containing a bed of an appropriate absorbent or a cryogenic trap.

- The vial/trap is then placed in line with a GC column, heated and the vaporised sample swept onto the column and the components separated.

- Used to identify spoiled food, fragrances from botanical material, the determination of plasticizers in plastics, solvents in paints and varnishes and for forensic samples involving arson.
**Gas Chromatography**

**Basics**

**Sample Injection techniques**

- 5030C can be used for most volatile organic compounds that boiling points below 200 °C and are insoluble or slightly soluble in water.
- Multiple sample aliquots are collected in sealed containers with minimum headspace and stored at 4 °C or less in solvent free area.
- An inert gas is bubbled through aqueous sample and room or elevated temperature depending on the target analytes.
- The vapour is swept through a sorbent column where the analytes are captured.
- After purging, the sorbent column is heated (thermal extraction) and back-flushed with inert gas to desorb the components onto a GC column.

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**Gas Chromatography**

**Basics**

**Solid Phase Microextraction (SPME)**

- Solid phase microextraction (SPME) is suitable for sampling environmental contaminants with a wide range of physical properties in air, water and soil.
- A fused silica fibre with a polymer coating is exposed to the sample or the headspace above the sample.
- Organic analytes adsorb to the coating on the fibre. After adsorption equilibrium is attained, usually in 2 to 30 minutes, the fibre is withdrawn.
- The fibre is introduced into a GC injector, where the adsorbed analytes are thermally desorbed and delivered to the GC column.
- The amount of analyte adsorbed by the fibre depends on the thickness of the polymer coating and on the distribution constant for the analyte.
- Fibres with a range of different polarities are now commercially available.
Gas Chromatography

Basics

Direct Thermal Extraction

- Permits the direct thermal extraction of volatile and semi-volatile organics directly from small sample sizes (mg) without the need for solvent extraction or other sample preparation requirements.
- The sample is maybe trapped on sorbent resins or placed inside a preconditioned glass-lined stainless steel desorption tube.
- The desorption tube containing the sample is then connected to a short path thermal desorption system.
- The desorption tube is ballistically heated and carrier gas carries the analytes through the injection port and onto the GC column for analysis.

Comparison of Techniques

<table>
<thead>
<tr>
<th>Sample Matrix</th>
<th>Sample Size</th>
<th>Sensitivity</th>
<th>Range of Volatiles Analyzed</th>
<th>Sample Automated</th>
<th>Sample Prep Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Headspace L/S</td>
<td>0.1-10 ppm</td>
<td></td>
<td></td>
<td>Yes</td>
<td>5:10</td>
</tr>
<tr>
<td>P &amp; T D. G/L/3</td>
<td>5-1000 ppb</td>
<td></td>
<td></td>
<td>No</td>
<td>10:30</td>
</tr>
<tr>
<td>SPME G/L</td>
<td>0.1-10 ppt</td>
<td></td>
<td></td>
<td>Yes</td>
<td>5:15</td>
</tr>
<tr>
<td>DTE S</td>
<td>0.1-1.0 g</td>
<td></td>
<td></td>
<td>No</td>
<td>1:2</td>
</tr>
</tbody>
</table>

Gas Chromatography

Basics

Comparison of Techniques
Gas Chromatography Basics

Column

**Packed**
- \( \varnothing \) = from 3 to 6 mm
- \( L \) = from 0.5 m to 5 m
- Particle size = 50-150 µm
- Film thickness = -
- Carrier gas \( V \) = from 2 to 6 cm/s
- Amount of each solute = from 100 to 1000 µg
- Mostly used for gas phase analysis

**Capillary**
- \( \varnothing \) = from 0.1 to 0.5 mm
- \( L \) = from 5 m to 100 m
- Particle size = -
- Film thickness = 0.1 – 0.5 µm
- Carrier gas \( V \) = from 10 to 30 cm/s
- Amount of each solute = from 0.001 to 0.1 µg
- Sharper peaks

Gas Chromatography Basics

Column

Types

**Siloxane polymers**: Methyl, phenyl, cyanopropyl, trifluoropropyl

**Poly(ethylene) glycols**

Columns developed for particular applications
Gas Chromatography
Basics

Column

Selectivity: solute interactions and separation
Polarity: Physical characteristic of the stationary phase:

POLARITY

<table>
<thead>
<tr>
<th>POLARITY</th>
<th>SOLUBILITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>PHASE = SOLUTE</td>
<td>HIGH</td>
</tr>
<tr>
<td>PHASE ≠ SOLUTE</td>
<td>LOW</td>
</tr>
</tbody>
</table>

Non-polar columns
100% Methyl polysiloxane (1)
5% Phenyl methylpolysiloxane (5)

Intermediate polarity columns
35% Phenyl methylpolysiloxane (35)
50% Phenyl methylpolysiloxane (17)
6% cyanopropylphenyl methylpolysiloxane (1301)
14% cyanopropylphenyl methylpolysiloxane (1701)

Polar columns
50% cyanopropylphenyl methylpolysiloxane (225)
14% cyanopropyl methylpolysiloxane (23)
50% trifluoropropyl methylpolysiloxane (210)
Poly(ethylene) glycol (WAX)

High solubility = High retention and capacity

Gas Chromatography
Basics

Column

SELECTIVITY

<table>
<thead>
<tr>
<th>PHASE</th>
<th>DISPERSION</th>
<th>DIPOLE</th>
<th>H BONDING</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methyl</td>
<td>Strong</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>Phenyl</td>
<td>Strong</td>
<td>None</td>
<td>Weak</td>
</tr>
<tr>
<td>Cyanopropyl</td>
<td>Strong</td>
<td>Strong</td>
<td>Moderate</td>
</tr>
<tr>
<td>Trifluoropropyl</td>
<td>Strong</td>
<td>Moderate</td>
<td>Weak</td>
</tr>
<tr>
<td>PEG</td>
<td>Strong</td>
<td>Strong</td>
<td>Moderate</td>
</tr>
</tbody>
</table>

Separation by differences in solute heat vaporization. Vapour Pressure
Separation by differences in solute dipole moments. Smaller differences require a stronger dipole phase
Separation by differences in solute hydrogen bonding character.
Strong: alcohols, carboxylic acids, amines
Moderate: aldehydes, esters, ketones
Weak: hydrocarbons, halocarbons, ethers
Smaller differences require a stronger hydrogen bonding phase
Gas Chromatography

Basics

Column

Stationary Phase selection

Existing information
Selectivity
Polarity
Critical separations
Temperature limits
Capacity
Analysis Time
Bleed
Versatility
Selective detectors

<table>
<thead>
<tr>
<th>Column dimensions (Capillary columns)</th>
<th>Comments</th>
<th>Most Common</th>
<th>Available</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>Smaller diameters for low flow situations (e.g., GC/MS) Larger diameters for high flow situations (e.g., purge &amp; trap, headspace)</td>
<td>0.25 mm 0.32 mm 0.45 mm 0.53 mm</td>
<td>0.10-0.53 mm</td>
</tr>
<tr>
<td>Length</td>
<td>Larger length better resolution, but also higher retention times and more expensive</td>
<td>15 m 30 m 60 m</td>
<td>5-150 m</td>
</tr>
<tr>
<td>Film Thickness</td>
<td>Larger thickness for higher capacities, but also more bleed</td>
<td>0.1-3.0 µm</td>
<td>0.1-10.0 µm</td>
</tr>
</tbody>
</table>
Gas Chromatography Basics

**Column**
What are the best dimensions?

<table>
<thead>
<tr>
<th>To increase</th>
<th>Make diameter</th>
<th>Make Length</th>
<th>Make Film</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution</td>
<td>Smaller</td>
<td>Longer</td>
<td>depend</td>
</tr>
<tr>
<td>Retention</td>
<td>Smaller</td>
<td>Longer</td>
<td>Thicker</td>
</tr>
<tr>
<td>Pressure</td>
<td>Smaller</td>
<td>Longer</td>
<td>-</td>
</tr>
<tr>
<td>Flow rate</td>
<td>Larger</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Capacity</td>
<td>Larger</td>
<td>-</td>
<td>Thicker</td>
</tr>
<tr>
<td>Cost</td>
<td>-</td>
<td>Longer</td>
<td>-</td>
</tr>
<tr>
<td>Bleed</td>
<td>-</td>
<td>-</td>
<td>Thicker</td>
</tr>
</tbody>
</table>

Gas Chromatography Basics

**Temperature Control**

**Injector:** High enough to volatalize the sample without decompostion

**Column:** Resolution and retention time

**Detector:** It depends on the type of detector used
Gas Chromatography Basics

Temperature Control

The rate at which a sample passes through the column is directly proportional to the temperature of the column.

The higher the column temperature, the faster the sample moves through the column.

However, the faster a sample moves through the column, the less it interacts with the stationary phase, and the less the analytes are separated.

- Column Temperature

Isothermal. Holds the column at the same temperature for the entire analysis.

Temperature Programmed: allows analytes that elute early in the analysis to separate adequately, while shortening the time it takes for late-eluting analytes to pass through the column.
Gas Chromatography Basics

Detectors

The GC detector is designed to respond to very small quantities of vapor contained in a permanent gas.

The detector is selected based on its sensitivity for the compound/s of interest and its selectivity to minimize response to the sample matrix.

Two functions:

• To indicate the exact point at which a analyte passes, and thus to define the retention time for the qualitative analysis.

• To cause a signal proportional to the amount of solute that passes through him, with quantitative purposes.
Gas Chromatography
Basics
Detectors

Characteristics of Ideal GC Detector

- Good stability and reproducibility.
- Linear response to analytes that extends over several orders of magnitude.
- Similarity in response toward all analytes.
- Temperature range from room temperature to 400 °C.
- A short response time that is independent of flow rate.
- Non-destructive.
- High reliability and ease of use.

No one detector exhibits all of these characteristics.

Gas Chromatography
Basics
Detectors

Different classifications

- **Bulk property detectors** and **solute property detectors**.
  The bulk property detector measures some bulk physical property of the eluent (such as dielectric constant or refractive index) and the solvent property detector, measures some physical or chemical property that is unique to the solute (such as heat of combustion or fluorescence).

- **Destructive or non-destructive**

  - **Concentration sensitive devices** such as TCD or **mass sensitive devices** such as the flame ionization detector (FID).

  \[
  A = \int_{t_1}^{t_2} \frac{v}{v_i} \, dt = \int \frac{v}{v_i} \, \frac{dm}{dt} \, dt = \int \frac{m}{v_i} \, \frac{dm}{dt} \, dt = k \cdot m
  \]

  \[
  A = \int v_i \, dt = \int \frac{dm}{dt} \, dt = k \cdot m
  \]

- **Specific or non-specific.** An example of a specific detector would be the nitrogen phosphorous detector (NPD), which as its name implies detects only those substances that contain nitrogen or phosphorous. In general (though not always), non specific detectors have lower sensitivities than the specific detectors.
**Gas Chromatography**

**Basics**

**Detectors**

**Thermal Conductivity Detector (TCD)**

- Bulk property detector. Differential
- Non-destructive
- Non-selective. Universal
- Sensitivity: $10^{-6}$ g/mL (low)
- Linear dynamic Range: $10^4$
- Carrier gas: He or H₂

A filament carrying a current is situated in a tubular cavity through which flows the column eluent. When there is only carrier gas flow, the heat lost from the filament is constant, the parameters will change in the presence of a different gas or solute vapor and as a result the temperature of the filament changes, causing a change in potential across the filament (decrease of the thermal conductivity). This potential change is amplified and either fed to a suitable recorder or passed to an appropriate data acquisition system. Extremely flow and pressure sensitive.

**Gas Chromatography**

**Basics**

**Detectors**

**Electron Capture Detector (ECD)**

- Absolute
- Non-destructive
- Selective. Halogens, organometallic, nitriles, nitro
- Concentration sensitivity
- Sensitivity: $10^{-8}$ g/mL
- Linear dynamic Range: 500-10000
- Carrier gas: N₂ or 5% Methane and 95% Ar

The ECD uses a radioactive Beta-particles (electrons) emitter -- a typical source contains a metal foil holding 10 millicuries of $^{63}$Nickel. The electrons formed are attracted to a positively charged anode generating a steady current. As the sample is carried into the detector by a stream of carrier gas, analyte molecules capture the electrons (for example a molecule containing a halogen atom which has only seven electrons in its outer shell) and reduce the current between the collector anode and a cathode.
Gas Chromatography

Basics

Detectors

Flame Ionization Detector (FID)

Absolute
Destructive
Non-Selective. Universal.
Insensitive to: H₂O, N₂, O₂, CO, CO₂, NO₂,
NH₃, SO₂, CS₂, Noble gases
Sensitivity: 1 pg/s
Linear dynamic Range: 10⁶
Carrier gas: N₂, He, H₂
Fuel gas H₂,
Oxidant gas: O₂, or Air

A FID consists of a hydrogen/air flame and a collector plate. A potential of a few hundred volts is applied across the burner tip and a collector electrode located above the flame. The resulting current is amplified and proportional to the number of carbon atoms in the flame. The effluent from the GC column passes through the flame, which breaks down organic molecules and produces ions. The ions are collected on a biased electrode and produce an electrical signal.

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### Gas Chromatography

**Basics**

**Detectors**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>TCD</th>
<th>ECD</th>
<th>FID</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement</td>
<td>Diferencial</td>
<td>Absolute</td>
<td>Absolute</td>
</tr>
<tr>
<td>Treatment</td>
<td>Non destructive</td>
<td>Non destructive</td>
<td>Destructive</td>
</tr>
<tr>
<td>Response Type</td>
<td>Universal</td>
<td>Selective</td>
<td>Universal</td>
</tr>
<tr>
<td>Response Sensitivity</td>
<td>Concentration</td>
<td>Concentration</td>
<td>mass</td>
</tr>
<tr>
<td>Temperature limit</td>
<td>400</td>
<td>350</td>
<td>400</td>
</tr>
<tr>
<td>Carrier gas</td>
<td>He, H₂</td>
<td>N₂, Ar + 5% Methane</td>
<td>N₂, He, H₂</td>
</tr>
<tr>
<td>Minimum detectable amount (g/s)</td>
<td>10⁻⁸</td>
<td>10⁻¹²</td>
<td>5 x 10⁻¹²</td>
</tr>
<tr>
<td>Linearity</td>
<td>10⁴</td>
<td>500-10⁴</td>
<td>10⁶</td>
</tr>
</tbody>
</table>
Gas Chromatography

Basics

Detectors

Nitrogen Phosphorus Detector (NPD)

Emission of an electrical charge from a solid surface that is heated.

Ions are generated from a hydrogen and air plasma in the presence of heat and an alkaline catalyst embedded in a ceramics source.

They are collected at the anode and provides background current through the electrode system. When a solute that contains nitrogen or phosphorus is eluted, the partially combusted nitrogen and phosphorus materials are adsorbed on the surface of the bead.

Selective to CN and P

Selective and Sensitive

Destructive

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Photoionization Detector (PID)

Uses an UV light source to break molecules to positively charged ion that can easily be counted with a detector.

Ionization occurs when a molecule absorbs high energy UV light, which excites the molecule, and results in temporary loss of a negatively charged electron and the formation of positively charged ion. The gas becomes electrically charged.

In the PID, the charged particles produce a current that is amplified and recorded.

The ions recombine after passing the detector to reform their original molecules.

They will only detect components which have ionization energies similar to the energy of the photons the detector uses. Common lamps: 9 eV, 10eV, 11.6 eV. SELECTIVE

Absolute

Non-destructive

Sensitivity: 0.01 pg/s

Linear dynamic Range: $10^6$
Gas Chromatography
Basics

Detectors

Other Detectors

HALL: Electrolytic Conductivity Detector.
Analysis of compounds containing halogen, sulfur or nitrogen

FPD: Flame photometric detector.
Analysis of compounds containing sulfur and phosphorus

Hyphenated Techniques: combination of GC with other analytical techniques:
• MS
• FTIR
• AE
• AA
• NMR

etc