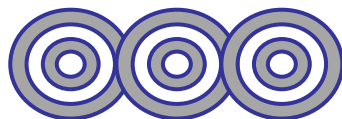




# Instrumental Methods of Analysis



## Gas Chromatography Fundamentals & Applications

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Advanced Materials  
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# Gas Chromatography: An Overview

Gas chromatography (**GC**) is a common type of chromatography in which the mobile phase is **gas** used in analytical chemistry for separating and analyzing compounds that can be vaporized without decomposition.

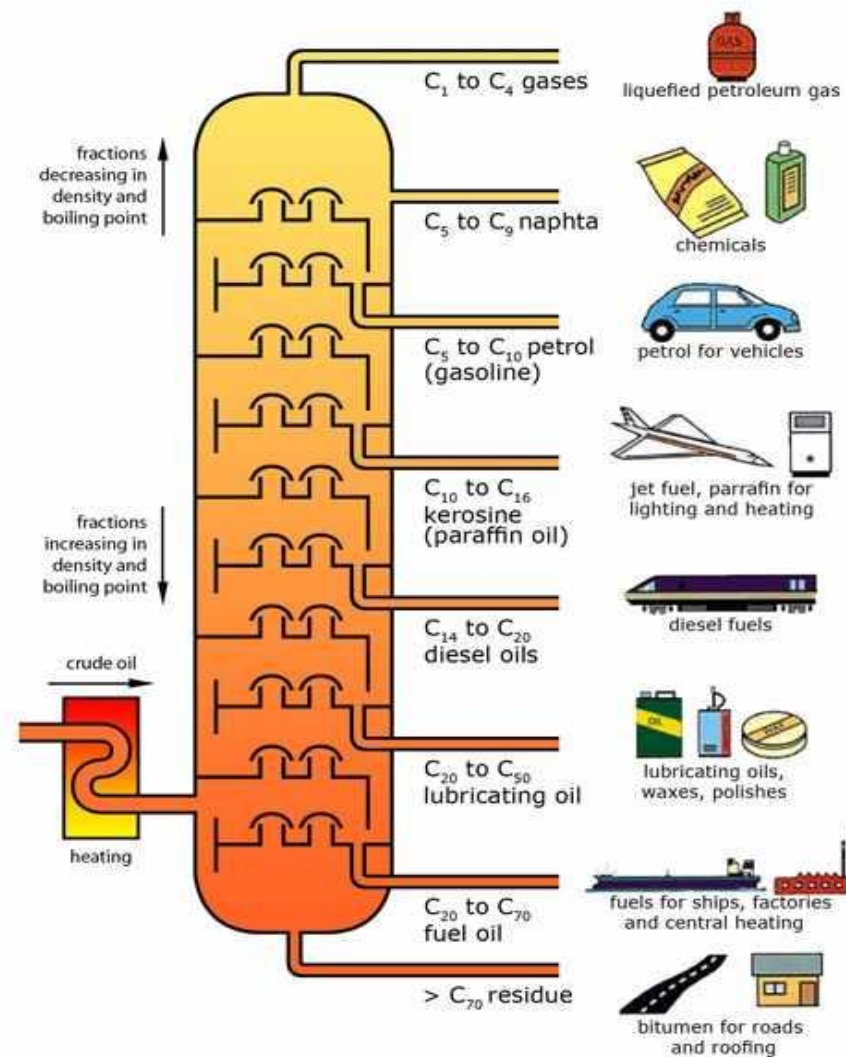
## Separation by

- Partition** between gaseous mobile phase and liquid stationary phase supported by inert packing (**GLC**).
- Adsorption** between gaseous mobile phase and solid stationary phase (**GSC**).



## Petroleum Refinery

Separation depends on **temperature**.  
Based on a wide range of **boiling points** and **polarity**.



## Fractional Distillation

# Gas Chromatography Analysis

In GC, the sample is **vaporized** and **injected** onto the head of a chromatographic column.

Elution is brought about by the flow of an **inert gaseous** mobile phase, the mobile phase does not interact with molecules of the analyte (i.e., carrier gas); its only function is to transport the analyte through the column.

After separation, the compounds are respectively detecting using suitable detectors.

**Vaporization**



**Injection**



**Elution**



**Separation**



**Detection**

GC used for **volatile** and **non degradable** compounds by temperature.

Occasionally, it is also possible to **derivatize** (chemically modify) non-volatile and heat degradable target chemicals prior to analysis.

**Derivatization** in GC is the process of chemically modifying a compound to produce a new compound which has properties that are suitable for analysis using a GC. Bulky and nonpolar groups are often used for this purposes.

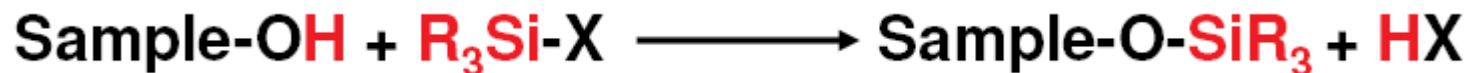
### **What does derivatization accomplish?**

- Increases volatility.
- Increases stability.
- Increases detectability.
- Enhances sensitivity for detection.

# Main types of derivatization

## Silylation

-Replaces active hydrogens (from alcohol) with e.g. TMS (trimethylsilyl group). Readily volatilizes the sample. Most prevalent method.



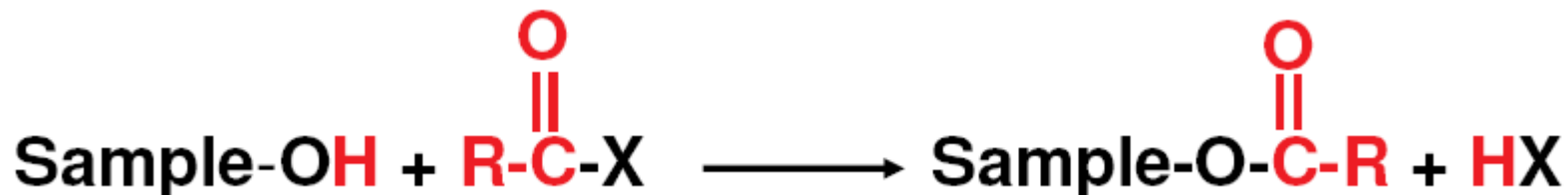
## Alkylation

-Reduces molecular polarity by replacing active hydrogens with an alkyl group.

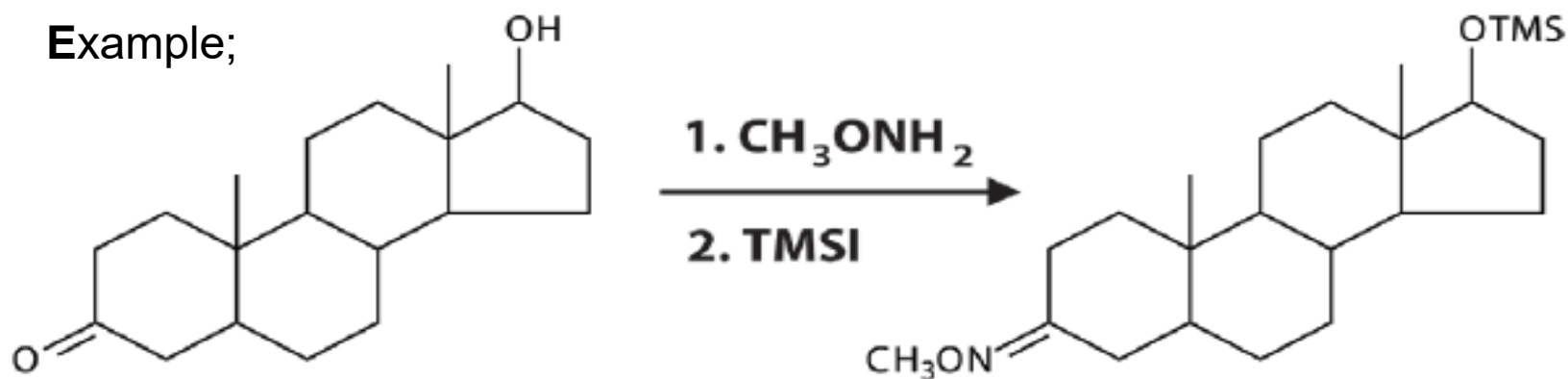
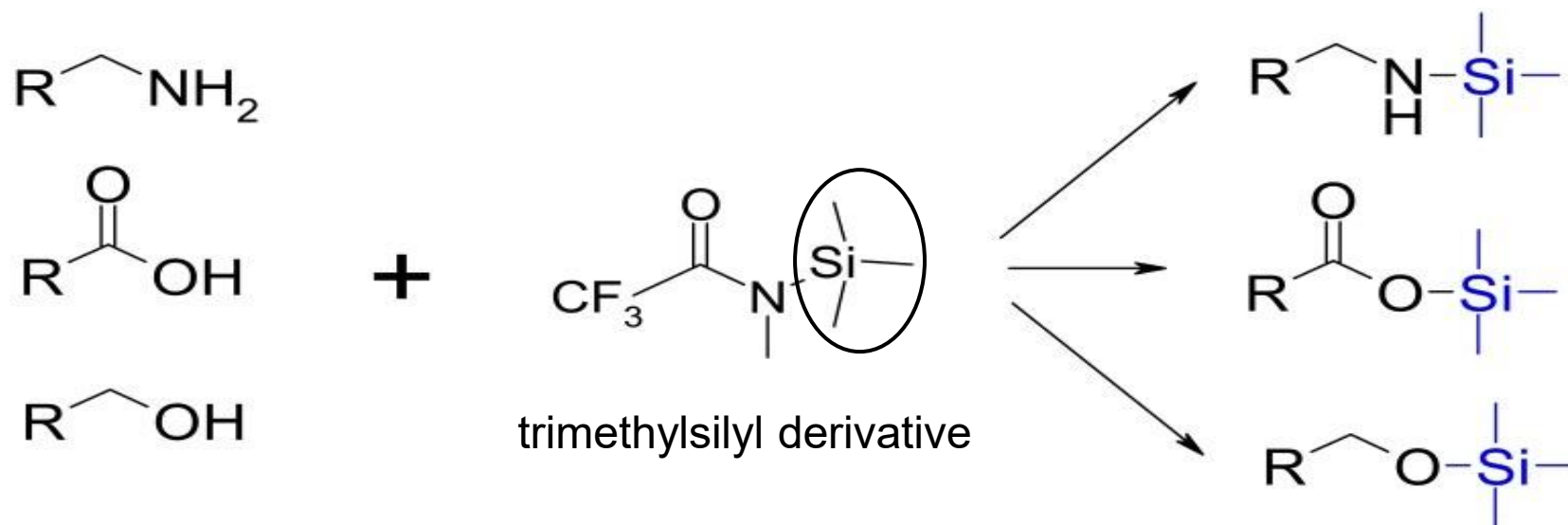


## Acylation

-Reduces the polarity of amino, hydroxyl and thiol groups and adds halogenated functionalities to enhance the sensitivity for GC detectors such as electron capture detector (ECD).



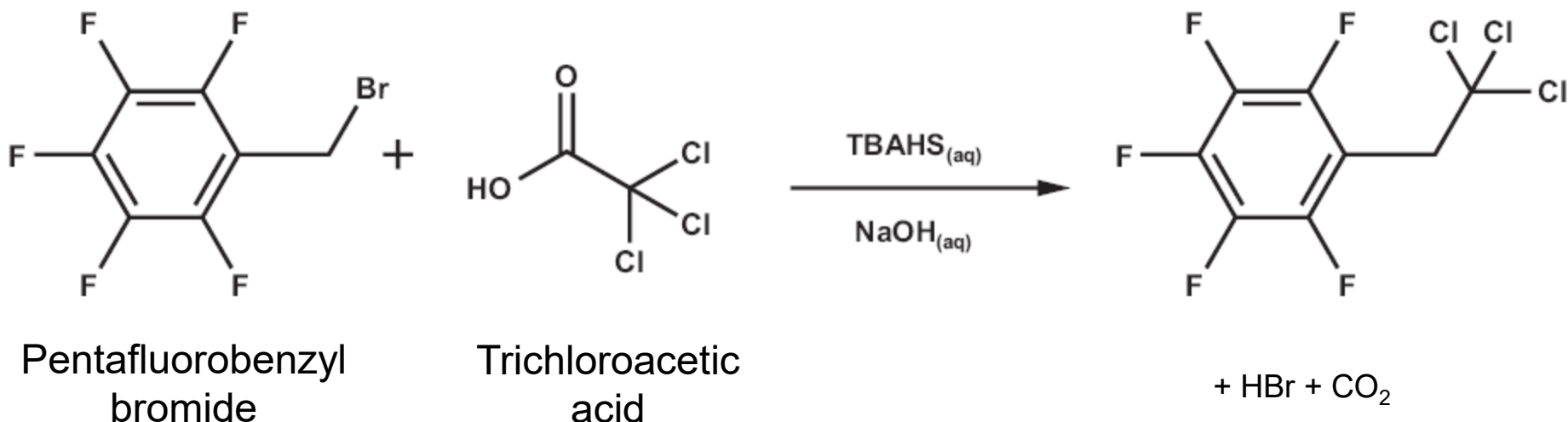
## General silylation reaction



Derivatization reaction of androsterone using TMSI/methoxyamine.

## Alkylation of Chlorinated Acetic Acids

Chloroacetic acid, Dichloroacetic acid and Trichloroacetic acid are difficult to analyze by GC due to highly polar acidic groups and therefore must be derivatized to increase their detectability.



This reaction demonstrates derivatization of the chlorinated acetic acids using the alkylation reagent pentafluorobenzyl bromide to form their corresponding fluorinated derivatives.



# GC Physical Components

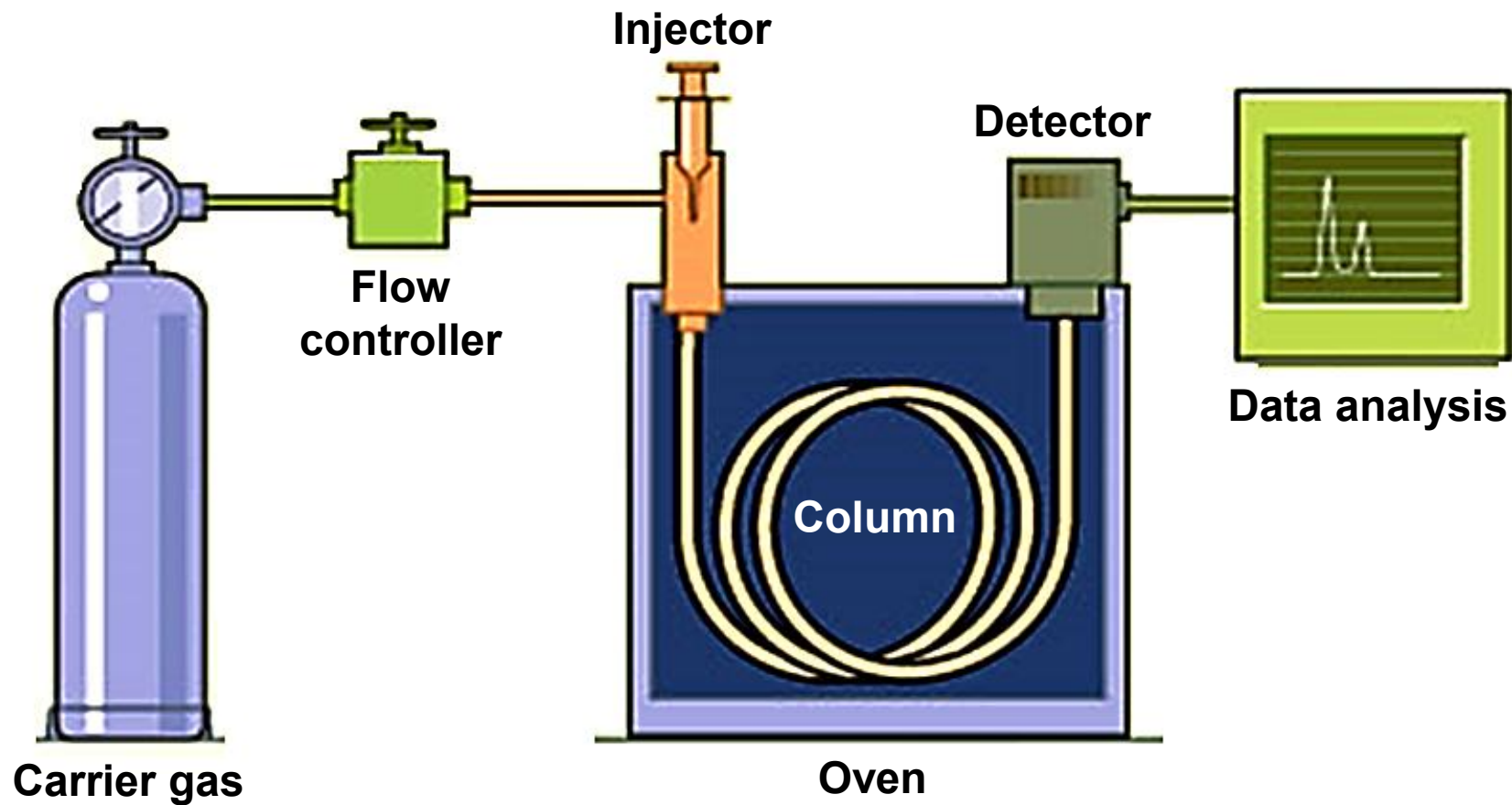
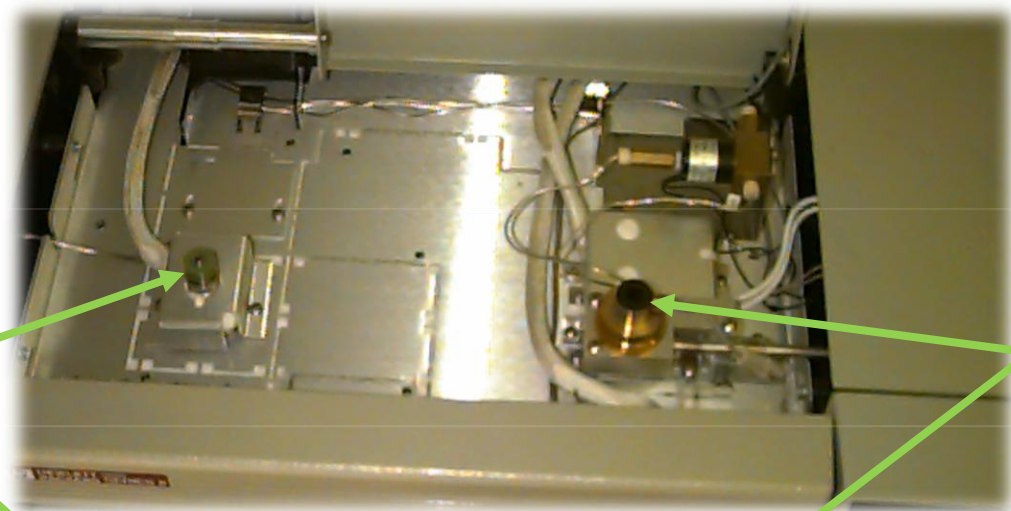


Diagram of a GC

## Top & front views of gas chromatograph

**Top view**

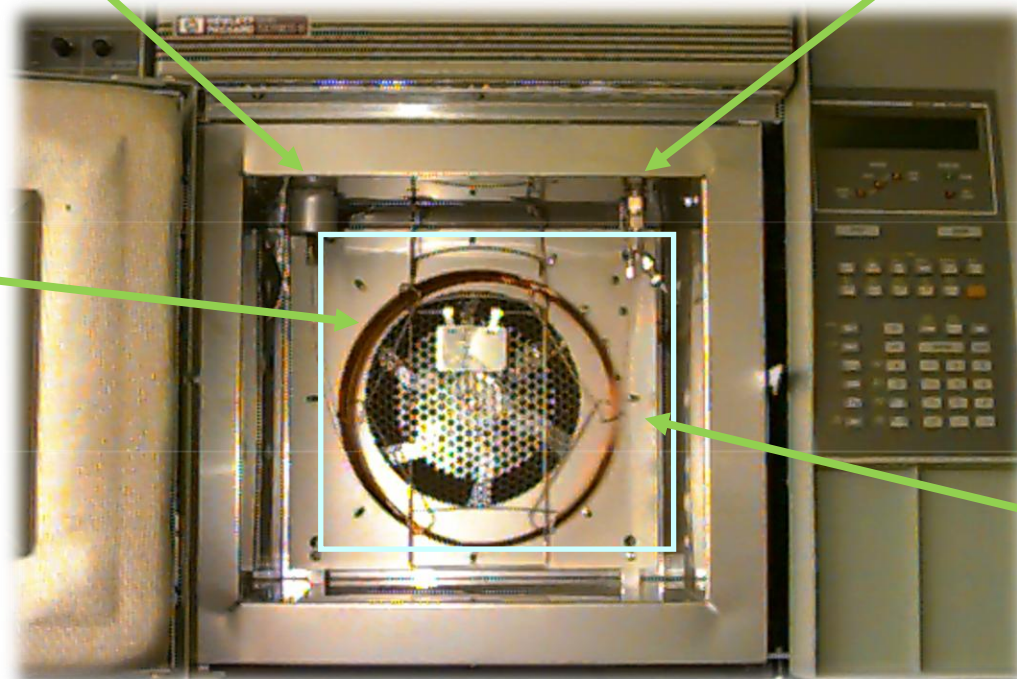


**flame  
ionization  
detector**

**injection port**



**column**

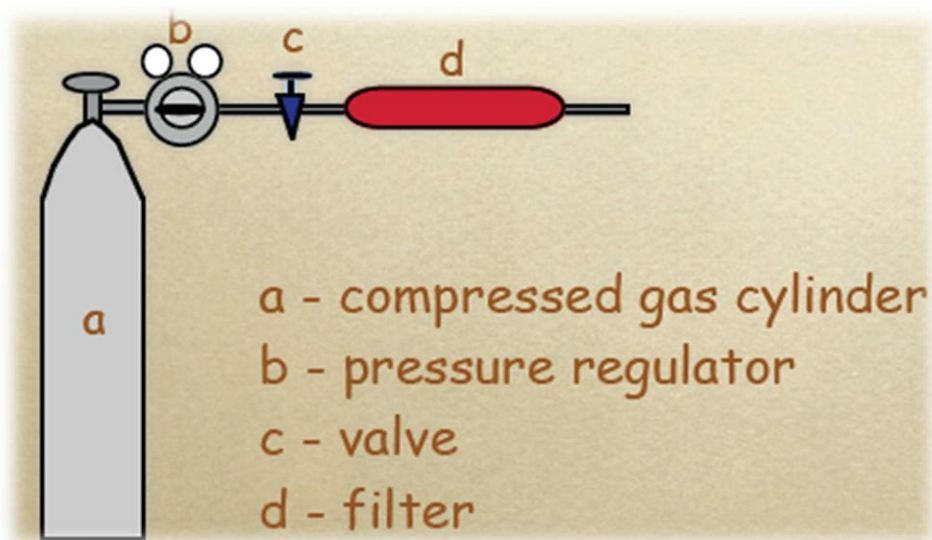


**oven**

**Front view**

# Carrier Gas

- Chemically inert gases ( $\text{He}$ ,  $\text{H}_2$ ,  $\text{N}_2$  &  $\text{CO}_2$ )
- High purity, 99.9995% pure or better
- Free from water and oxygen
- Detector compatibility
- Economic / safety reasons
- Efficiency / speed
- Gas filter used to eliminate impurities such as: water, oxygen and hydrocarbons



**H<sub>2</sub>**: efficient, cheap and rapid but not safe.

**N<sub>2</sub>**: cheap and safe but less efficient and not inert.

**He**: efficient, rapid, inert and safe but relatively expensive.

# Carrier gas supply

Pressure regulators:

- Reduce pressure of gas
- Control the flow rate

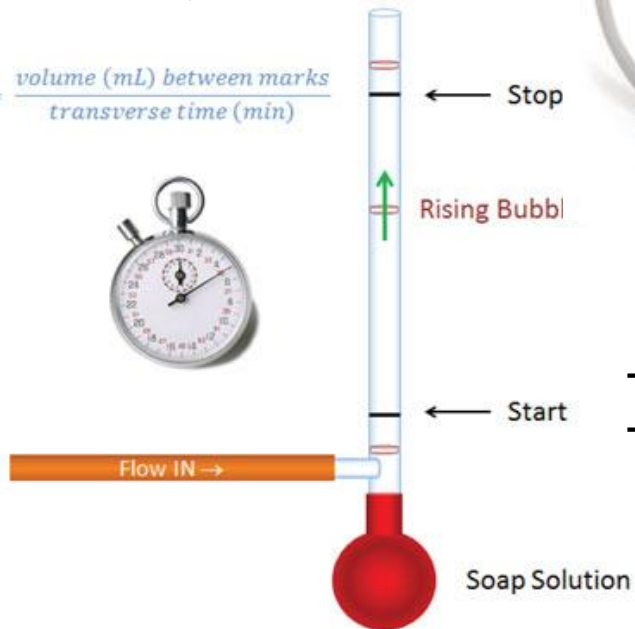


**Double stage pressure regulator**



# Flow meters

$$\text{Flow rate} = \frac{\text{volume (mL) between marks}}{\text{transverse time (min)}}$$



soap bubble flow meter



Electronic pressure control  
or electronic flow meter

- Constant flow
- Constant pressure



rotameter

# Injector

## Role of injectors

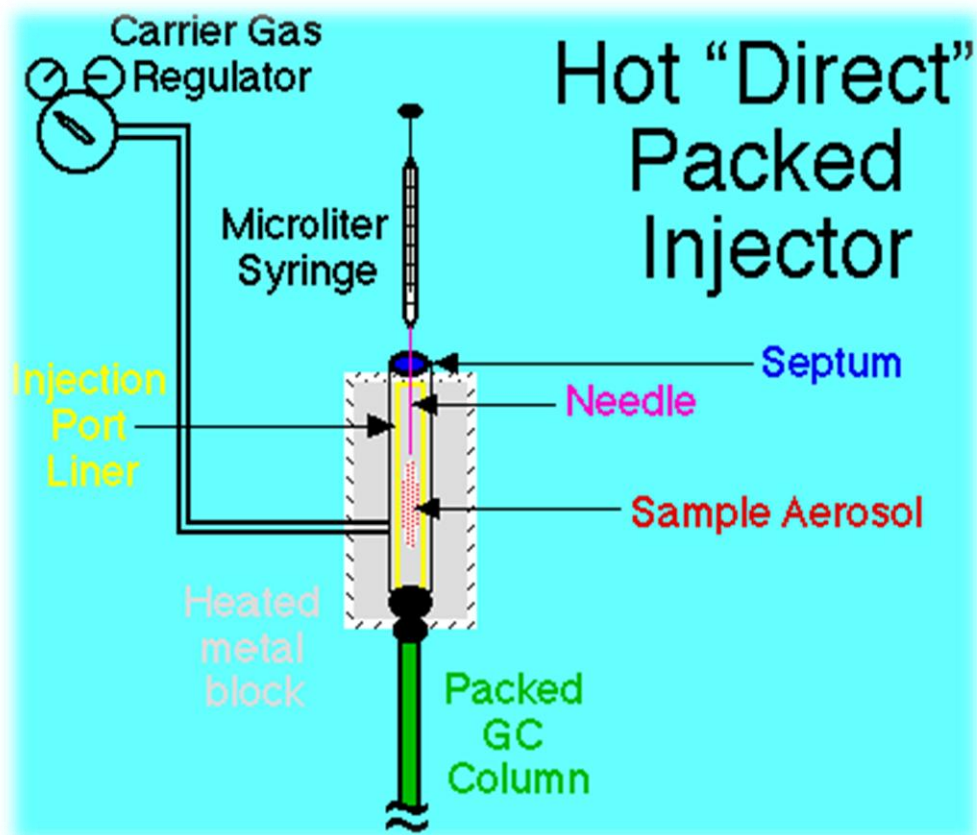
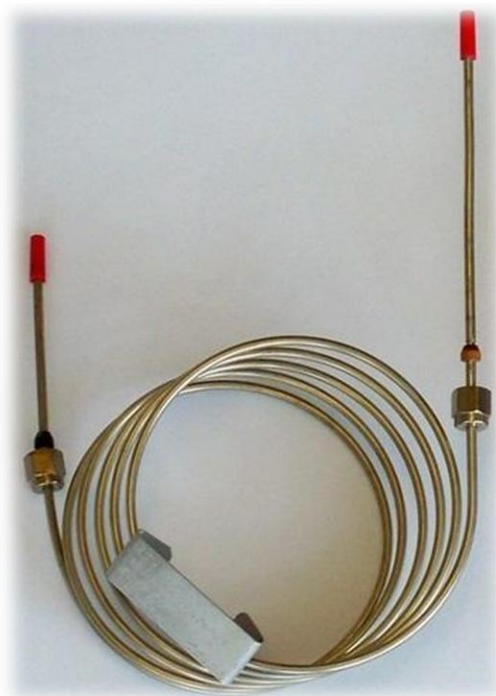
- Works as an inlet for the sample.
- It vaporizes and mix the sample with the carrier gas before the sample enters the head of the column.

The injection volume has a great effect on the quality of the separation.

The type of column used in the analysis sets the mode of injection.

# Direct vaporization injector

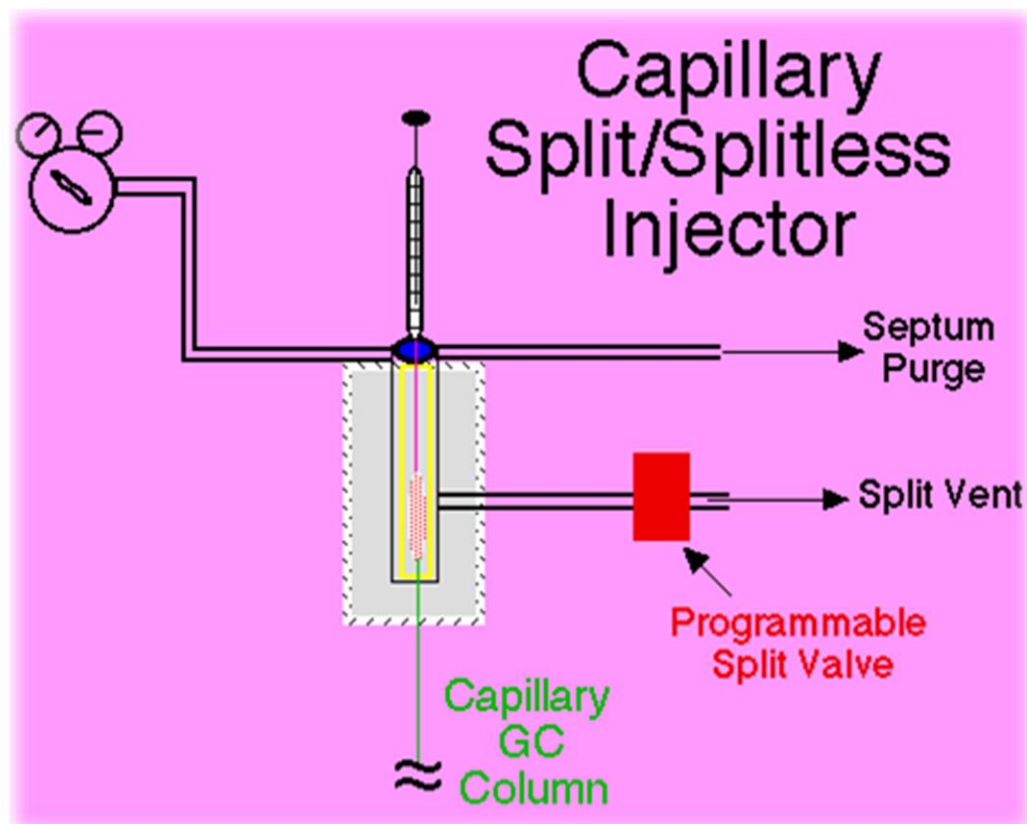
For packed columns



# Split / Splitless injector

## For capillary columns

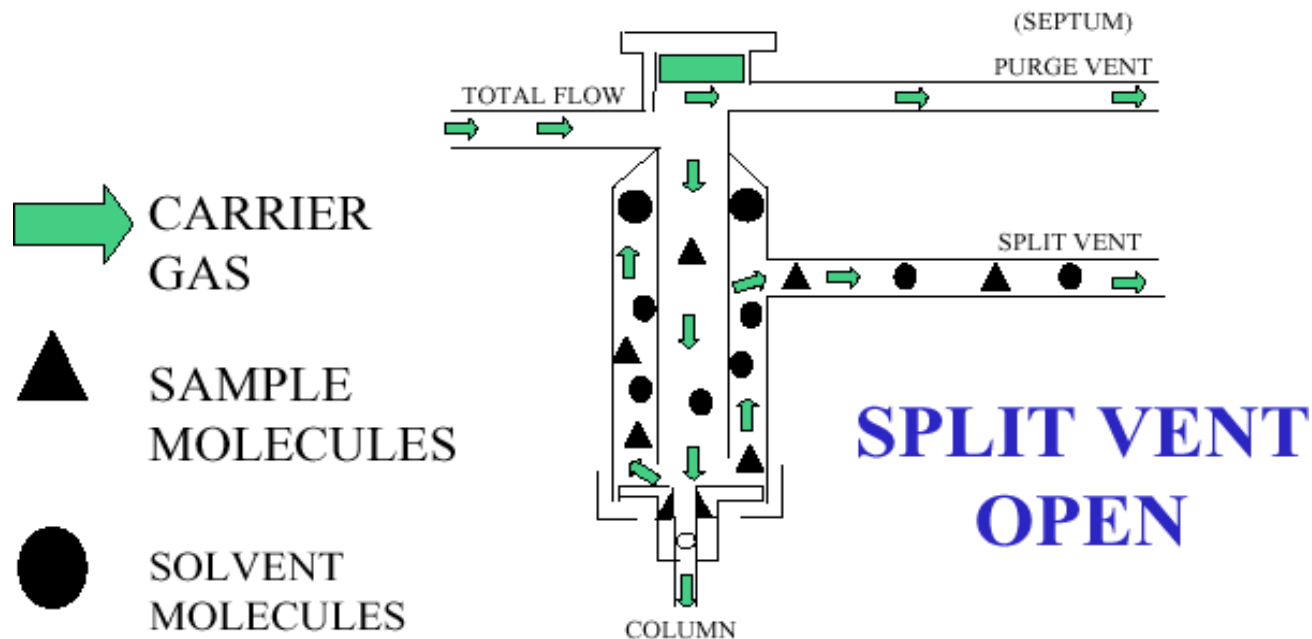
-Operate in two modes, with or without flow splitting.





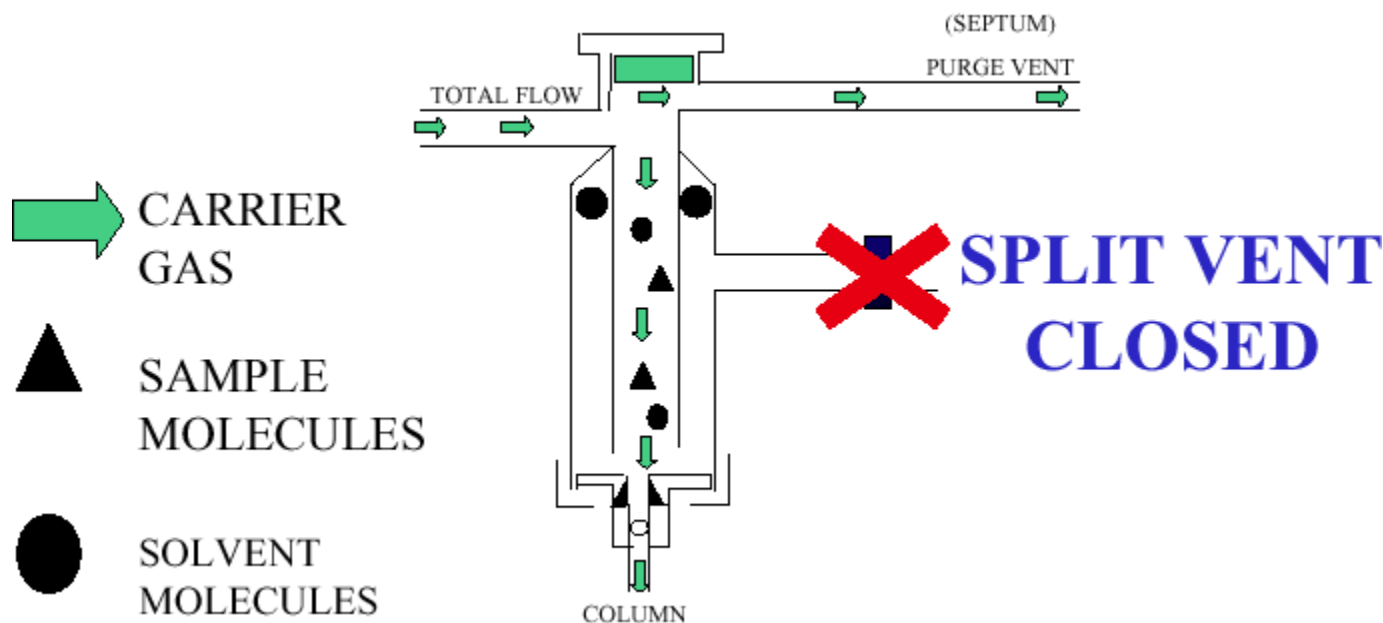
# Split mode

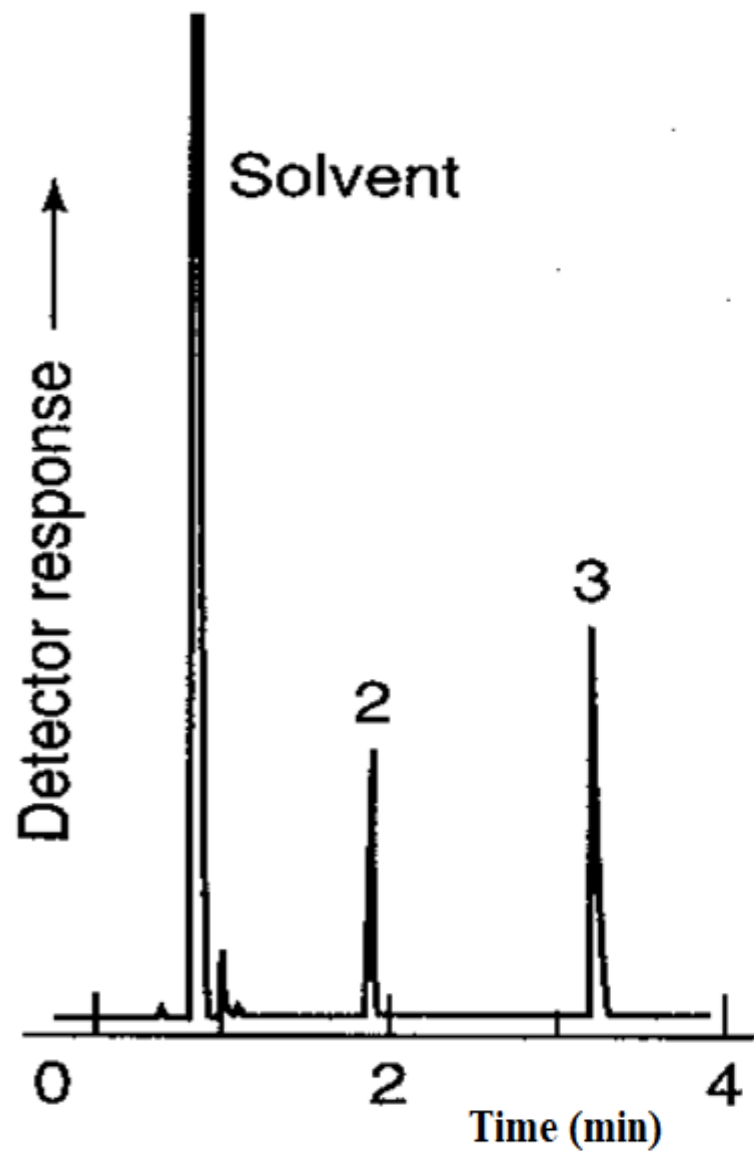
- Carrier gas arrives in the vaporization chamber with a relatively large flow.
- A vent valve separates the carrier gas flow into two parts of which the smallest enters the column.
- The split ratio varies between 1:10 and 1:500.



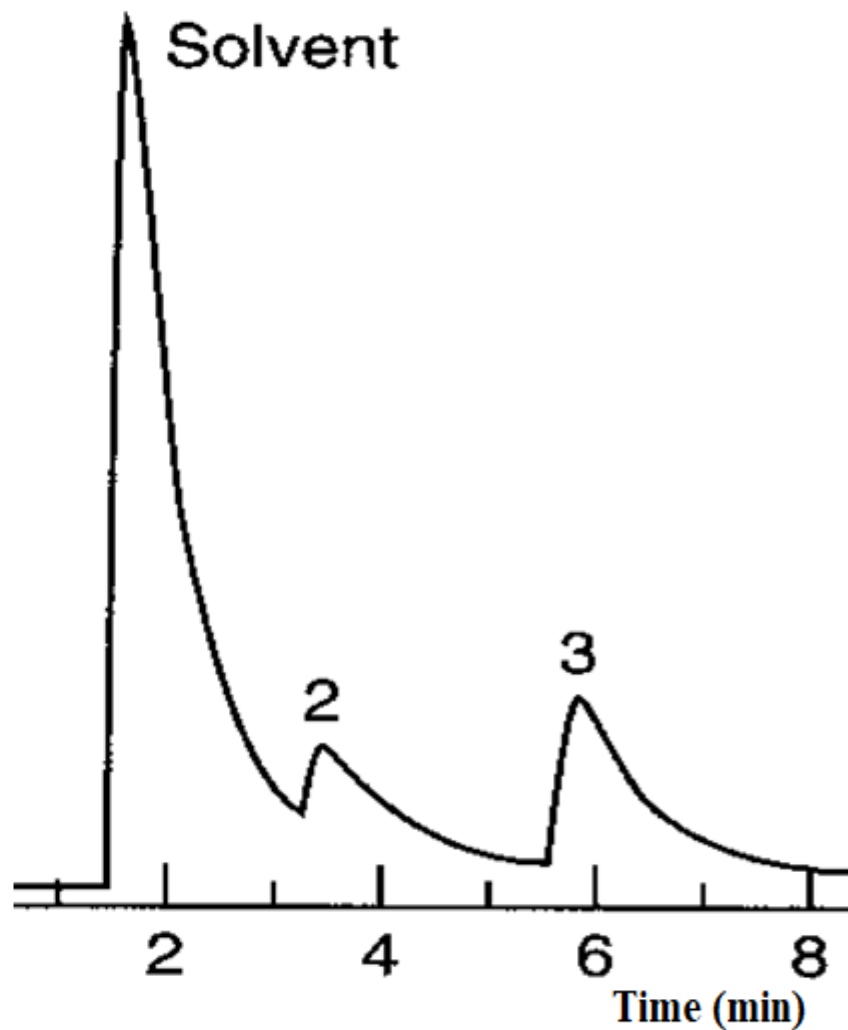
# Splitless mode

- All sample to column.
- Best for quantitative analysis.
- For trace analysis.





**Split mode injection**



**Splitless mode injection**

# Sample injection

---

It allows a rapid and simple introduction of the sample to be analysed in the gas chromatograph.

There are two main injection systems, depending on the nature of sample:

- Injection port:** for introduction of liquids and solutions.
- Sampling loop injection:** for introduction of gas samples.

# Injection port

For rapid introduction and volatilisation of the liquid and solution samples.

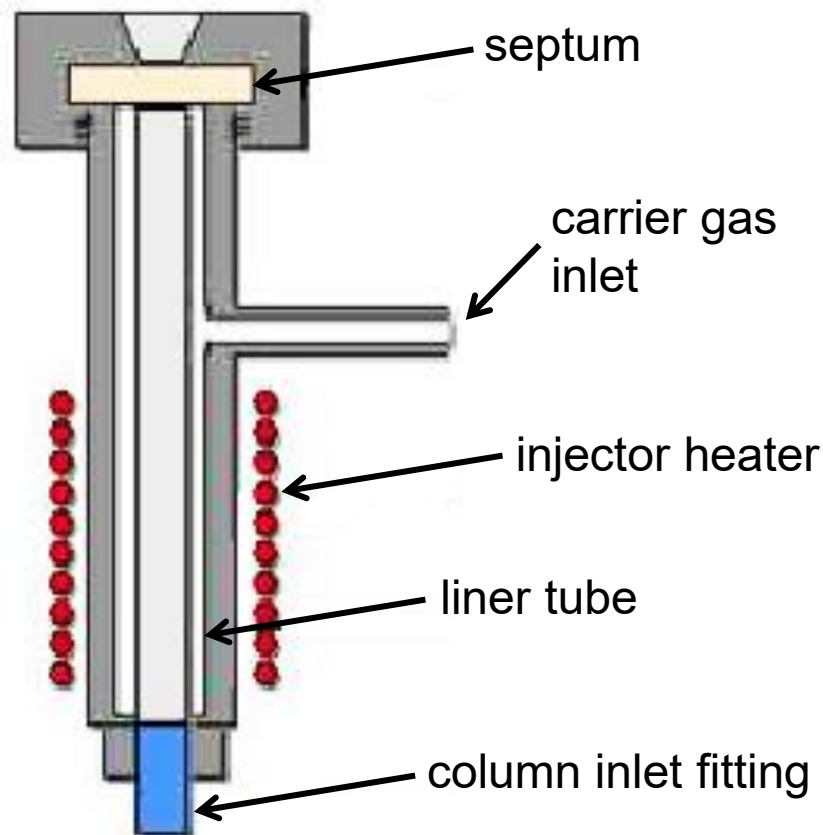
- Injection port temperature must be high enough to quickly evaporate the sample without thermal degradation.

- The injection temperature is 20° higher than the boiling point of the less volatile constituent of the mixture.

- To avoid condensation of the sample in the injection port, the injector temperature must be higher than column temperature:

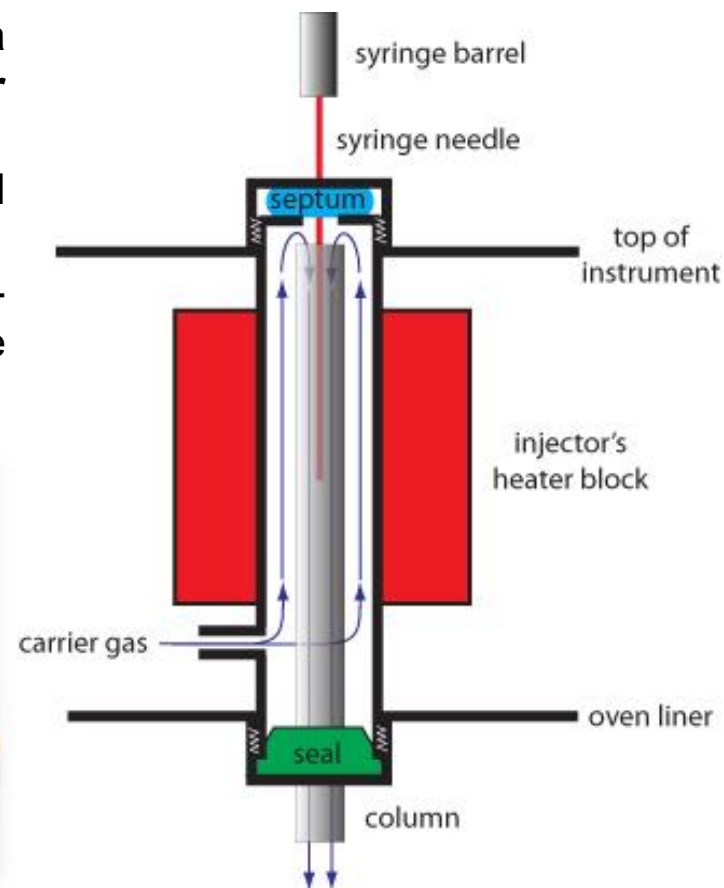
$$T_{\text{injector}} > T_{\text{column}} \text{ (about } 50^{\circ}\text{C)}$$

- The volume of the injection port must allow the volatilisation of the liquid sample and avoid the excessive dead volume.



# The septum

- The most common injection method is where a microsyringe is used to inject sample through a **rubber septum** into a vapouriser port at the head of the column.
- It allows an easy injection with a simple syringe and maintains seal to avoid gas leaks.
- The septum material is an elastomere temperature-resistant with a maximum temperature limit which should be respected during operation.



- The septum should be checked and changed periodically.
- The injection port temperature should be less than the limit temperature of the septum.
- A decrease of carrier gas pressure or an increase of retention times could be due to a damaged septum.



# The liner

-Presents a hot and inert surface and a suitable volume for a rapid vaporisation of the sample.

-The liner is generally made of glass but some models may have also steel liners.

-In some chromatographs, the injection port does not have liner, the sample is directly injected in the column then vaporized.

-The liner must be regularly checked or changed, it can be contaminated by degradation or non-volatile products.



Straight tube liner.



Gooseneck design. Useful in trace analysis.



Double gooseneck design. Useful in trace analysis with active compounds and helps prevent backflash.



Cyclo double gooseneck design. Useful for trace, active, and dirty samples.



Recessed gooseneck design. Useful for use with dirty samples.

# Injection techniques

Bad injection techniques are one of the major sources of errors in gas chromatography, both qualitatively and quantitatively.

If an automatic injector (auto-injector or auto-sampler) is available, it is better than manual injection

- Syringe injection.
- Gas sampling loop/valve.
- Purge and trap.
- Solid phase microextraction (**SPME**).

# Syringe injection

The syringe used to introduce an accurate volume of the liquid or gas sample in the injector.

Several syringe models are available: from 1 mL to several millilitres, with various options: fixed or removable needle, adaptor, sharp or round needle.



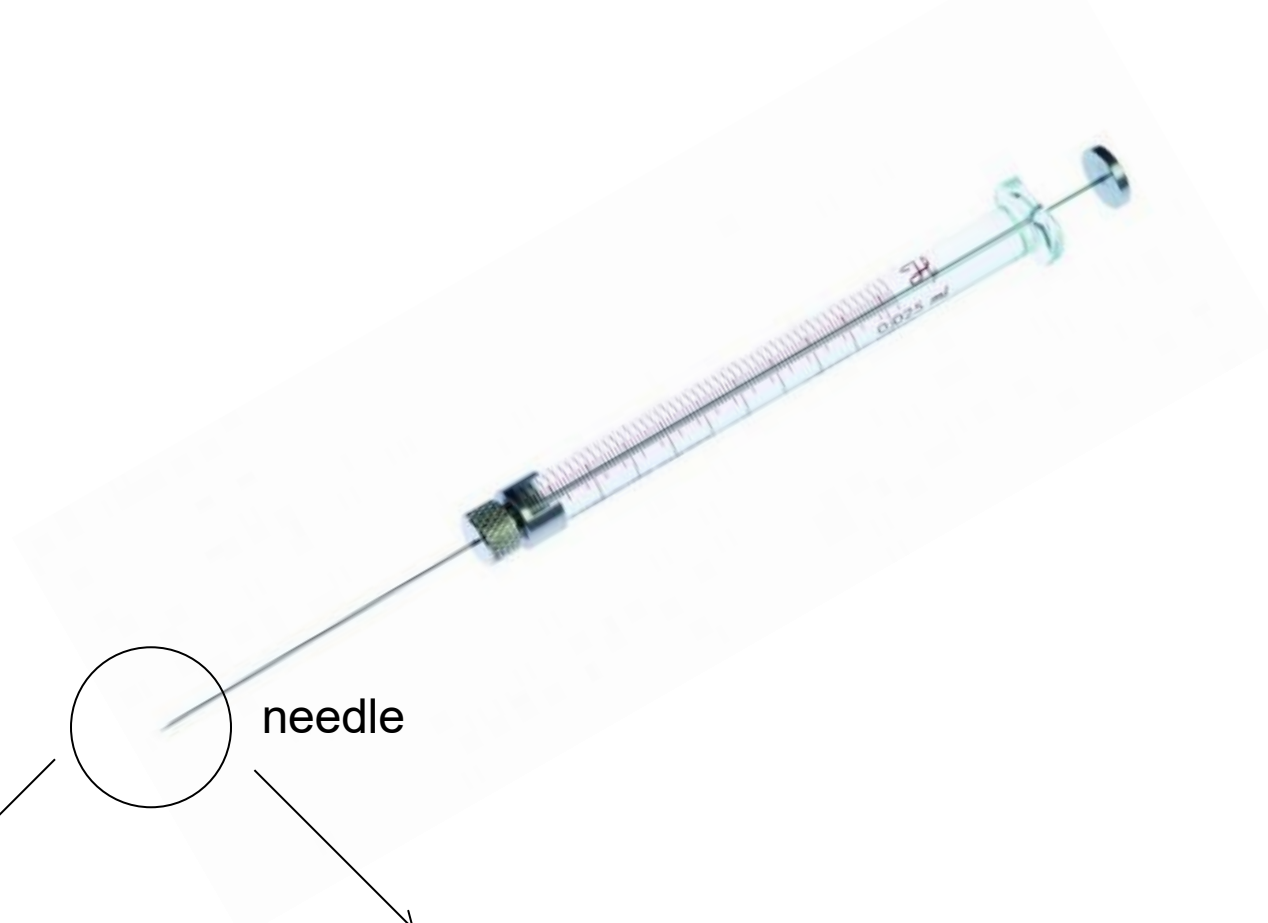
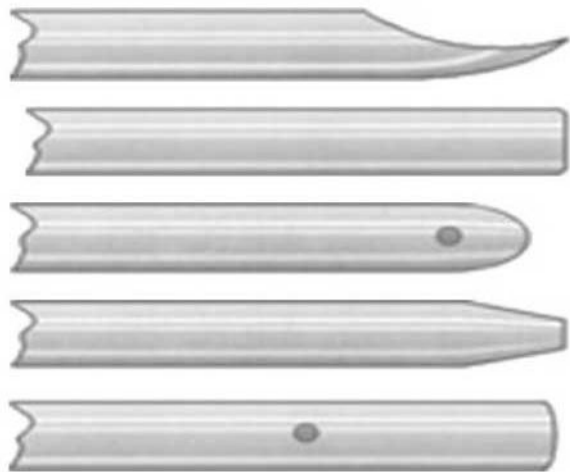
Microsyringe

## Injection volume:

- Liquids 0.1–10  $\mu\text{L}$  (typical).
- Gases 0.5–5 mL (typical).



Adaptor, can be used to help control the injected volume



needle

Bevel

Blunt



## Example (effect on the separation quality)

- Samples should be injected as a plug, rapid and consistent in order to obtain sharp peaks and acceptable precision
- The sample should be injected and the needle removed from septum as quickly as possible



**slow injection**



**fast injection**

# Needle volume correction

**Because of the remaining sample in the syringe needle**

Load sample into syringe



Read the volume



Rapidly inject the sample – remove the syringe

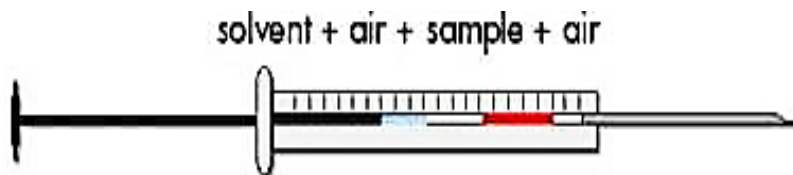
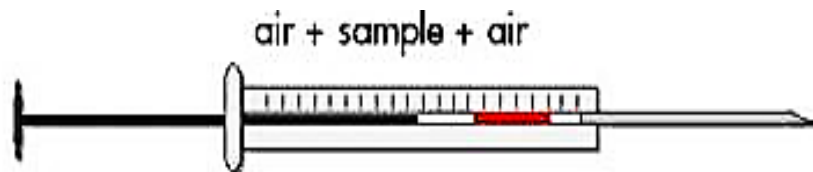
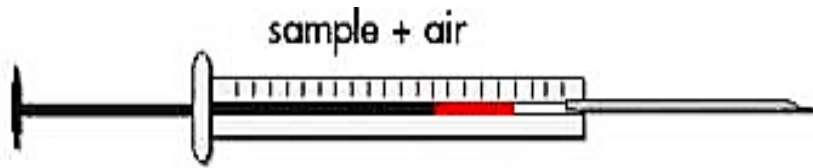
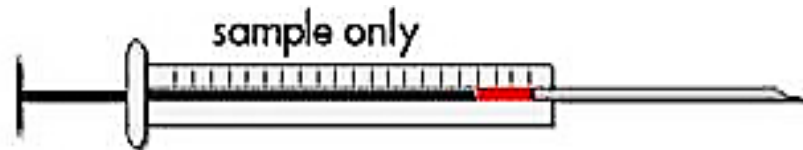


Start run then pull back on syringe



Subtract the needle volume from the total

# Syringe loading methods



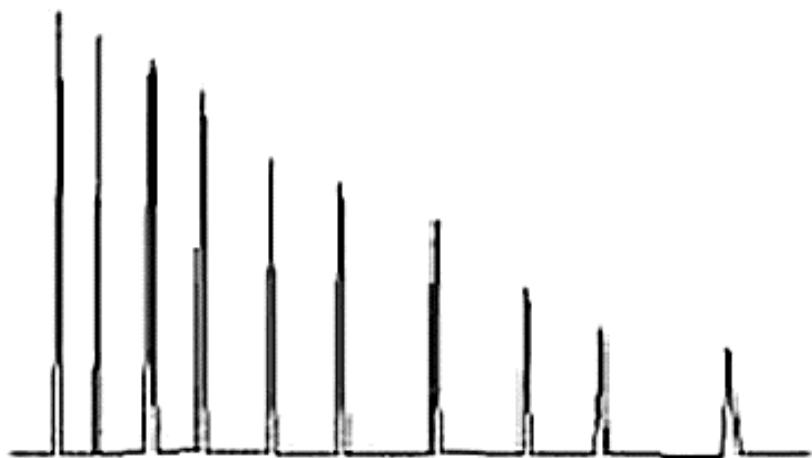


# Hot needle method

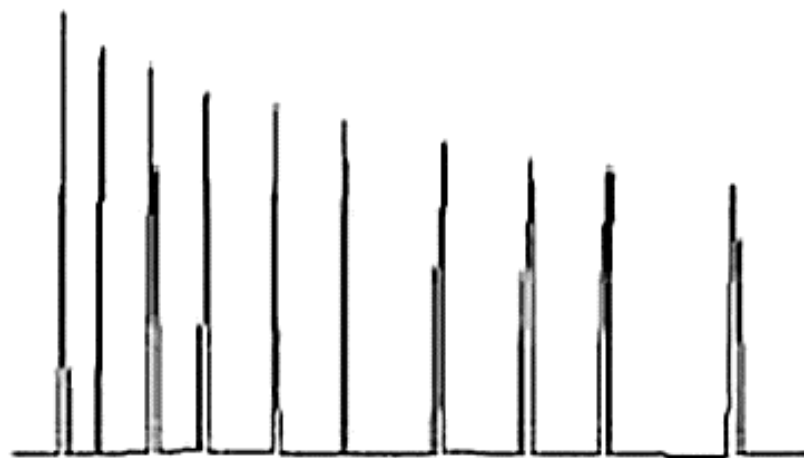
- Draw sample into syringe barrel.
- Draw 2–3  $\mu\text{L}$  air into barrel.
- Insert needle into injection port and allow to heat for a few seconds.
- Rapidly inject sample and withdraw the needle.
- Sample should be injected as a plug.

This insures that all sample is injected and the hot needle assist in solvent volatilization.

Injection of a mixture of n-alkanes ( $\text{C}_{10} - \text{C}_{19}$ )



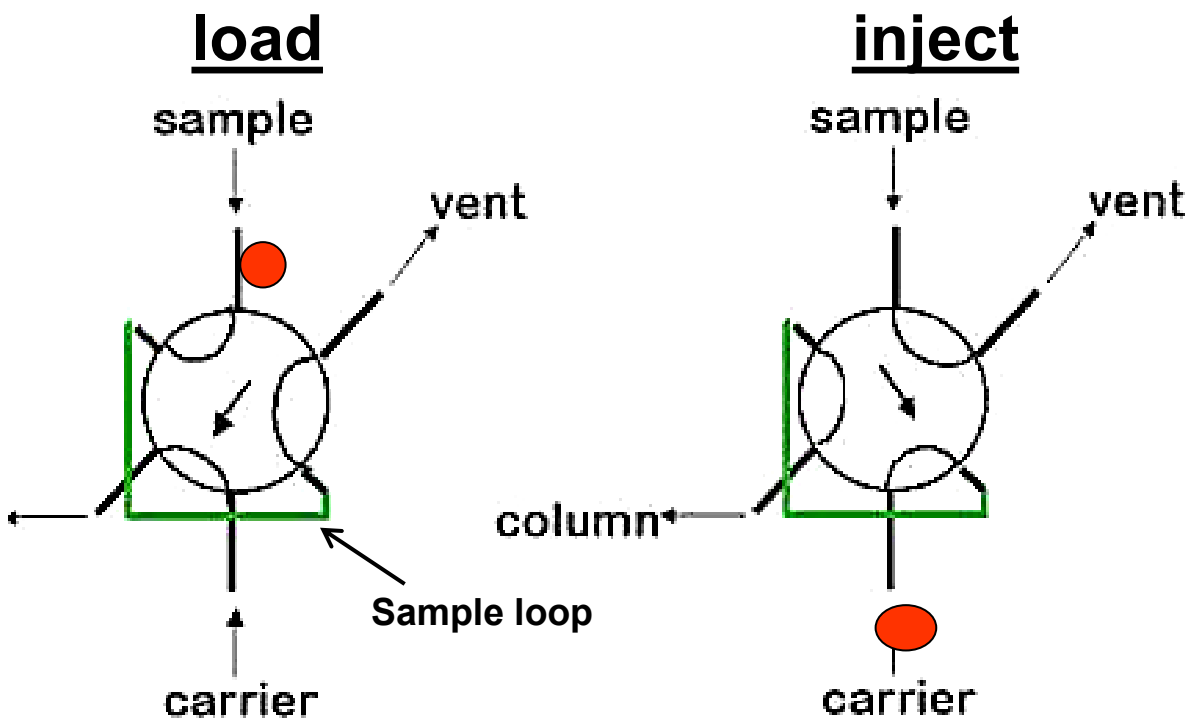
Filled needle method



Hot needle method

# Gas sampling loop / valve

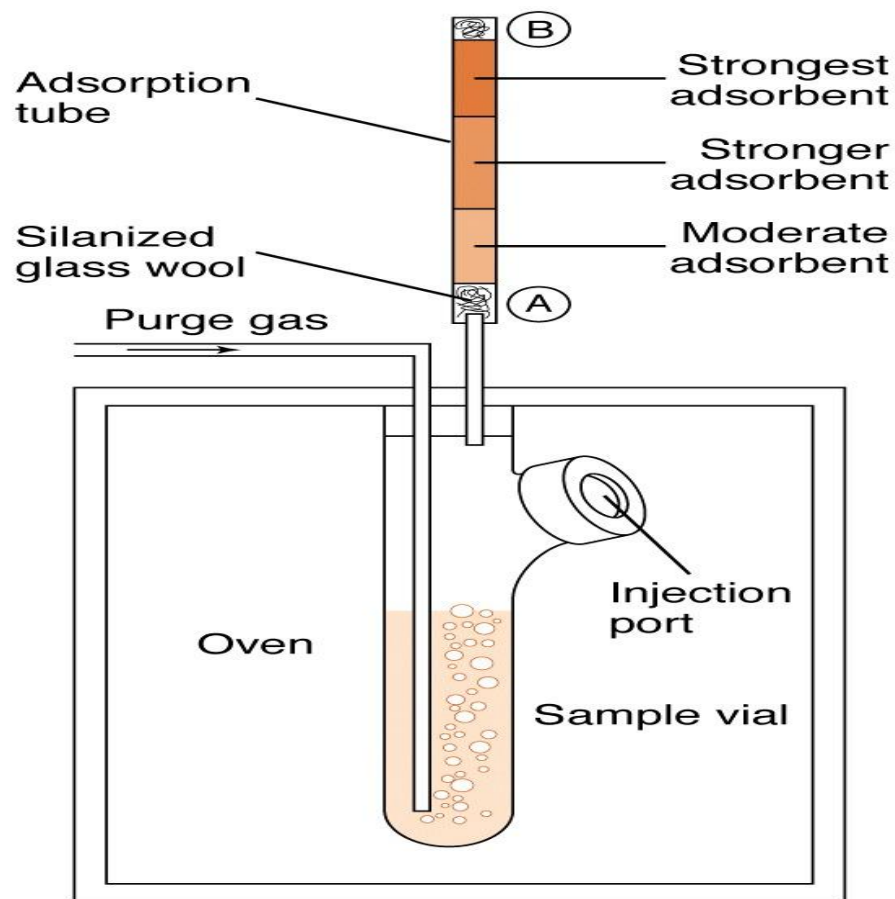
- Introducing a constant amount of a gas can be difficult with a syringe.
- Valves give better reproducibility.
- Require less skill.
- Can be easily automated.



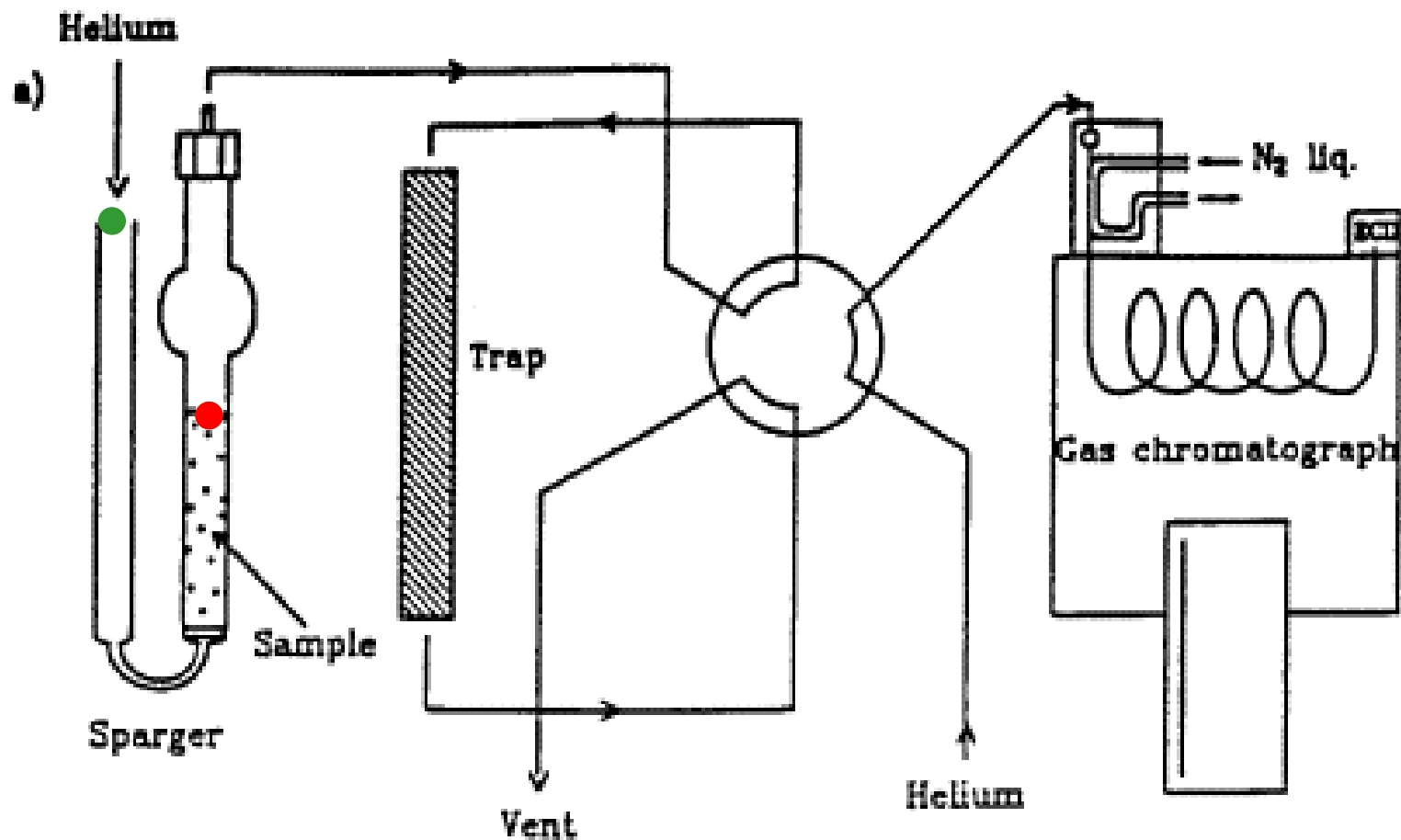
6 port valve

# Purge and Trap

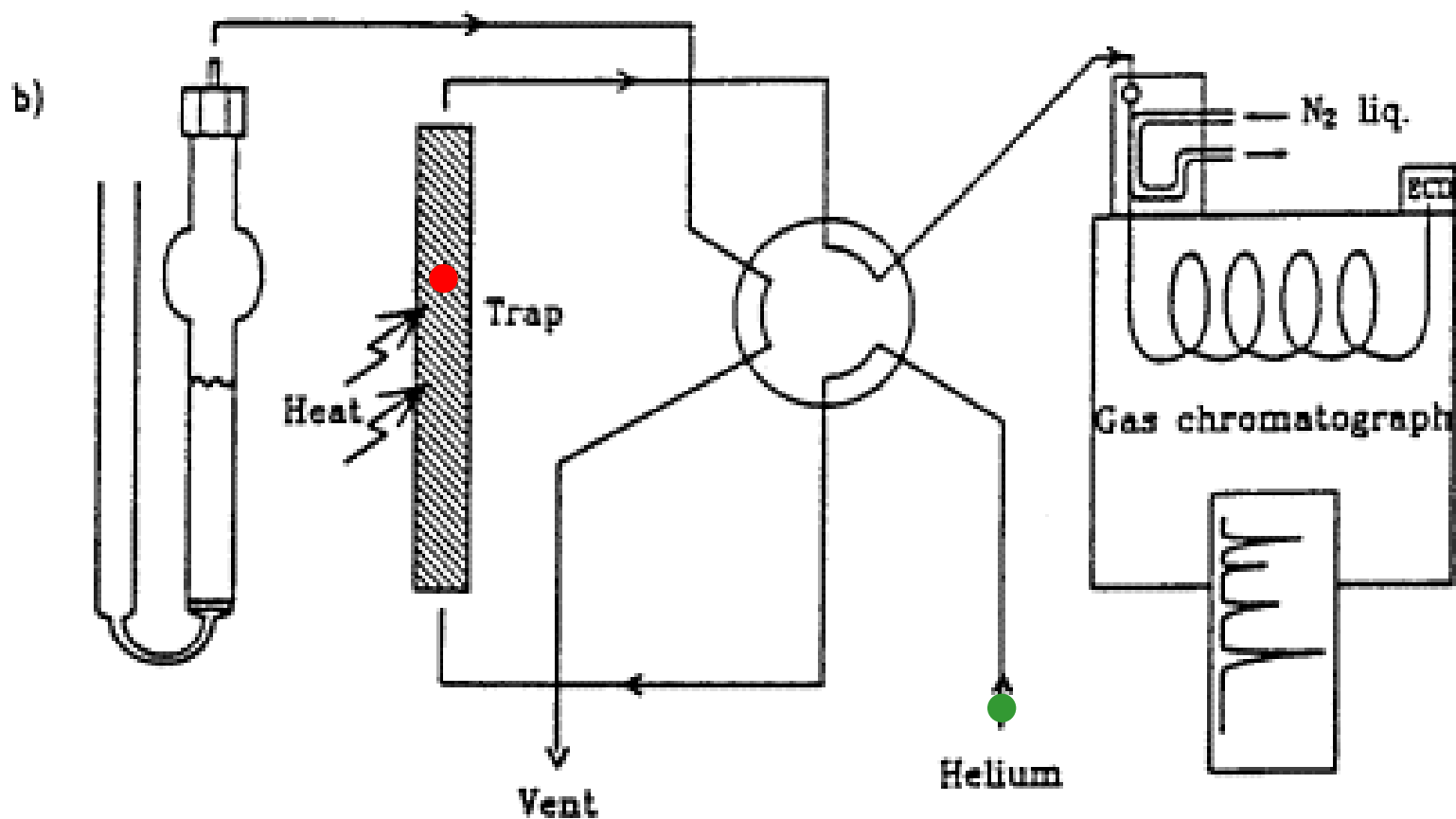
- The sample is permanently purged with carrier gas, which carries the analytes to the trapping medium.
- Useful for concentrating insoluble or poorly soluble volatile organic compounds (VOCs).
- Lower detection limit.



# Purge and trap step



# Desorption step

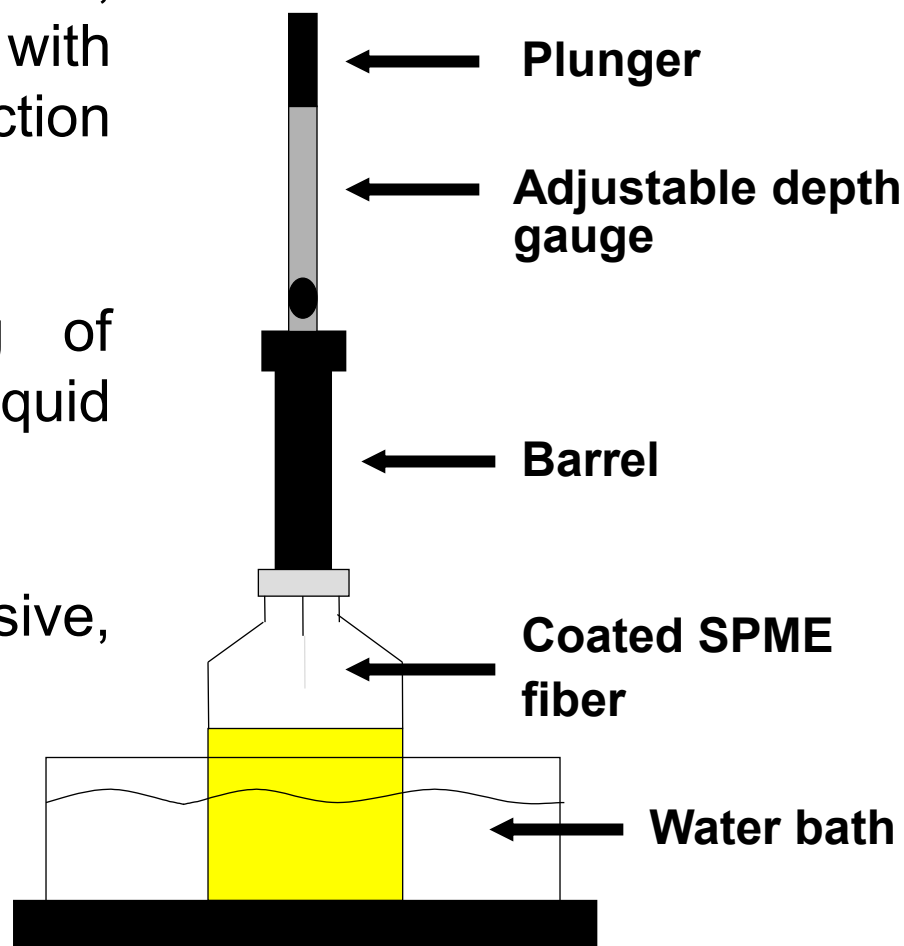


# Solid Phase Microextraction (SPME)

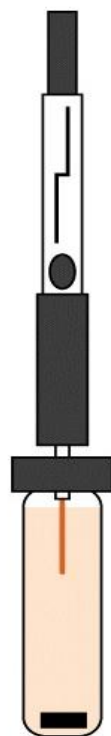
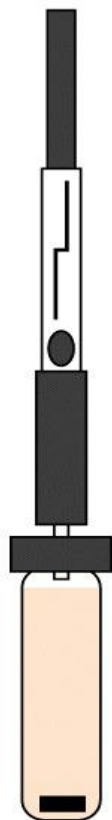
A technique that uses a short, thin, silica fused rod which is coated with absorbent polymer (fiber) for extraction of compounds.

Principle: Equilibrium partitioning of compounds between the fiber and liquid sample.

It is fast, sensitive, inexpensive, portable and solvent-free.



Pierce sample  
septum with  
metal needle

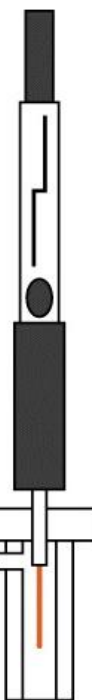
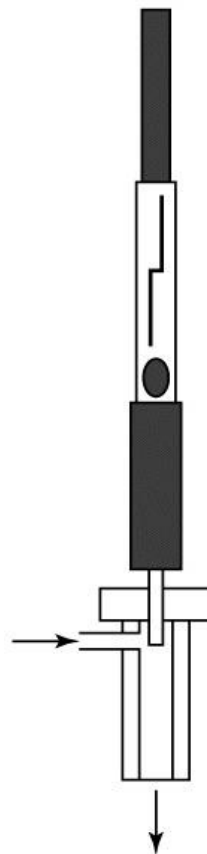


Retract fiber  
and withdraw  
needle

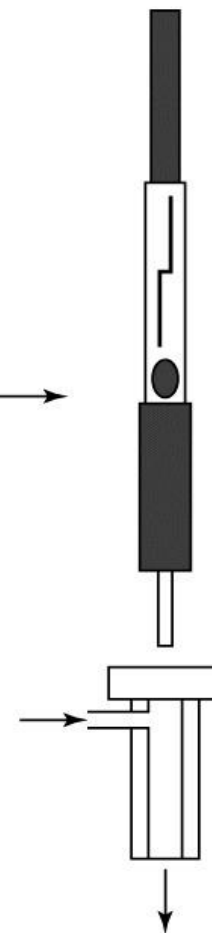


Expose fiber  
to solution or  
headspace for  
fixed time with  
stirring

Pierce  
chromatography  
septum with  
metal needle



Retract fiber  
and withdraw  
needle



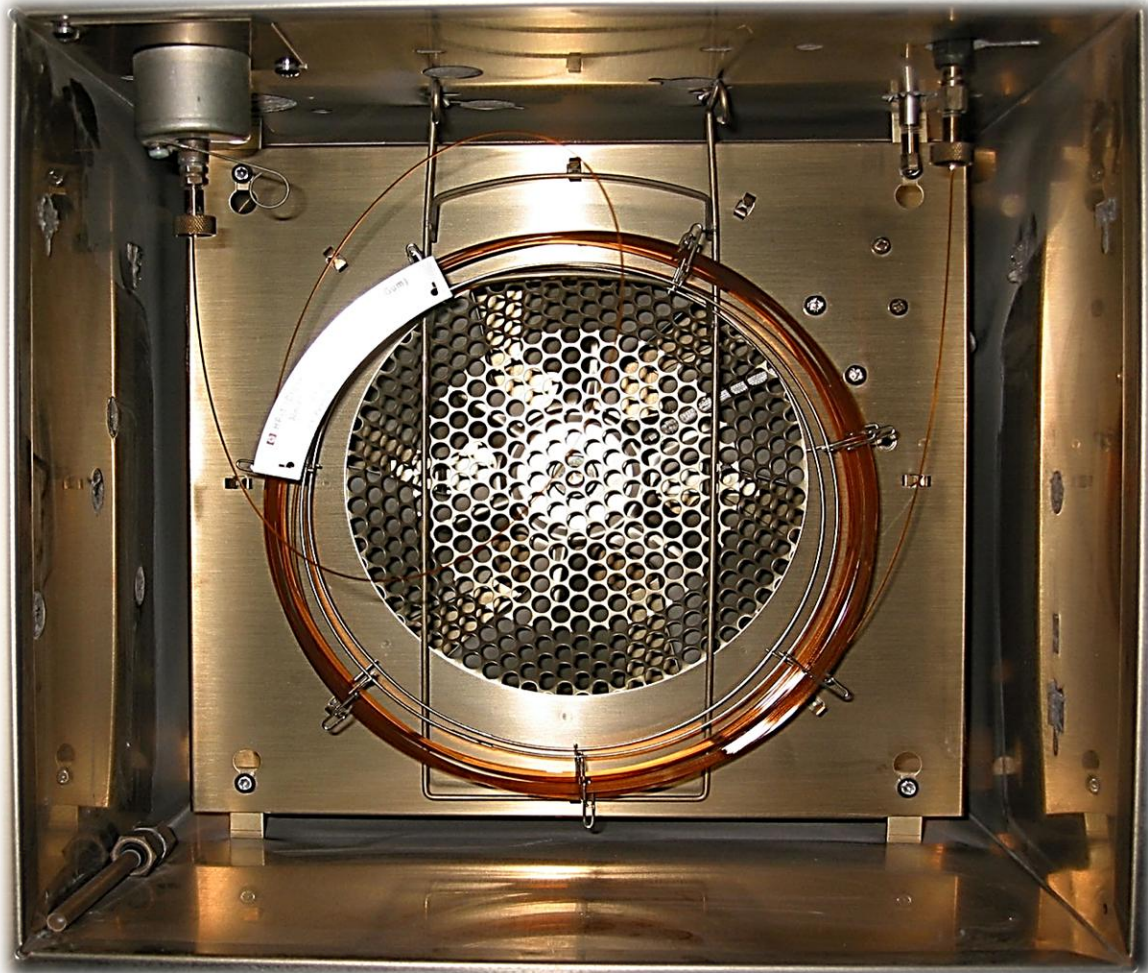
Expose hot fiber  
to carrier gas  
for fixed time



# Oven

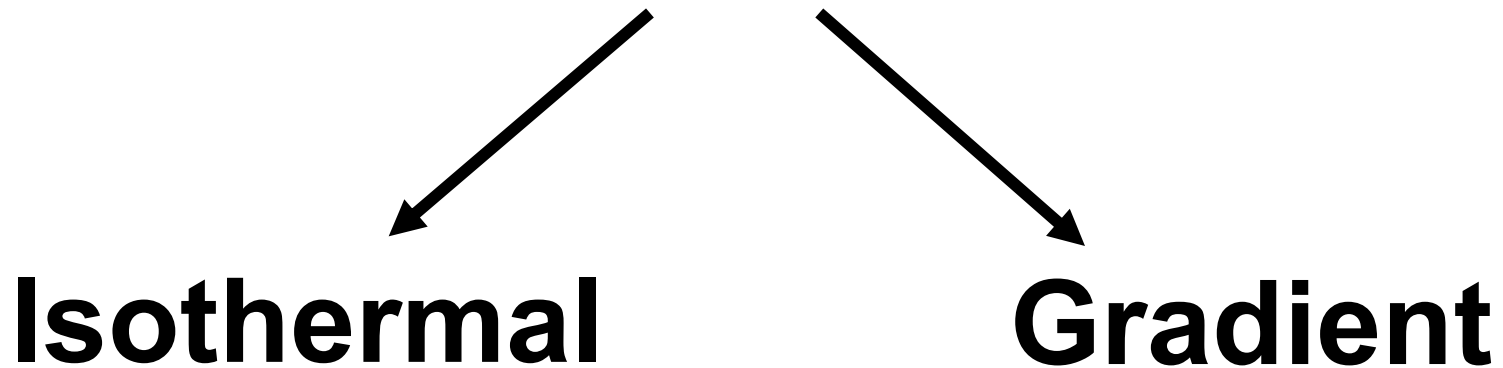
Column temperature is an important variable that must be controlled for precise work, so the column is ordinarily housed in a thermostated oven.

The optimum column temperature depends upon the **boiling point** of the sample and the degree of separation required.





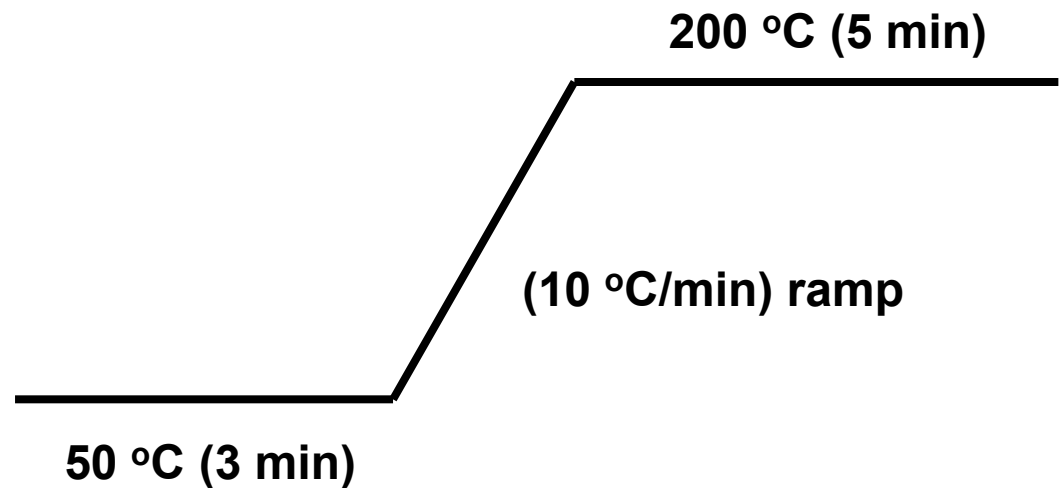
# Temperature control



150 °C



A horizontal line representing a constant temperature of 150 °C.

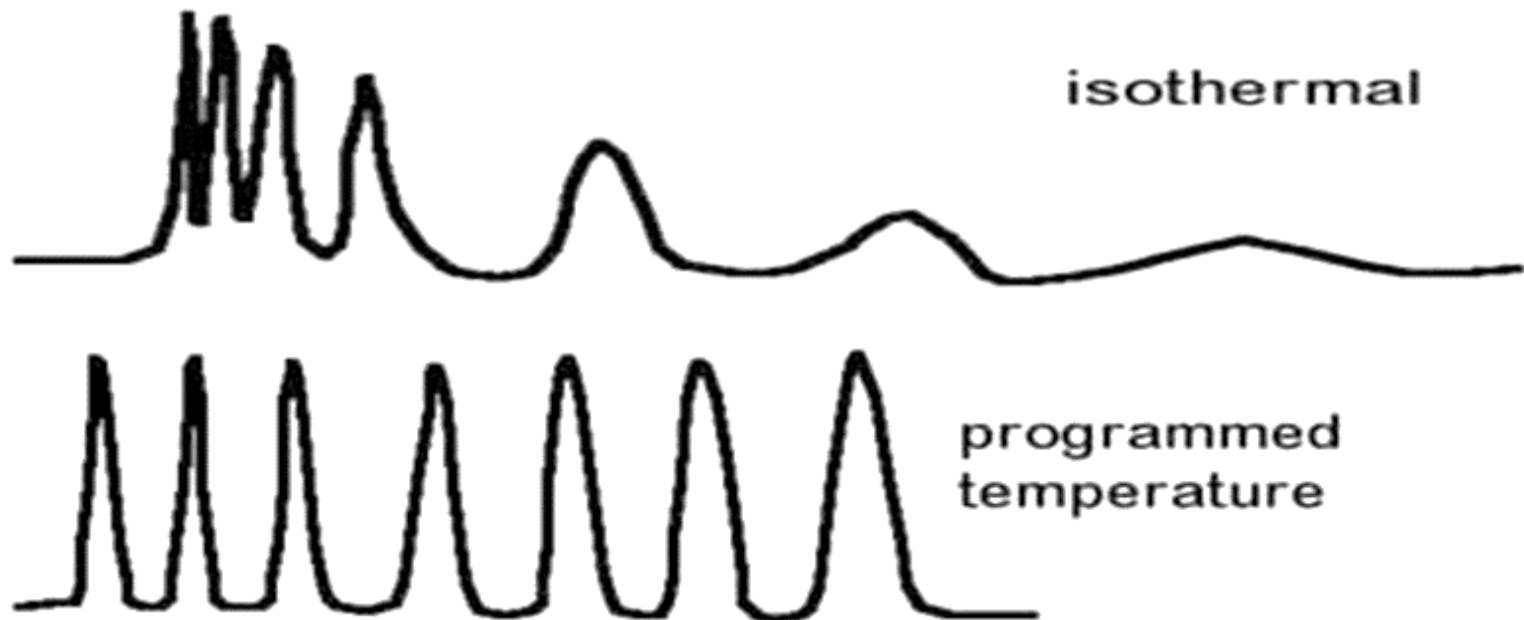


Some GC's allow for a more complex program

# Temperature program

## Factors to consider

- Changes in volatility of solutes.
- Stability of solutes.
- Flow rate changes.
- Stability of stationary phase.



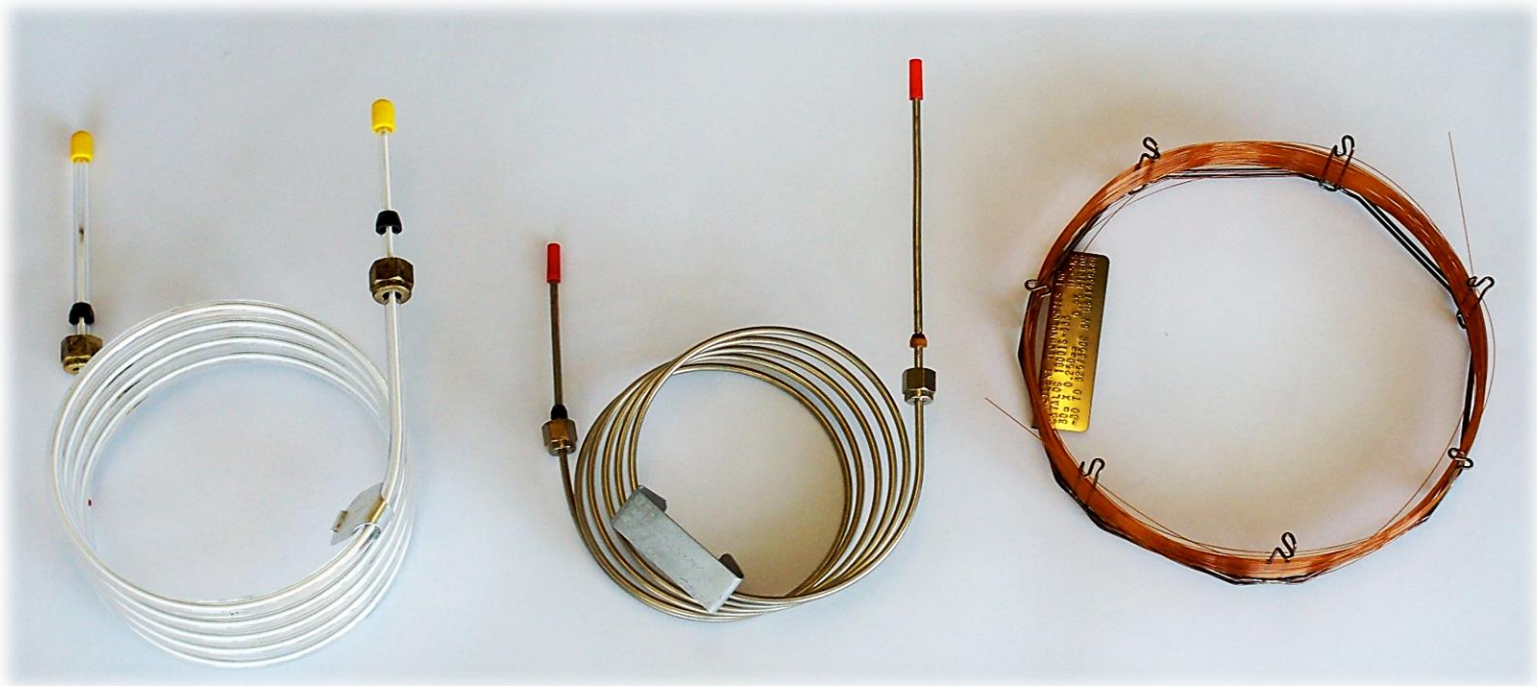
# Column

Although it's usually the smallest part, the column is considered the most important component in any column chromatographic system (heart of chromatographic system).

Columns can be classified by tubing diameter and packing type.

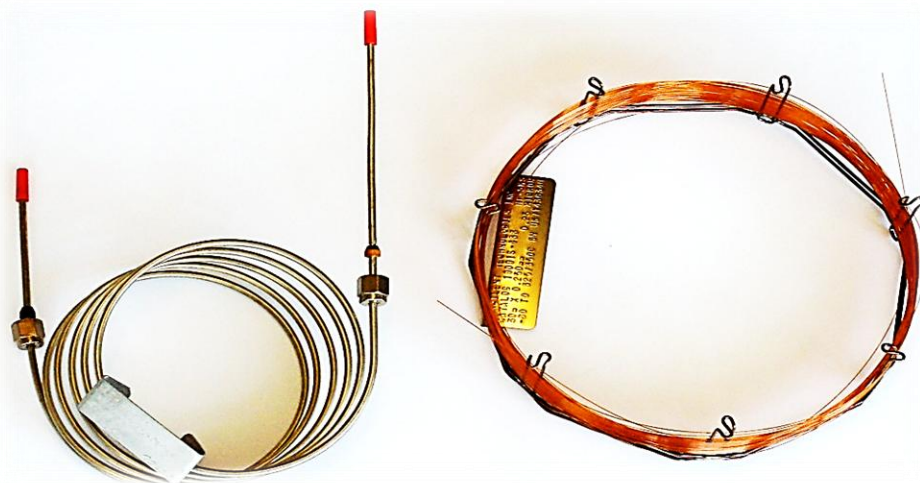
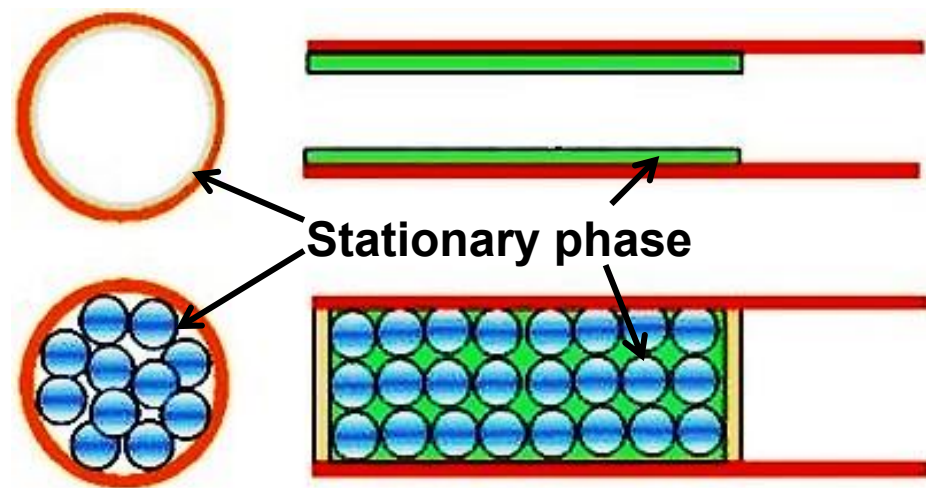
- **Packed columns.**
- **Open tubular capillary columns.**
  - Wall-coated open tubular (WCOT)
  - Support-coated open tubular (SCOT)
  - Porous layer open tubular (PLOT)

A broad variety of tube sizes (dimensions) and materials, such as stainless steel, fused silica and glass tubes, have been used as molds for GC.

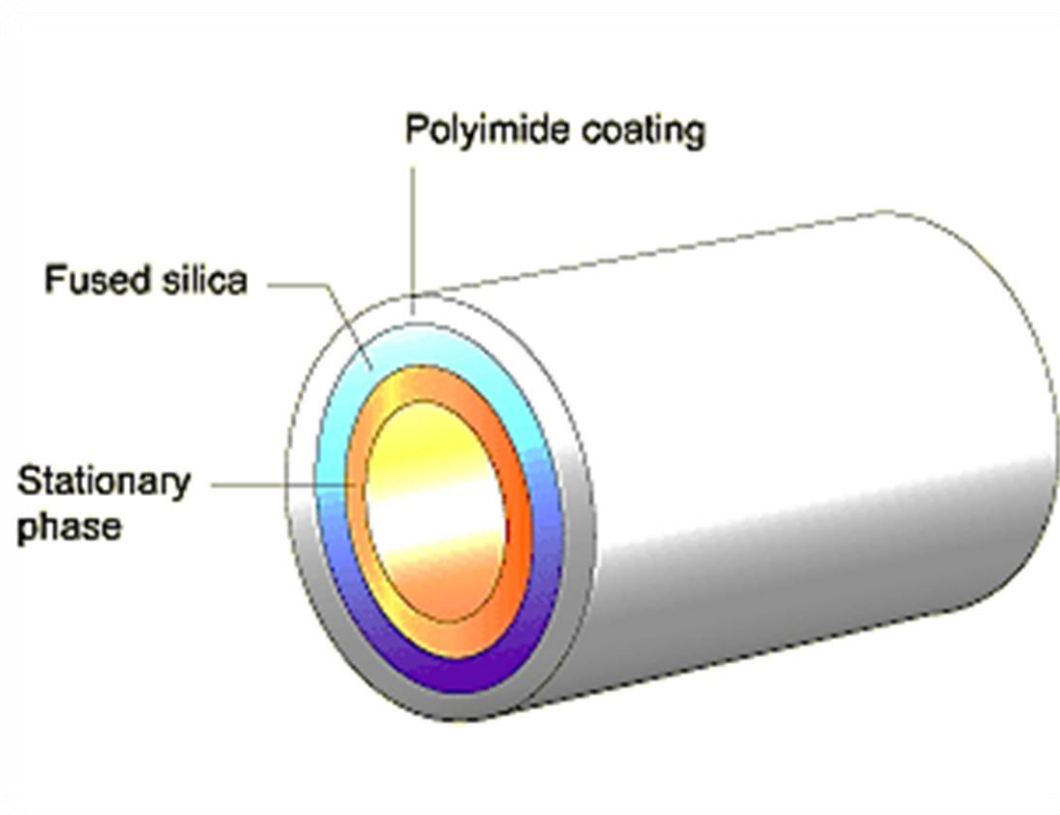


# Packed vs. Open tubular capillary columns

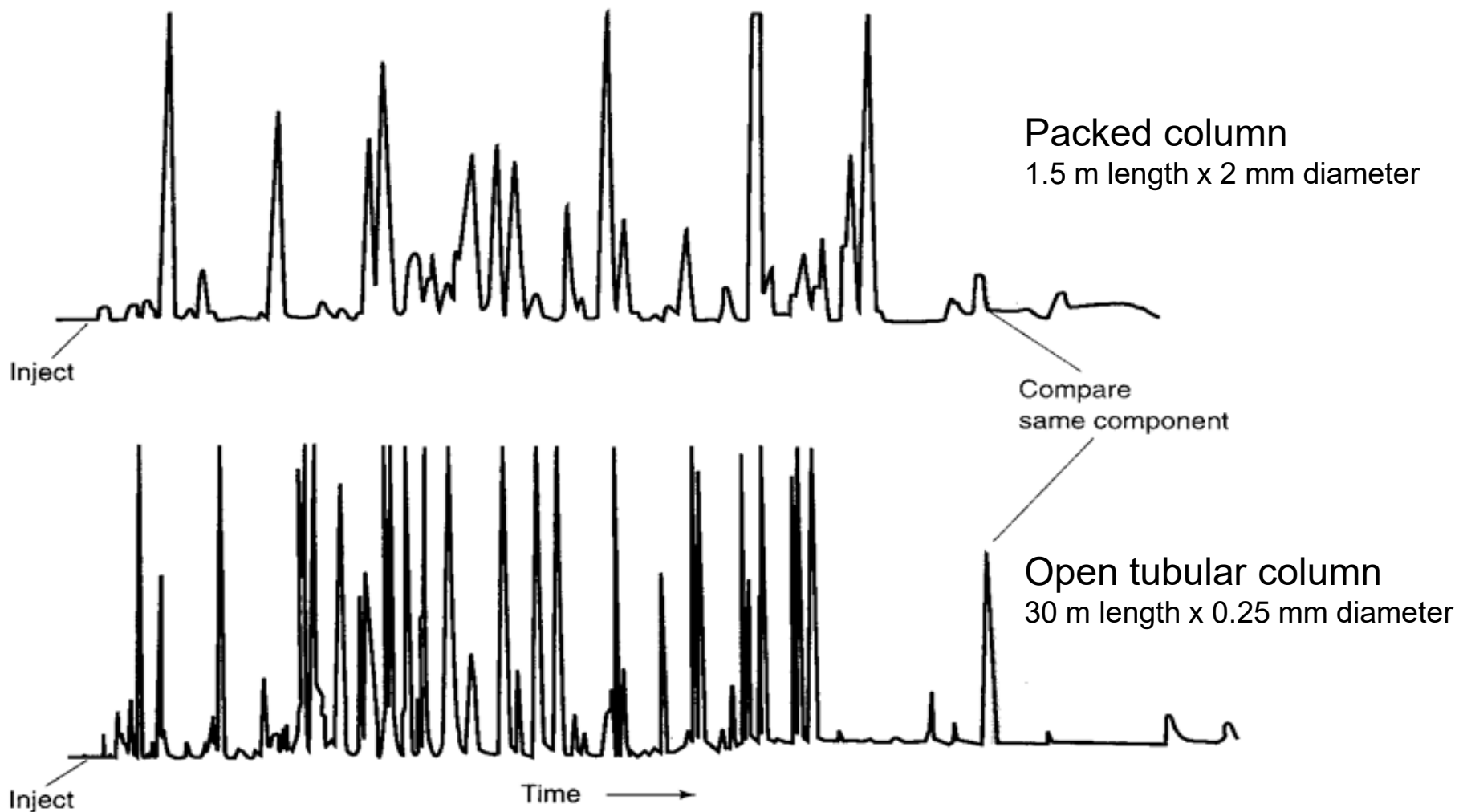
|                               | Packed column | Open tubular column |
|-------------------------------|---------------|---------------------|
| Length, m                     | 0.5–5         | 5–100               |
| Internal diameter, mm         | 2–4           | 0.1–0.5             |
| Flow rate, mL/min             | 10–60         | 0.5–10              |
| Head pressure, psig           | 10–40         | 3–40                |
| Total plates                  | 5,000         | 250,000             |
| Capacity, $\mu\text{g/peak}$  | 10            | 0.1                 |
| Film thickness, $\mu\text{m}$ | 1–10          | 0.1–8               |



Fused silica is a synthetic quartz of high purity. A protective coating is applied to the outer surface with polyimide being the most common coating material. The polyimide coating is responsible for the brownish color of fused silica capillary columns.

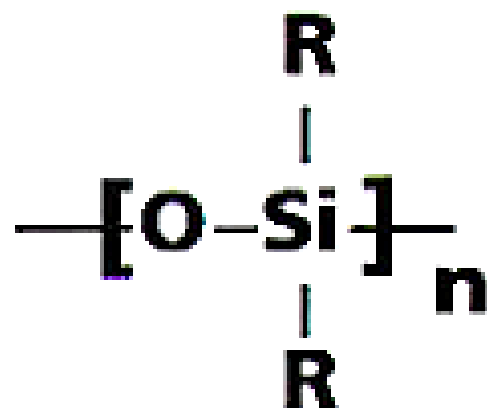


# Packed vs. Open tubular columns



GC separation of a perfume oil

# Polysiloxanes ...



R = CH<sub>3</sub>

methyl

CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CN

cyanopropyl

CH<sub>2</sub>CH<sub>2</sub>CF<sub>3</sub>

trifluoropropyl

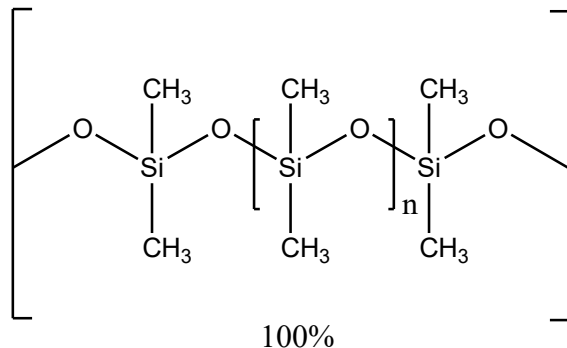


phenyl

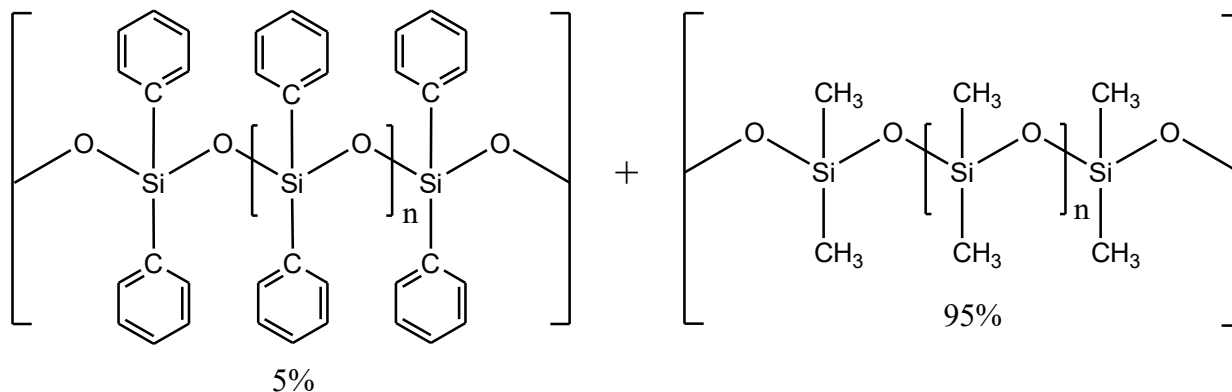


# Stationary phases

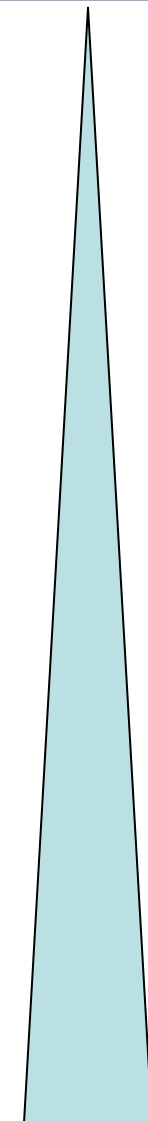
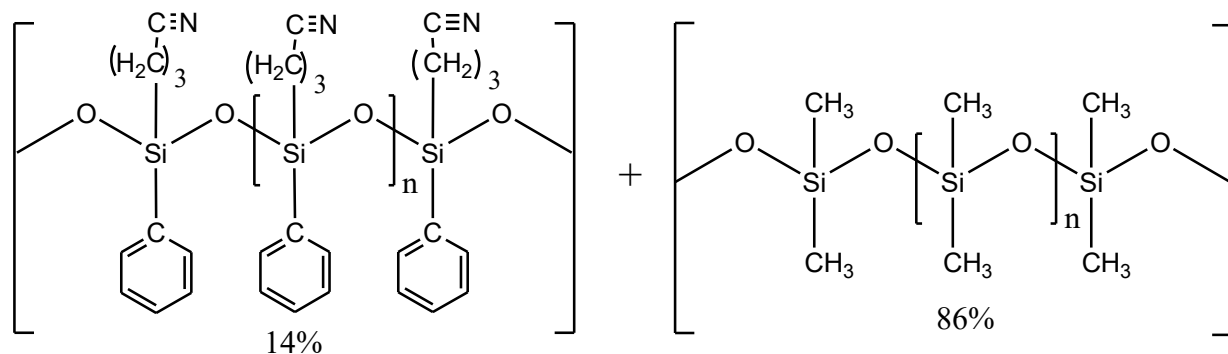
100% dimethyl  
polysiloxane  
*Least polar phase*



5% diphenyl  
95% dimethyl  
polysiloxane  
*Non-polar phase*



14%  
cyanopropylphenyl  
86% dimethyl  
polysiloxane  
*polar phase*



**Polarity**

# Principle of separation

Like dissolve like (like attract like)

Non-polar stationary phases best for non-polar analytes

Polar stationary phases best for polar analytes

| Stationary Phase                                 | Common Trade Name | Maximum Temperature, °C | Common Applications                                                                        |
|--------------------------------------------------|-------------------|-------------------------|--------------------------------------------------------------------------------------------|
| Polydimethyl siloxane                            | OV-1, SE-30       | 350                     | General-purpose nonpolar phase; hydrocarbons; polynuclear aromatics; drugs; steroids; PCBs |
| Poly(phenylmethyldimethyl) siloxane (10% phenyl) | OV-3, SE-52       | 350                     | Fatty acid methyl esters; alkaloids; drugs; halogenated compounds                          |
| Poly(phenylmethyl) siloxane (50% phenyl)         | OV-17             | 250                     | Drugs; steroids; pesticides; glycols                                                       |
| Poly(trifluoropropyldimethyl) siloxane           | OV-210            | 200                     | Chlorinated aromatics; nitroaromatics; alkyl-substituted benzenes                          |
| Polyethylene glycol                              | Carbowax 20M      | 250                     | Free acids; alcohols; ethers; essential oils; glycols                                      |
| Poly(dicyanoallyldimethyl) siloxane              | OV-275            | 240                     | Polyunsaturated fatty acids; rosin acids; free acids; alcohols                             |

# Commercial columns certificate

Example..

## TRACE™ TR-5 GC Columns

Thermo Scientific™

### Description:

- Nonpolar phase, 5% phenyl methyl polysiloxane
- High operating temperature and extremely low bleed
- Widely used in a variety of applications
- Similar to: DB-5, BP5, Rtx-5, HP-5, Ultra-2, PTE-5, SPB5, MDN-5, CP-Sil 8CB, SPB-5, AT-5, ZB-5, 007-2(MPS-5), SE-52, SE-54

### Recommended applications:

- Alcohols
- Free fatty acids
- Aromatics
- Flavors
- Low polarity pesticides

### Specifications ...

|                   |                       |
|-------------------|-----------------------|
| Catalog number    | 260E130P              |
| For Use With      | GC/MS                 |
| Max. Temperature  | 340/350°C             |
| Diameter          | 0.25mm                |
| USP Type          | G27, G36              |
| Film Thickness    | 0.25µm                |
| Length (Metric)   | 15m                   |
| Stationary Phase  | Trace TR-5            |
| Item Description  | 15m x 0.25mm x 0.25µm |
| Diameter (Metric) | 0.25mm                |



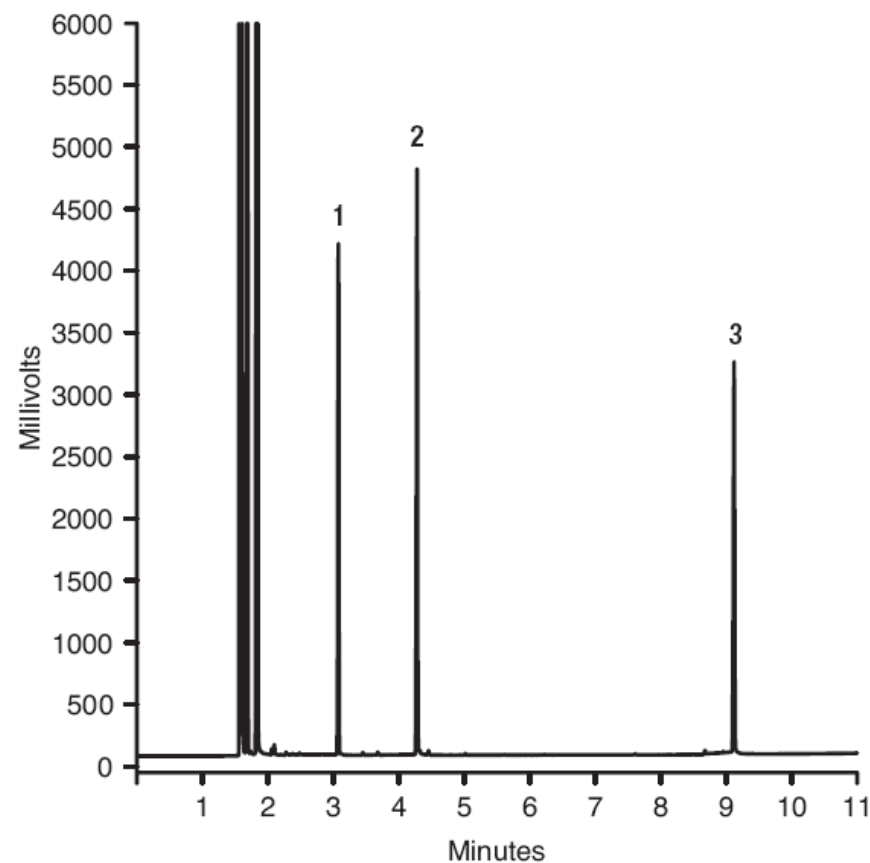
## Application note (20560)

### GC Analysis of Derivatized Amino Acids

**Abstract** The derivatization of amino acids was achieved using the N-methyl-N-(trimethylsilyl) trifluoroacetamide silylation reagent. The trifluoroacetamide derivatized compounds were then analyzed on a TRACE TR-5 column using FID detector.

#### Separation Conditions

|                        |                                        |
|------------------------|----------------------------------------|
| Column                 | TRACE TR-5<br>30 m × 0.25 mm × 0.25 µm |
| Carrier gas            | Helium                                 |
| Column flow            | 1.2 mL/min, Constant flow              |
| Split ratio            | 50:1                                   |
| Oven temperature       | 100 °C, 15 °C/min, 300 °C              |
| Injector temperature   | 240 °C                                 |
| Detector type          | FID                                    |
| Detector temperature   | 280 °C                                 |
| Detector Hydrogen flow | 35 mL/min                              |
| Detector Air flow      | 350 mL/min                             |
| Injection Volume       | 1 µL                                   |



| Peak no. | Derivatized Amino acid | t <sub>R</sub> (min) |
|----------|------------------------|----------------------|
| 1        | L-alanine              | 3.1                  |
| 2        | L-leucine              | 4.3                  |
| 3        | L-lysine               | 9.1                  |

# Detector

Modern detectors use a sensitive **transducer** to convert a chemical or physical property, such as pH or photon intensity, to an easily measured electrical signal, such as a voltage or current.



- In chromatography, the detector serve to detect the appearance of analytes at the end of the column.
- Generates an electrical signal proportional to the sample concentration.
- Provide information about the identity of analytes.
- Must be hot enough (20 to 30 °C above the column temperature).

# Properties of ideal detector

## Characteristics of the ideal GC detectors:

- Adequate sensitivity.
- Good stability and reproducibility.
- Rapidly respond to concentration changes (short response time).
- Low sensitivity to variation in flow, pressure and temperature.
- Large linear range response.
- A temperature range from room temperature to at least 400°C.
- Produces an easily handled signal.
- Stable with respect to noise and drift.
- Nondestructive of sample.

**No detector exhibits all these characteristics.**

# GC detectors

GC and detector in the same instrument.

## Examples...

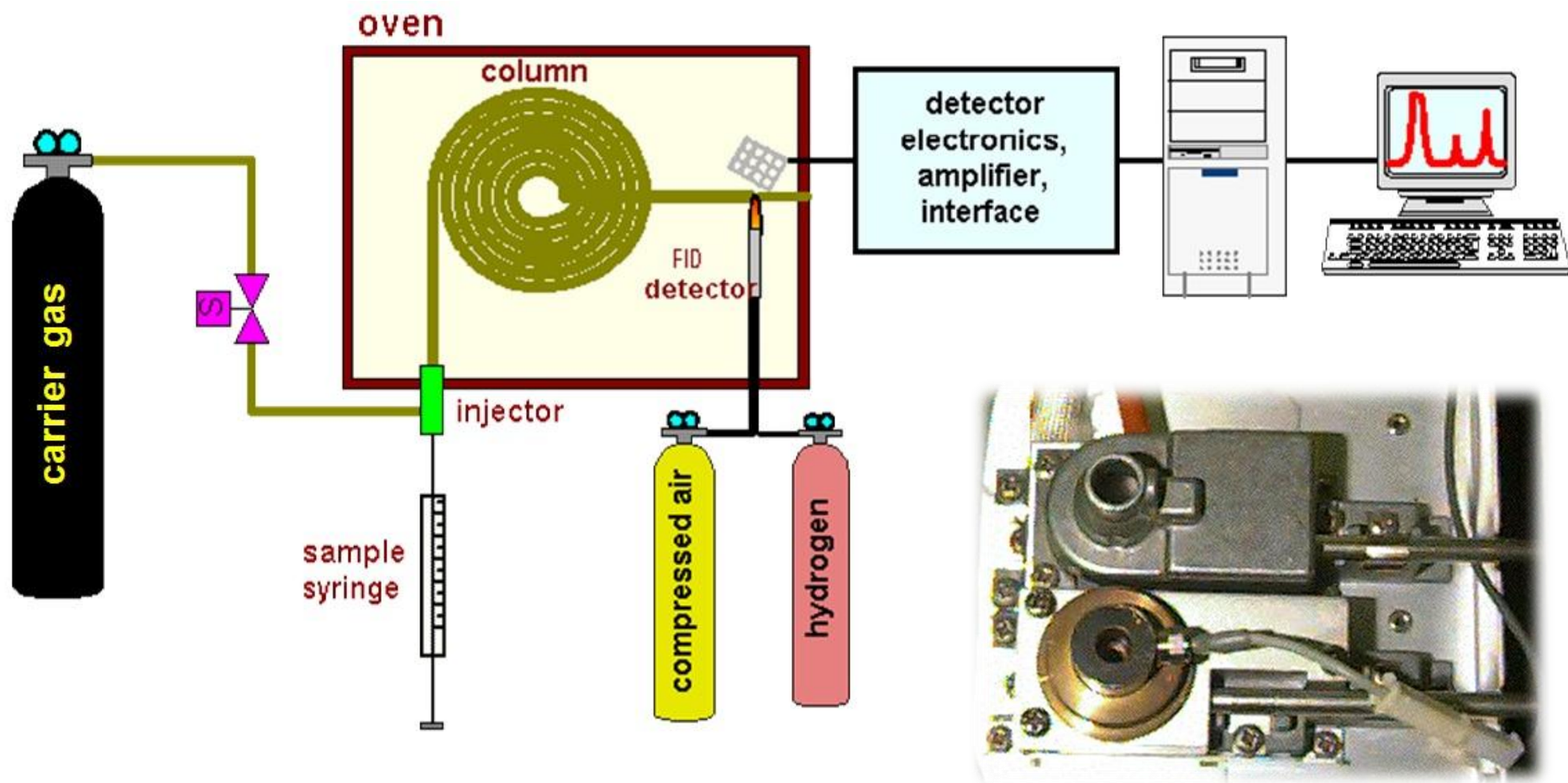
- Flame Ionization Detector (**FID**)
  - Thermal Conductivity Detector (**TCD**)
  - Electron Capture Detector (**ECD**)
  - Nitrogen-Phosphorous Detector (**NPD**)
- 

**Hyphenated GC methods;** GC attached to a second instrument.  
Exploit advantage of each method.

## Examples...

- Mass Spectrometry (**GC-MS**)
- Infrared Spectrometry (**GC-FTIR**)
- Nuclear Magnetic Resonance (**GC-NMR**)
- Atomic Absorption Spectroscopy (**GC-AAS**)
- Atomic Emission Spectroscopy (**GC-AES**)
- Inductively Coupled Plasma-Mass Spectrometry (**GC-ICP-MS**)

# Flame Ionization Detector (FID)



The effluent from the column is mixed with hydrogen and air and ignited.

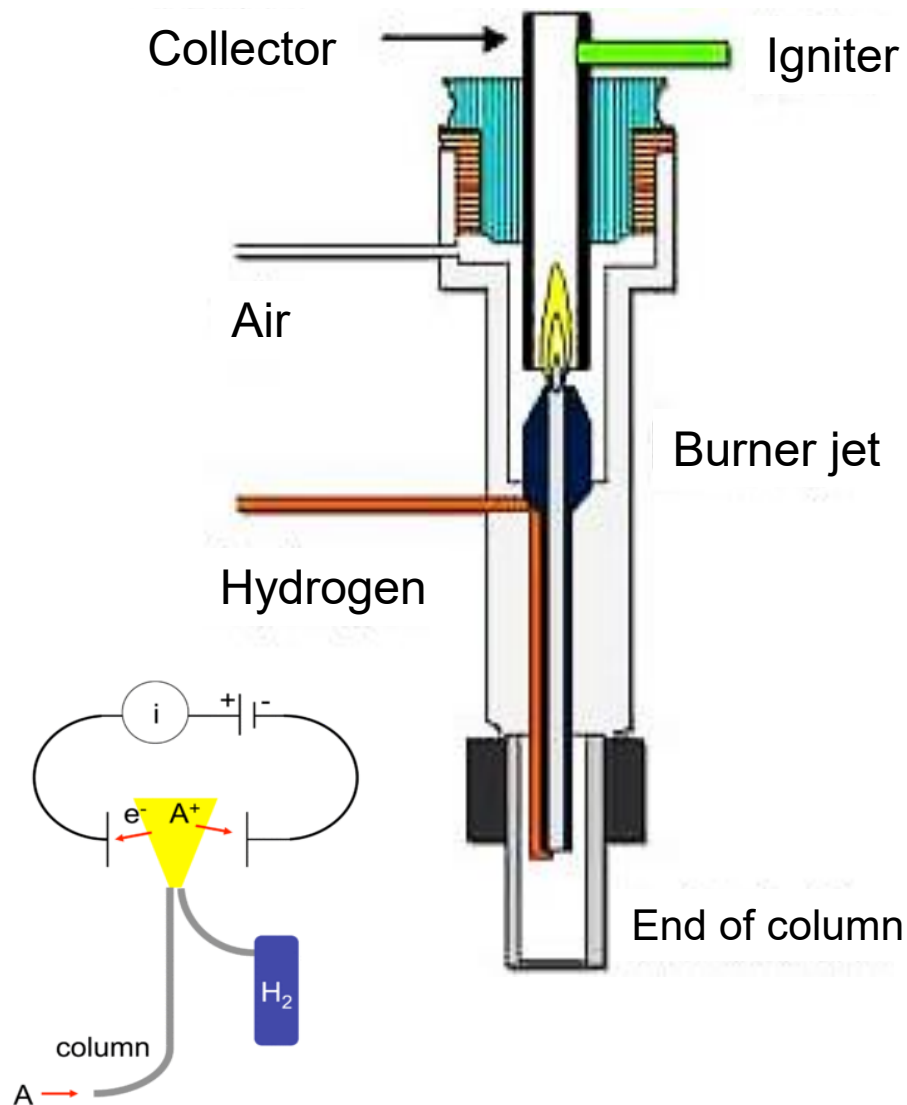


# Mode of detection

-Organic compounds burning in the flame produce ions and electrons (current) which can conduct electricity through the flame.

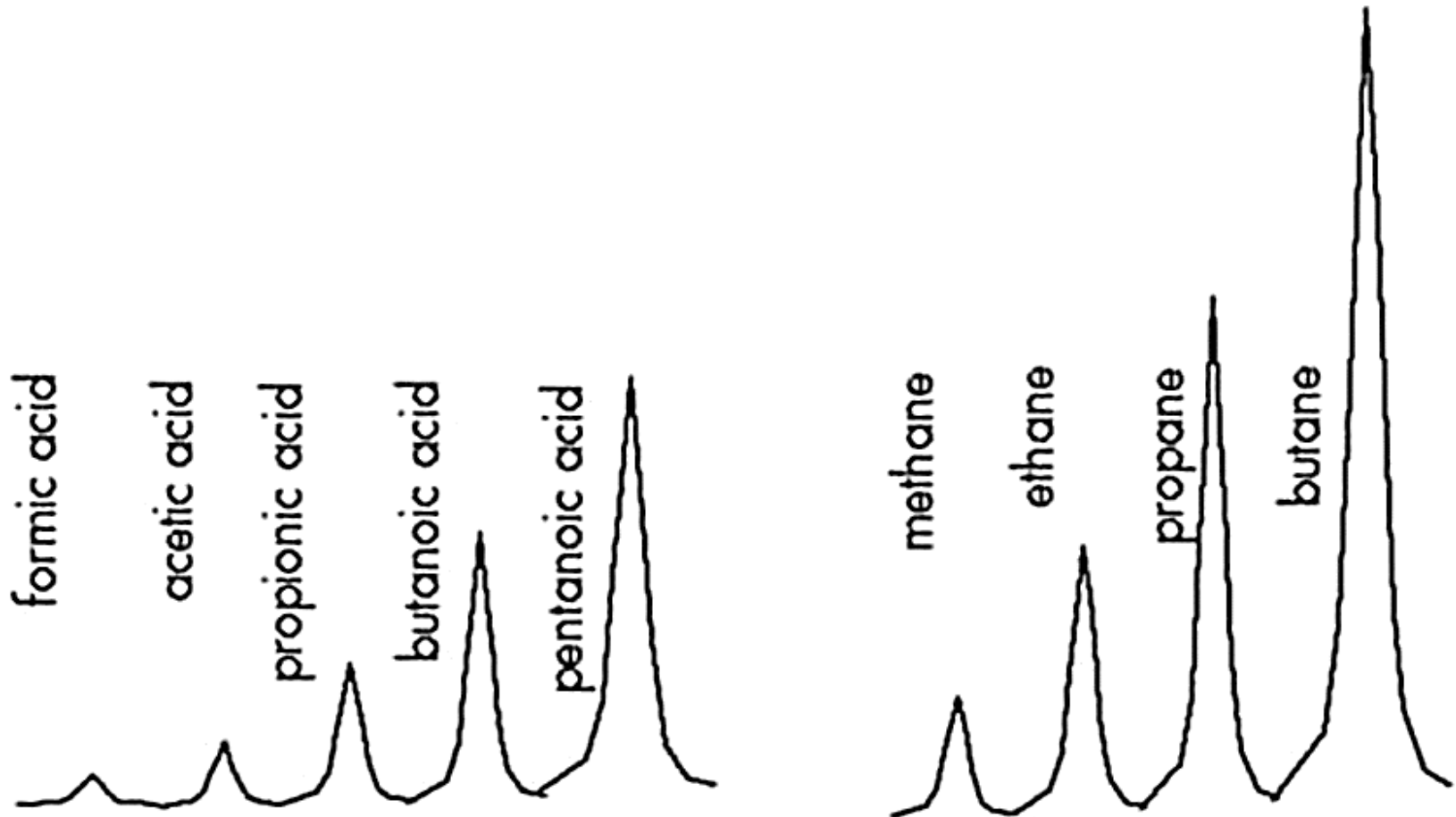
-Two electrodes are used to provide a potential difference. A large electrical potential is applied at the burner tip, and a collector electrode is located above the flame.

-The current resulting from the pyrolysis of any organic compound is measured which is proportional to the carbon content of the molecule entering.



# FID response

Response is based on the number of carbon and if other elements like halogens or oxygen are present which reduce combustion.



# Compounds with little or no FID response

-Noble gases (He, Ne, Ar, ... etc)

-NH<sub>3</sub>

-NO<sub>x</sub>

-H<sub>2</sub>O

-CO<sub>2</sub>

-CO

-CS<sub>2</sub>

-O<sub>2</sub>

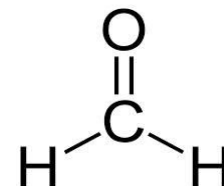
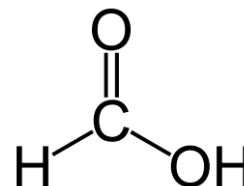
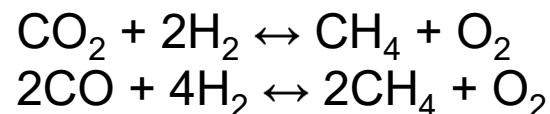
-N<sub>2</sub>

-Perhalogenated compounds, CHCl<sub>3</sub>, CCl<sub>4</sub>,  
chlorofluorocarbon (CFCs)

-Formic acid

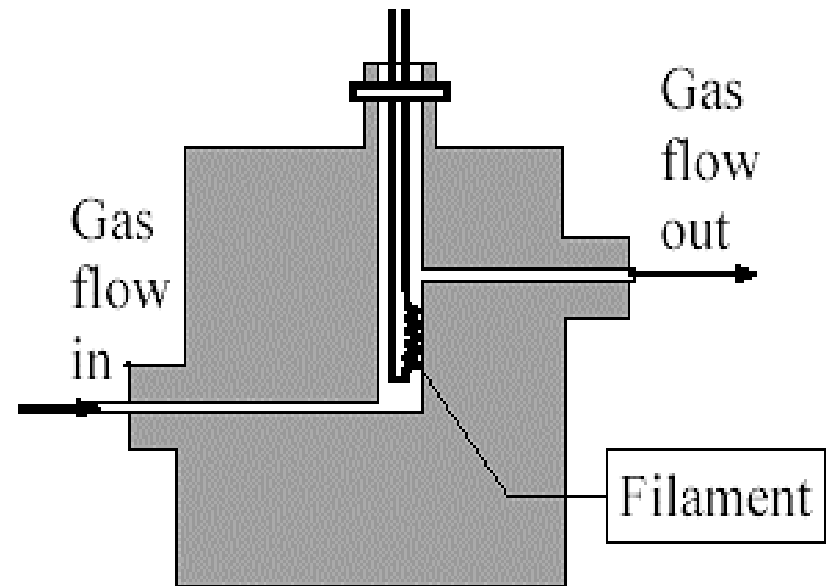
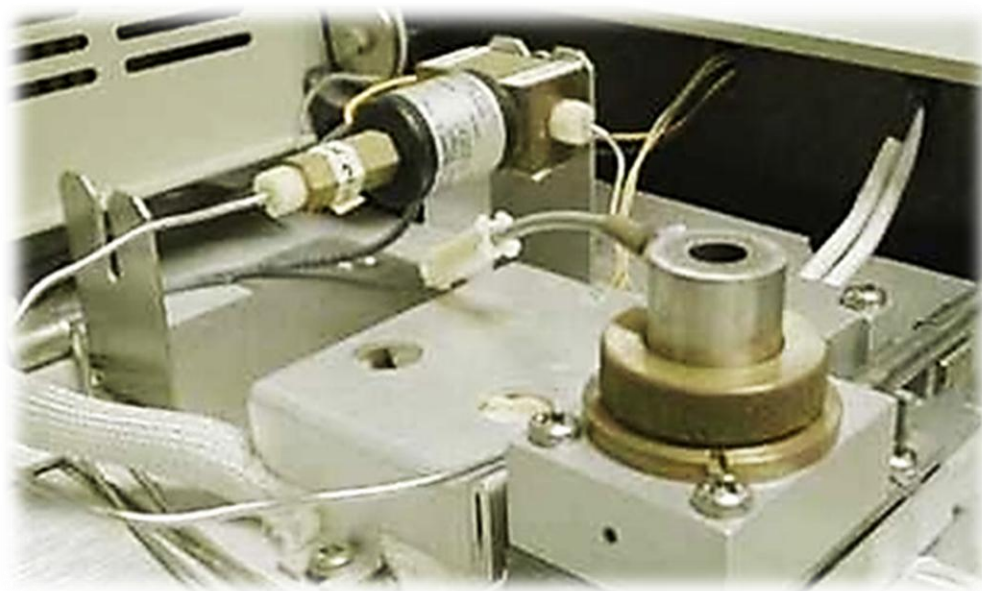
-Formaldehyde

In some systems, CO and CO<sub>2</sub> can be detected in the FID using a methanizer, which is a bed of Ni catalyst that reduces CO and CO<sub>2</sub> to methane, which can be in turn detected by the FID.



# Thermal Conductivity Detector (TCD)

The thermal conductivity detector (TCD), also known as a **Katharometer**, is a bulk property detector and a chemical specific detector commonly used in gas chromatography.



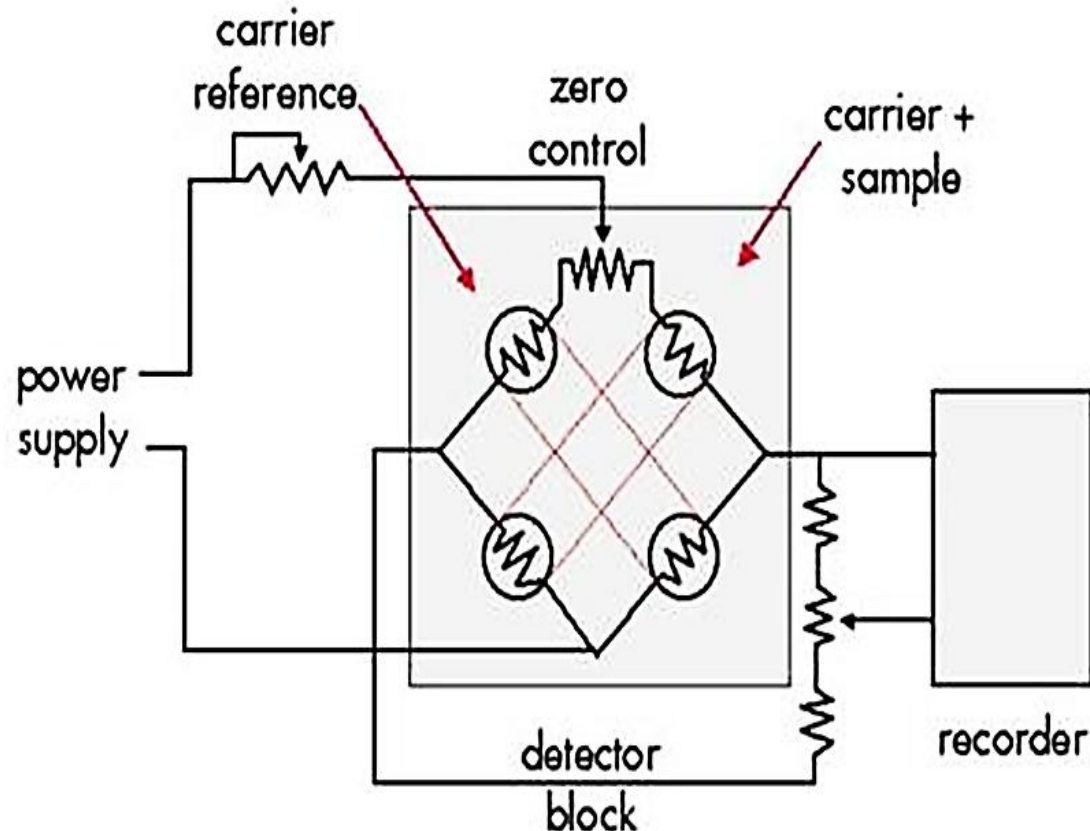
# Mode of detection

The TCD consists of an electrically heated filament in a temperature-controlled cell.

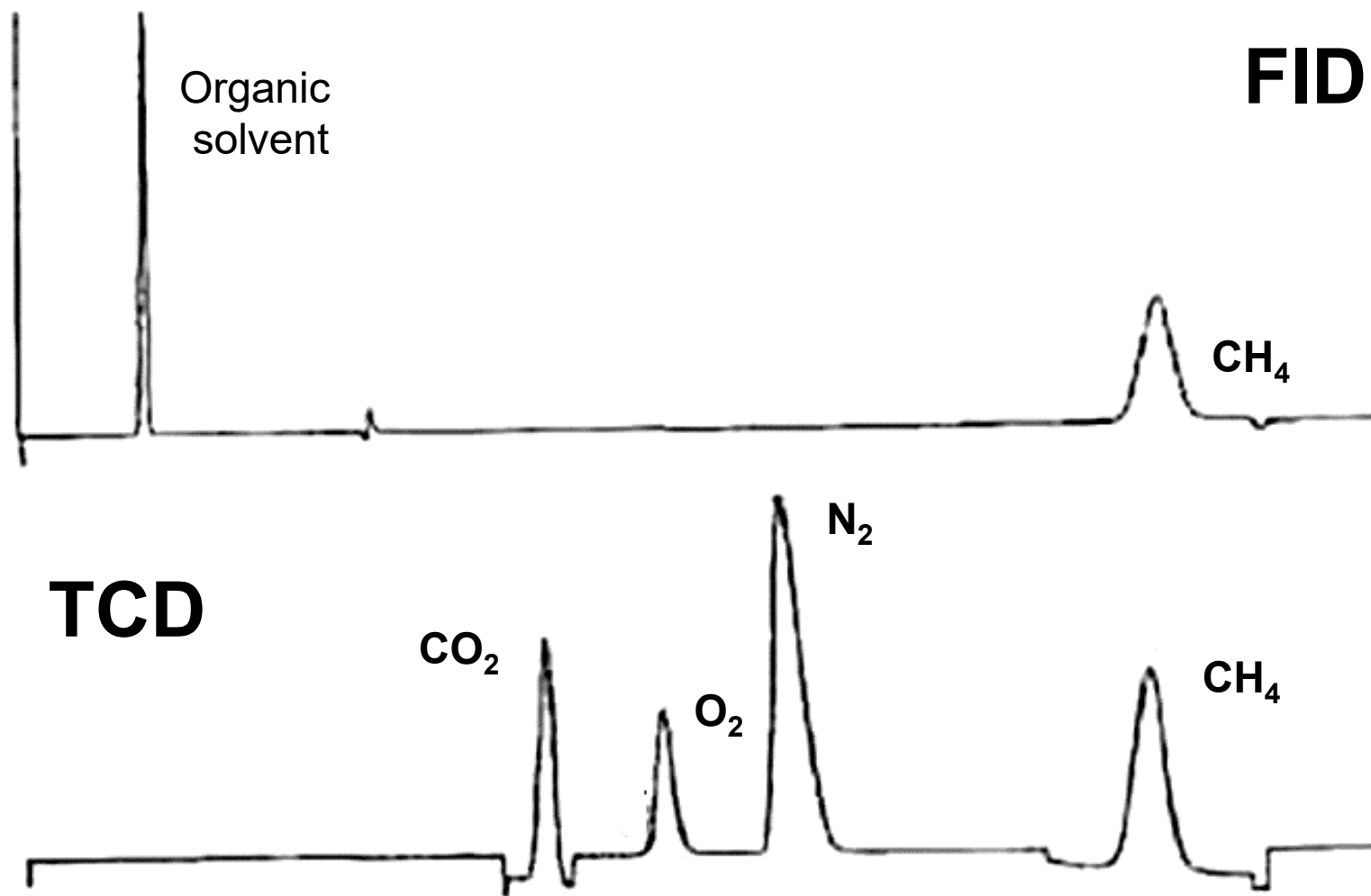
Change in resistance of a wire based on variations in the thermal conductivity of the gas evolving from a column.

This detector senses changes in the thermal conductivity of the column effluent and compares it to a reference flow of carrier gas.

Since most compounds have a thermal conductivity much less than that of the common carrier gases of He or H<sub>2</sub>, when an analyte elutes from the column the effluent thermal conductivity is reduced, and a detectable signal is produced.

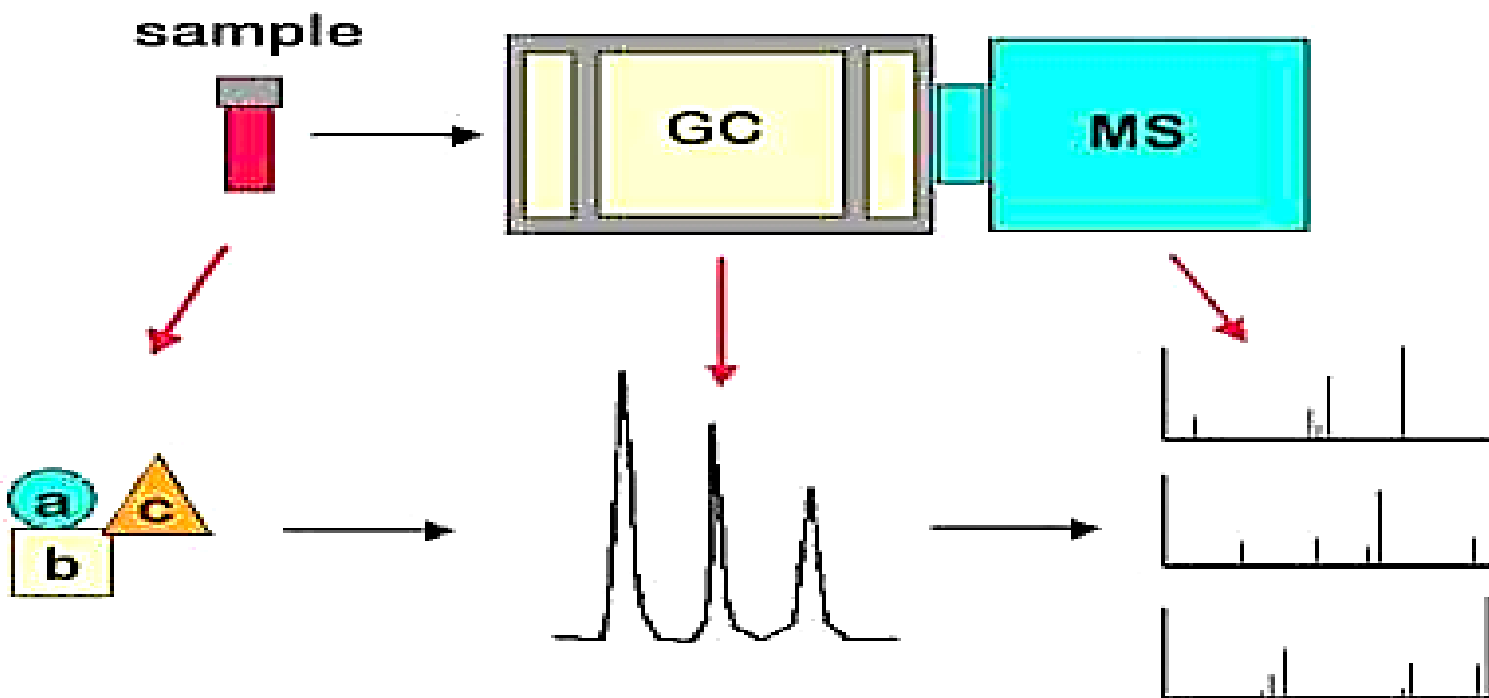


# TCD vs. FID response



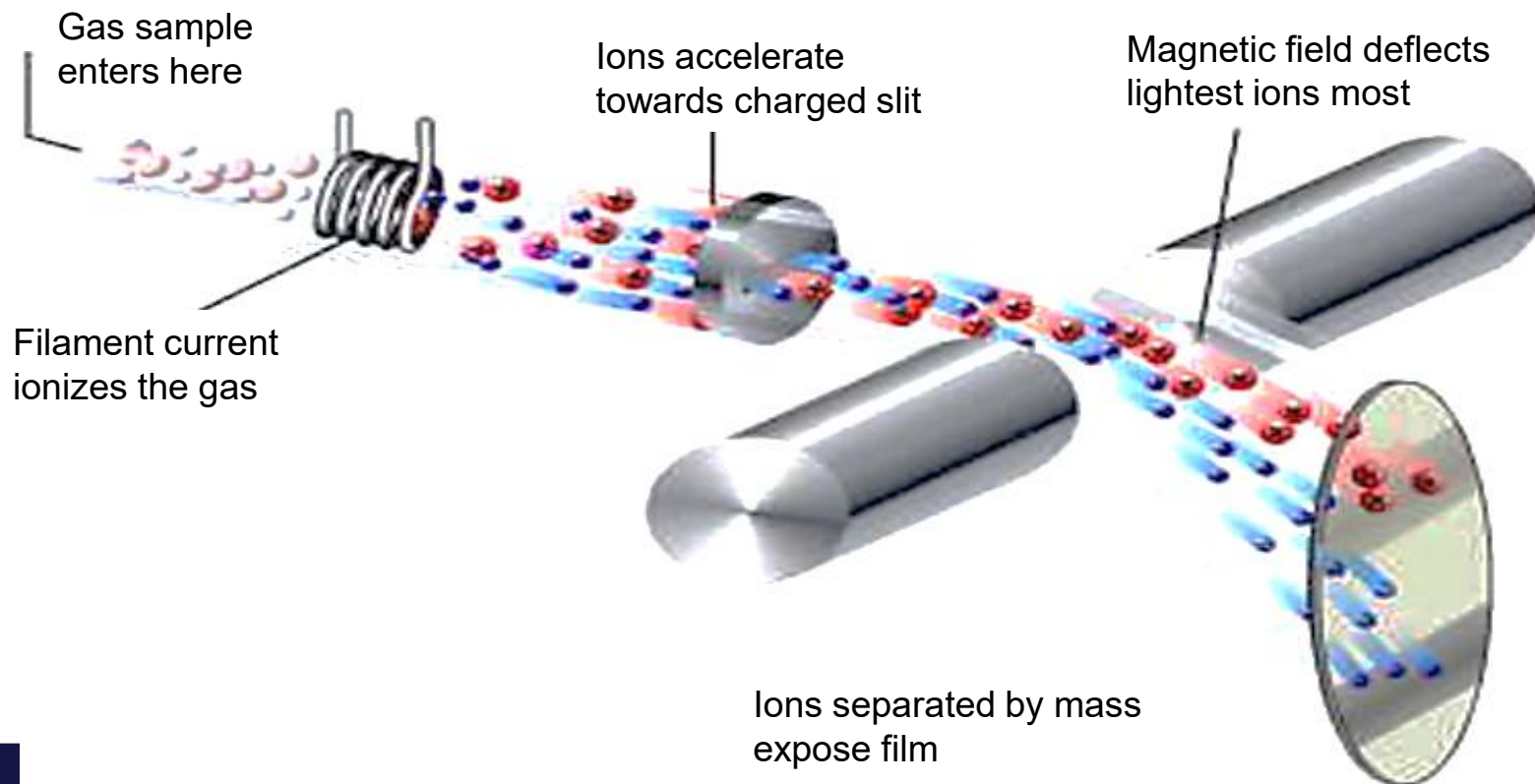
# Mass Spectrometry (GC-MS)

- Synergistic combination of two powerful analytical techniques.
- The GC separates the components of a mixture in time.
- The MS provides information that aids in the structural identification of each component.
- Uses the difference in mass-to-charge ratio ( $m/z$ ) of ionized atoms or molecules to separate them from each other.



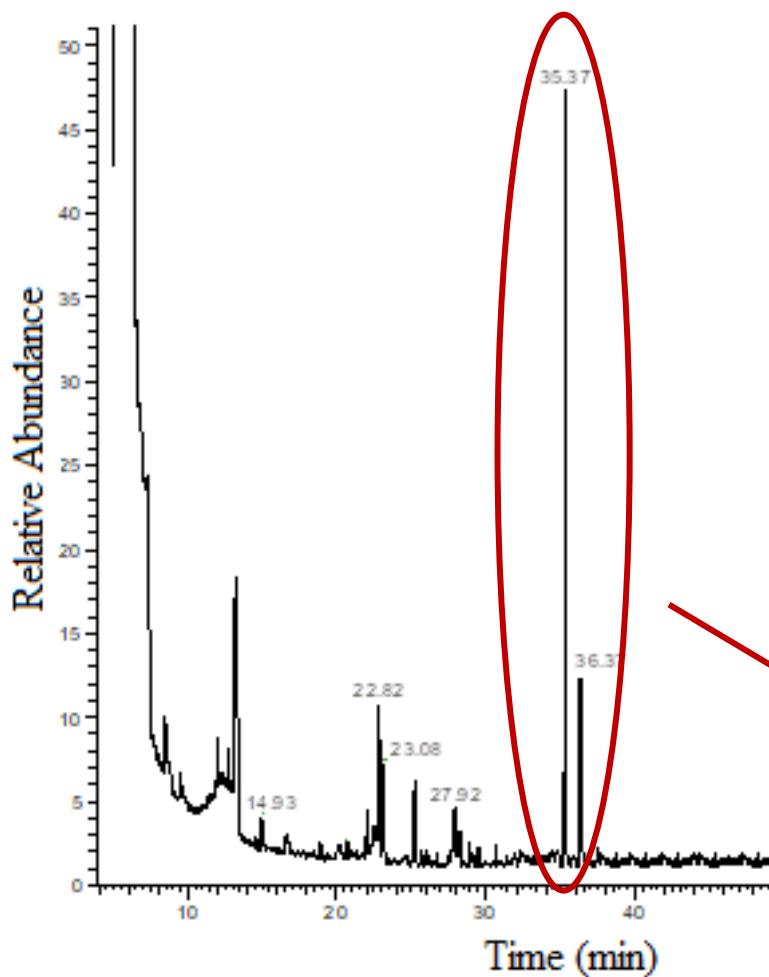
# The general operation of a mass spectrometer is:

- Result of the GC goes through an ionizer where it is bombarded by a high energy electron beam.
- This beam breaks the complex molecules into a standard set of fragments.
- The ionized samples then go through magnetic field which deflects ion according to mass to charge ratio.
- A detector picks up the fragments of a certain mass.
- Each peak of a chromatogram becomes a “fingerprint” of the compound.
- The fingerprints are compared with a library to identify the compounds.



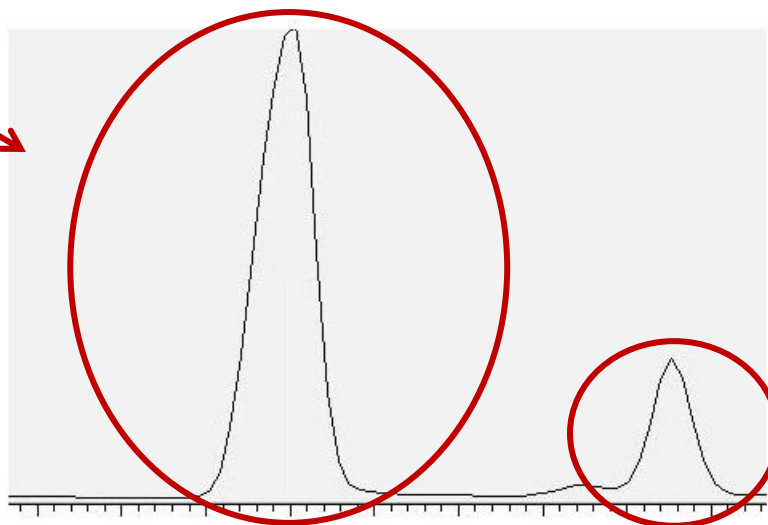


# GC-MS example



## GC-MS Conditions

|                   |                                              |
|-------------------|----------------------------------------------|
| Column            | DB-5MS (Agilent)<br>30 m × 0.25 mm × 0.25 μm |
| Carrier gas       | Helium                                       |
| Column flow       | 1.0 mL/min                                   |
| Injection mode    | Splitless mode (1 μL)                        |
| Oven temp.        | 70 °C (2 min) 20 °C/min, 230 °C              |
| Injector temp.    | 280 °C                                       |
| Interface temp.   | 280 °C                                       |
| Ion source temp.  | 230 °C                                       |
| Ionization energy | 70 eV (EI)                                   |



# GC-MS Library Search

1 (Spec. List) Caffeine

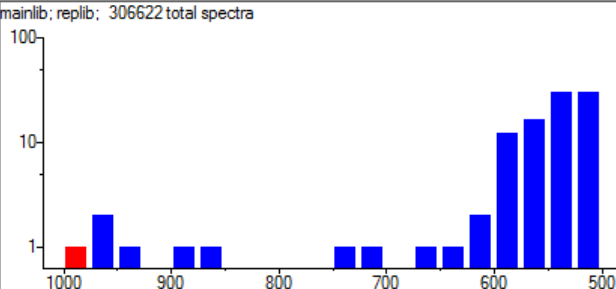
CN1C=NC2=C1C(=O)N(C)C(=O)N2C

2 (Spec. List) Theobromine

CN1C=NC2=C1C(=O)NC(=O)N2C

Names Structures Spec List

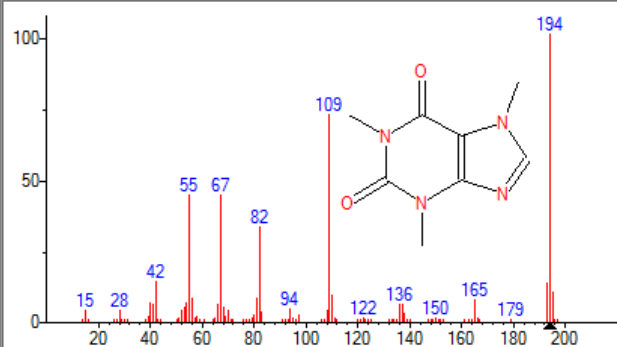
mainlib: replib: 306622 total spectra



| #  | Lib. | Match | R.Match | Prob. (%) | RI   | Syn | DBs | Name     |
|----|------|-------|---------|-----------|------|-----|-----|----------|
| 1  | M    | 999   | 999     | 98.8      | 1... | 65  | 9   | Caffeine |
| 2  | R    | 974   | 974     | 98.8      | 1... | 65  | 9   | Caffeine |
| 3  | R    | 952   | 956     | 98.8      | 1... | 65  | 9   | Caffeine |
| 4  | R    | 928   | 930     | 98.8      | 1... | 65  | 9   | Caffeine |
| 5  | R    | 882   | 882     | 98.8      | 1... | 65  | 9   | Caffeine |
| 6  | R    | 873   | 884     | 98.8      | 1... | 65  | 9   | Caffeine |
| 7  | R    | 748   | 810     | 0.98      | -    | 1   | 0   | 1,4-D.   |
| 8  | M    | 705   | 709     | 0.98      | -    | 1   | 0   | 1,4-D.   |
| 9  | M    | 656   | 679     | 0.08      | -    | 0   | 0   | 6-Ami.   |
| 10 | R    | 627   | 627     | 0.02      | 2... | 27  | 5   | Proxy.   |
| 11 | M    | 614   | 614     | 0.02      | 2... | 27  | 5   | Proxy.   |
| 12 | M    | 604   | 801     | 0.00      | -    | 0   | 0   | 2-Flu... |
| 13 | M    | 595   | 758     | 0.00      | -    | 0   | 0   | 4-Flu... |
| 14 | R    | 592   | 596     | 0.00      | 1... | 7   | 2   | 9-Tet.   |
| 15 | M    | 591   | 591     | 0.00      | -    | 0   | 0   | Dyph.    |
| 16 | M    | 591   | 591     | 0.00      | -    | 1   | 0   | Dode.    |
| 17 | M    | 590   | 603     | 0.00      | -    | 0   | 0   | 3,3'...  |
| 18 | M    | 589   | 603     | 0.00      | -    | 2   | 0   | 2H-1.    |
| 19 | R    | 586   | 592     | 0.00      | 1... | 0   | 0   | 1,8-D.   |
| 20 | M    | 585   | 588     | 0.00      | -    | 0   | 0   | Valer    |

Names Structures InLib = 856, Hit List

(Spec. List) Caffeine



Plot/Text of Search Spectrum

Plot of Search Spectrum

Spec List

Name: Caffeine

Formula: C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>

MW: 194 Exact Mass: 194.080376 CAS#: 58-08-2 NIST#: 290714 ID#: 1 DB: S

Other DBs: Fine, TSCA, RTECS, EPA, USP, HODOC, NIH, EINECS, IRDB

Comment: NIST Mass Spectrometry Data Center, 1998.

Related CAS#: 71701-02-5; 95789-13-2

InChIKey: RYYVLZVUVJVGH-UHFFFAOYSA-N Non-stereo

10 largest peaks:

194 999 | 109 721 | 55 439 | 67 438 | 82 328 |

42 138 | 193 135 | 195 103 | 110 92 | 81 81 |

Synonyms:

1. 1H-Purine-2,6-dione, 3,7-dihydro-1,3,7-trimethyl-

2. Alert-Pep

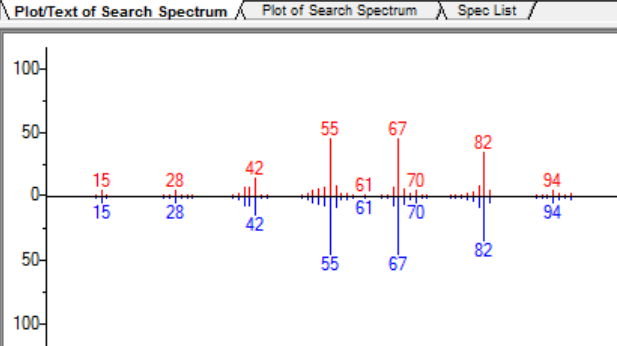
3. Caffeina

4. Caffein

5. Caffeine

6. Cafipel

(mainlib) Caffeine



Plot/Text of Hit

Plot of Hit

Difference

Head to Tail

Side by Side

Subtraction

Caffeine

Head to Tail MF=999 RMF=999

Caffeine

999 999R 98.8P 1835RI

Name: Caffeine

Formula: C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>

MW: 194 Exact Mass: 194.080376 CAS#: 58-08-2 NIST#: 290714 ID#: 198382

Other DBs: Fine, TSCA, RTECS, EPA, USP, HODOC, NIH, EINECS, IRDB

Contributor: NIST Mass Spectrometry Data Center, 1998.

Related CAS#: 71701-02-5; 95789-13-2

InChIKey: RYYVLZVUVJVGH-UHFFFAOYSA-N Non-stereo

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Synonyms:

1. 1H-Purine-2,6-dione, 3,7-dihydro-1,3,7-trimethyl-

2. Alert-Pep

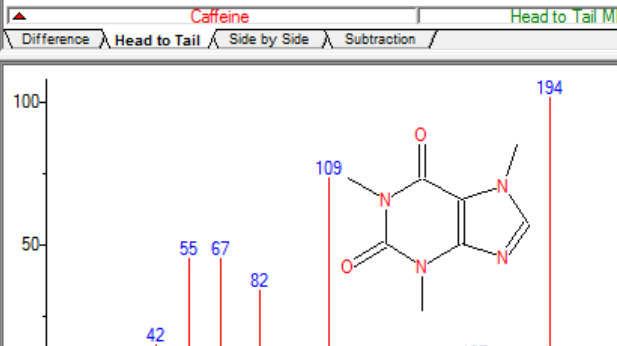
3. Caffeina

4. Caffein

5. Caffeine

6. Cafipel

(mainlib) Caffeine



Plot/Text of Hit

Plot of Hit

Lib. Search

Other Search

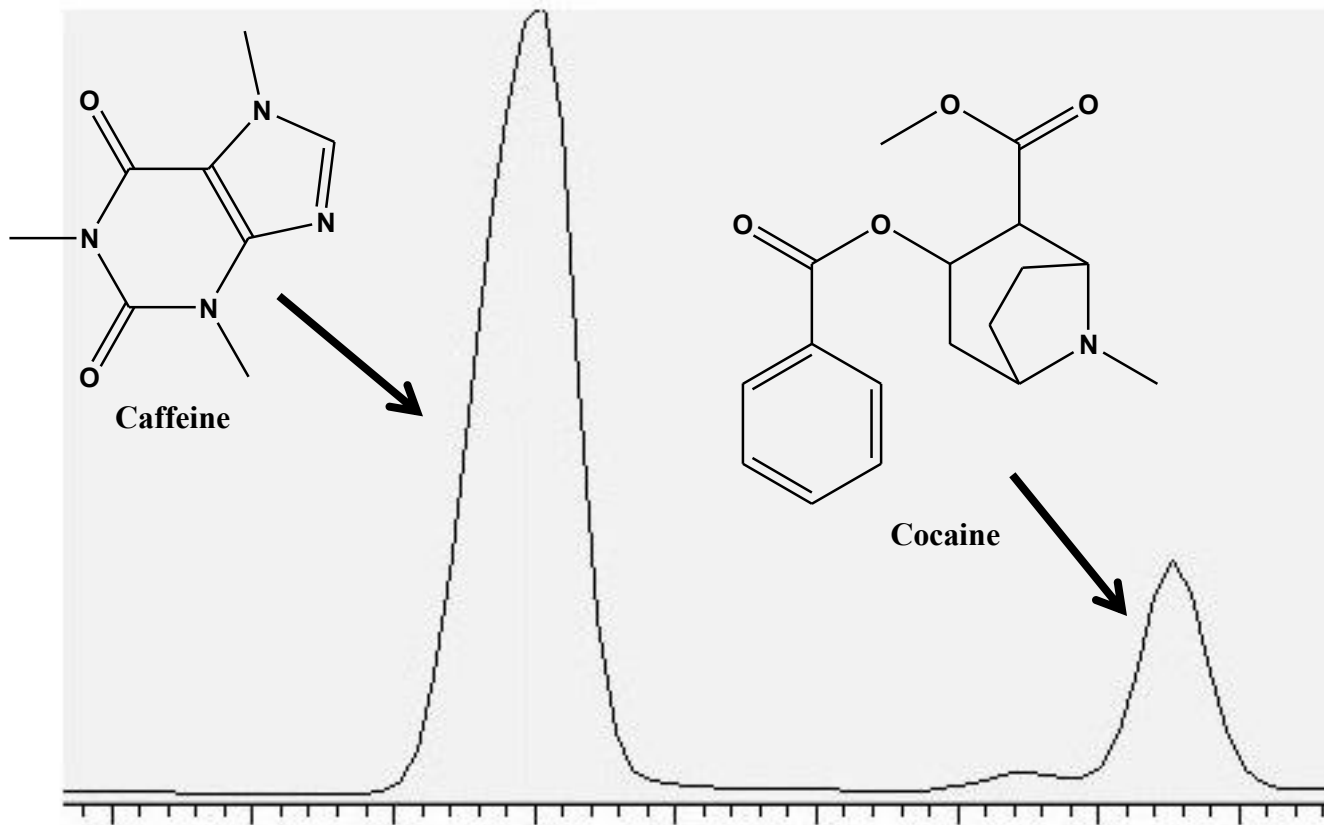
Names

Compare

Librarian

MSMS

NIST library search



- Identification of the mixtures constituents
- Check the purity of the compound

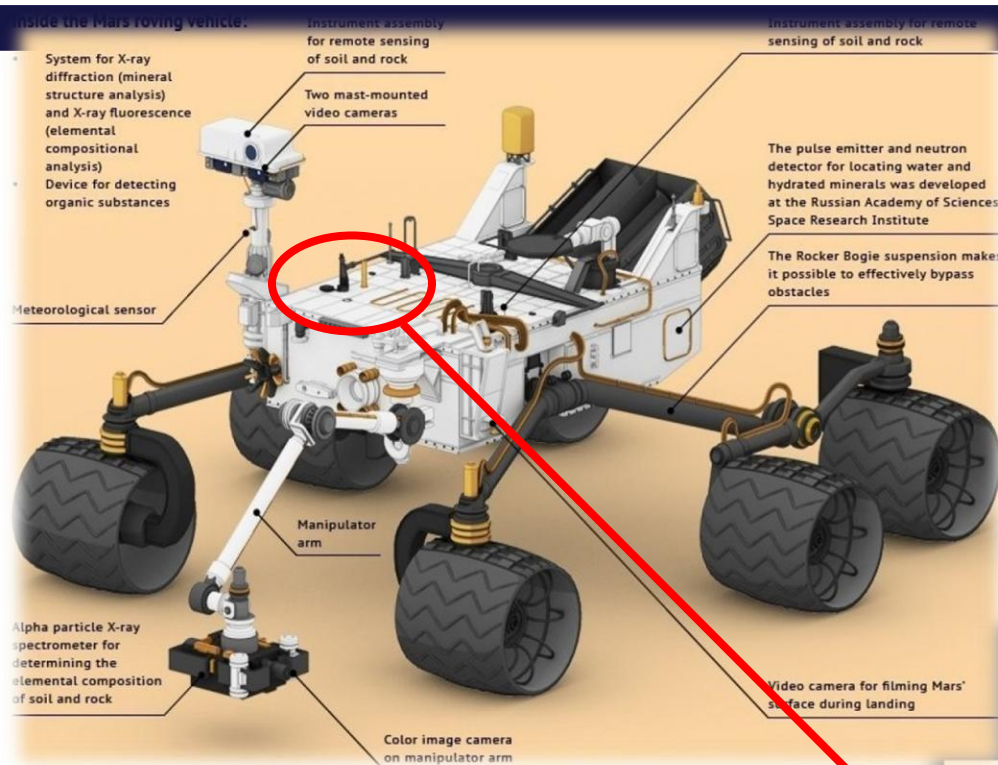
# GC detectors; applications and sensitivity ranges

| Gas Chromatographic Detectors    |                                                    |                                     |
|----------------------------------|----------------------------------------------------|-------------------------------------|
| Type                             | Applicable Samples                                 | Typical Detection Limit             |
| Flame ionization                 | Hydrocarbons                                       | 0.2 pg/s                            |
| Thermal conductivity             | Universal detector                                 | 500 pg/mL                           |
| Electron capture                 | Halogenated compounds                              | 5 fg/s                              |
| Mass spectrometer                | Tunable for any species                            | 0.25–100 pg                         |
| Thermionic                       | Nitrogen and phosphorous compounds                 | 0.1 pg/s (P)<br>1 pg/s (N)          |
| Electrolytic conductivity (Hall) | Compounds containing halogens, sulfur, or nitrogen | 0.5 pg Cl/s<br>2 pg S/s<br>4 pg N/s |
| Photoionization                  | Compounds ionized by UV radiation                  | 2 pg C/s                            |
| Fourier transform IR             | Organic compounds                                  | 0.2 to 40 ng                        |

# Factors Influencing the GC Separation

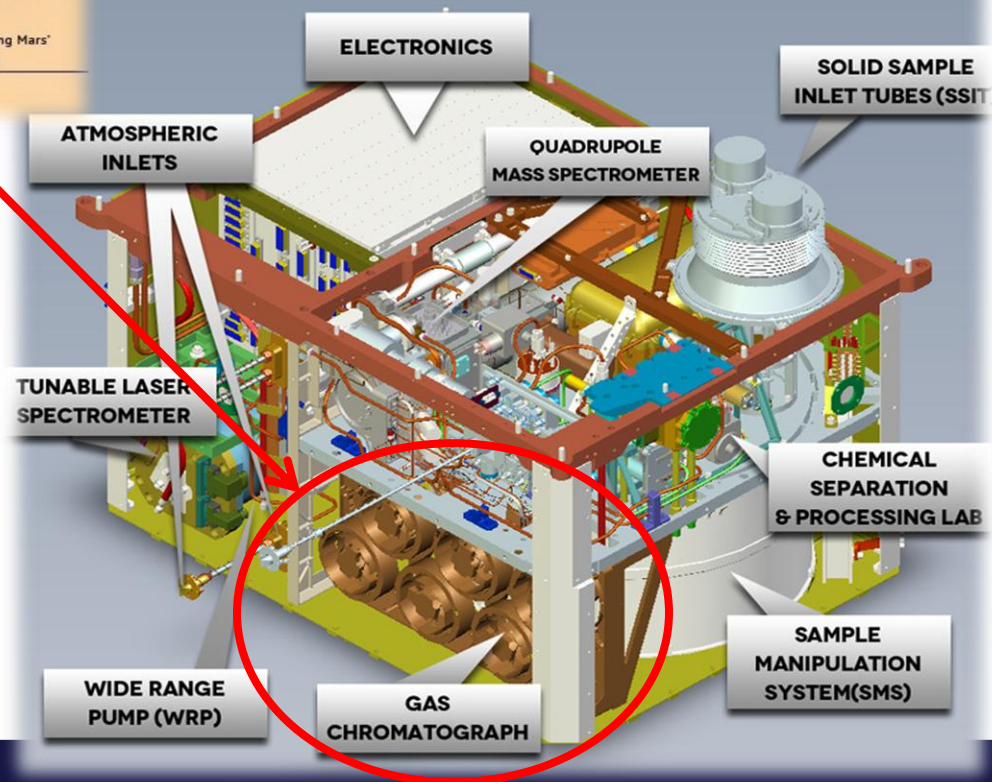
## The major interrelated factors to consider

- Carrier gas type and purity
- Carrier gas velocity (flow rate)
- Injection method (manual, automatic, speed, syringe, headspace, SPME, ...)
- Injector temperature
- Injection mode (split/splitless)
- Injection volume
- Column stationary phase
- Column length
- Column internal diameter
- Film thickness for stationary phase (open tubular columns)
- Column (oven) temperature; isothermal or temperature program
- Pressure
- Type of detector
- Detector temperature
- Detector conditions



The curiosity rover  
Landing on Mars: Aug 2012

## The Sample Analysis at Mars (SAM) instrument







*Thank You!*

