Experiment-10

Gas Chromatography

الدكتوره قرأت قراءه تم اللجوء الى chat GPT

Ayaayyash

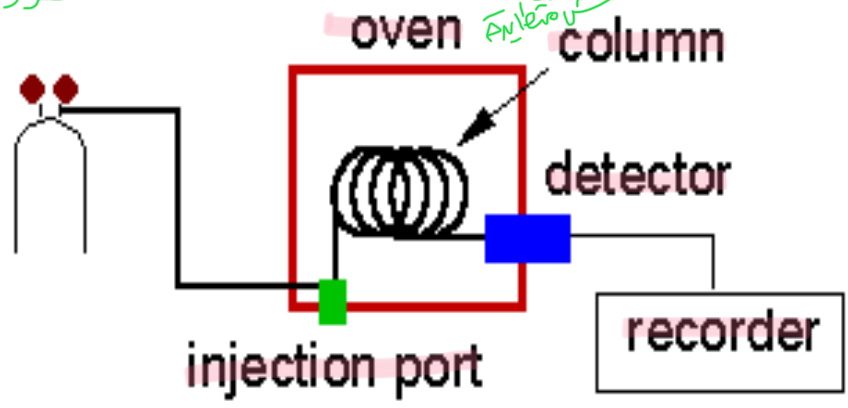
Gas Chromatography (GC)

الكروماتوغرافيا الغازية (Gas Chromatography) هي تقنية تحليلية تُستخدم لفصل المركبات العضوية المتطايرة (volatile organic compounds) بناءً على خصائصها الفيزيائية والكيميائية، مثل القطب والتفاعل مع الأطوار المختلفة

Gas chromatography is a chromatographic technique that can be used to separate volatile organic compounds.

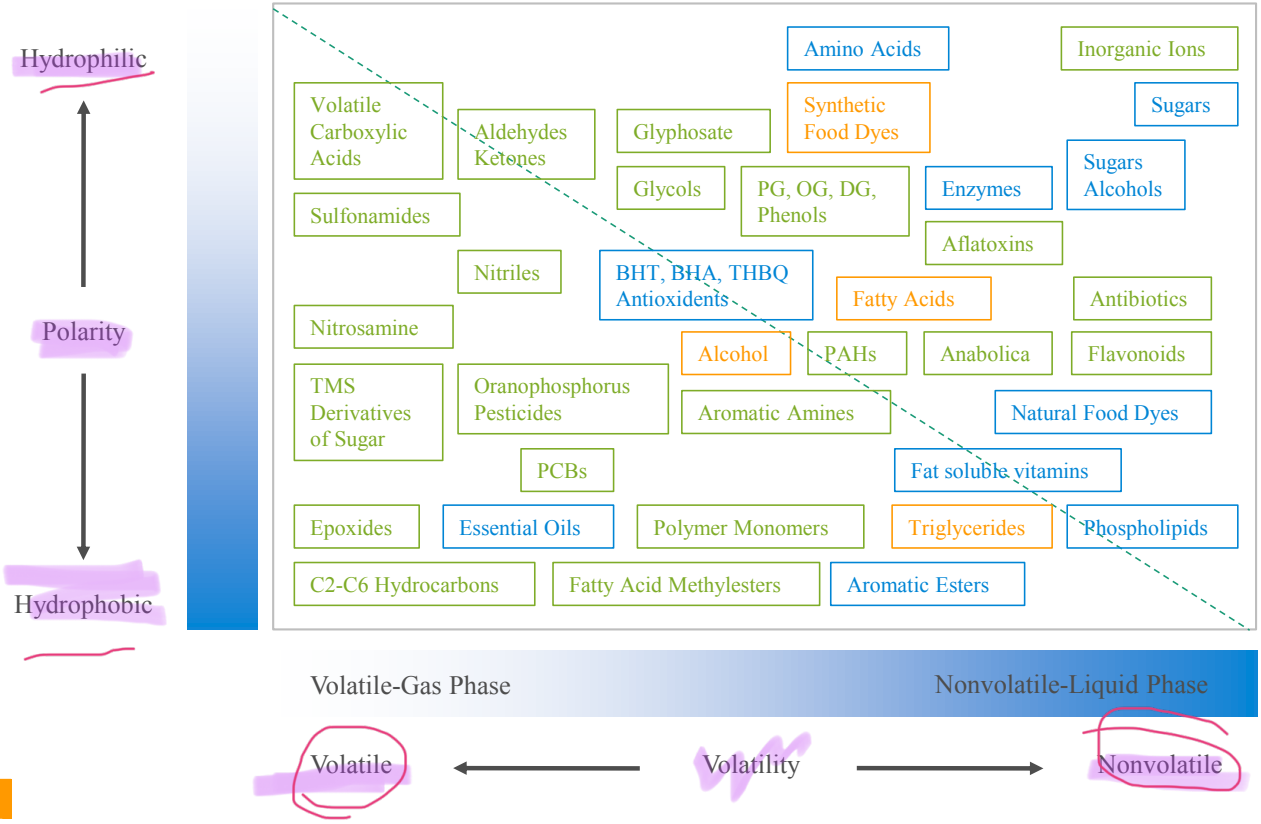
It consists of:

- ✓ a flowing mobile phase
- ✓ an injection port
- ✓ a separation column (the stationary phase)
- ✓ an oven
- ✓ a detector.



Introduction

Which Separation Technique for Which Compound?



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Introduction

What Is Gas Chromatography?

- Gas chromatography (GC) is a technique to separate the individual components of a given mixture so that each can be identified and quantified.
- To be suitable for GC analysis a compound must have sufficient volatility and thermal stability.
- If all or some of the components of a sample are volatile at around 400°C or below, and do not decompose at these temperatures, the compound can probably be analyzed using a gas chromatograph.
- The instrument vaporizes a sample of the compound and transports it via a carrier gas into a column. The components of the sample travel through the column at varying rates depending on their physical properties.
- The eluted components enter a heated detector that generates an electronic signal based on its interaction with the component.
- A data system records the size of the signal and plots it against elapsed time to produce a chromatogram.

شروط التحليل في GC:
التطاير الكافي (Volatility): يجب أن يكون المركب قابلاً للتبخر عند درجات حرارة تصل إلى حوالي 400 درجة مئوية.
الثبات الحراري (Thermal Stability): لا يجب أن يتحلل المركب عند درجة الحرارة المستخدمة في التحليل.

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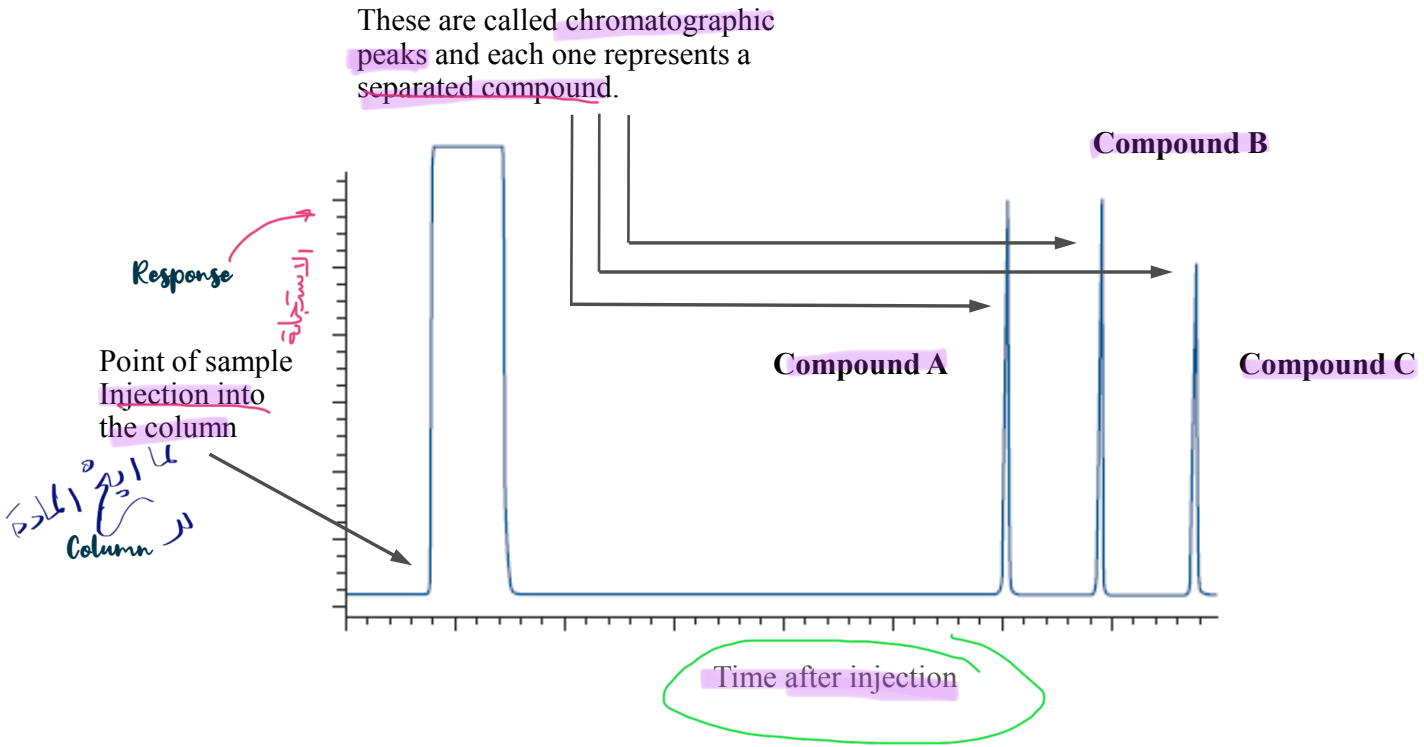
Introduction

What Does a Chromatogram Look Like?



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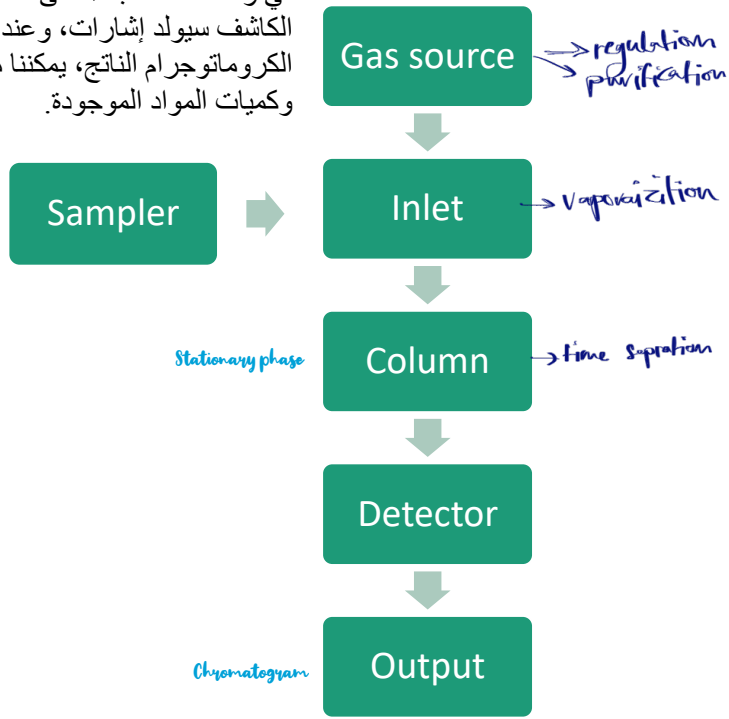
Configuration of a GC System

General Overview

إذا أدخلنا خليطًا يحتوي على مادتين في الجهاز، فإن كل مادة ستخرج من العمود في وقت مختلف بناءً على خصائصها. الكاشف سيولد إشارات، وعند تحليل الكروماتوجرام الناتج، يمكننا معرفة نوع وكميات المواد الموجودة.

- A gas chromatograph consists of
- A regulated and purified carrier gas source, which moves the sample through the instrument
- An inlet, which also acts as a vaporizer for liquid samples
- A column, in which the time separation occurs
- A detector, which responds to the components as they elute from the column by changing its electrical output
- Output: Data interpretation of some sort



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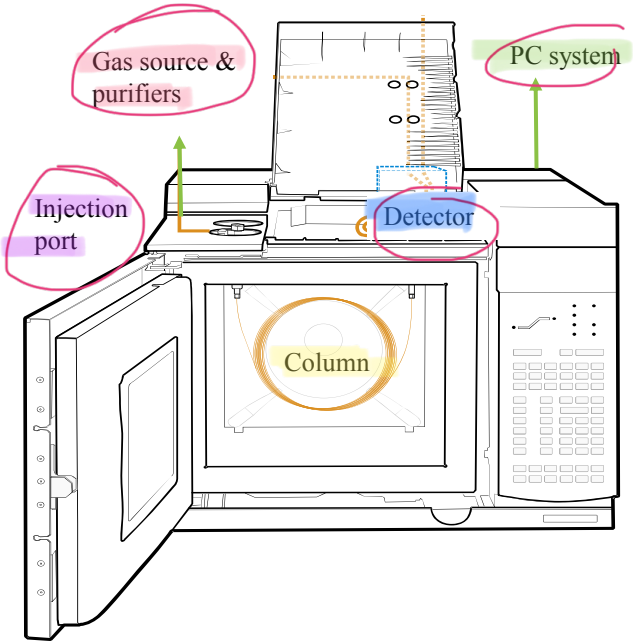
Configuration of a GC System



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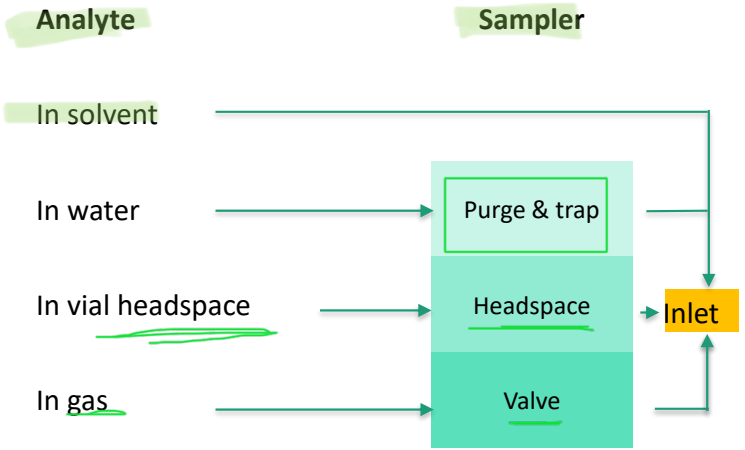
Configuring a GC System

The Sampler

- The choice of the sampler depends on the analyte matrix.



GC autosampler

GC headspace sampler

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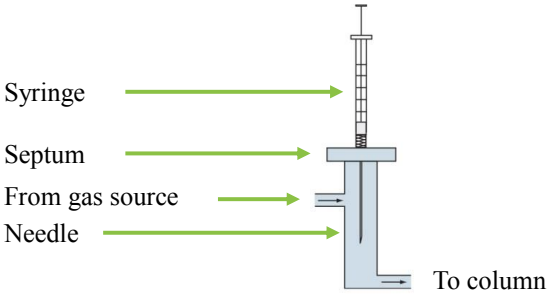
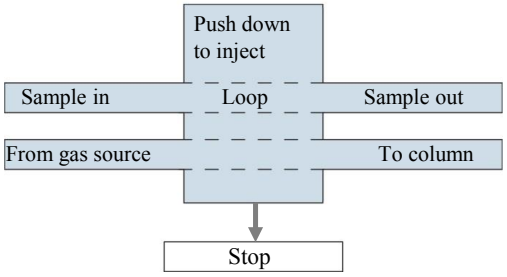
Configuring a GC System

The Inlet

- The inlet introduces the vaporized sample into the carrier gas stream. The most common inlets are injection ports and sampling valves.
- Injection ports
  - Handle gas or liquid samples
  - Often heated to vaporize liquid samples
- Liquid or gas syringes are used to insert the sample through a septum into the carrier gas stream.
- Sampling valves
- The sample is flushed from a loop that is mechanically inserted into the carrier gas stream. Different valves are used for liquids and gases due to different sample volumes



Scheme of injection port



Scheme of sampling valves

Source: Fundamentals of Gas Chromatography
Publication #: G1176-90000

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Configuring a GC System

The Different Inlet Types

Split / Splitless

- This is the most common inlet
- In splitless mode, all the sample goes on column
- Inlet heated to vaporize sample

اكثر استخدام ببخر الماده اذا كانت سائله بوديها على splitless ← column ال

Cool-on-Column

- Whole sample introduced directly into column
- High precision
- Eliminates sample discrimination
- Eliminates sample degradation

كل العينه بتروح على ال column كثير دقيقه بتزيل العينه بس تميزها وتكسرها

Programmable Temperature

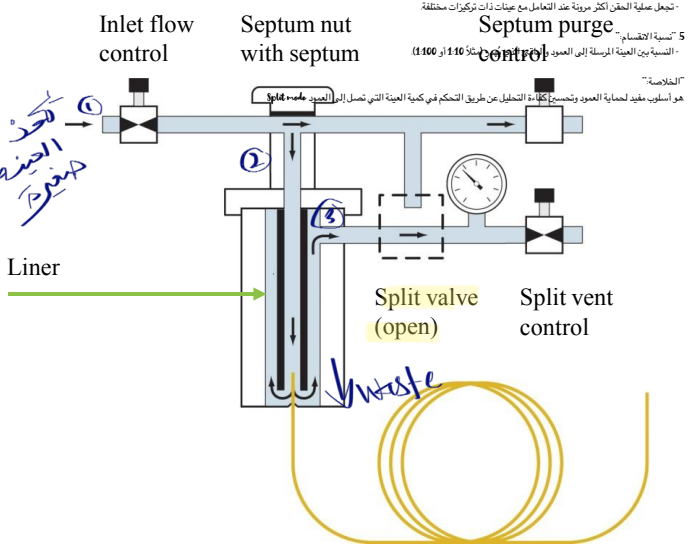
- Sample injected into cool liner
- Inlet heated to vaporize sample

العينه بتنحقن بال cool liner بتدخل بالمدخل التسخين عشان تتبخر

Configuring a GC System

The Different Inlet Types – Split/Splitless Port

- Split mode
- Capillary columns have low sample capacities. Small sample sizes (µl) must be used to avoid overloading the column.
- The split mode provides a way to inject a larger sample, vaporize it, and then transfer only a part of it to the column. The rest is vented as waste.
- The split valve remains open. The sample is injected into the liner, where it vaporizes. The vaporized sample divides between the column and the split vent.



A typical split/splitless port in split mode.

Source: Fundamentals of Gas Chromatography
Publication #: G1176-90000

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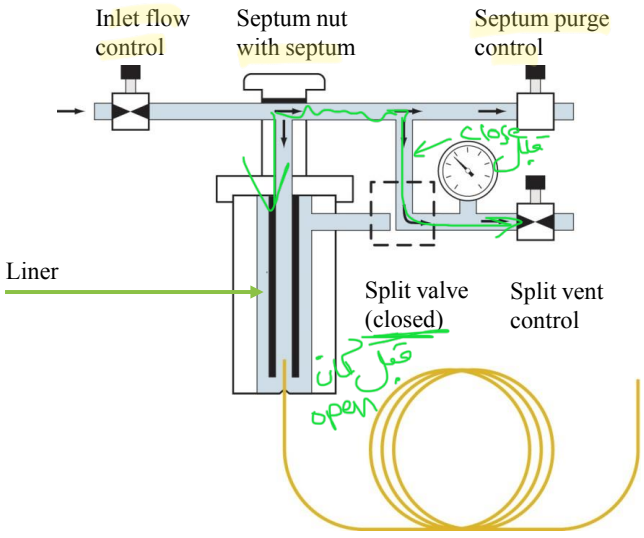
Configuring GC System

The Different Inlet Types – Split/Splitless Port

- Splitless mode
- This mode is well suited to low concentration samples. It traps the sample at the head of the column while venting residual solvent vapor.
- Step 1: Split valve closed, sample injected. The solvent (the major component) creates a saturated zone at the head of the column, which traps the sample components.
- Step 2: Once the sample is trapped on column, open the split valve. The residual vapor in the inlet, now mostly solvent, is swept out the vent.
- The flows are now the same as in the split mode.



Splitless mode in injection.

Source: Fundamentals of Gas Chromatography
Publication #: G1176-90000

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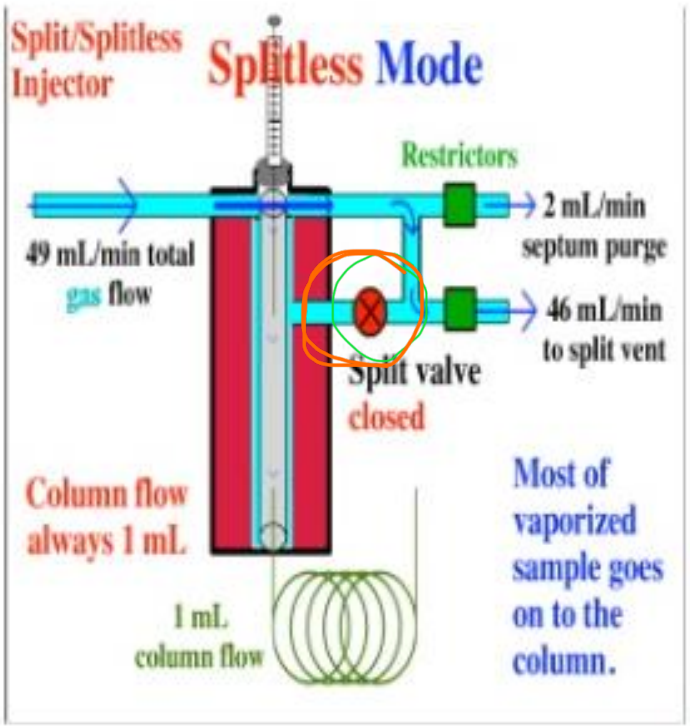
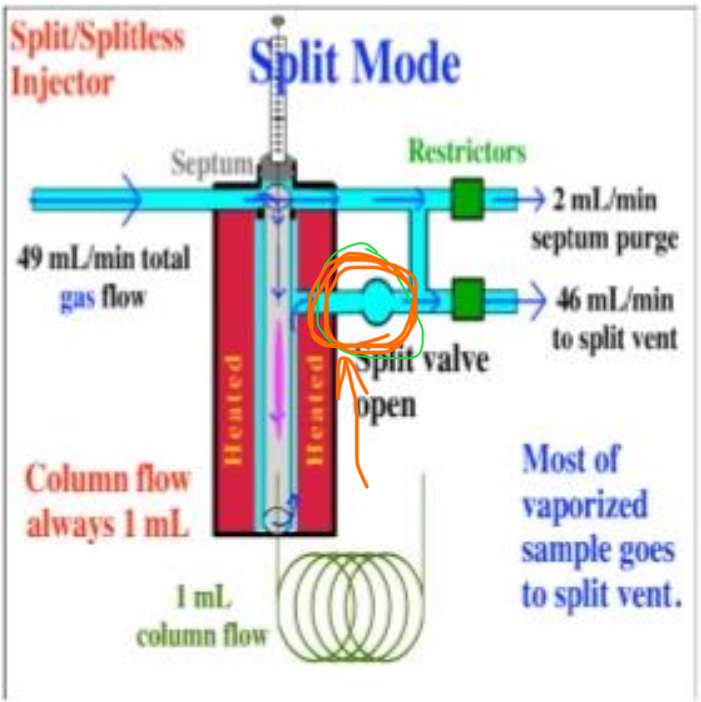
GC – Split/Splitless Injections

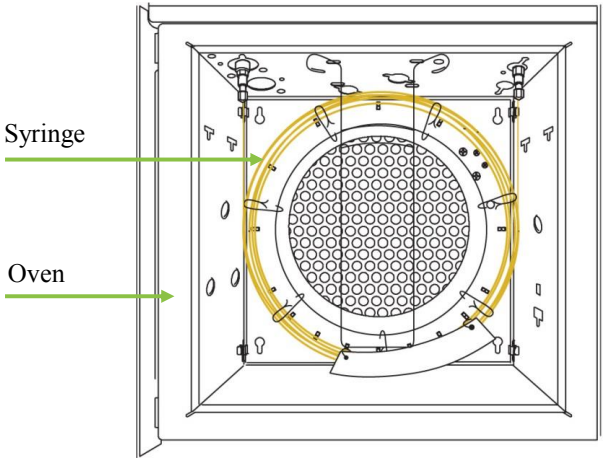




Configuring a GC System

The Column

- The separation happens here.
- Most separations are highly temperature-dependent, so the column is placed in a well-controlled oven.
- The sample vapor is directed into a column by a carrier gas. Compounds selectively partition between stationary phase (coating) and mobile phase (carrier gas).
- The oven temperature may be ramped to elute all compounds.
  - Isothermal: temperature stays the same for run
  - Ramped: temperature is raised during run



Column and oven

Source: Fundamentals of Gas Chromatography
Publication #: G1176-90000

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- "الفصل في GC": "يعتمد على حركة المركبات بين "المرحلة الثابتة" (داخل العمود) و"المرحلة المتنقلة" (غاز الناقل).
- "الحرارة": "العمود داخل فرن يُسخّن لفصل المركبات حسب درجة غليانها.
- "وضعان للحرارة":
  1. "متساوي الحرارة": "حرارة ثابتة، مناسب لمركبات قريبة الغليان.
  2. "منحدر": "حرارة ترتفع تدريجيًا، مناسب لمركبات مختلفة الغليان.

GC Columns

Packed columns

- Typically a glass or stainless steel coil.
- 1-5 total length and 5 mm inner diameter.
- Filled with the Stationary phase or a packing coated with the Stationary phase.

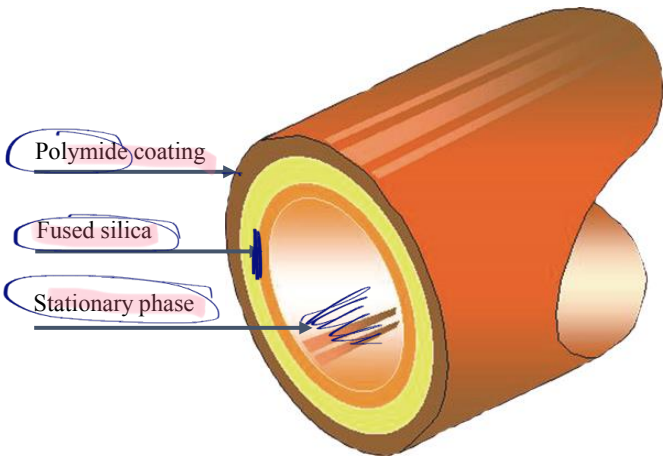
Capillary columns

- Thin fused-silica.
- Typically 10-100 m in length and 250 μm inner diameter.
- Stationary phase coated on the inner surface.
- Provide much higher separation efficiency
- But more easily overloaded by too much sample.

Configuring a GC System

Inside a Capillary Column

- A capillary GC column is composed of narrow tubing (0.05 to 0.53 mm ID) with a thin polymer coating (0.1 – 10.0 µm) inside.
- Selecting the right capillary column is critical and depends on factors such as selectivity, polarity, and phenyl content.
- Column diameter influences efficiency, solute retention, head pressure, and carrier gas flow rate.
- Column length affects solute retention, head pressure, bleeding, and costs).



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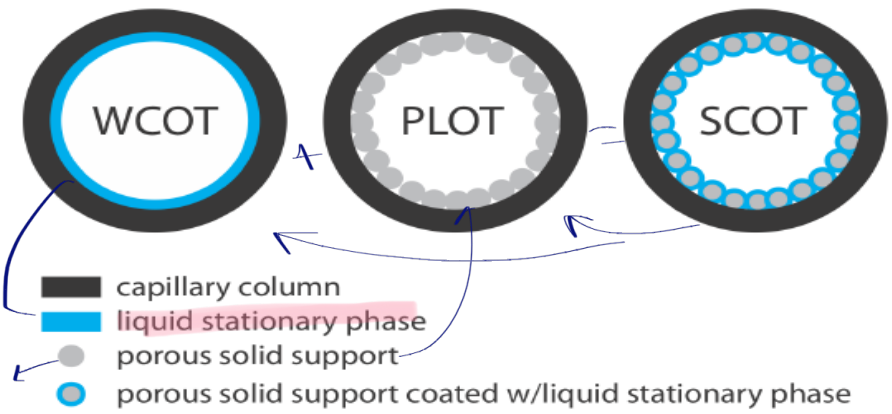
Capillary columns are of three principal types.
In a ***wall-coated open tubular column*** (WCOT) a thin layer of stationary phase, typically 0.25 nm thick,
is coated on the capillary's inner wall.
In a ***porous-layer open tubular column*** (PLOT), a porous solid support—alumina, silica gel, and molecular sieves are typical examples—is attached to the capillary's inner wall
. A ***support-coated open tubular column*** (SCOT) is a PLOT column that includes a liquid stationary phase. Figure 12.4.4 shows the differences between these types of capillary columns.



Configuring a GC System

The Detector

- The gas stream from the column, which contains the separated compounds, passes through a detector. The output from the detector becomes the chromatogram.
- Several detector types are available but all perform the same tasks:
  - Produce a stable electronic signal (the baseline) when pure carrier gas (no components) is in the detector
  - Produce a different signal when a component is passing through the detector.

GC detector

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Configuring a GC System

Common Detectors

Thermal conductivity detector	• Detects compounds with thermal conductivity that differs from carrier gas
Flame ionization detector	• Detects compounds that burn or ionize in a flame
Electron capture detector	• Detects electron-capturing compounds (for example, halogenated compounds)
Nitrogen-phosphorus detector	• Detects compounds that contain nitrogen and phosphorus
Flame photometric detector	• Detects compounds that contain sulfur and phosphorus
Atomic emission detector	• Tunable to many elements
Mass selective detector	• Identifies components from mass spectra (when combined with GC, the most powerful identification tool available)

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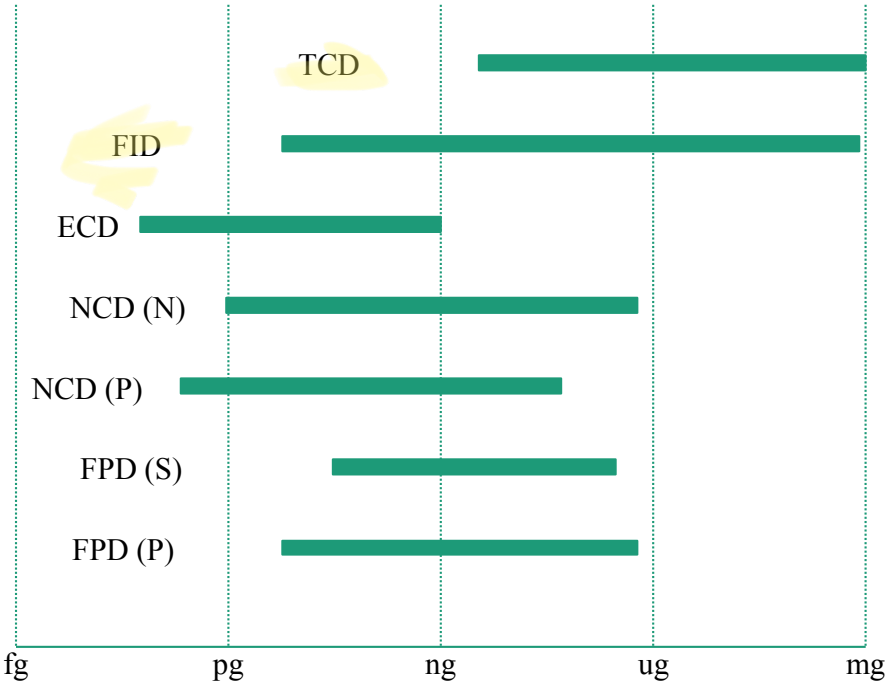
Configuring a GC System

Detector Sensitivity



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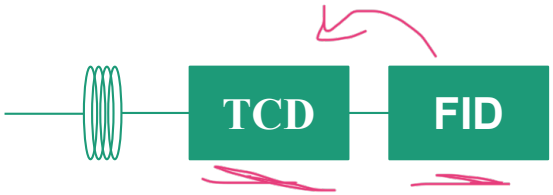
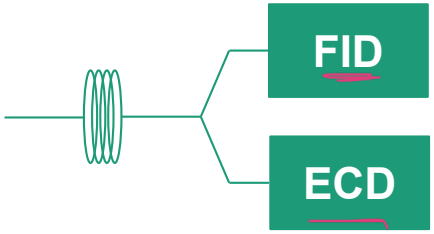
Detector Arrangement



Serial

Place non-destructive detector before other detector



Parallel

Split column effluent to different detectors

Thermal Conductivity Detector (TCD) and Flame Ionization Detector (FID) detectors are the two most common detectors on commercial GCs.

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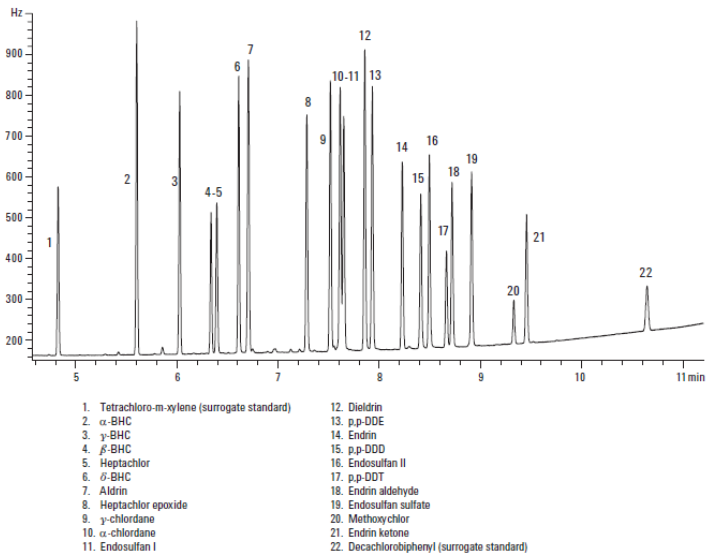
Configuring a GC System

GC Output

- The chromatogram plots abundance against time.
- Peak size corresponds to the amount of compound in the sample. As the compound`s concentration increases, a larger peak is obtained.
- Retention time (t_R) is the time it takes of a compound to travel through the column.
- If the column and all operating conditions are kept constant, a given compound will always have the same retention time.



1. Tetrachloro-m-xylene (surrogate standard)
2. α-BHC
3. γ-BHC
4. β-BHC
5. Heptachlor
6. δ-BHC
7. Aldrin
8. Heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. Endosulfan I
12. Dieldrin
13. p,p-DDE
14. Endrin
15. p,p-DDD
16. Endosulfan II
17. p,p-DDT
18. Endrin aldehyde
19. Endosulfan sulfate
20. Methoxychlor
21. Endrin ketone
22. Decachlorobiphenyl (surrogate standard)

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The Capabilities of GC

Key Points to Remember

Strengths

- Easy to use
- Robust
- Many detectors
- Low cost

Limitations

- Lack of confirming data other than retention time except for mass spectrometer detection
- Compounds must be thermally stable

لا يوجد بيانات مؤكدة غير وقت الاحتباس (إلا باستخدام جهاز قياس الطيف الكتلي).
المركبات يجب أن تكون مستقرة حرارياً.

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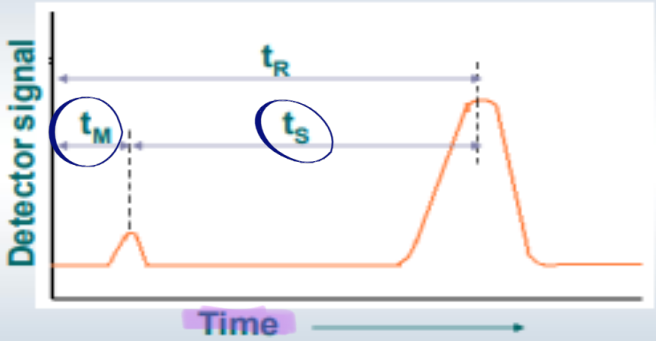
Theory of gas chromatography :

Retention time (t_R) :

The time gap between the sample injection & the appearance of chromatographic peak for a particular analyte is called as retention time.



Retention time is then :

$$t_R = t_M + t_S$$

Retention time يعتمد على

Resolution (R) :

Resolution is the degree of separation between the two adjacent peaks i.e. the distance between the two adjacent peaks divided by the mean of the peak widths.

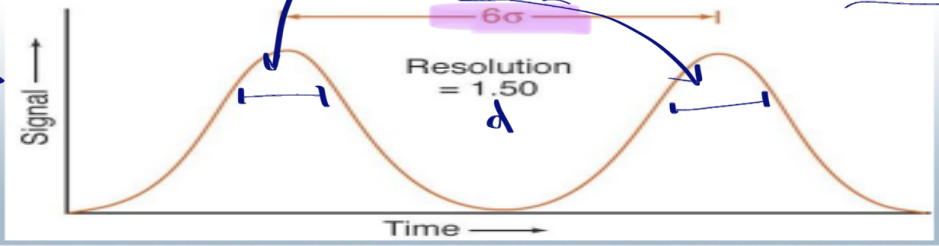
$$R = \frac{2d}{tw_1 + tw_2}$$

where, d = distance between two peaks.

tw1 = width of first peak.

tw2 = width of second peak.

If R = 1.5, baseline separation (99.7% resolution) is achieved.



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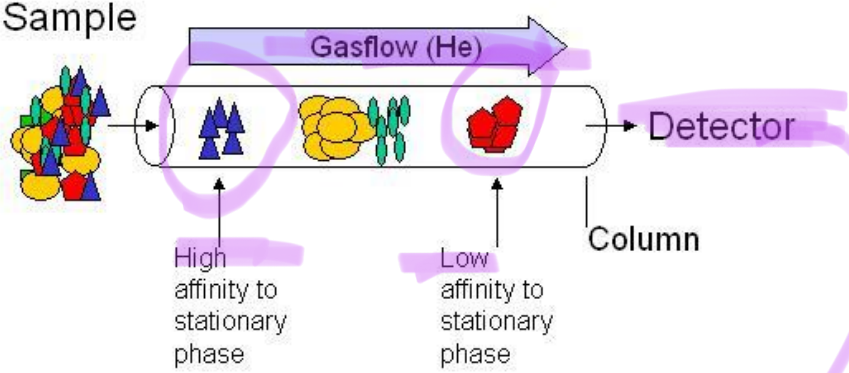
Information Obtained from the Analysis

- Results The time (retention time) until the injected sample reaches the detector is a characteristic value of each component. Investigating the retention time under given analysis conditions makes it possible to determine what a component is (qualitative analysis).

- the size of the component peak, in other words its area and height, makes it possible to determine how much of the component there is (quantitative analysis

مبدأ العمل

A Sample



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