Fundamentals of Gas Chromatography: Hardware

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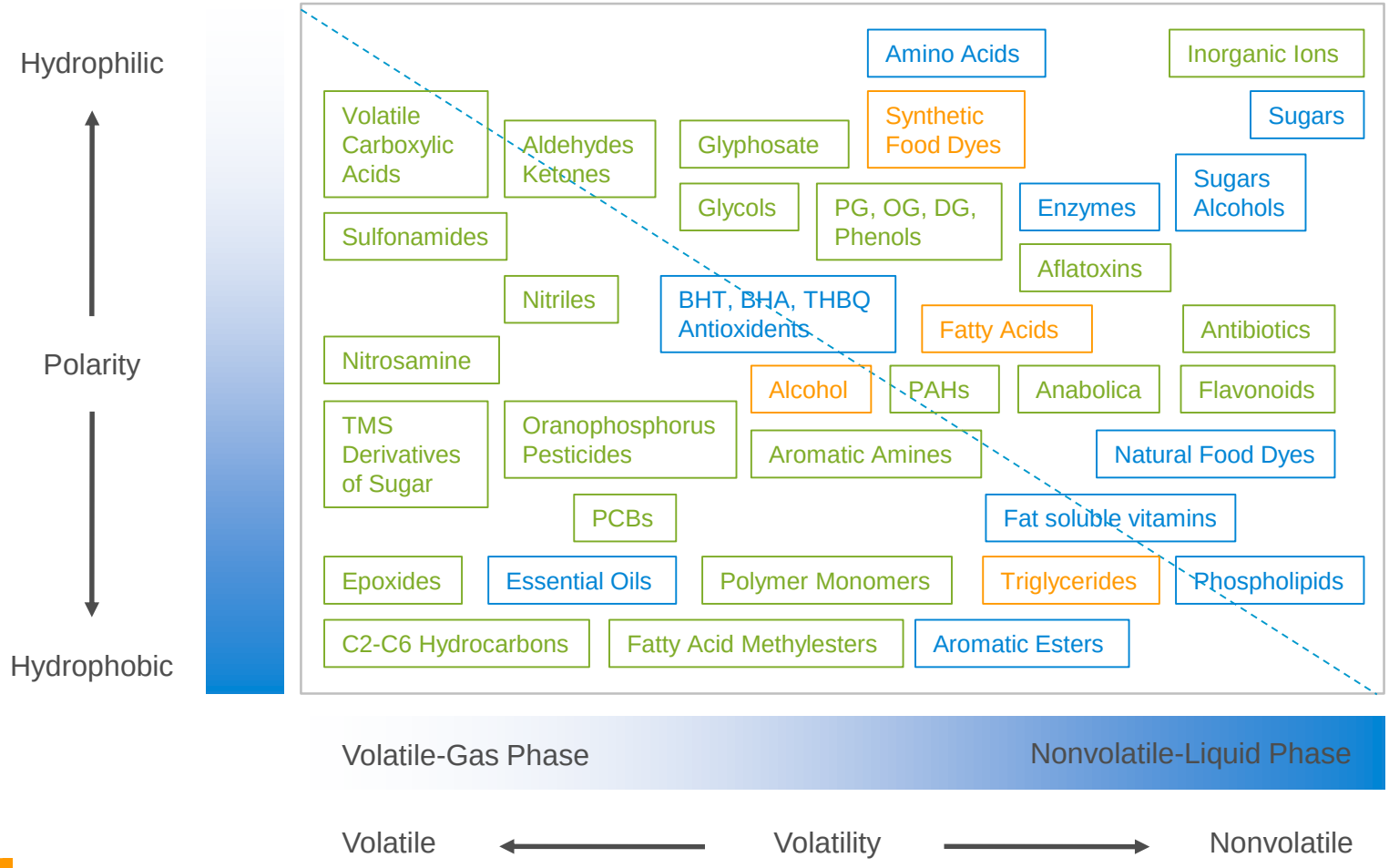
Which Separation Technique for Which Compound?



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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.

Introduction

What Is GC Used For?

GC is used to separate polar and nonpolar compounds that are volatile.

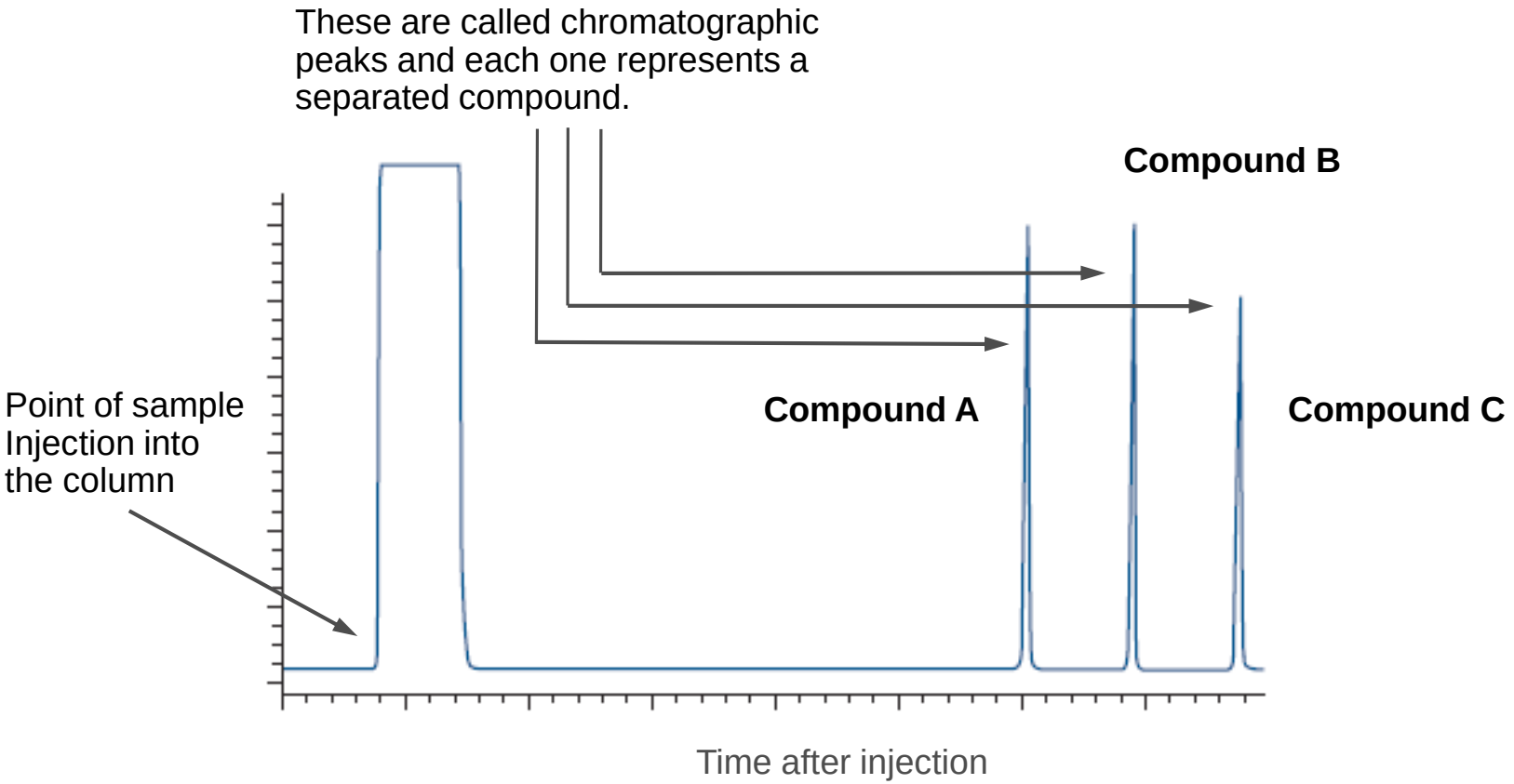
Typical applications:

- Food and flavor analysis
- Environmental analysis (PAH, pesticide, herbicides, benzene)
- Industrial chemical analysis (alcohol, halogenated hydrocarbons, aromatic solvents, phenols)
- Petroleum industry analysis (gasoline, volatile sulfur compounds, refinery gases)

If a compound is nonvolatile (for example, proteins, salts, polymers), then liquid chromatography is a better separation technique.

Introduction

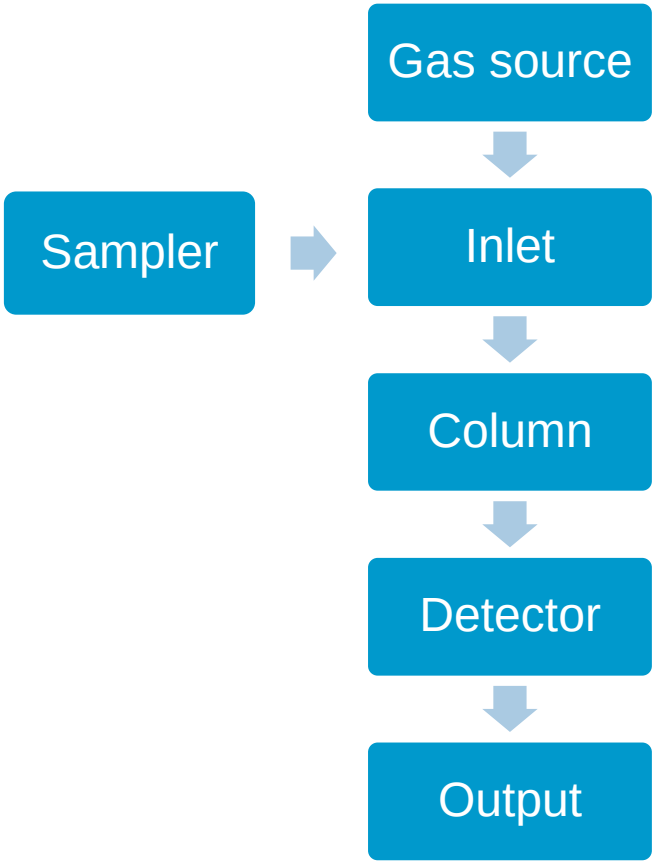
What Does a Chromatogram Look Like?



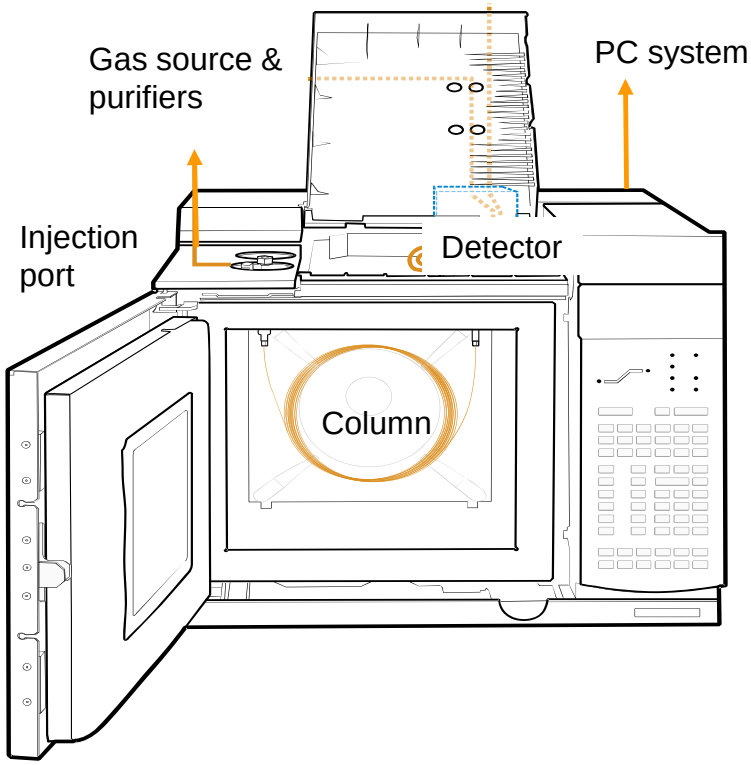
Configuration of a GC System
General Overview

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



Configuration of a GC System



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Configuring a GC System
The Gas Source

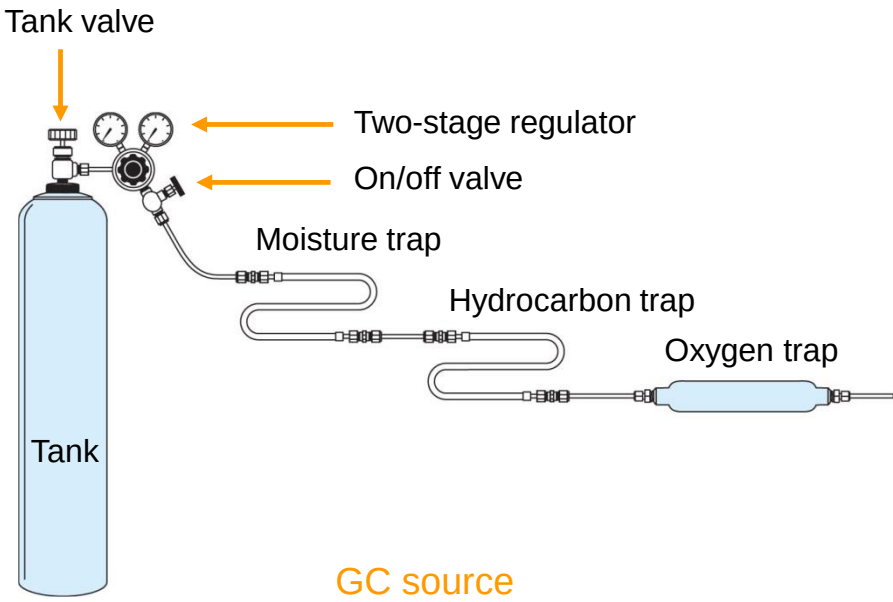
The carrier gas such as helium, nitrogen, hydrogen, or a mixture of argon and methane must be pure (>99.9995%). Contaminants may react with the sample and the column, create spurious peaks, load the detector and raise the baseline, and so on.

The function of the carrier gas is to transport the sample through the system.

A high-purity gas with traps for water, hydrocarbons, and oxygen is recommended.

Specific detector gases support certain detectors (FID, for example).

Compressed gas cylinders or gas generators supply the gas.



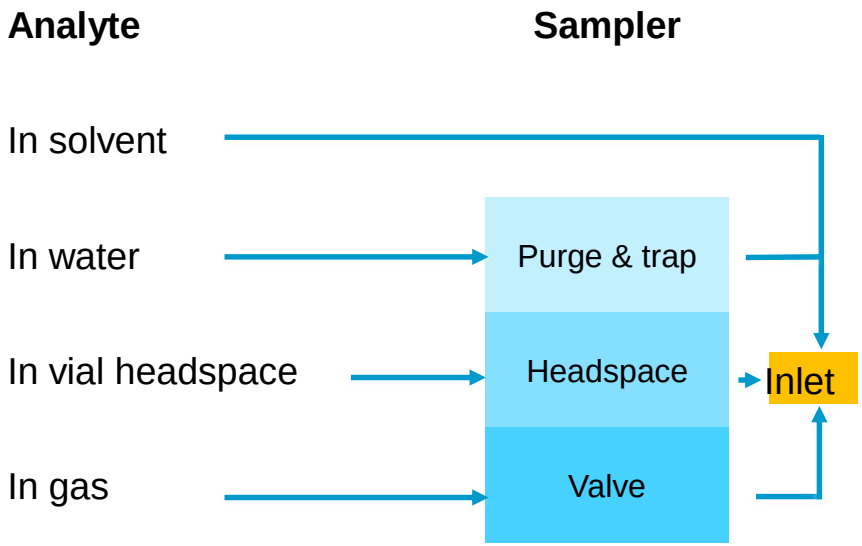
GC source

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

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

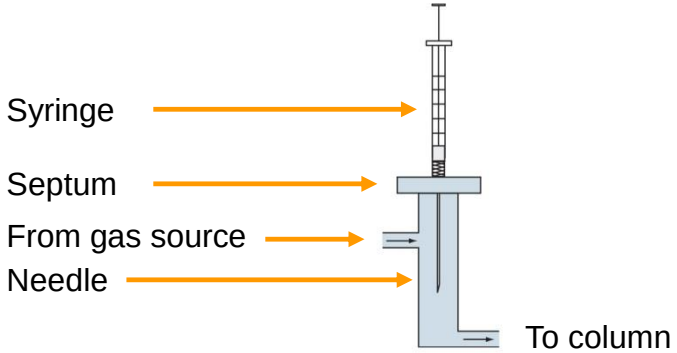
The inlet introduces the vaporized sample into the carrier gas stream. The most common inlets are injection ports and sampling valves.

- Injection ports
  - Handle gas or liquid samples
  - Often heated to vaporize liquid samples

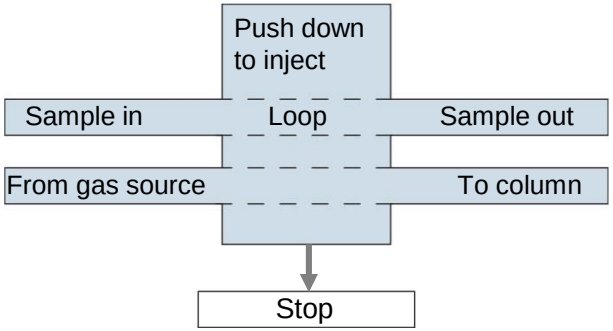
Liquid or gas syringes are used to insert the sample through a septum into the carrier gas stream.

- 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
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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

Cool-on-Column

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

Programmable Temperature

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

Configuring a GC System

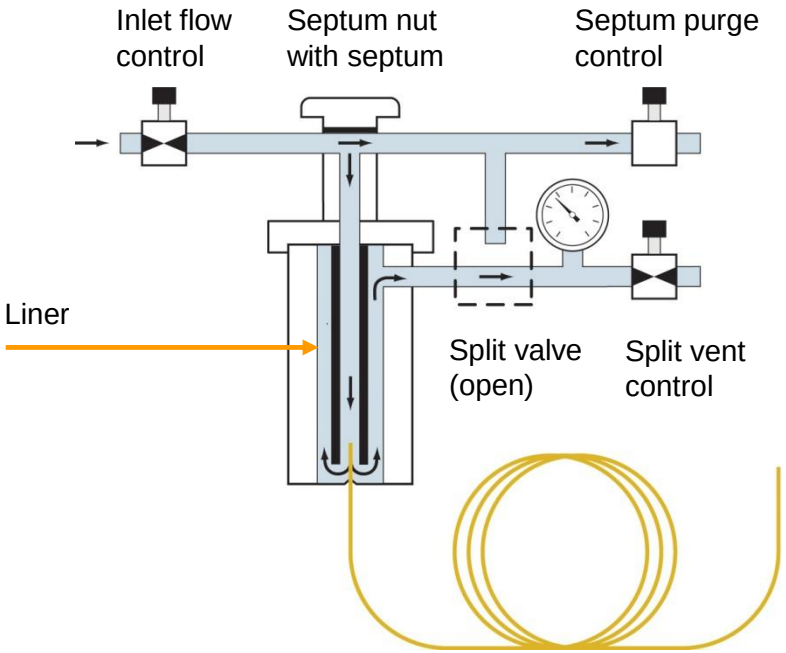
The Different Inlet Types – Split/Splitless Port

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

Configuring GC System

The Different Inlet Types – Split/Splitless Port

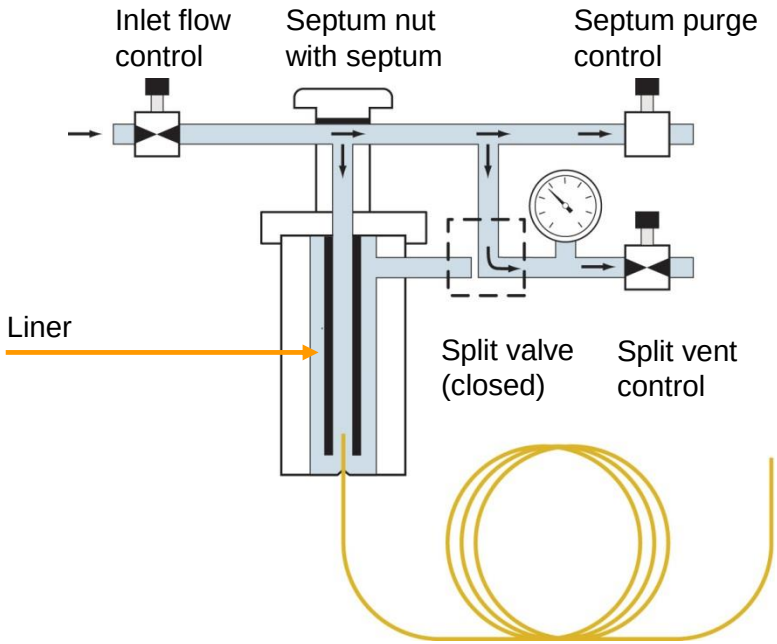
Splitless mode

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

Configuring a GC System
The Column

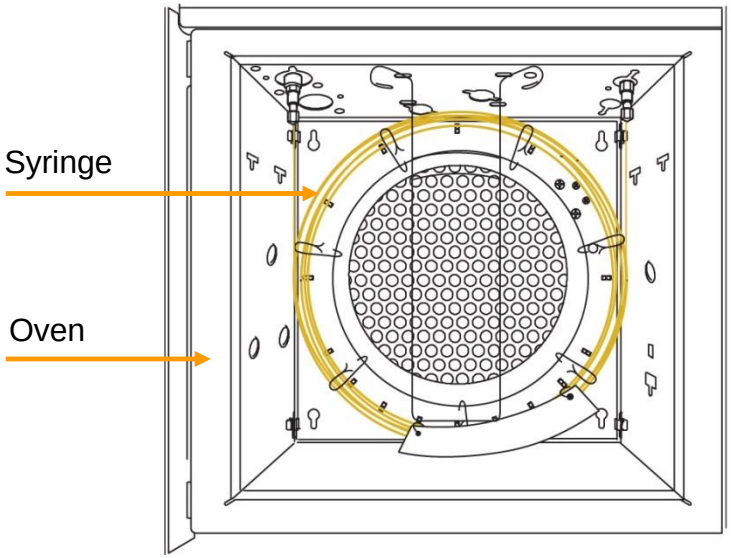
The separation happens here.

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

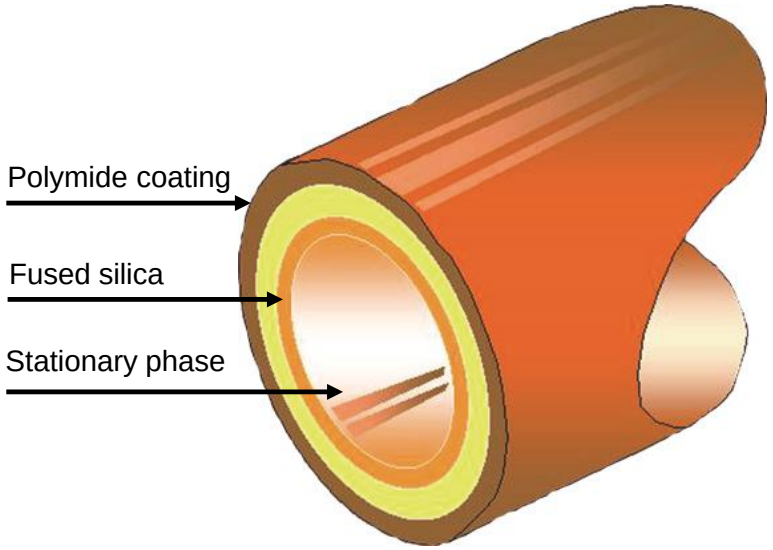
Configuring a GC System
Inside a Capillary Column

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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Configuring a GC System
Column Selection Summary

1. If no information is available about which column to use, start with a DB-1 or DB-5.
2. Low bleed (“ms”) columns are usually more inert and have higher temperature limits.
3. Use the least polar stationary phase that provides satisfactory resolution and analysis times. Non-polar stationary phases have superior lifetimes compared to polar phases.
4. Use a stationary phase with a polarity similar to that of the solutes. This approach works more times than not; however, the best stationary phase is not always found using this technique.
5. If poorly separated solutes possess different dipoles or hydrogen bonding strengths, change to a stationary phase with a different amount of the dipole or hydrogen bonding interaction. Other co-elutions may occur upon changing the stationary phase, thus the new stationary phase may not provide better overall resolution.
6. If possible, avoid using a stationary phase that contains a functionality that generates a large response with a selective detector. For example, cyanopropyl containing stationary phases exhibit a disproportionately large baseline rise (due to column bleed) with NPDs.
7. A DB-1 or DB-5, DB-1701, DB-17, and DB-WAX cover the widest range of selectivities with the smallest number columns.
8. PLOT columns are used for the analysis of gaseous samples at above ambient column temperatures.

Source: Agilent J&W GC Column Selection Guide
Publication #: 5990-9867EN
For Research Use Only. Not for diagnostic procedures.

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

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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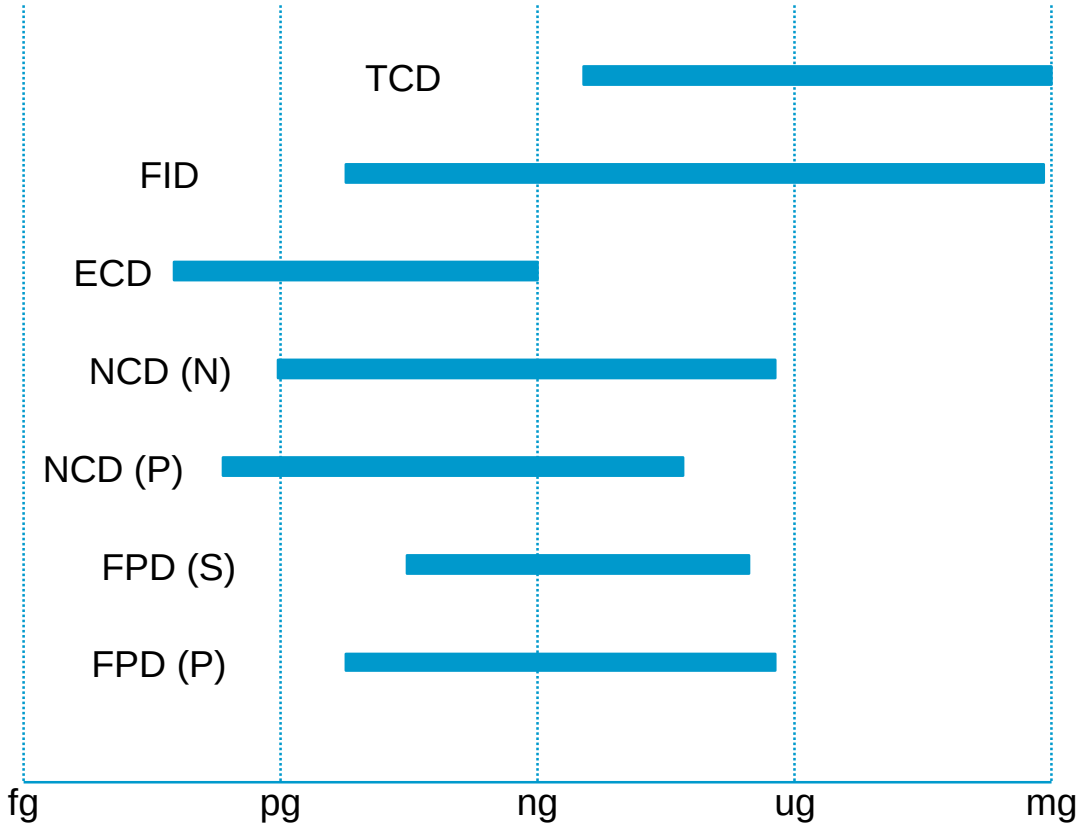
Configuring a GC System

Detector Sensitivity



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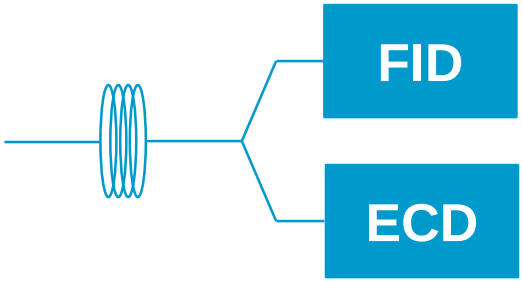
Detector Arrangement

Serial

Place non-destructive detector before other detector



Parallel

Split column effluent to different detectors

Configuring a GC System

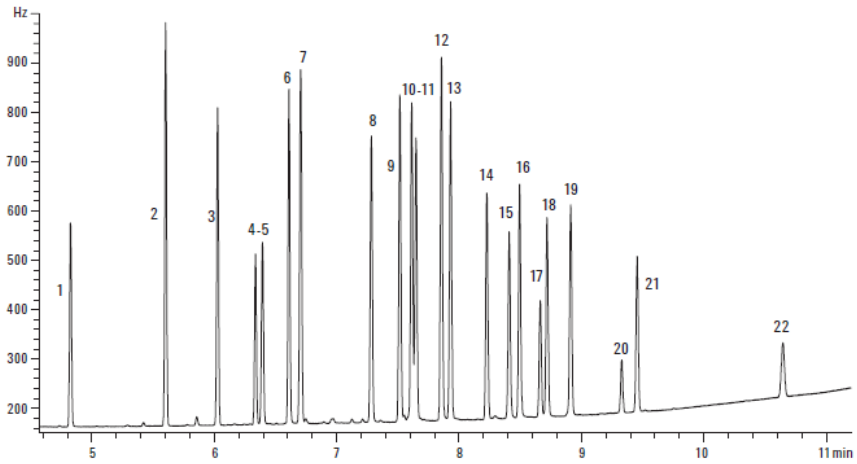
GC Output

The chromatogram plots abundance against time.

Peak size corresponds to the amount of compound in the ample. 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)

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

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Publication	Title	Pub. No.
Primer	Fundamentals of Gas Chromatography	G1176-90000
Video	Fundamentals of Gas Chromatography (14 min)	
Guide	Agilent J&W GC Column Selection Guide For Research Use Only. Not for diagnostic procedures.	5990-9867EN
Web	CHROMacademy – free access for students and university staff to online courses	
Application compendium	A compilation of Application Notes (22MB)	5991-3592EN

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