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Theory and Key Principles Series

Gas Chromatography (GC)

Session 3 – The Split/Splitless Inlet

Introduction

Welcome to Shimadzu's Gas Chromatography Theory and Key Principles Series!

Presenter

Andrew Clissold

GC/GCMS
Business
Manager

Theory & Key Principles Series – GC

– *Introduction to Gas Chromatography* *

– *GC Columns* *

– **The Split/Splitless Inlet**

– Advanced Liquid Injection Techniques

– Alternatives to Liquid Injection

– Choices of Detectors for GC

– Processing GC Data

– Maintenance & Troubleshooting

* *Now available on demand at www.shimadzu.co.uk/webinars*

The Split/Splitless Inlet

In this presentation:

- The GC Inlet
  - The packed injector – let's start simple!
- The Split/Splitless Inlet
  - Why can't we just use a packed inlet?
- Split Mode
  - The split ratio
- Splitless Mode
  - Why, when & how?
  - High pressure splitless injection
- Carrier Gas Saver

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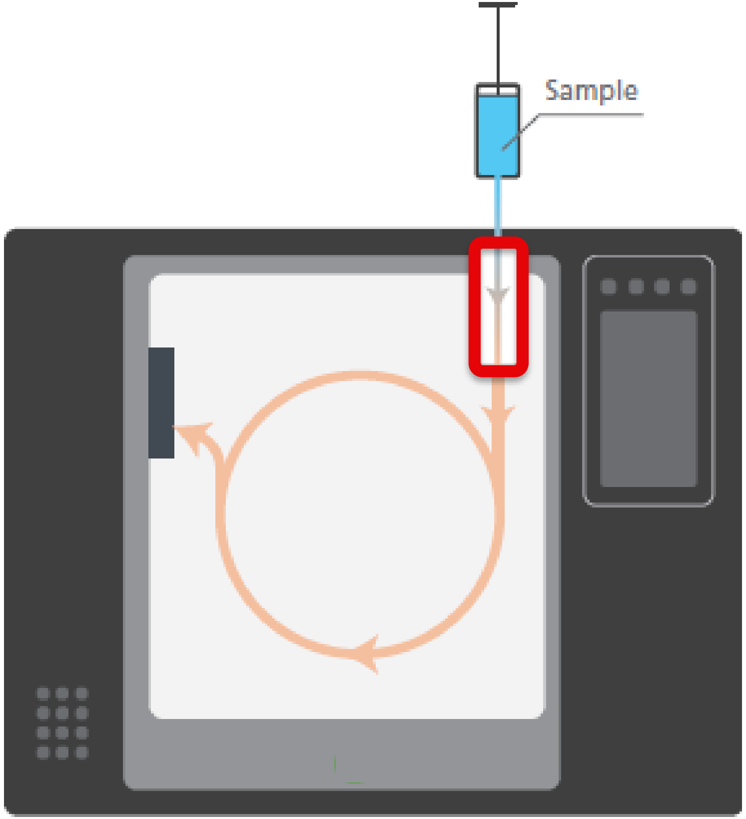
The GC Inlet

The GC inlet

Sometimes called a GC Injector

"Entry system" for sample & carrier gas onto the column.

A **flow controller** manages the pressure/speed/flow of gas down the column.

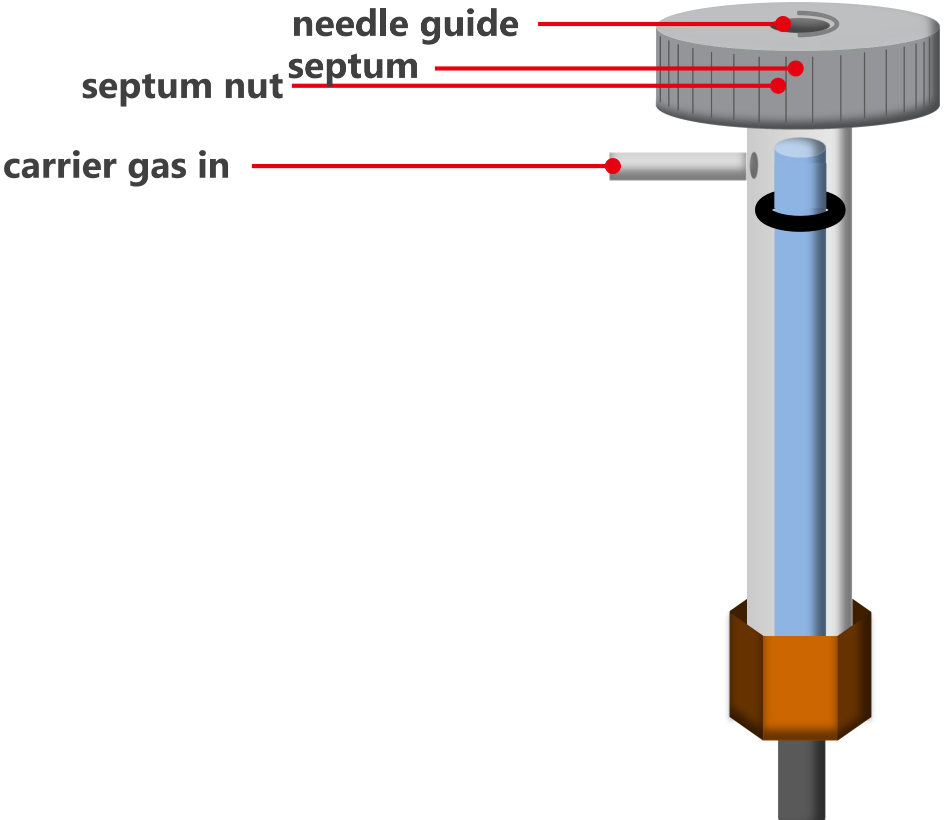


The GC inlet

A metal tube brings the **carrier gas** from the flow controller.

A **septum nut**, with a **needle guide**, is screwed on the top.

A rubber **septum** allows the needle to inject sample inside.



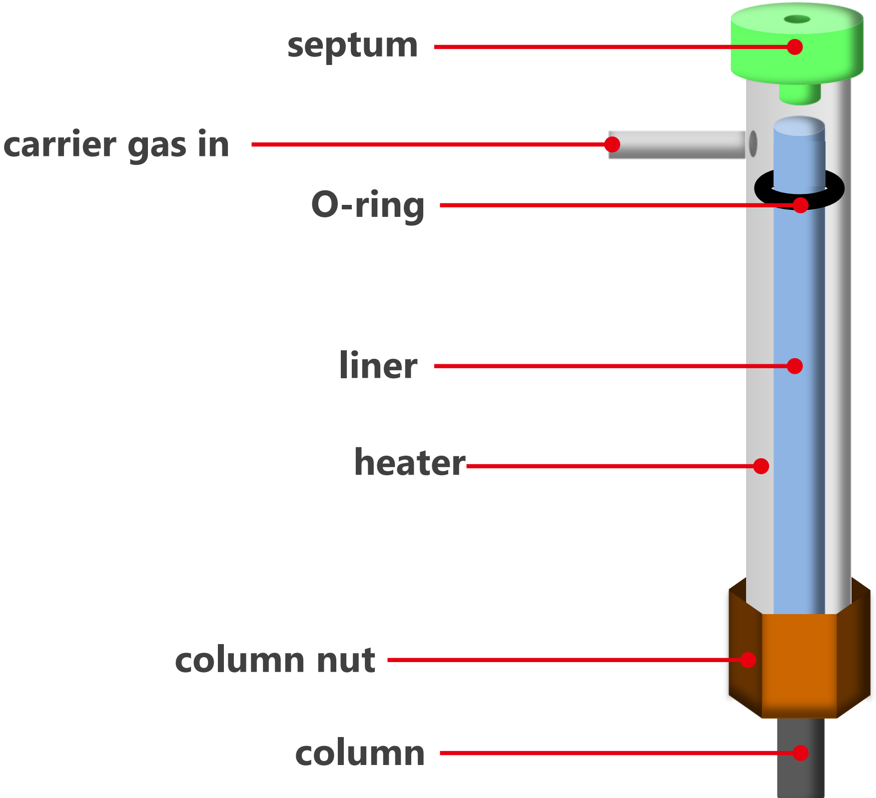
The GC inlet

The **liner** is a hollow, glass tube where a sample mixes with the carrier gas.

The **O-ring** forces carrier gas to travel through the liner.

The **column** connects to the bottom of the inlet, held in place with a **column nut**.

The inlet is **heated**, to around 250 °C, to vaporise the sample.



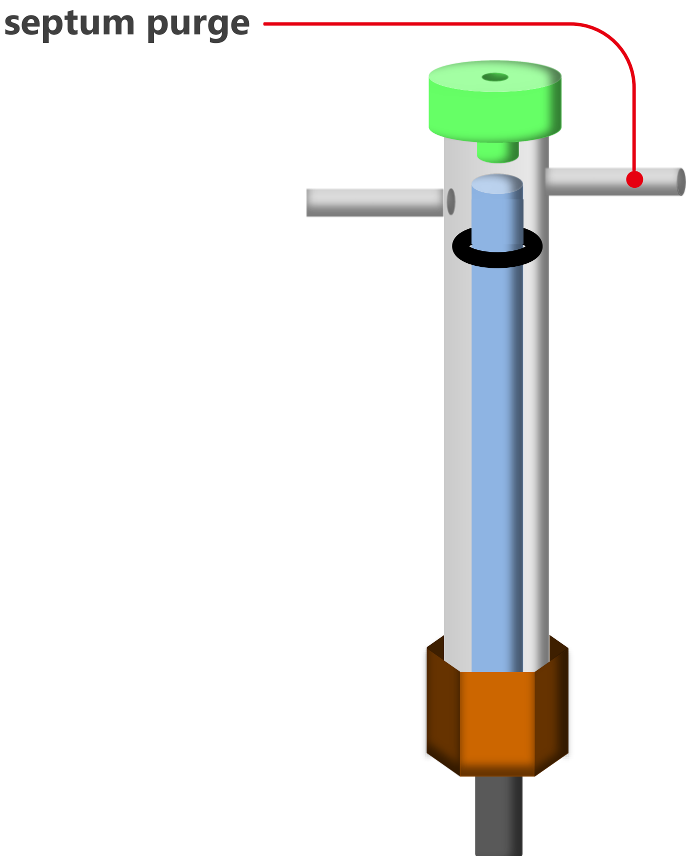
Septum purge

At high temperatures, chemicals can be released from the septum and appear as **ghost peaks** on a chromatogram.

To remove this contamination, most inlets are fitted with a **septum purge**.

A small flows prevents the off-gassed chemicals going down the liner.

The septum purge is connected to the **flow controller**, where the flow rate is regulated to around 3 mL/min.



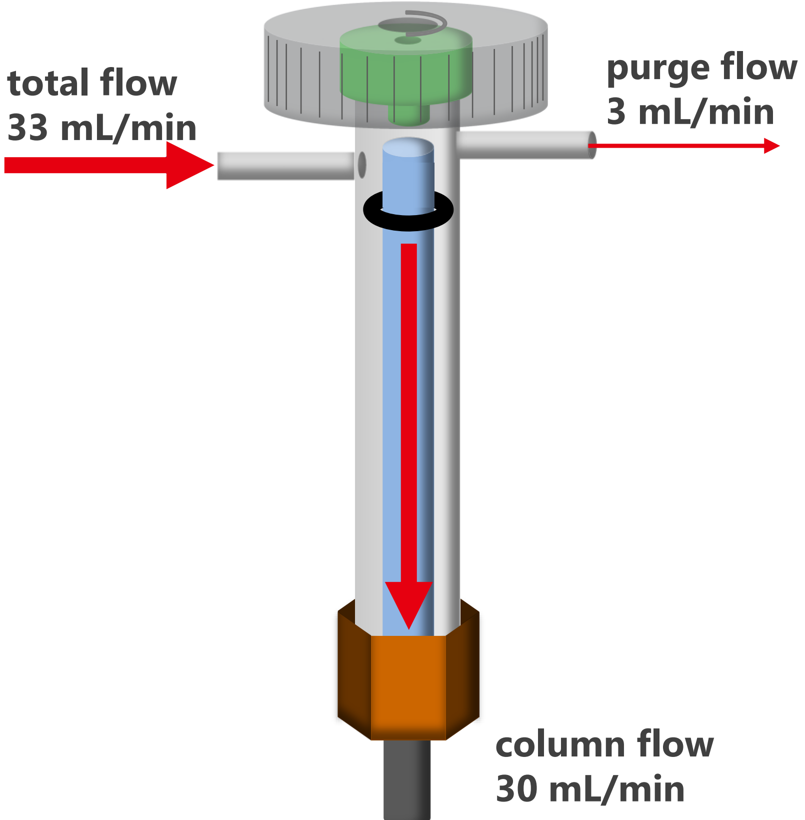
Packed injector

This completes the components of a **packed injector**.

For **packed columns**, or **wide-bore capillary columns** (>0.53 mm i.d.).

For a packed column, a typical **column flow** is 30 mL/min.

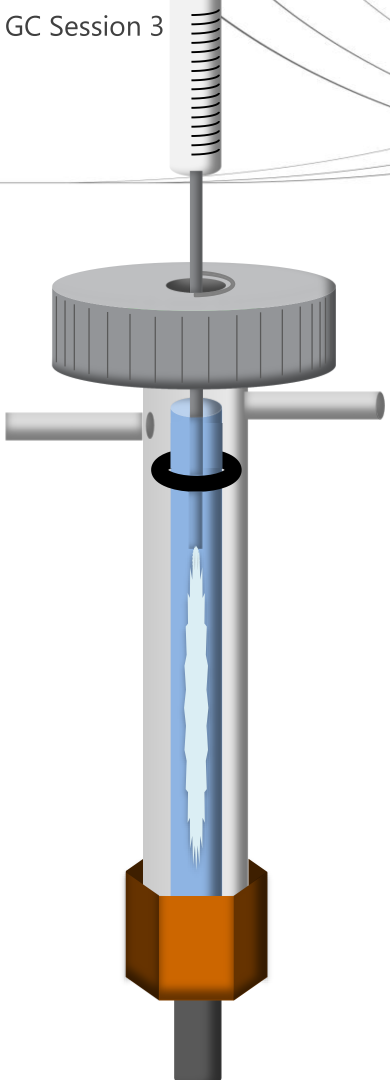
With a **purge flow** of 3 mL/min, this gives a **total flow** of 33 mL/min.



Injection process

With a typical liquid injection, 1 µL of a diluted sample is injected.

Under common conditions, 1 µL of liquid expands to about 250 µL of gas!



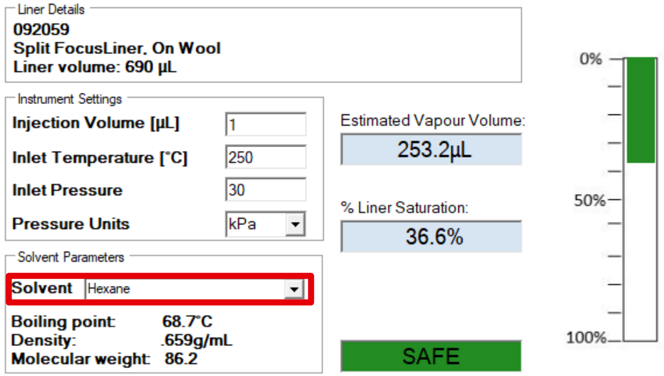
Injection process

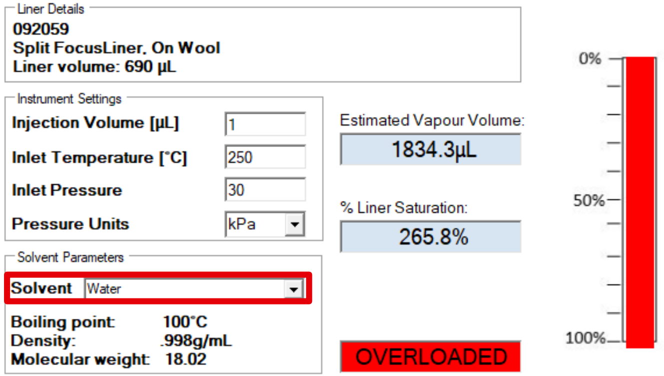
1 µL of liquid expands to about 250 µL of gas!

Volume of gas must be less than the volume of the liner. Liner volume is approx. 500uL.

Injecting too much can result in **backflash**.

Highly polar solvents have a much greater expansion volume – the injection volume must be reduced.





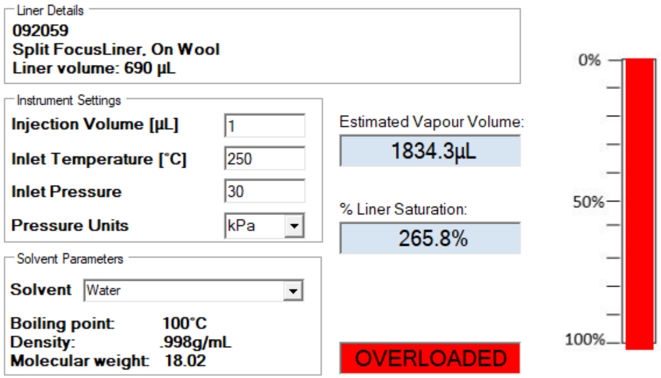
Solvent expansion calculators

Google ‘**Liner Selection Tool**’:

www.trajanscimed.com › products › mn-1024-g

Liner selection tool - Trajan Scientific and Medical

The liner selection tool helps you select the right inlet liner for your analysis. The tool also includes a handy Vapor Volume Calculator that checks if you are ...



Liner volume

The liner volume has an important effect on **peak shape**.

At 30 mL/min, the sample is flushed from the 0.5 mL liner in 1 second.

Sample bandwidth is 1 second wide at the head of the column.

Remember: **peaks typically only get wider** over time!

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The Split/Splitless Inlet

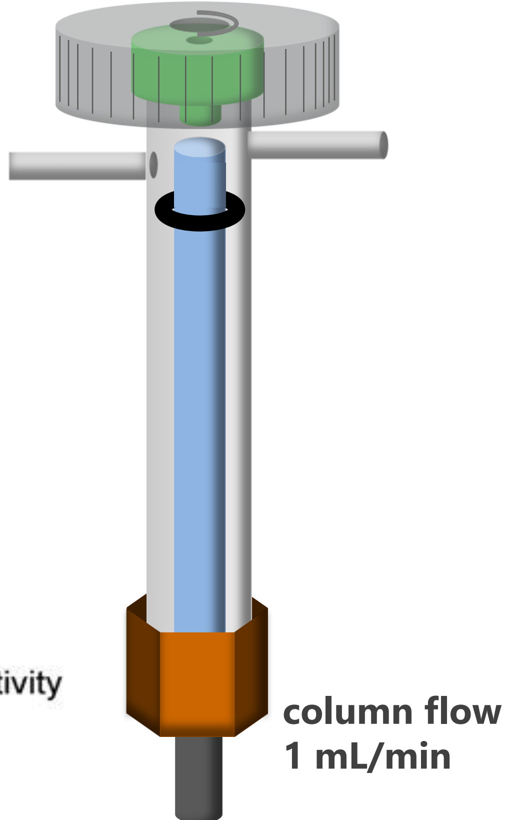
Problems with capillary columns

Much narrower inner diameter than packed columns.

To maintain the optimal linear velocity, column flow is very low.

To flush the 0.5 mL liner volume now takes 30 seconds!
This is far too long.

Peak widths need to be approx. 3 seconds.

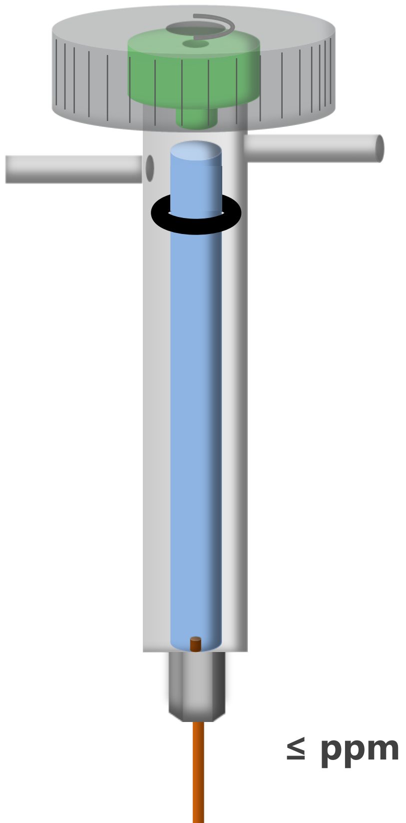


Problems with capillary columns

Packed columns are ‘packed’ with stationary phase.

Capillary columns have just **mg amounts of stationary phase**.

Injecting too much sample causes **column overload,** which leads to very poor peak shape.



Split flow

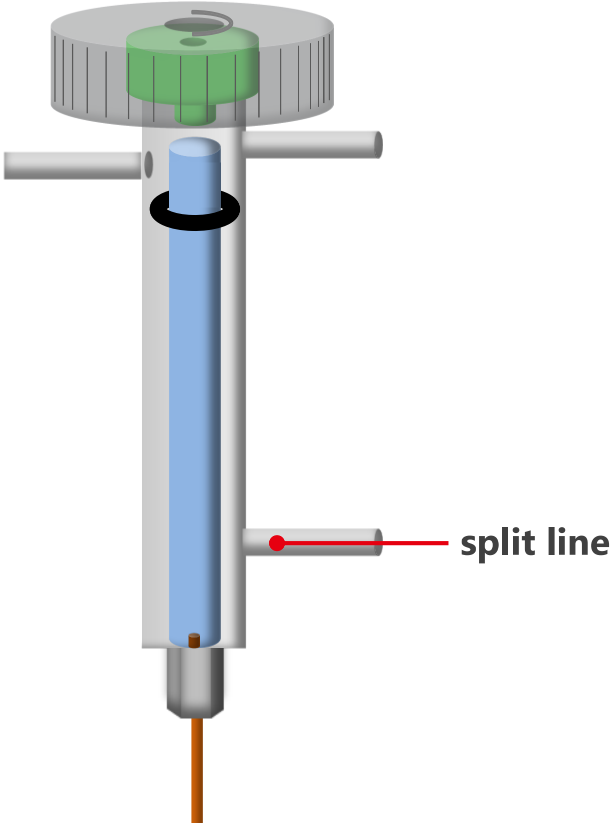
The solution – add a **split line!**

This is an 'escape route' for the excess flow that can no longer go down the column.

This allows:

- **Optimised column flow**
- **High flow rate through the liner**
- **Sample dilution**

Split line flow is regulated by the flow controller.



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Split Mode

Split ratio

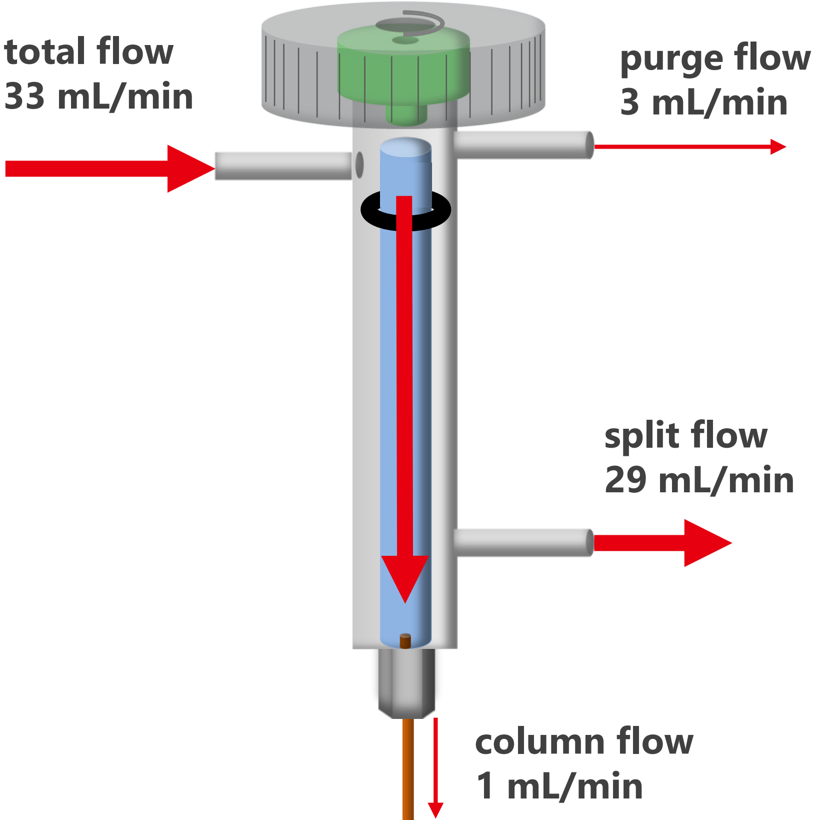
The **split ratio** determines the dilution factor of the sample onto the column.

1 mL/min **column flow**

29 mL/min **split flow**

For every 30 portions of sample, 1 is analysed.

Split ratio = 30:1

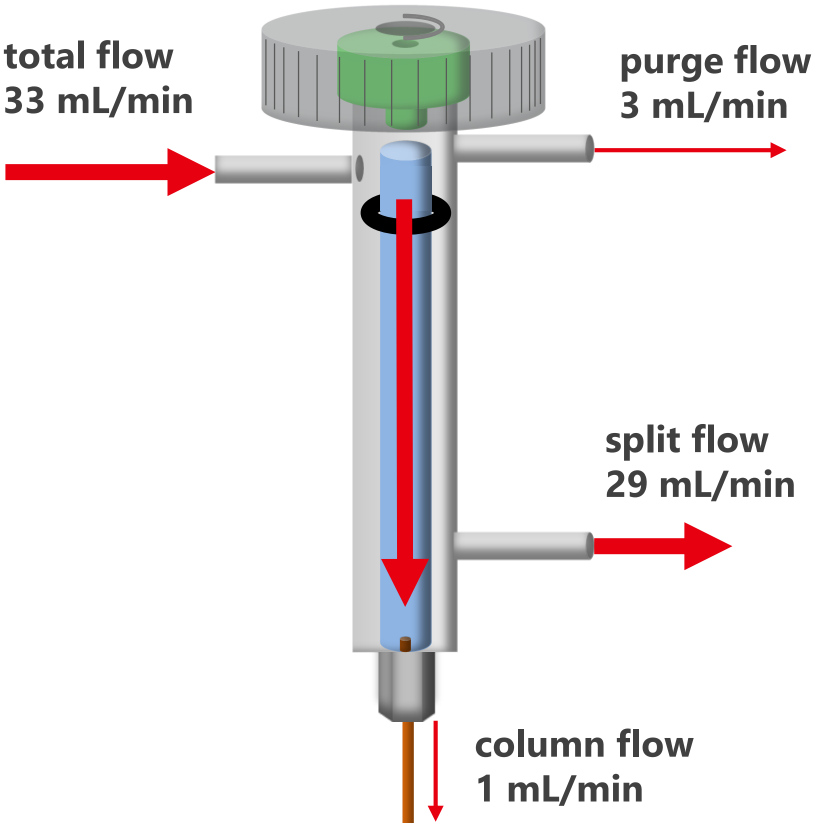


Split ratio

What effect does split ratio have on peak shape and size?

Higher split ratio =
- **Narrower** peaks
- **Smaller** peak area

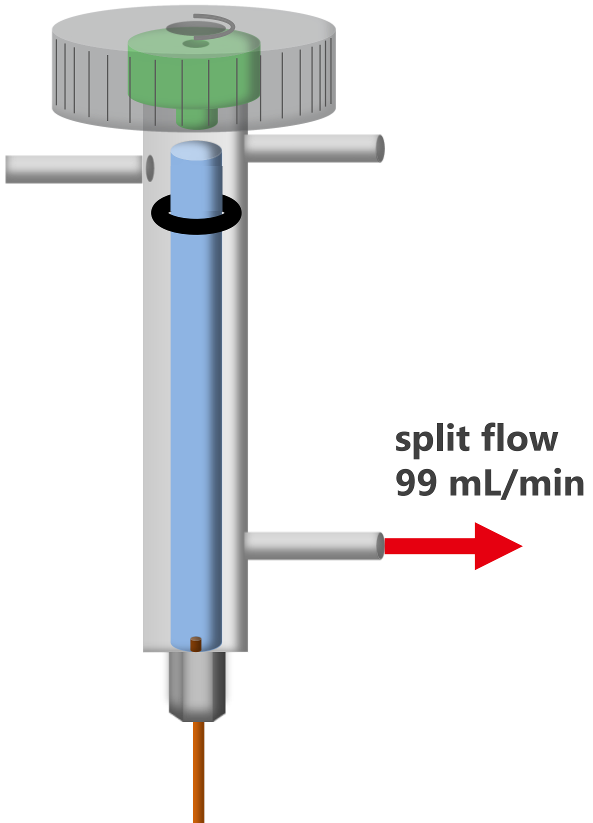
Lower split ratio =
- **Wider** peaks
- **Larger** peak area



Drawbacks of split mode

A high split flow also results in a high carrier gas consumption rate.

Relies on high sample concentrations to enable further dilution.



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Splitless Mode

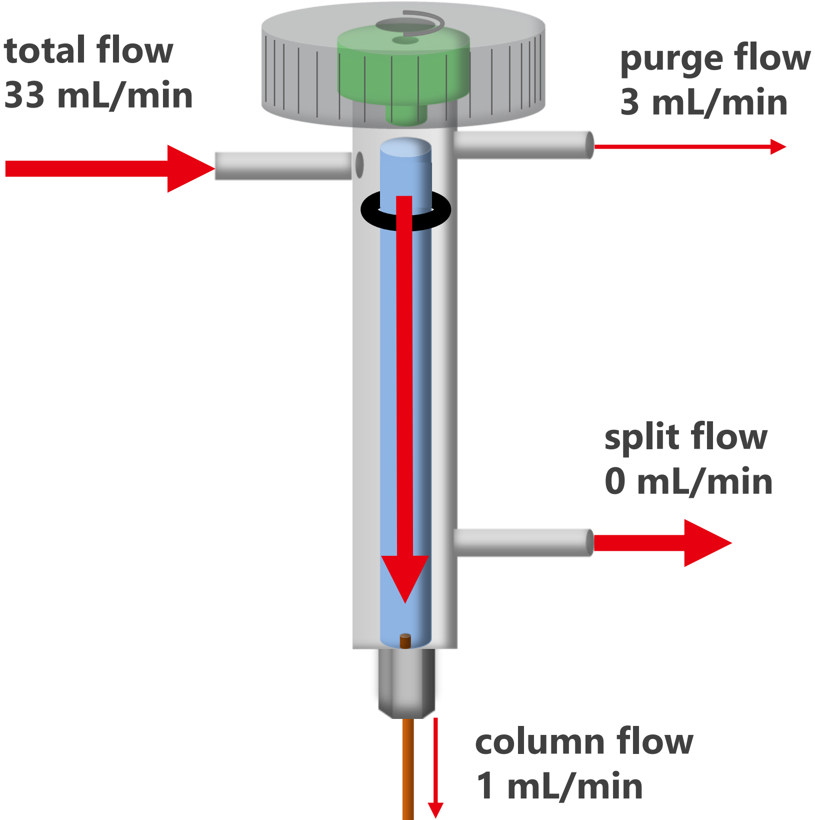
Splitless mode

Used when **sample concentration is too low** for split analysis.

Split flow is off so everything transfers onto the column.

Column flow is still 1 mL/min.

Sample bandwidth is around 1 minute.

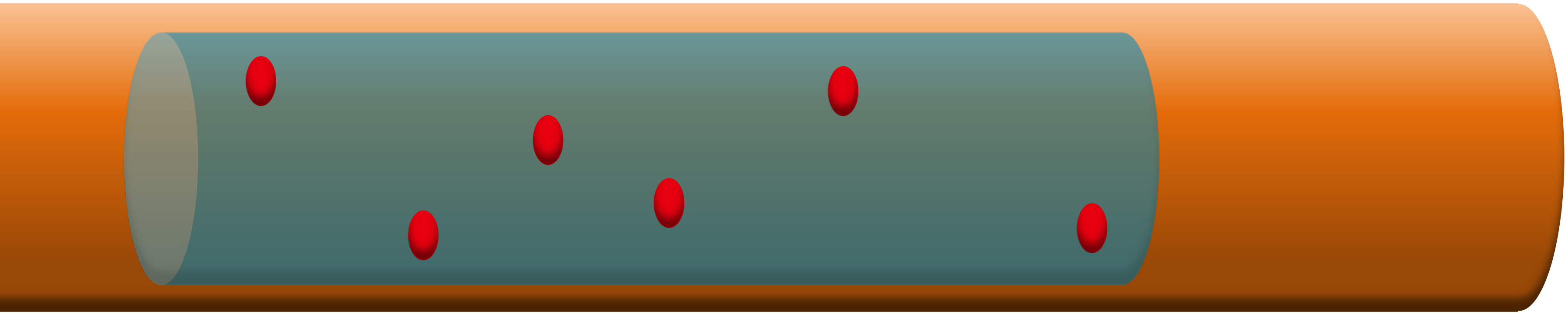


Solvent effect

The vaporised sample transfers from inlet to column.

The column inside the oven is kept at least 20 °C below the solvent boiling point.

Sample condenses on the head of the column.



Solvent effect

The vaporised sample transfers from inlet to column.

The column inside the oven is kept at least 20 °C below the solvent boiling point.

Sample condenses on the head of the column.

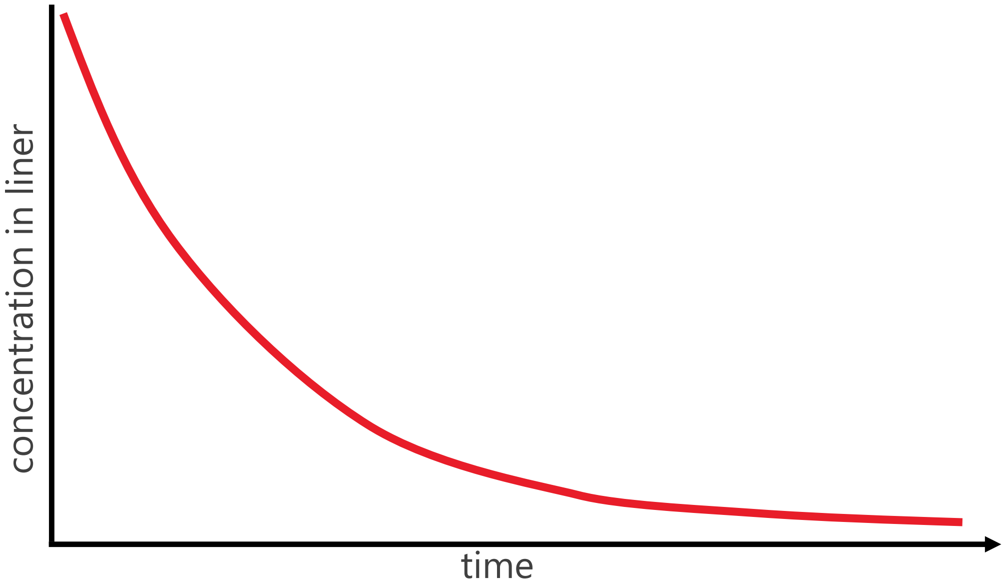
Oven heats, evaporating solvent, which leaves behind a sharp band of analytes.

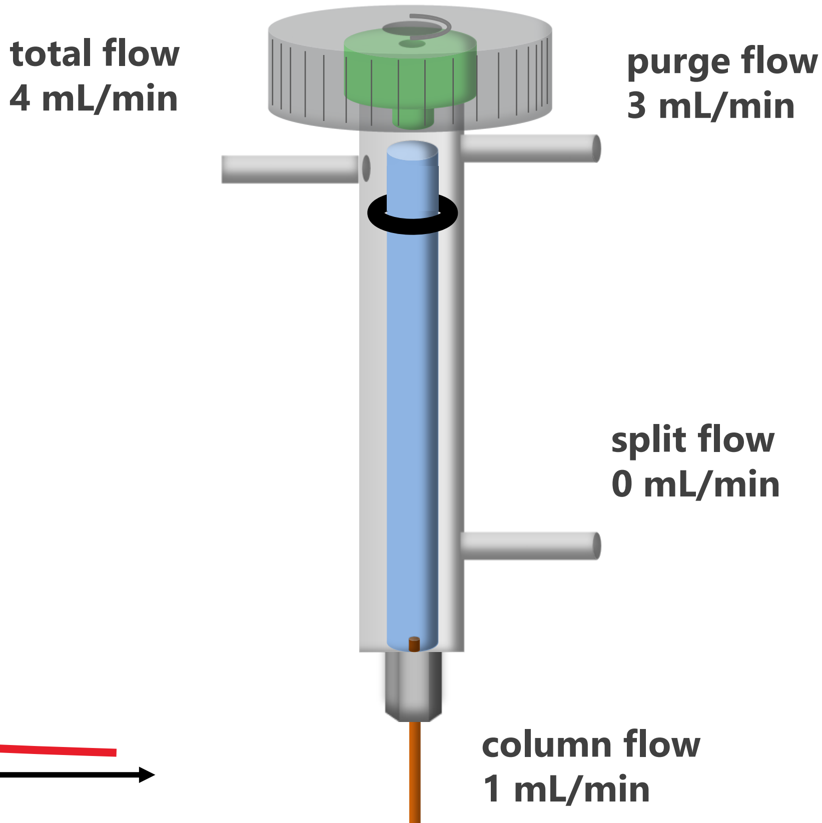
- These analytes must have a **boiling point at least 20 °C above solvent boiling point**, or they'll evaporate with the solvent.

Sampling time

The sampling time is:
time taken to flush the liner 1.5 – 2x

$$\frac{1\ \text{mL/min}}{0.5\ \text{mL volume}} \times 2 = 1\ \text{min}$$





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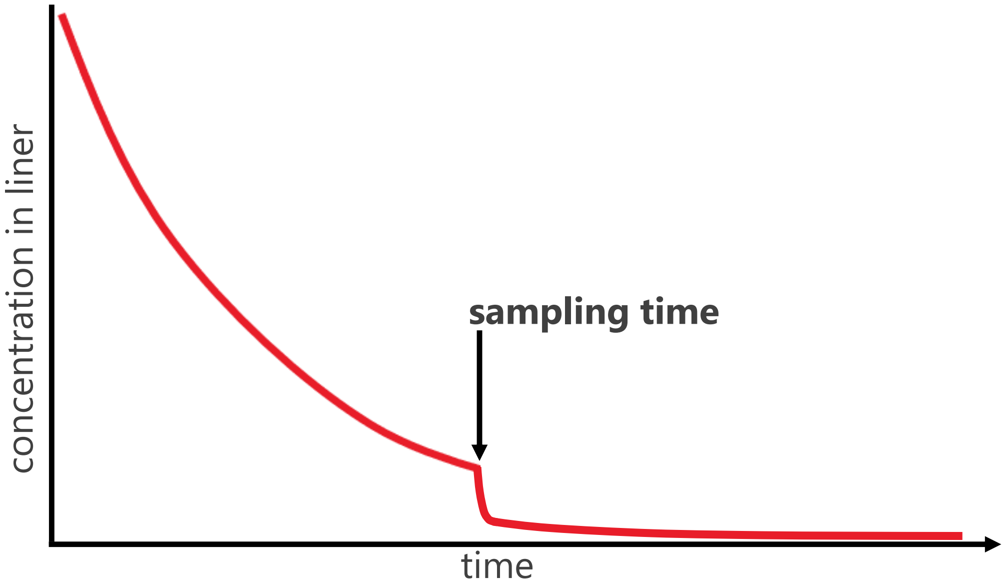

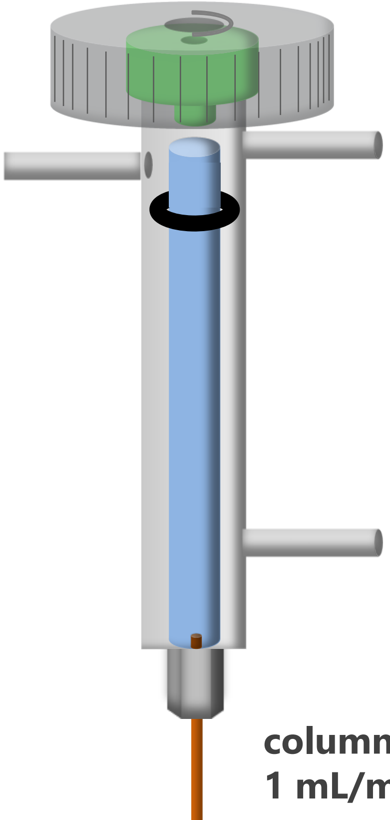

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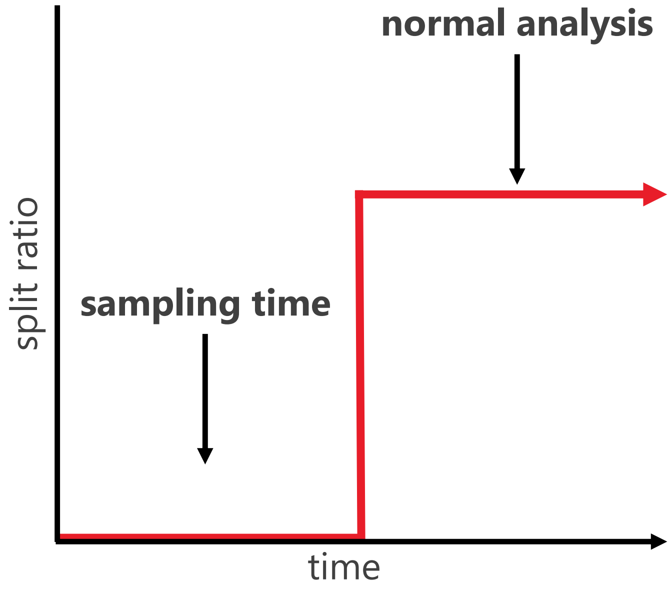
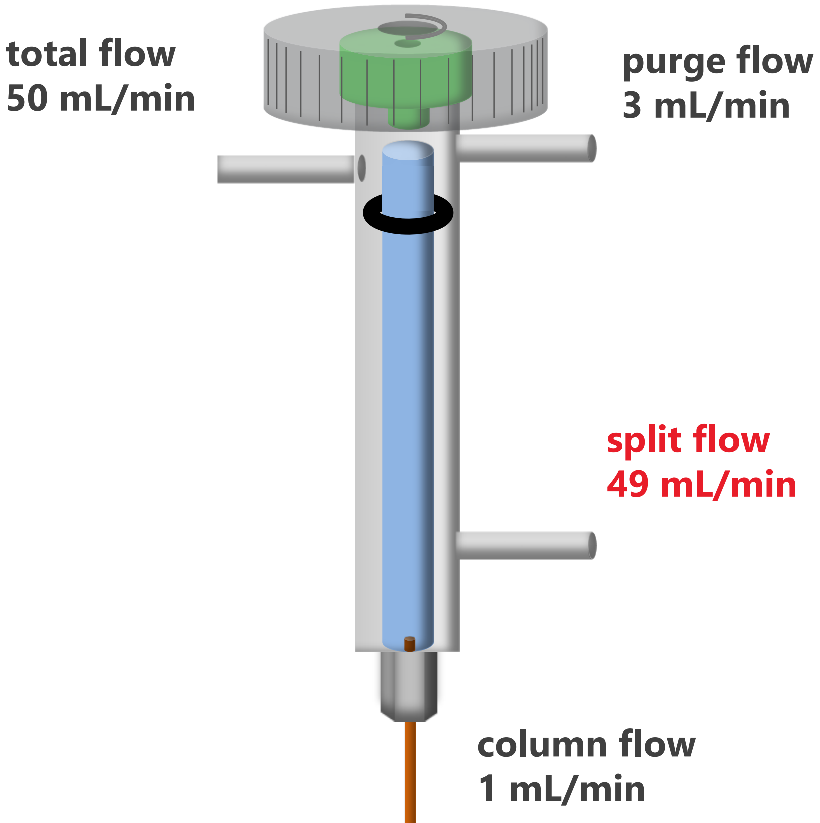
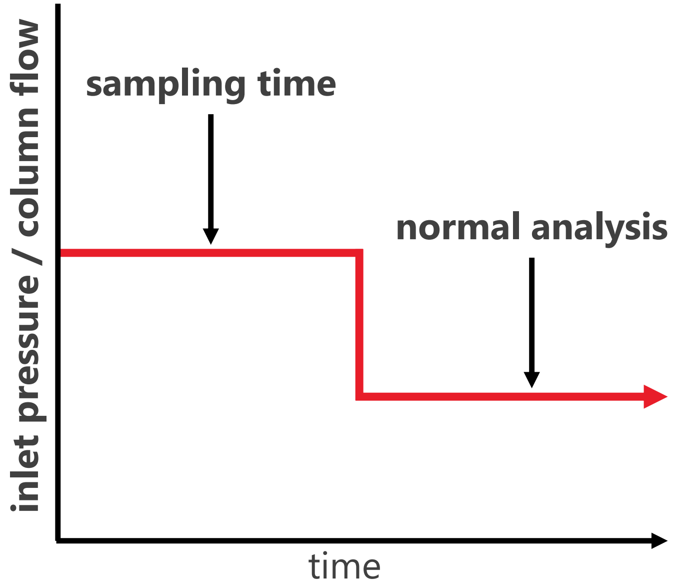


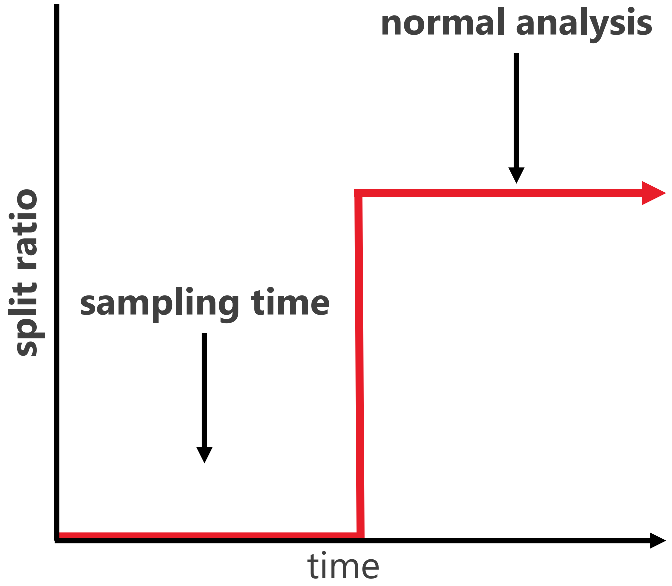


High pressure injection

Sometimes called **pulsed splitless**.

Inlet pressure is increased during sampling time to speed up transfer to column.





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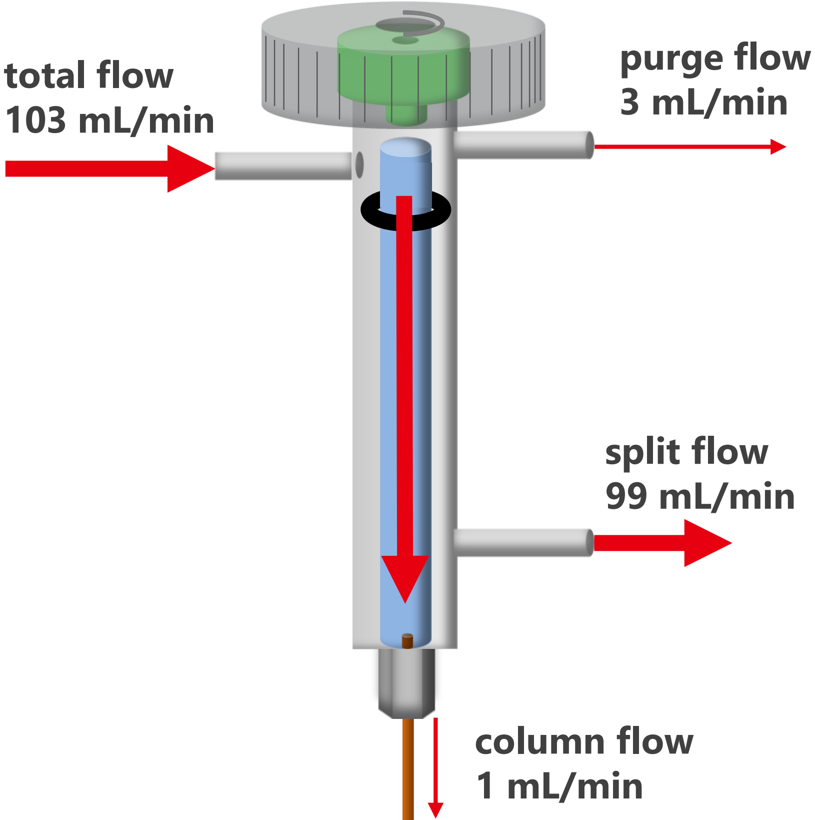
Carrier gas saver mode

High carrier gas flow rates

High gas consumption costs time & money.

Both split & splitless modes can consume very large quantities of gas (>100 mL/min).

Modern GC hardware and software has a built-in **gas saver mode** to minimise consumption.



When to turn down the flow

In **split mode**, a high split dilutes the sample and increases transfer rate.

But what does the high split flow do after all the sample is transferred? **Not much!**

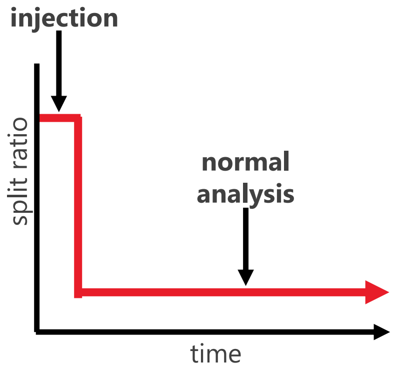
In **splitless mode**, a high split flow helps remove the final traces of the sample from the inlet.

But for how long does the inlet need to be flushed?

Carrier gas saver mode settings

All of these extra tubes on the inlet make it easy for air to diffuse inside, so always maintain a low flow rate through the split line (except in the sampling time for splitless mode).

A split ratio of 5:1 is usually sufficient.

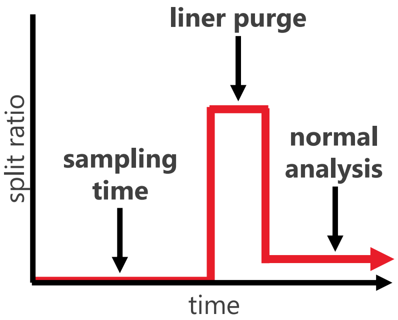


Split mode

Enable carrier gas saver mode after 1 minute.

Splitless mode

Enable carrier gas saver mode 1 minute after the sampling time has finished.



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Summary

Summary

- **The split/splitless inlet is optimised for use with capillary columns.**
- It is comprised of:
  - **Septum nut** and **septum**
  - **Liner** with **O-ring** (with a fixed internal volume, making **backflash** possible if the injection volume is too high)
  - A **heated** body
  - **Septum purge** (to remove contaminants caused by a heated septum)
  - **Split line** (to dilute sample flow and increase sample transfer)

- **Split mode** is the most common technique, and is the go-to mode.
  - The split line speeds up transfer of the sample onto the column and dilutes it down prior to column transfer.
  - The **split ratio** defines the sample dilution – the higher the ratio, the less sample is transferred to the column.
  - Higher split ratios give thinner, sharper peaks

- **Splitless mode** is used only when sample concentration is too low to split it.
  - The split line is closed to facilitate full sample transfer to the column.
  - It relies on **solvent focussing**, where the analytes need to be significantly less volatile than the solvent.

- **Carrier gas saver** helps reduce gas consumption by reducing the split ratio after the injection.

Next time

The next session will be on…

Advanced Liquid Injection Techniques

This will cover:

- *When is split/splitless unsuitable*
- *Programmable Temperature Vaporisation (PTV) technique*
- *On-Column Injection (OCI) technique*
- *Large Volume Injection (LVI) using a Multi-Mode Inlet (MMI)*

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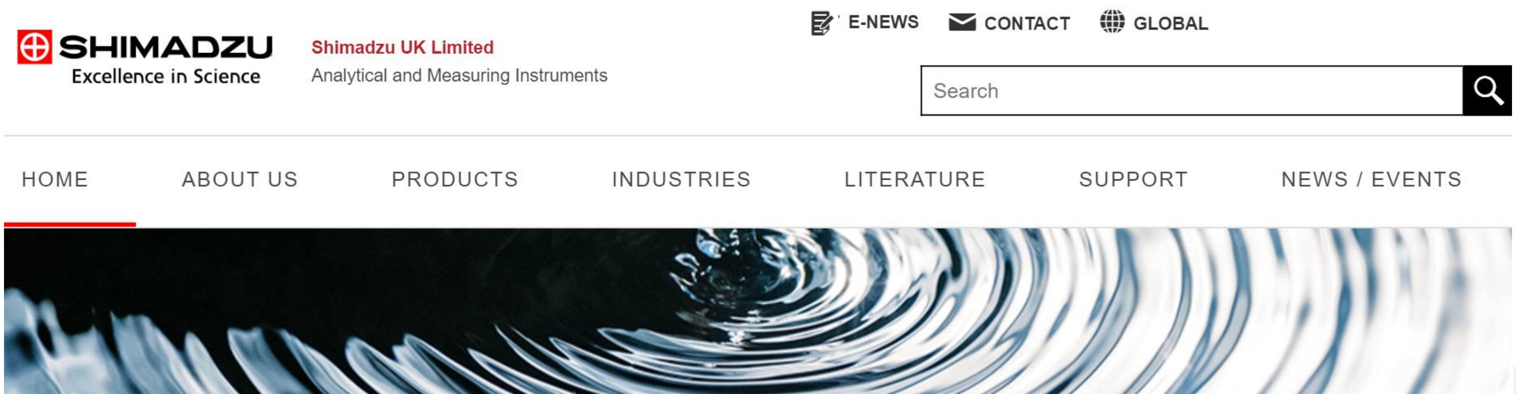

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UK Contact Details

www.shimadzu.co.uk *info@shimadzu.co.uk* *01908 552209*

@ShimadzuUK

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