Injection Techniques for Capillary GC

SUPELCO
Solutions within.

Updated: 28-May-2014

Agenda

Overview

Practical Considerations

Split Injection

Splitless Injection

On-Column Injection

Direct Injection

sigma-aldrich.com/analytical

Overview

- Convert extract or sample to a gas cloud that the GC system can handle
- Four primary injection techniques are used in GC
  - Split
  - Splitless
  - On-column
  - Direct





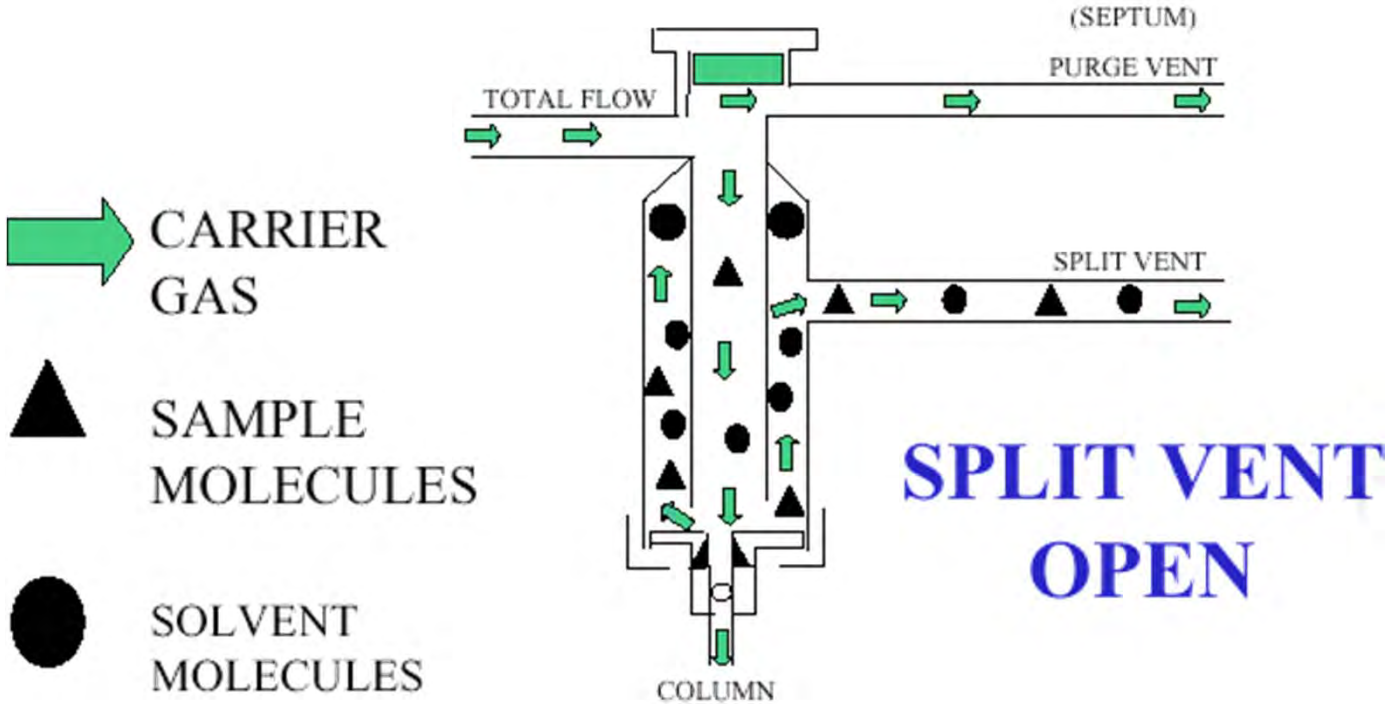
Practical Considerations

- Syringes
  - Large enough so it is not filled to capacity
  - Extract/sample should occupy at least 10% of the syringe volume
  - Use gas tight design for gas phase samples
- Reproducibility
  - Use an autosampler when able
  - For manual injections…
    - Use a Chaney adapter
    - Be smooth and rapid
- Inlet Liner
  - Use the correct design for the injection technique
  - Match the deactivation with the application

Split Injection

Useful for High or Unknown Concentrations

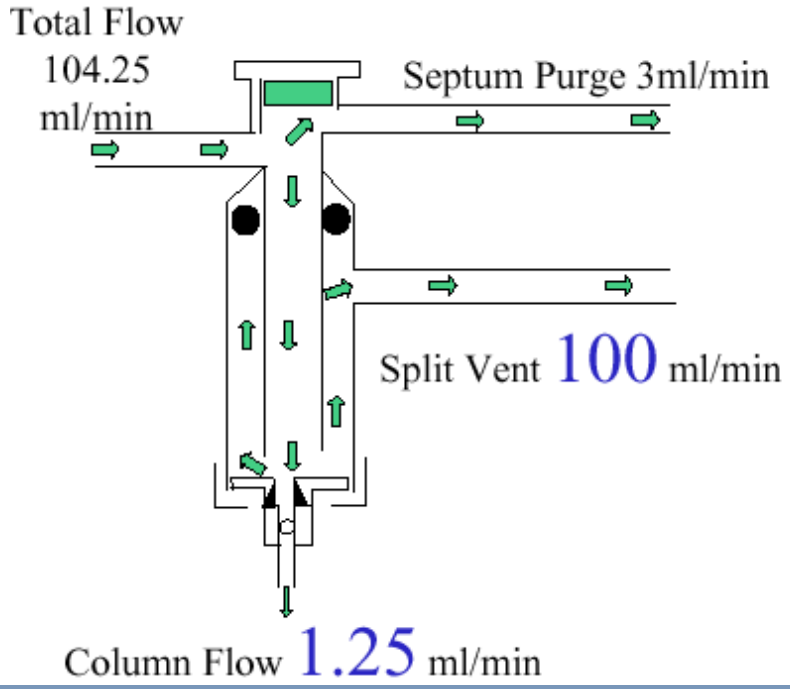
- Extract or sample is vaporized, mixed with carrier gas, then split in the inlet
- Limit amount of material reaches column (prevents overload + fronting peaks)
- A small portion flows to the column while the bulk is vented away
- Can be used with isothermal or temperature programmed analysis



Split Injection

Split Ratio

- Total flow = septum purge + column flow + split vent flow
- Split ratio = split vent flow / column flow



Split Ratio Calculation

$$= \frac{\text{Split Vent Flow}}{\text{Column Flow}}$$

$$= \frac{100}{1.25}$$

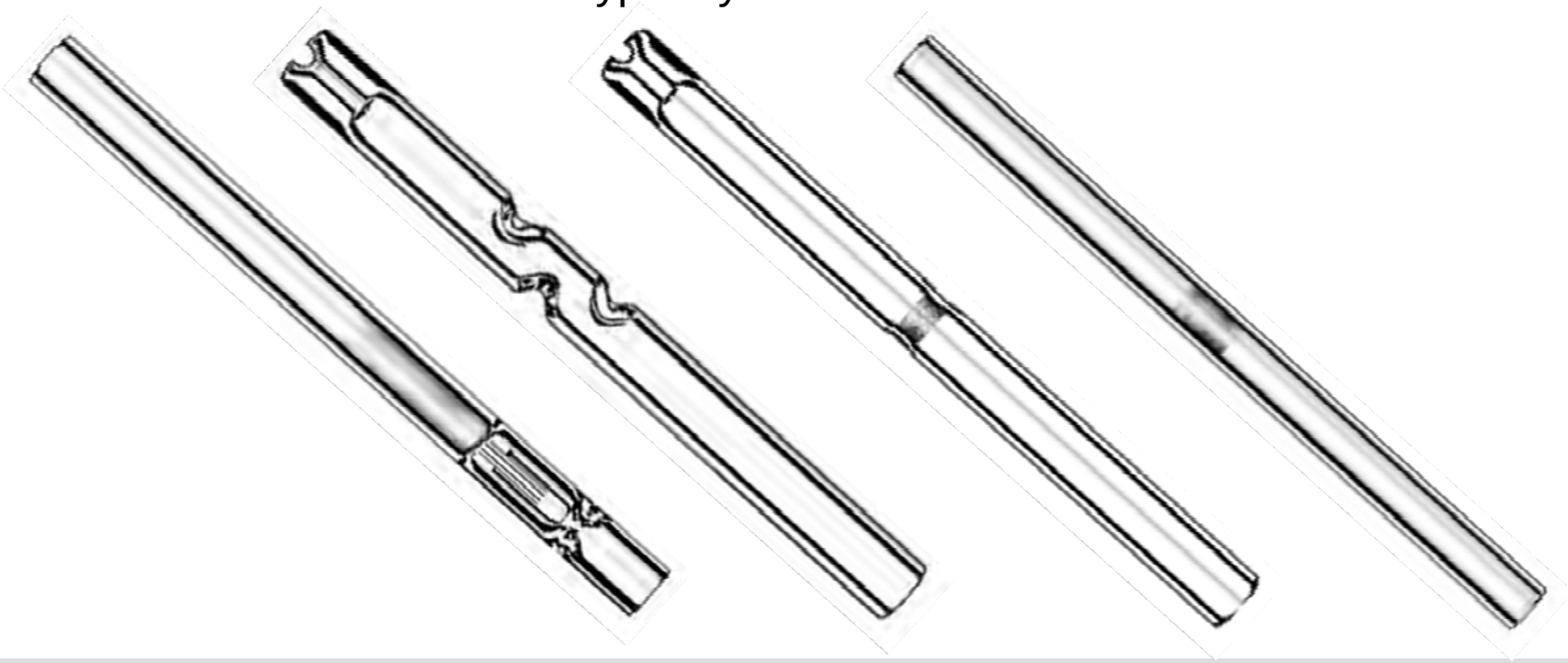
$$= 80 \text{ to } 1$$

Split ratios range from <5:1 (wide bore column applications) to >400:1 (Fast GC applications).
User should experimentally determine which split ratio works best for their application.

Split Injection

Liner Choices

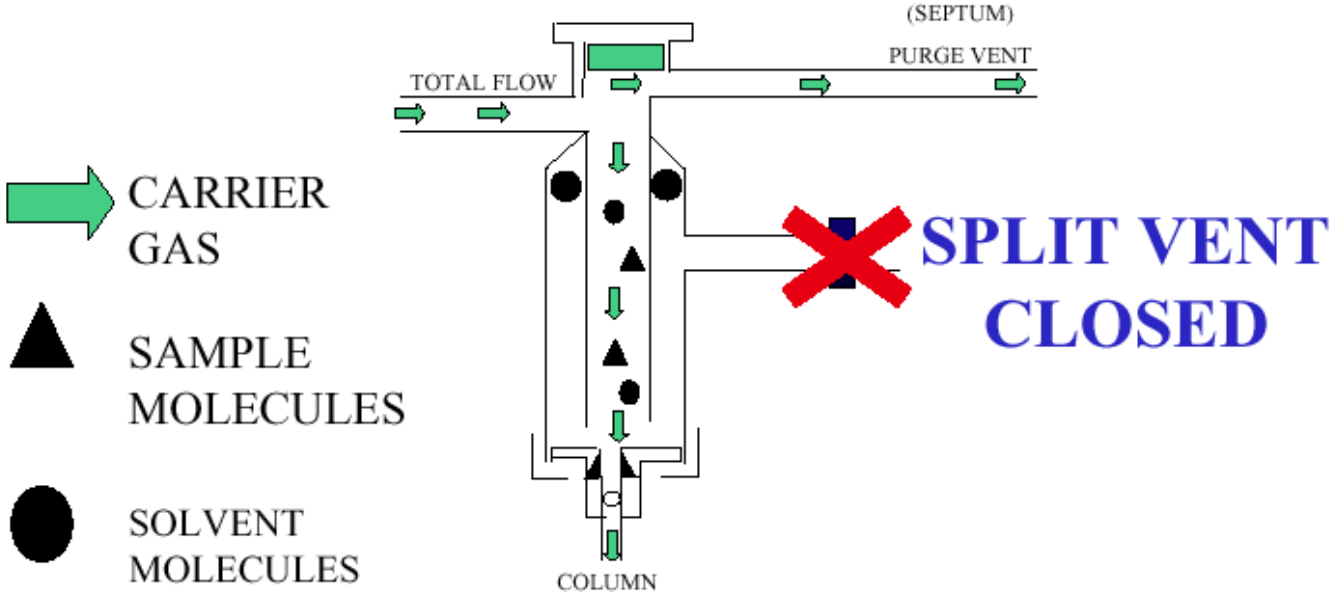
- Cups, baffles, twists, or frits establish turbulent flow rather than laminar flow
- Facilitate sample mixing prior to the point where the sample is split
- Wool may be used to improve vaporization
- 2-4 mm I.D. inlet liners are typically use



Splitless Injection

Useful for Trace Analysis

- Extract or sample is vaporized then mixed with carrier gas in the inlet
- Carrier gas transfers entire gas cloud into the column
- Analytes condense on column (oven 10-20 ºC below boiling point of solvent)
- Split vent opens to purge inlet after 1.5-2 inlet volumes have passed through
- Can be used with isothermal or temperature programmed analysis



Splitless Injection

When to Open the Split Vent?

- Split vent opens to purge inlet after 1.5-2 inlet volumes have passed through
- Open too soon…
  - Loss of response (especially for higher molecular weight analytes)
- Open too late…
  - Too much solvent enters column (may swamp early eluting analytes)

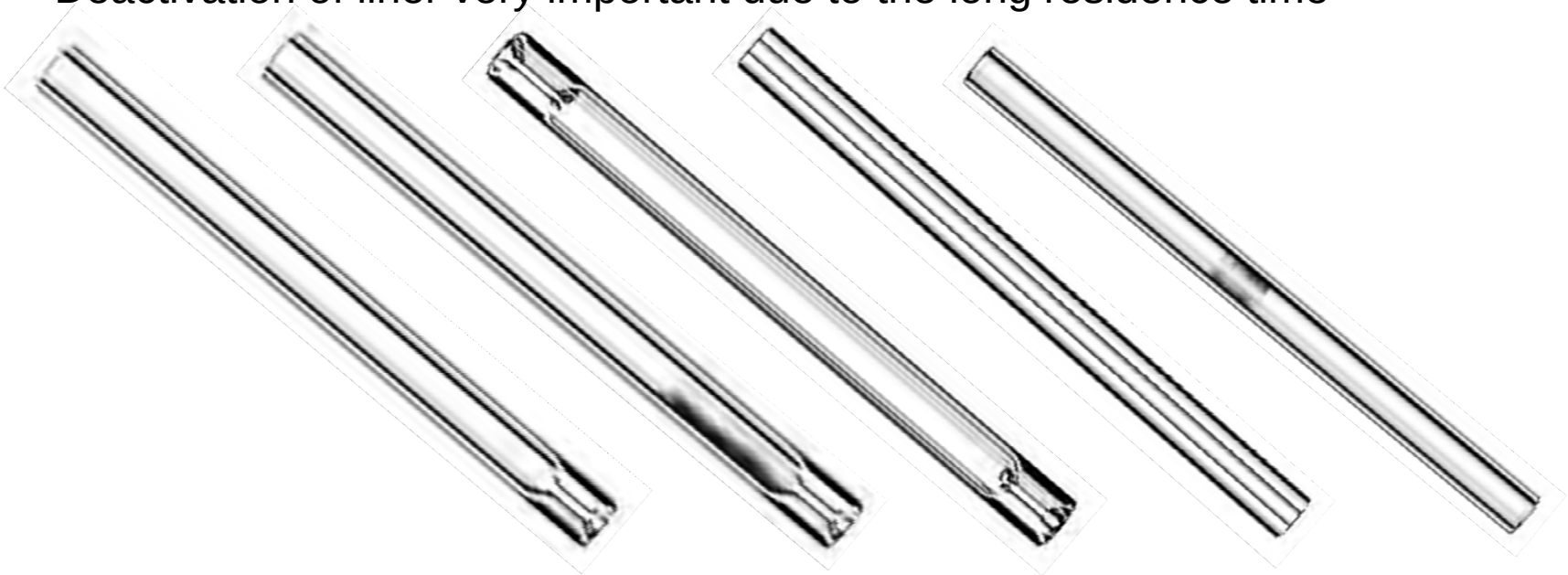
Typical times range from 30 seconds to 2 minutes.

User should experimentally determine which time works best for their application.

Splitless Injection

Liner Choices

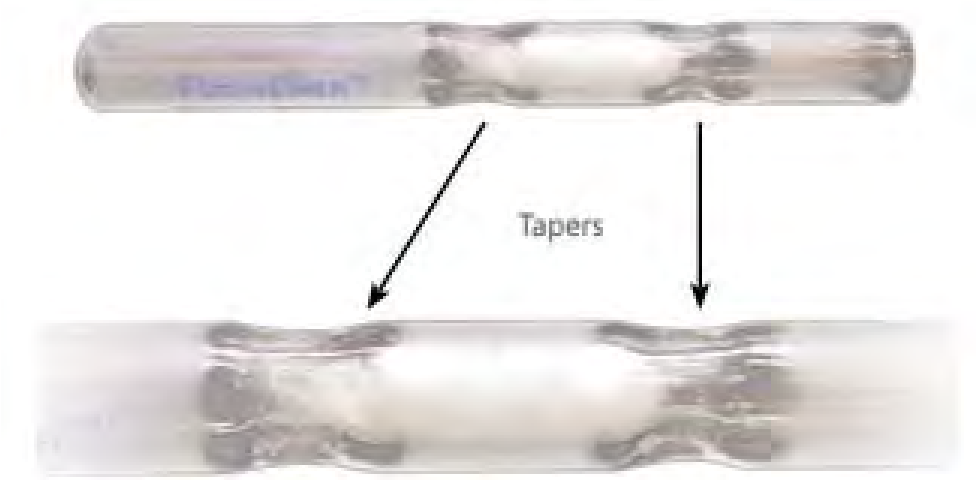
- Dwell time longer, so design does not need to create high turbulence
- Tapers may help focus analytes onto column
- Wool may be used to improve vaporization
- 2-4 mm I.D. inlet liners are necessary for solvent expansion
- Deactivation of liner very important due to the long residence time



Splitless Injection

FocusLiner™ Inlet Liners

- Wool plugs increases reproducibility
  - Increase surface area which facilitates maximum vaporization
  - Wipes off any droplets formed on the outside of the needle
- FocusLiner inlet liners incorporate a unique design to prevent shifting of the wool plug during repeated injections or sudden inlet pressure changes



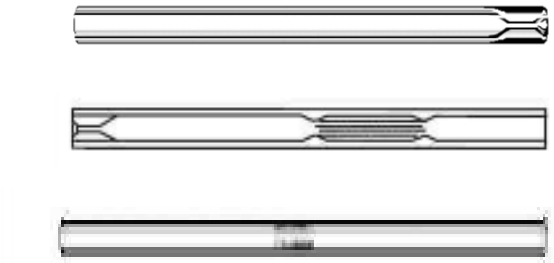
Suitable for split or splitless injection.

FocusLiner is a trademark of SGE Analytical Science Pty Ltd.

Splitless Injection

Inlet Liner Volume

- Inlet liner must contain the gas cloud formed after all the solvent vaporizes
- If not, can cause poor reproducibility, ghost peaks, and poor peak shapes



4 mm I.D., Single taper: 850 µL

4 mm I.D., FocusLiner w/taper 880 µL

4 mm I.D., Straight w/wool: 985 µL

Liner Volume

		200 ºC Inlet		300 ºC Inlet	
	B.P. (ºC)	10 psi	30 psi	10 psi	30 psi
Methylene chloride	40	360	200	437	241
Methanol	64.5	570	315	691	382
Water	100	1279	706	1548	855

Resulting vapor volume of solvent

Use an inlet liner with an internal volume equal to or larger than the expansion volume of the solvent.

On-Column Injection

Useful when Analytes have Vastly Different Boiling Points

- Liquid extract or sample deposited directly into a 0.53 mm I.D. column
- Inlet liner with tapered region
  - Creates seal between column and liner
  - Guides the needle into the column (special syringe required)
- Must be used with temperature programmed analysis
- Eliminates splitter discrimination (inaccurate quantification)

Direct Injection

Useful for Gas Phase Samples

- Use with headspace, purge and trap, and solid phase microextraction (SPME)
- Entire gas cloud is transferred to the column
- Can be used with isothermal or temperature programmed analysis
- No solvent, so little solvent expansion (large inlet liner volumes not needed)
- Narrow bore 0.5-1.5 mm I.D. inlet liners are used to maintain a high linear velocity through the injection port, minimizing band broadening

Supelco site
Bellefonte, Pennsylvania (USA)

Thank You!