Get Your GC Methods In-Line with the Correct Liner

Mark Sinnott
January 20, 2022

DE33650537

Agilent
Trusted Answers

Agenda

Liners

General Considerations

- Wool, no wool, tapers, frits etc.

Goals of sample introduction

Split injection over-view

- Liner selection

Splitless injection over-view

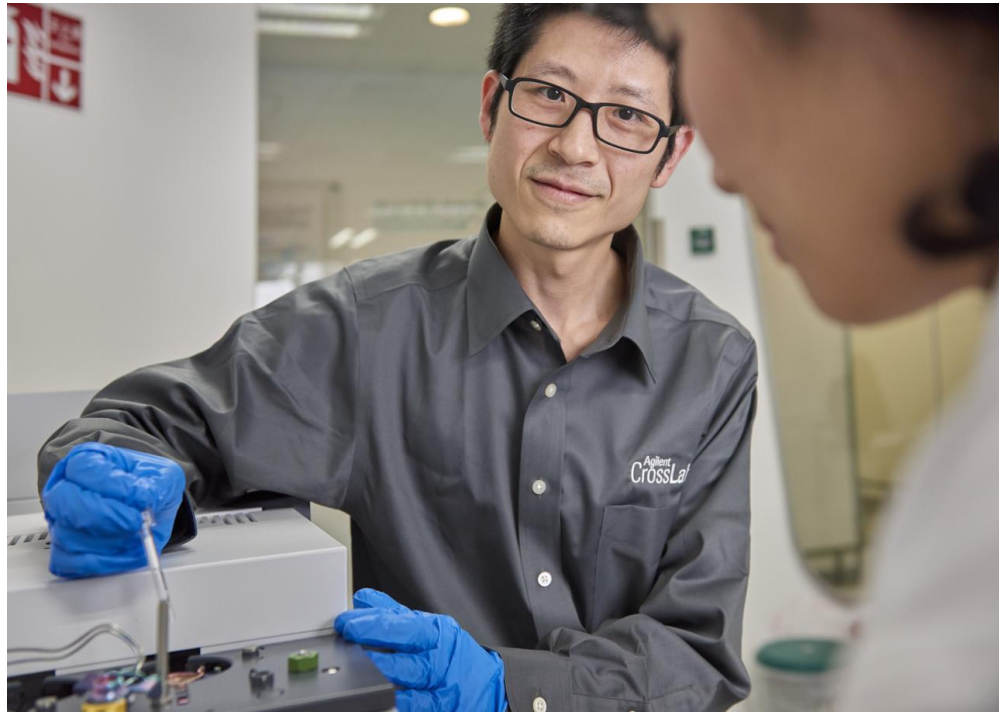
- Liner selection

Specialty Liners

Cross-Lab liners

Examples

Summary/conclusions



Liners - General

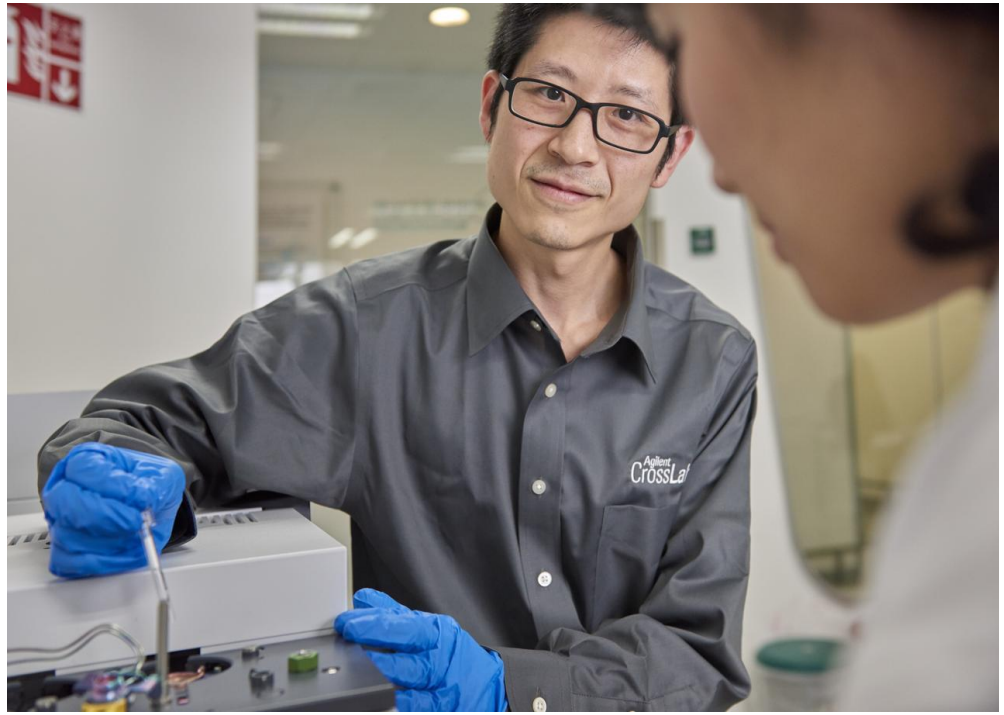
Liners

Purpose of liners

- Provide an “inert” space for liquid samples to vaporize

Key aspects

- Liner volume
- Treatment or deactivation
- Special characteristics (glass wool or frit, cup, taper)
- Type of injection



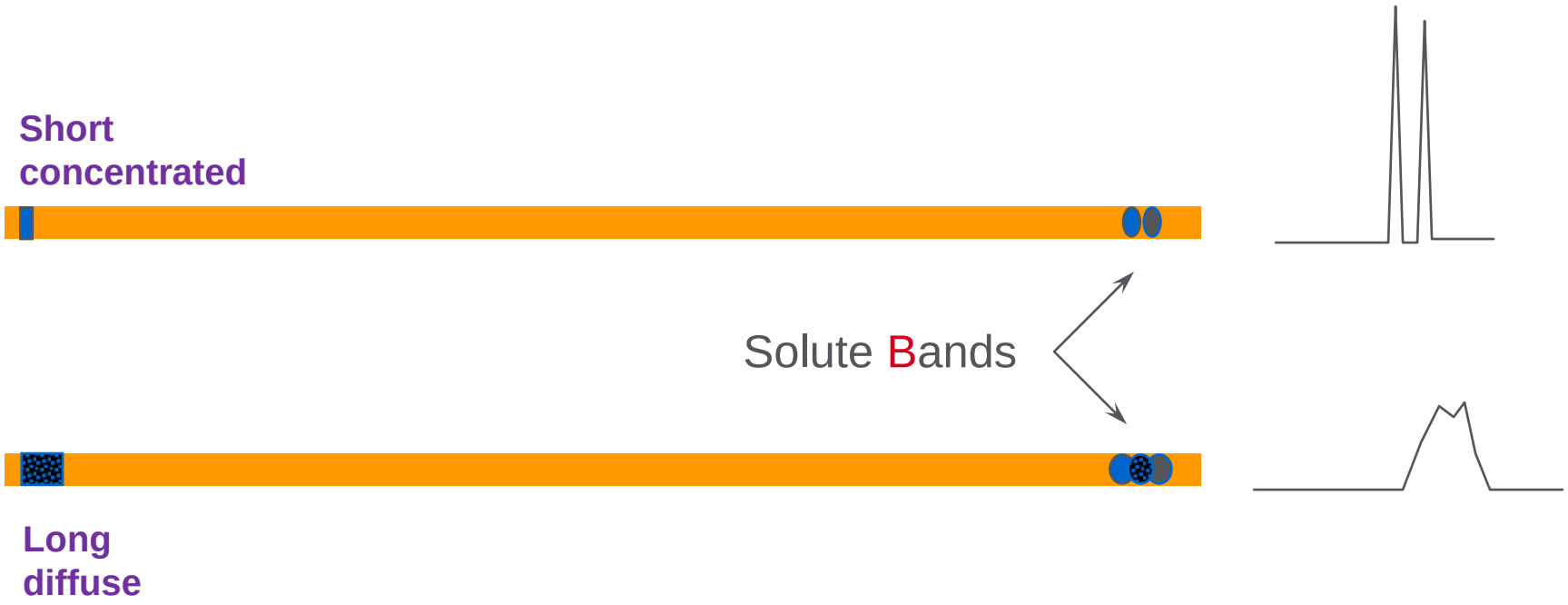
Inlet Choices

Inlet	Column	Mode	Sample Concentration	Comments	Sample to Column
Split/splitless	Capillary	Split Purged split Splitless Purged splitless	High High Low Low	Most commonly used inlet. Very flexible	Very little Very little All All
Multimode	Capillary	Split Pulsed split Splitless Pulsed splitless Solvent vent	High High Low Low Low	Flexibility of standard S/SL inlet and PTV	Very little Very little All All Most
Cool-on-column	Capillary	N/A	Low or labile	Minimal discrimination and decomposition	All
Packed	Packed Large Capillary	N/A N/A	Any Any	OK if resolution is not critical	All All
Programmed temperature vaporizaton	Capillary	Split Pulsed split Splitless Pulsed splitless Solvent vent	High High Low Low Low	Not great for hot injections Can concentrate analytes and vent solvent	Very little Very little All All Most
Volatiles interface	Capillary	Direct Split Splitless	Low High Low	Purge and trap / headspace	All Very little All

Sample Introduction Goals

- ***Efficiently*** introduce sample into the column
  - Equally as important as the column itself
- Needs to be reproducible
- Minimize efficiency losses
- Representative of sample

Influence of Injection Efficiency



Same column, same chromatographic conditions

Liners - General

Liners – Volume

Choose a liner with enough volume to accommodate the vaporized sample

- Especially important for polar solvents with large vapor volumes (i.e. water)
- If vapor volume exceeds liner volume, samples may backflash
  - May cause ghost peaks and reproducibility issues

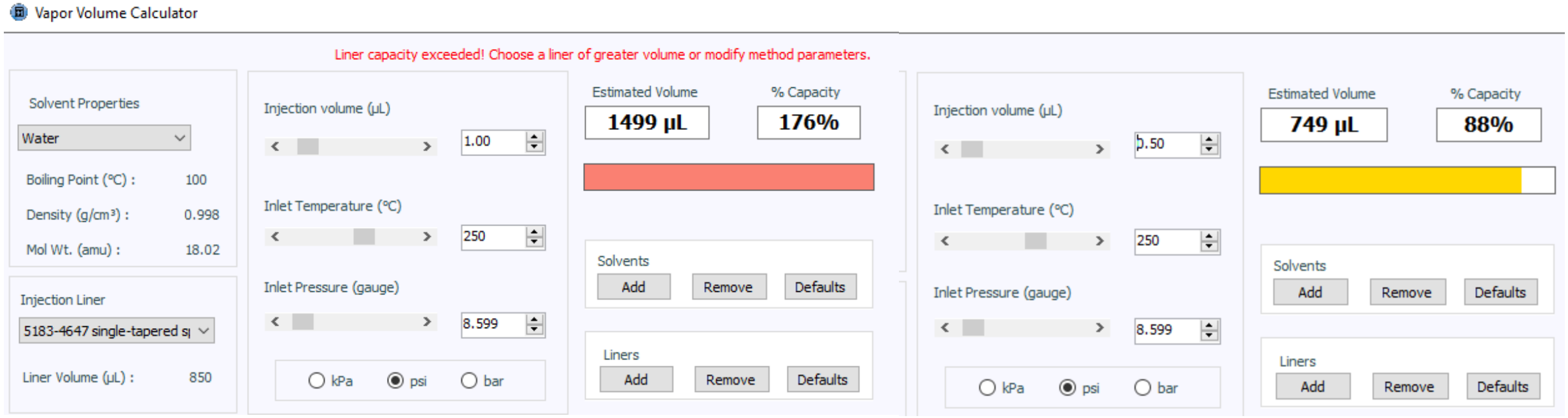
Agilent liners are primarily 2 or 4 mm in inner diameter and 78 mm long.

- Therefore:
  - 2 mm liners hold 245 µL of vapor
  - 4 mm liners hold 972 µL of vapor

Recommended injection volumes are 1 to 2 µL or less for organic solvents and 0.5 µL or less for water.

Liners - General

Backflash



Water as a solvent:

Watch injection volumes, keep at 0.5 µL or less

Best to calculate vapor volume

Liners - General

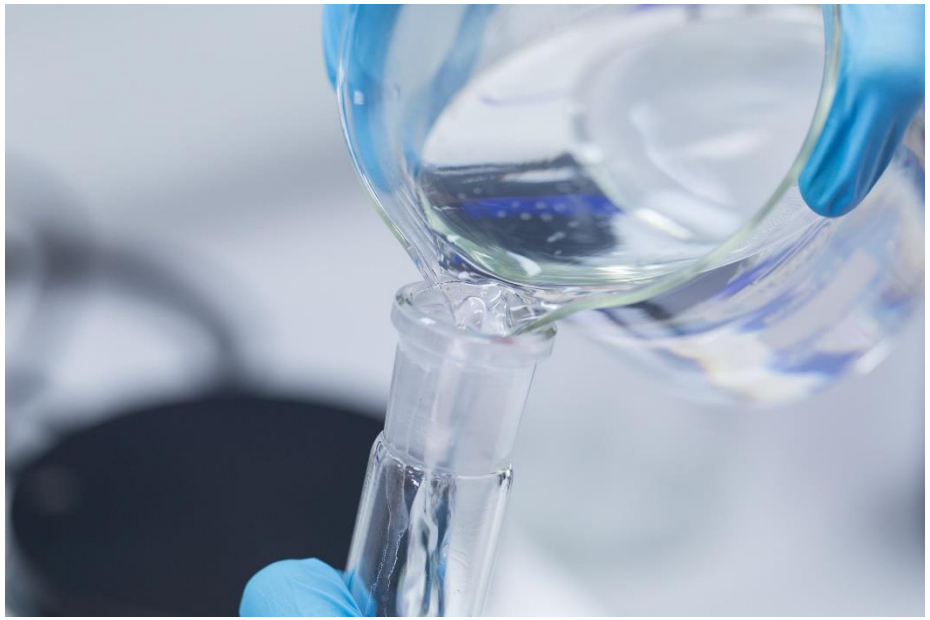
Liners – Deactivation

Minimize adsorption of active compounds to surfaces

- Unwanted adsorption can lead to poor peak shape and lower response
  - Deactivated liners are usually treated with a silylating reagent

Agilent has a few different deactivation options:

- Ultra inert
- Original
- None



Liners - General

Liners – Special characteristics

Some liners have special features required for different injection techniques

- **Taper (gooseneck)** – Minimizes sample contact with gold seal
- **Dual taper** – Minimizes contact with gold sea, inlet weldment, and reduces potential for backflash
- **Glass wool/frit** – Prevents nonvolatiles from reaching column, helps with vaporization of heavier compounds, and can help remove residual sample from the needle (split liners)
- **Jennings cup** – Used for sample mixing in split inlets, reduces sample discrimination, prevents nonvolatiles from reaching the column. For clean samples.
  - Good for manual Injections
- **Press fit (direct) connection** – Bottom is designed to hold capillary column firmly (almost all sample goes onto the column). Side hole required for EPC with Direct Connect liners.
- **Others**
  - Baffles, spiral paths, laminar cups, column packings with stationary phase
    - All provide a turbulent sample flow path for mixing, a way to collect high molecular weight sample components or particles, and surface area to allow efficient vaporization of sample components.

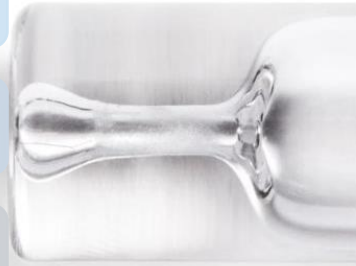
Liner Characteristics

What is glass wool used for?

Filtration	•Prevents nonvolatile matrix from entering column
Vaporization	•Provides volatilization surface for liquid injections, promotes mixing with carrier gas
Needle wiping	•Increases reproducibility by wiping needle after injection

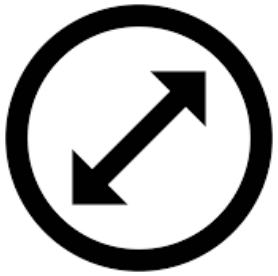
Straight or tapered?

Bottom taper	•Focuses sample on the head of the column •Minimizes contact with metal inlet parts
Center taper	•Holds wool in place
Top taper	•Reduces sample backflash •Minimizes sample exposure to inlet



Does liner diameter have an effect?

Inner diameter	•Small or large id for splitless injections •Large injections need large diameter
Outer diameter	•Large od ideal for splitless injections •Slower transfer, snug fit directs flow to the column •Smaller OD reduces pressure drop



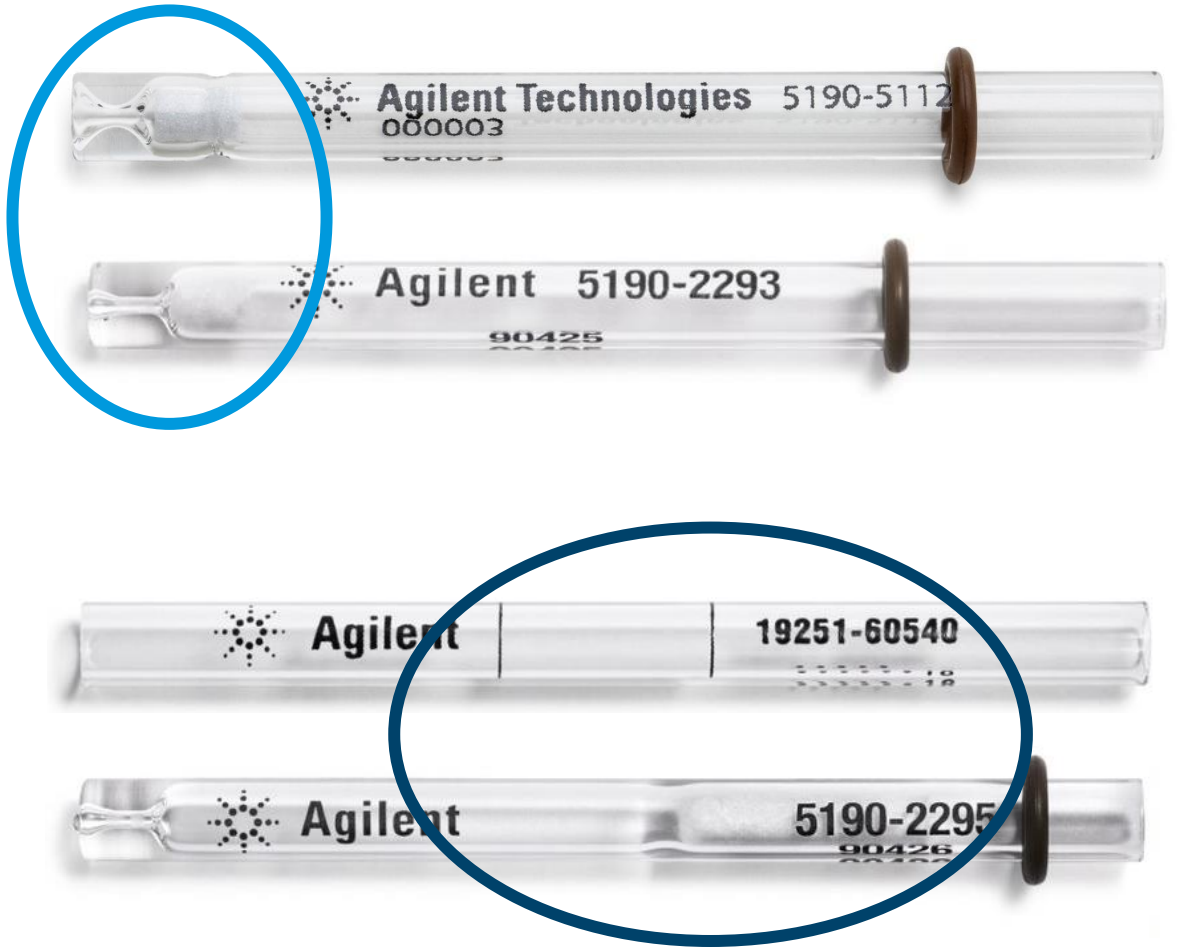
What does a glass frit do?

Equivalent vaporization	•Identical chemical performance as glass wool
Enhanced consistency	•Increased consistency of porosity while preventing foreign objects from entering the flowpath
Increased lifetime	• Allows for up to a 2x increase in liner lifetime

But How Do I Pick the Right Liner?

Splitless injections
- Ultra Inert deactivation
- Bottom taper
- Barrier (wool, dimpled, or frit)
  - Not always necessary w/splitless
  - Typically at the bottom

Split injections
- Ultra Inert deactivation
- Straight or tapered
- Barrier (wool or frit)
  - Much more critical
  - Typically at the top/middle
  - Wool wipes needle



Split Injection
Overview

Small fraction of the sample is introduced into the column

Used for high concentration samples

Superior injection efficiency = narrow peaks = high resolution

Split Injection
Major variables

Split ratio – Determines the fraction of sample on-column and efficiency of injection (sensitivity versus peak width)

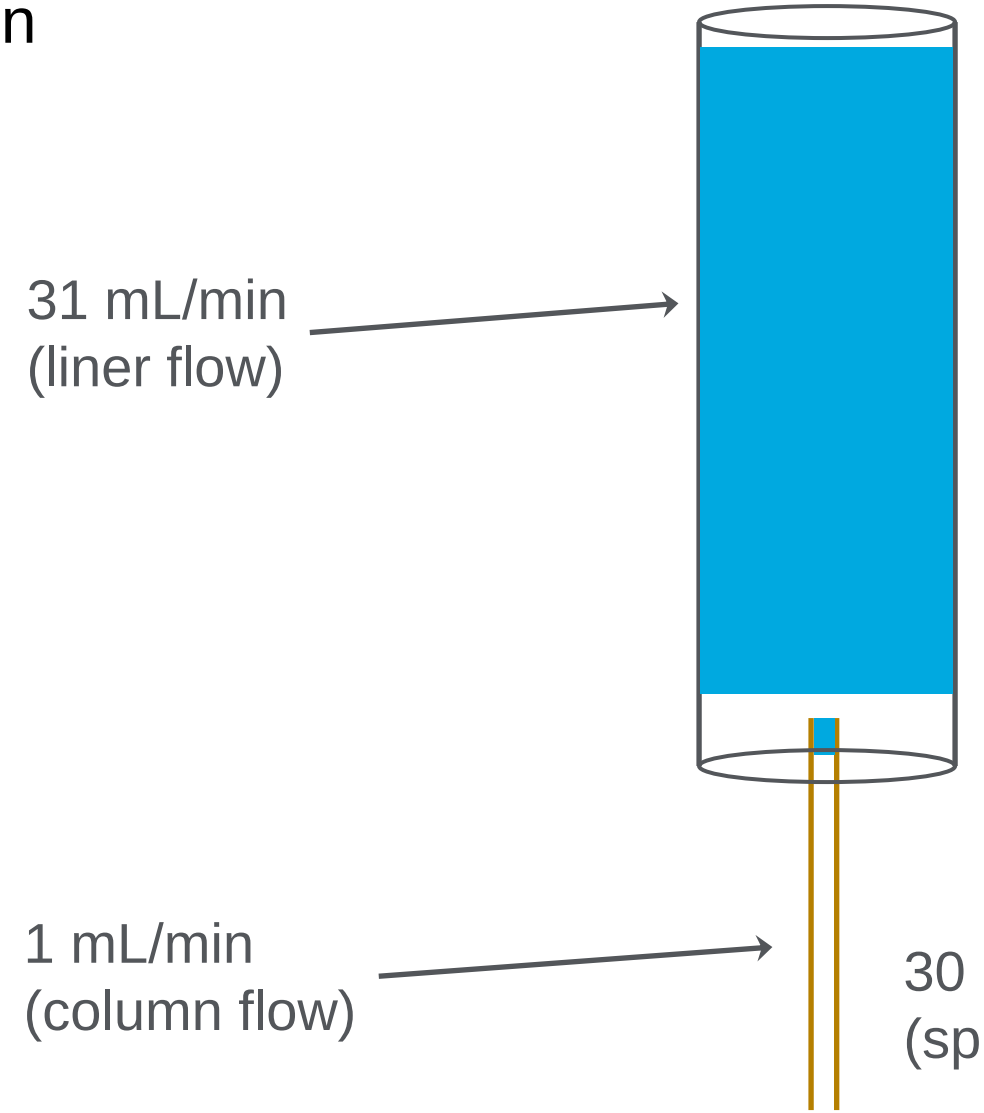
Liner – Influences efficiency of vaporization/discrimination

Temperature – Hot enough to vaporize sample without degradation or backflash

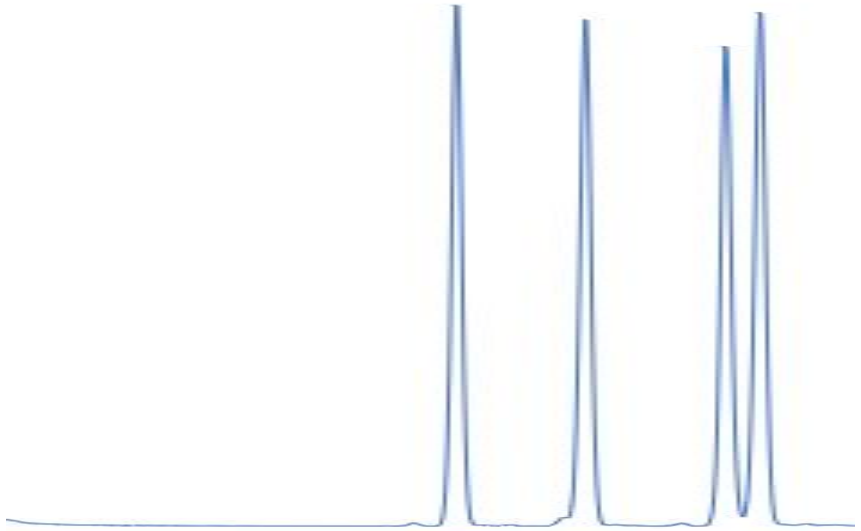
Injection volume – Typically 0.2 to 1 µL, increasing it does not have as much of an effect as you might expect (smaller is usually always better if you can meet RSD requirements)

Split Injection

Animation

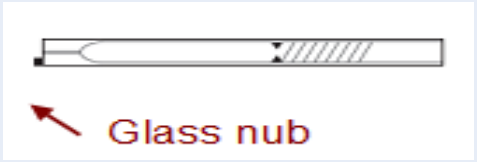
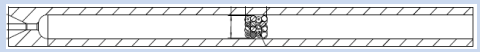


30:1 split ratio



Inlet

Liners – Split injection

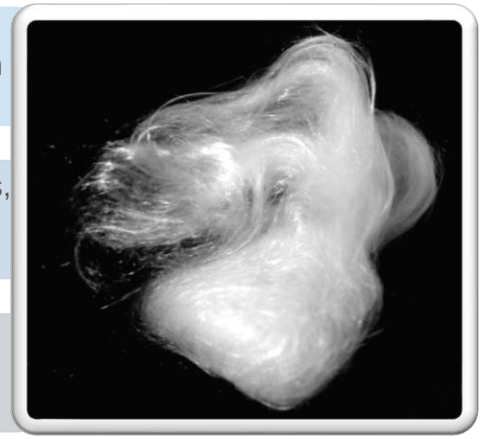
Liner	Part Number Each 5/pk 25/pk	Comments
	5190-2294 5190-3164 5190-3168	Simplest split liner, glass wool, UI deactivation, large volume (990 µL). Use for general purpose, can be used in splitless mode
	5190-2295 5190-3165 5190-3169	Glass wool, UI deactivation, 870 µL volume. Glass nub ensures that a gap remains below liner for split injection. Efficient for most applications
	5190-5105 5190-5105-005 5190-5105-025	Sintered glass frit, UI deactivation. Ideal for actives. Sintered glass frit more reproducible than glass wool
	5188-5396 5188-5398 5188-5397	Helix – Spiral feature creates turbulence; high surface area ($$)
	18740-80190	Liner with Jennings cup, no wool. 800 µL volume. Reduces inlet discrimination. Manual injections

Why a Barrier (Glass Wool or Frit)?

Filtration
- Prevents nonvolatile matrix from entering column

Vaporization
- Provides volatilization surface for liquid injections, promotes mixing with carrier gas
- Less inlet discrimination

Needle wiping
- Increases reproducibility by wiping needle after injection



Frit

Glass wool

When should I use a liner with a frit or glass wool?

- Complex (dirty) matrices
- Viscous matrices
- High split ratio

Split liners:

Split/splitless liner with glass wool, low pressure drop

Split injections have a higher carrier gas flow through the liner to help split the sample

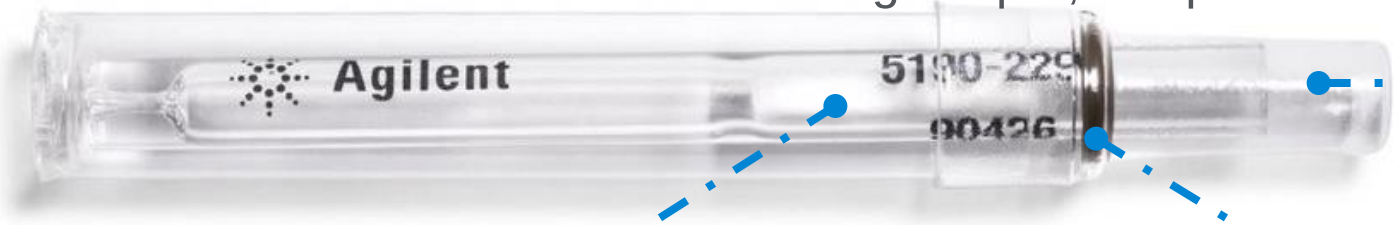
- Faster transfer onto column
- Split liners have a smaller outer diameter than splitless liners to help flow circulate
- Can also do splitless injections on a split liner (but not vice versa)

If potential exists for sample discrimination between low and high boiling components

- Use a liner with wool

Ultra Inert liners enable excellent peak shapes for tricky analytes

- 5190-2295 is a recommended liner – single taper, low pressure drop



Touchless packaging ensures contamination-free installation

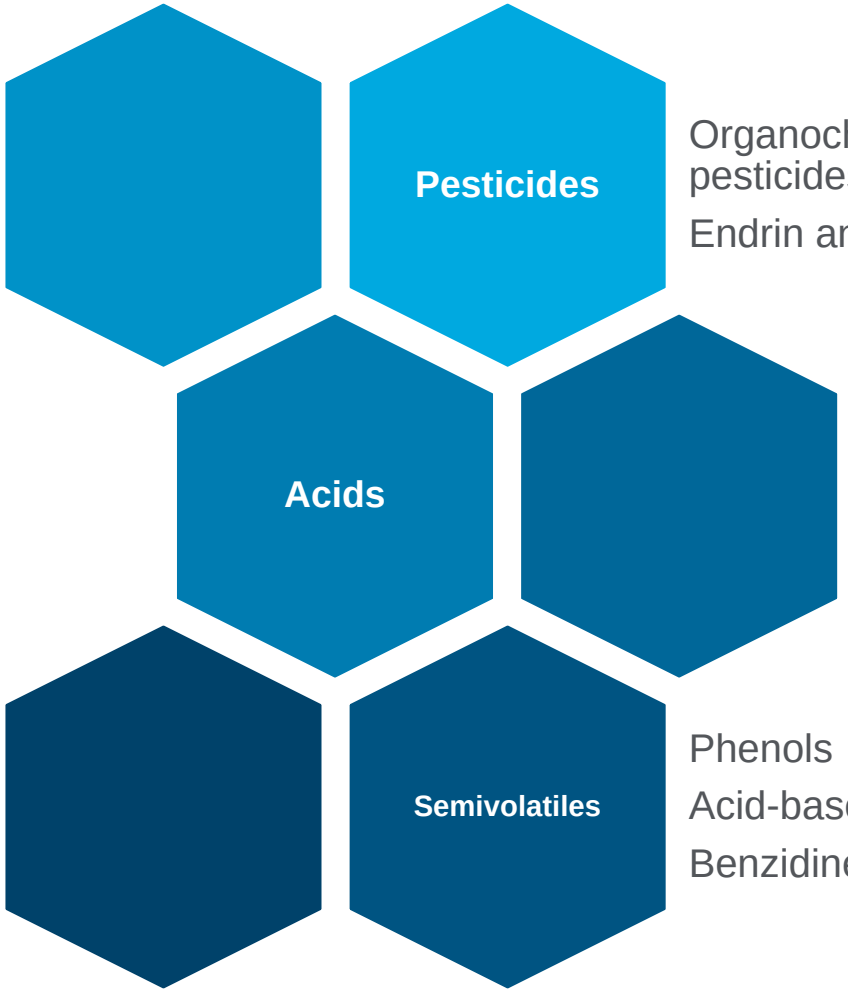
Preinstalled O-ring

Glass wool plug in upper position wipes needle, avoids sample discrimination, improves P&A, and collects septum particulate

Low pressure drop bead promotes better carrier gas flow for split injections

The Benefits of the Glass Frit

Dislodged glass wool fibers expose active sites that interact with sensitive analytes



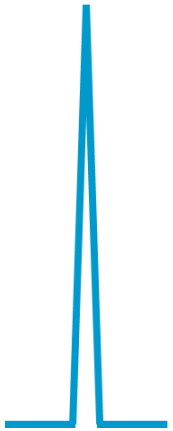
Organochlorine pesticides

Endrin and DDT

Phenols

Acid-base pairs

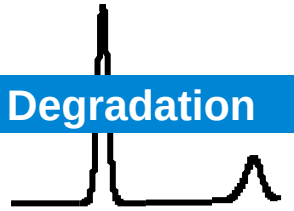
Benzidine



Interaction with active sites results in

Tailing

Degradation



Response loss

Split liners

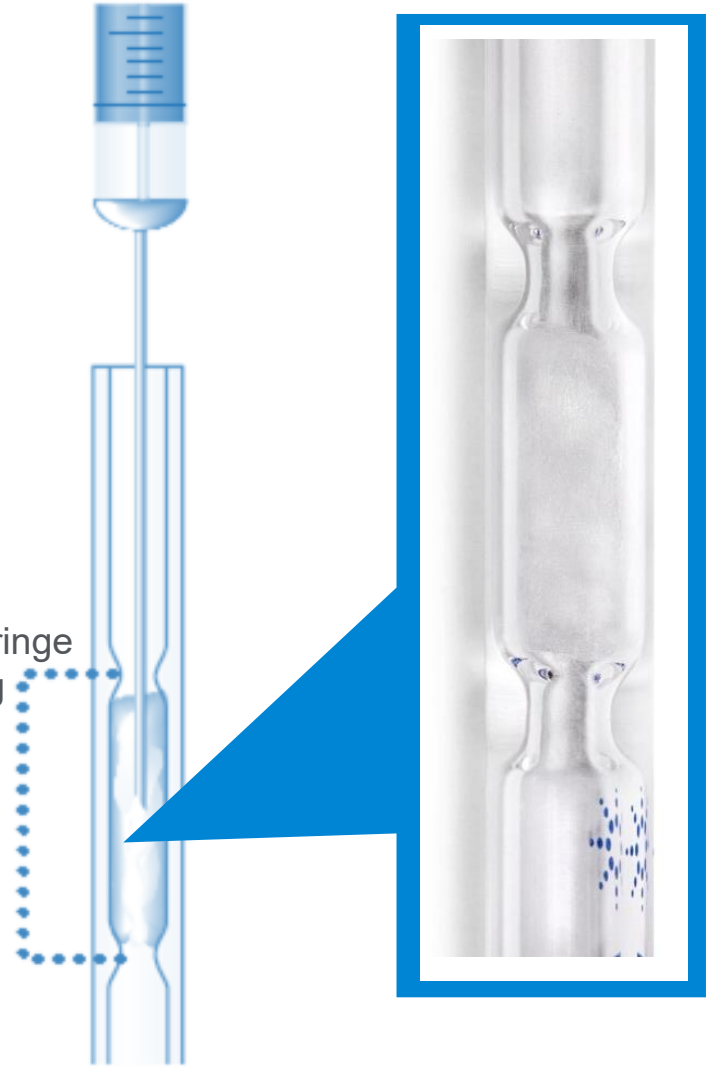
Focus liners always have wool

Geometric feature: Internal taper

- Positions wool high in liner body
  - Prevents wool shifting/migration
- “Focuses” syringe needle during injection
  - Wipes residual droplet from needle tip
  - **Improves injection reproducibility**

Ideal for split analysis

- Concentrated or high matrix samples
- Commonly used for TPH analysis



Straight Focus flow

flow Tapered Focus

Split liners

Straight liner with or without wool

Basic, general-purpose liner

Varying internal diameters

- Id changes the volume
- Smaller id → smaller injection size

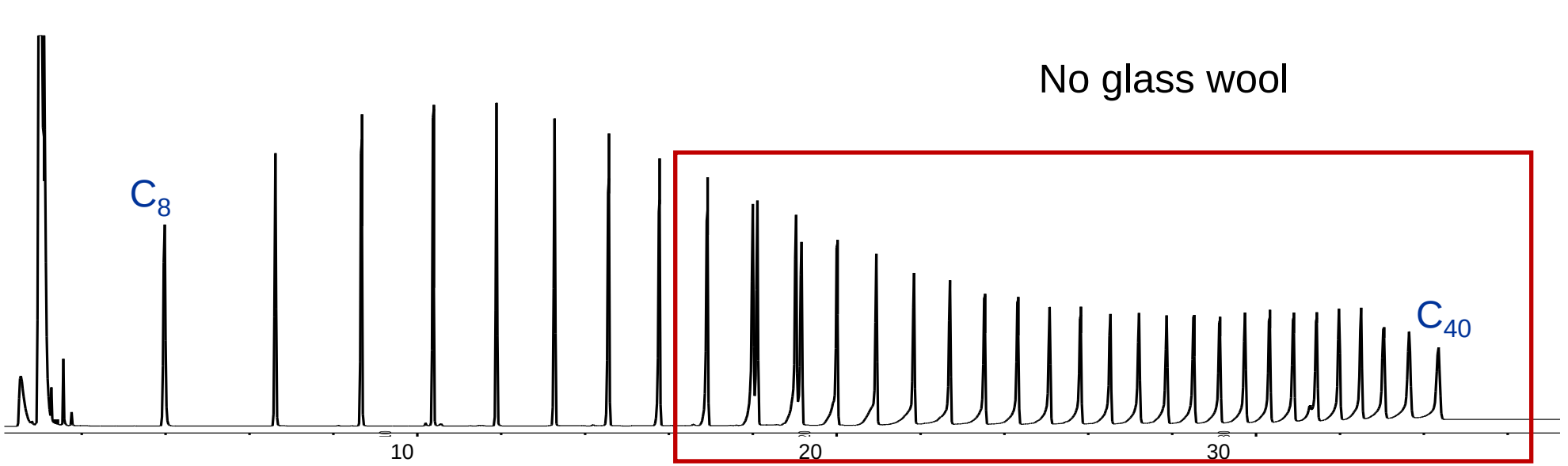
With or without wool

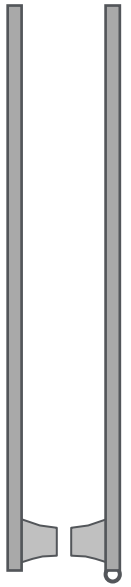
- May result in more maintenance if glass wool is not used with heavy matrix samples

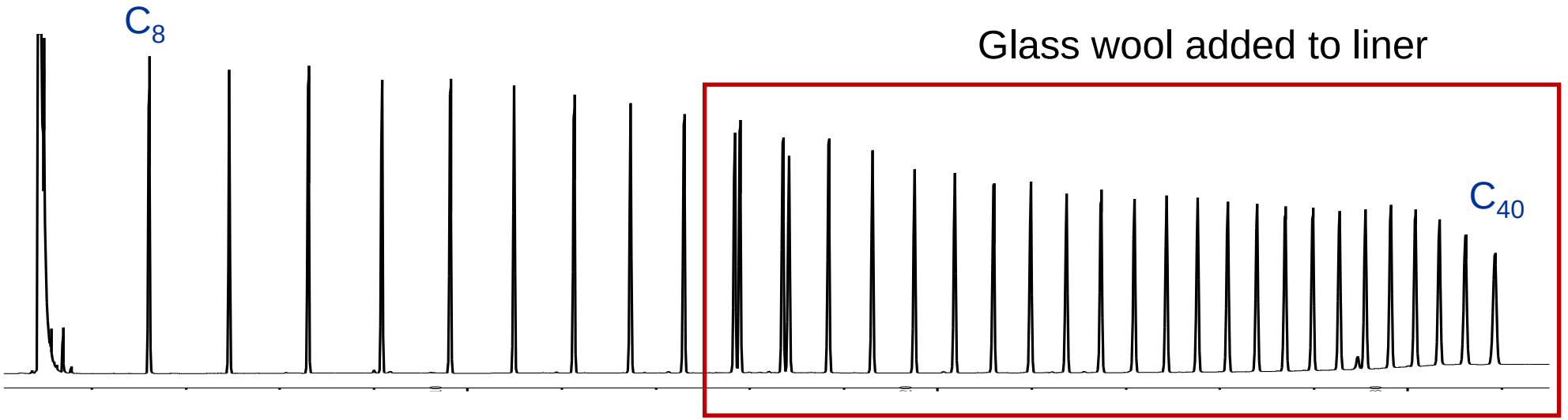
Use when

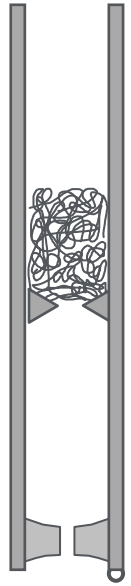
- Precision isn’t critical
- For higher concentration, nonactive analytes in clean matrices
- Absence of taper results in broader injection dispersion → can result in broader peaks
- Wool more likely to shift

What Does Mass Discrimination Look like?









Splitless Injection

Overview

More challenging the SPLIT

Most of the sample is introduced into the column

Used for low concentration samples

Poor injection efficiency = wider peaks = less resolution

Sample refocusing may be necessary

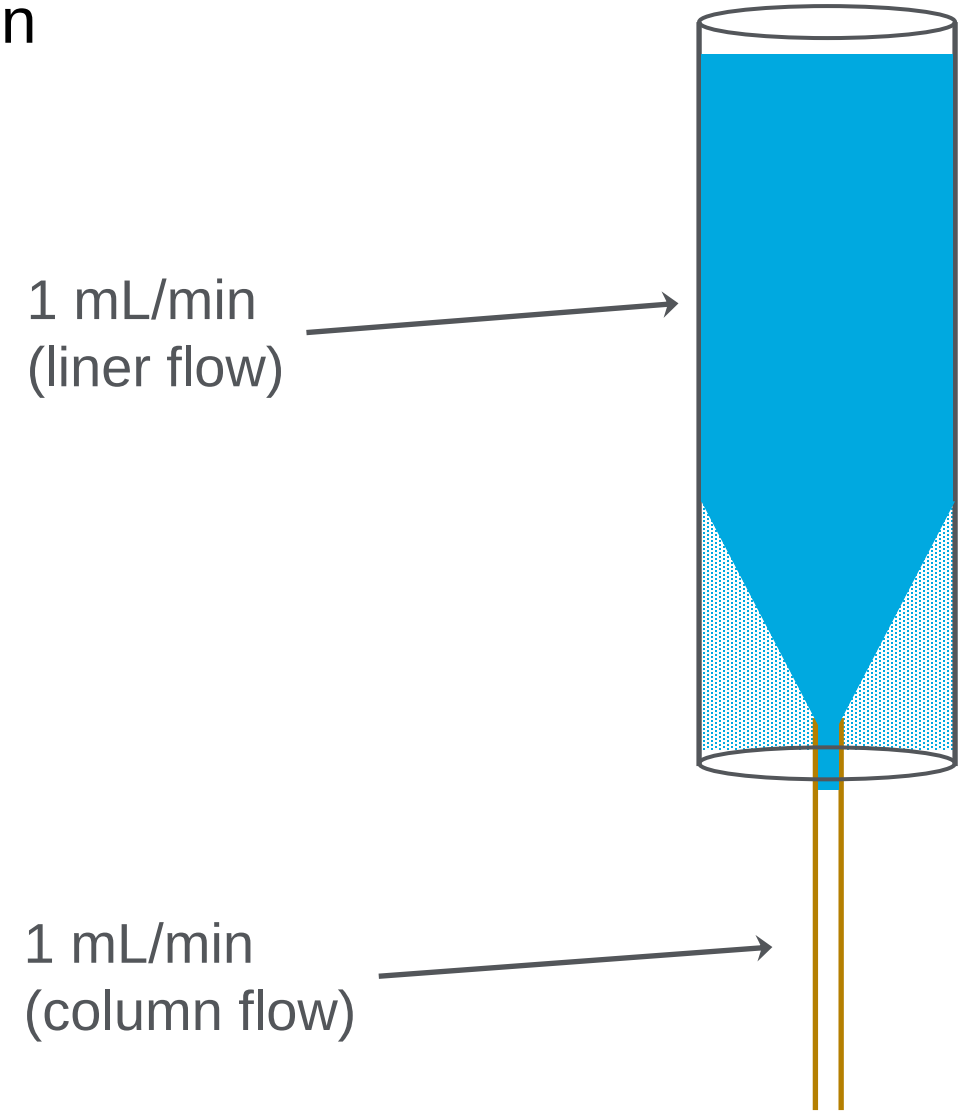
Splitless Injection

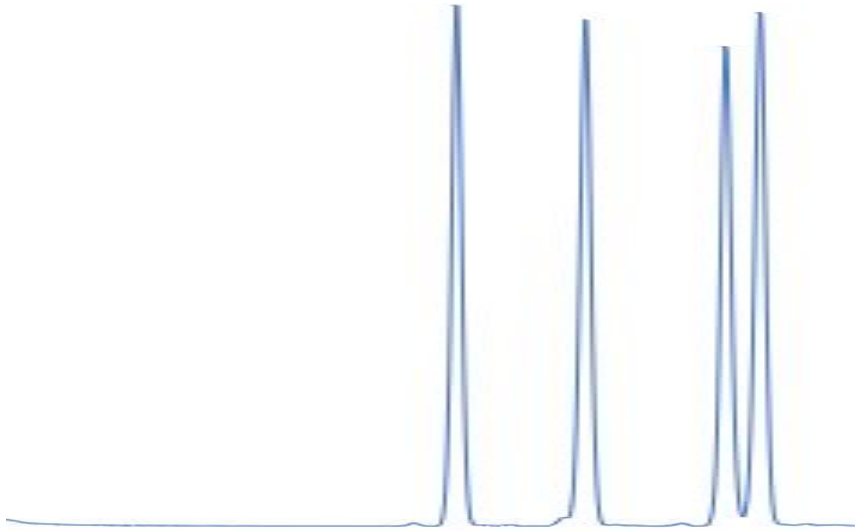
For trace level analysis

- Use split/splitless injection port or MMI in the splitless mode (split vent closed)
- The dilute sample is injected, the sample is volatilized, and most of the analytes and solvent are introduced to the column
- Later, the split vent is opened and the residual solvent is vented (purge time/flow)
- The timing, carrier/split vent flows, and oven temperature program are important
- The sample has longer residence time in the heated inlet giving more opportunity to vaporize high boiling sample components compared to split injection
- Typical splitless parameters:
  - Purge flow of 50 mL/min
  - Purge time of 0.5 to 2.0 minutes

Splitless Injection
Animation

Purge flow 30 mL/min
Purge Time 0.5 min





30 mL/min
after 0.5 min

Waste/split vent%

Splitless Injection

Major variables

Purge activation time – Determines amount of sample onto column and efficiency of injection (sensitivity versus peak shape)

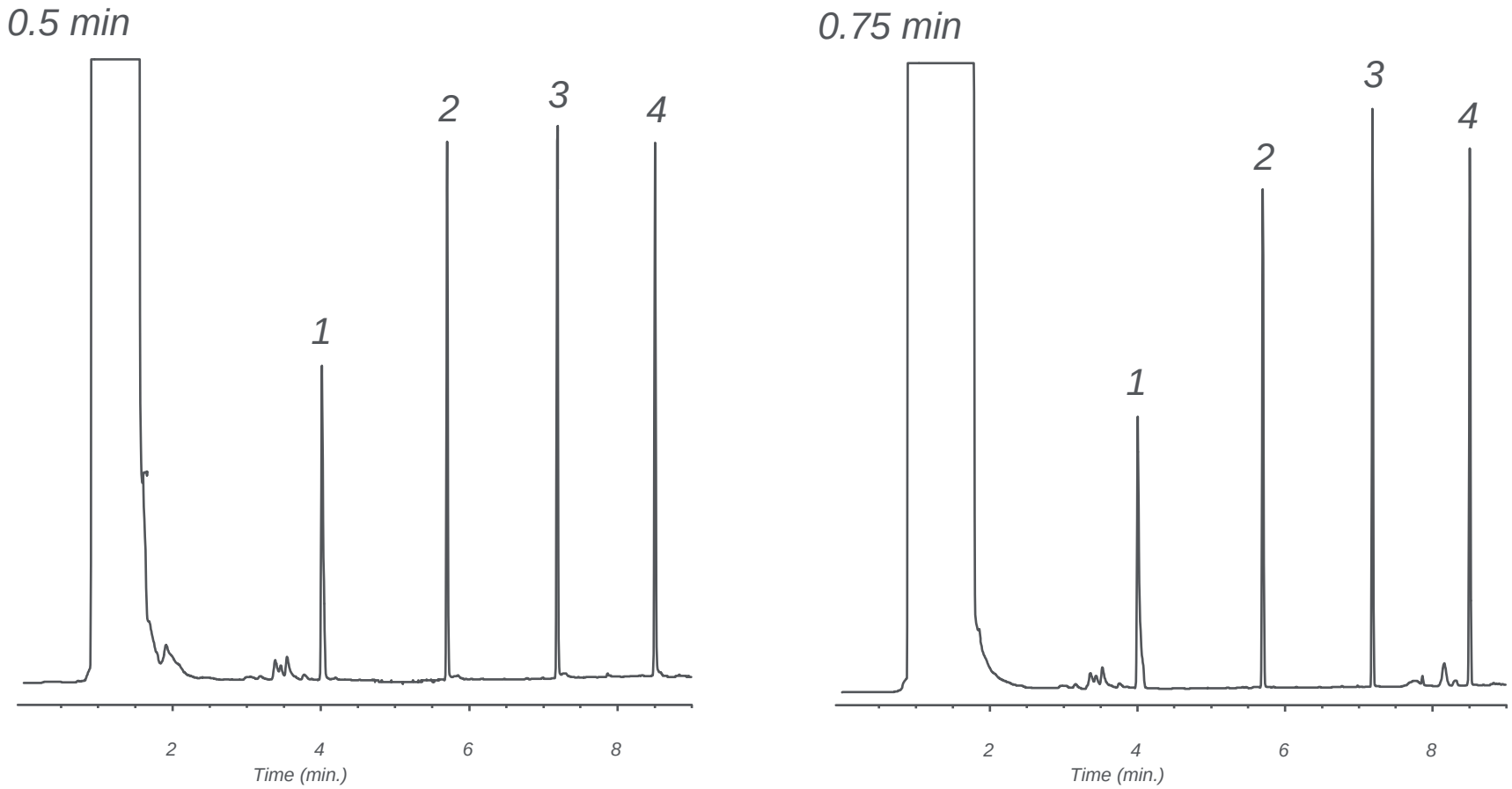
Liner – Preventing backflash is more critical than vaporization properties (liner volume, tapers, and wool are less important…)

Injection volume – Typically 1 µL or less (backflash: 0.5 µL max for water)

Temperature – Long residence times allow for lower temperatures

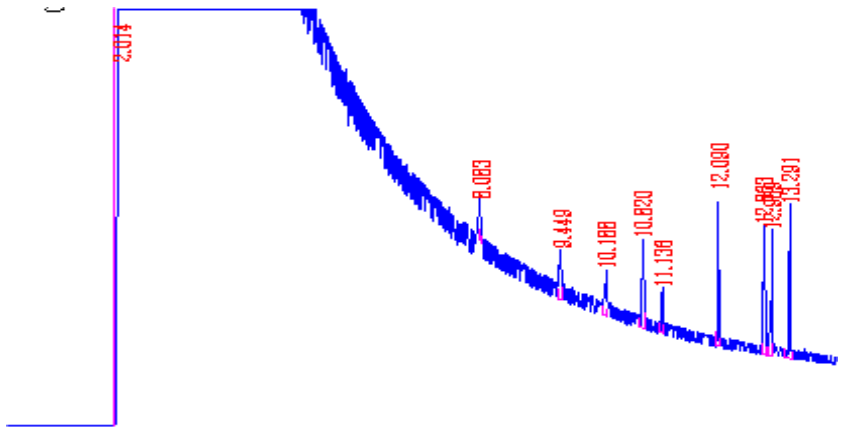
Splitless Injection

Purge activation time

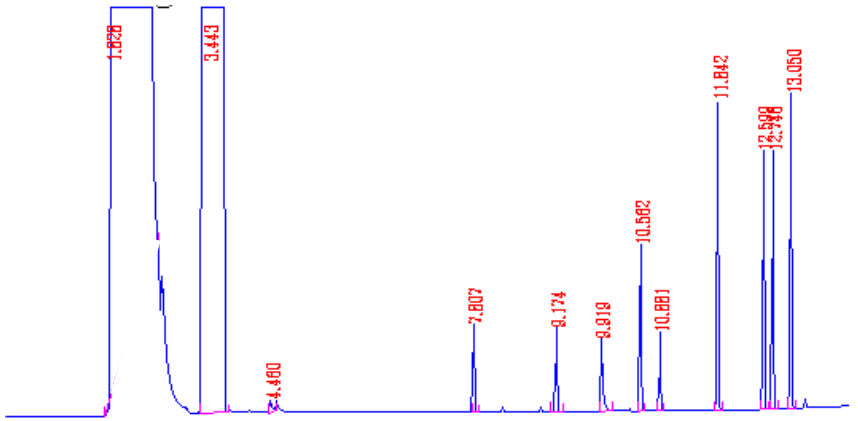


DB-1, 15 m x 0.25 mm id, 0.25 µm
60 °C for 1 min, 60-180 °C at 20°/min; Helium at 30 cm/s
1. n-decane 2. n-dodecane 3. n-tetradecane 4. n-hexadecane

Splitless Injections – Splitless Time (Purge Time On)



If the purge time is too long, it will results in large solvent tail



0.75 min purge time “clips” the solvent tail

Splitless: Sample Refocusing and the “Solvent Effect”

- Splitless injections are inherently inefficient
- Sample refocusing
  - Also known as the “solvent effect”
  - Condenses sample as a thin film on the head of the column
  - Initial oven temperature must be at least 10 °C below the solvent boiling point
  - Increases separation efficiency and resolution for better peak shape
    - Especially for low boiling analytes

- “Cold trapping” is a version of sample refocusing for high boiling analytes
  - Occurs when the starting oven temperature is ~150 °C below the boiling point of analytes of interest
  - Condenses the analytes on the head of the column
  - Results in better peak shapes

- Solvent effect and cold trapping can occur in the same sample
  - When looking at analytes with a wide distribution of boiling point

Splitless injection

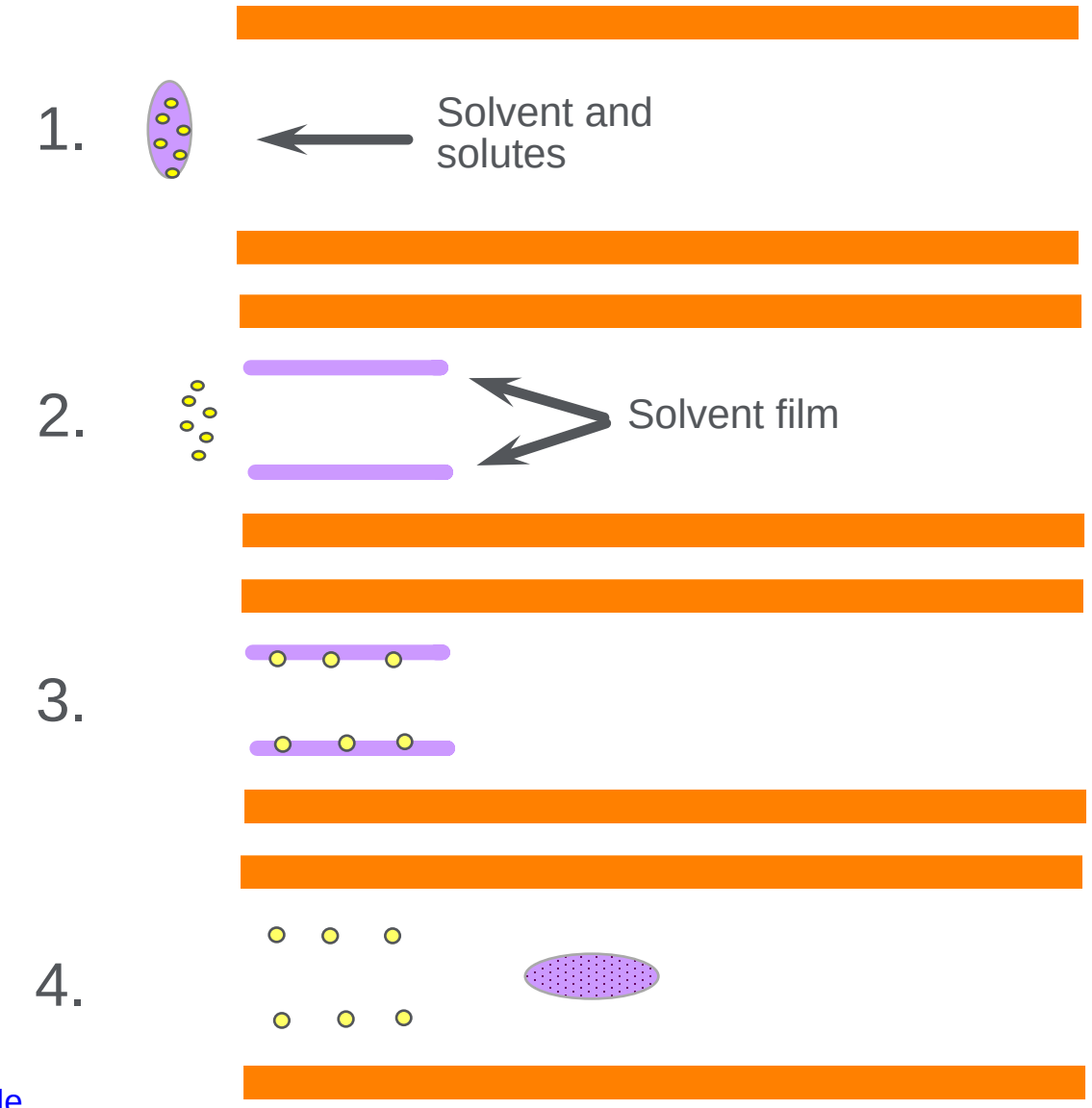
Solvent effect

Initial column temperature at least **10 to 20 °C below** sample solvent boiling point

Required to obtain good peak shapes unless cold trapping occurs

Rule of thumb, if solute boiling point is >150 °C above initial column temperature, the solute will cold trap

Cold trapping has greater efficiency than solvent effect



Solvent and stationary phase must be compatible

Inlet

Liners – Splitless injection

Liner	Part Number Each 5/pk 25/pk	Comments
	5190-2292 5190-3162 5190-3166	Single taper, UI deactivated, 900 µL volume. Taper isolates sample form gold seal, reducing breakdown of active compounds. Trace samples, general applications.
	5190-2293 5190-3163 5190-3167	Single taper, UI deactivated, glass wool, 900 µL volume. Glass wool aids volatilization of heavier compounds and protects the column. Trace, dirty samples.
	5190-5112 5190-5112-005 190-5112-025	Singer taper, UI deactivated, sintered glass frit. Glass frit acts like glass wool, but is more reproducible
	5190-3983 5190-4007 ****_****	Double taper, UI deactivated, 800 µL volume. Taper on inlet reduces backflash. High efficiency for trace, active samples.
	5190-7011 (5/pk) 5190-7012 (5/pk) 5190-7013 (5/pk) 5190-7014 (5/pk) 5190-7020 (5/pk)	Direct Connect liners, single and dual taper, original deactivation. Column press fits into liner. Focuses almost all sample onto column and reduces exposure to inlet. Ultimate for trace, active samples. Various hole placements for use with EPC.

Splitless Liners

Single taper with or without wool

Splitless has lower flows through liner

- Splitless liners are typically wider for a more snug fit
  - Ensures all available flow funnels through the liner, not around
- You can perform split injections with a **split** liner, as long as split ratio is not too high
  - Poor reproducibility, not enough room for high flows to the vent

In low carrier gas flow splitless analysis, a **bottom taper** helps focus analytes onto head of column

Agilent Ultra Inert liners enable excellent peak shapes for tricky analytes

- 5190-2293 is the recommended splitless liner – single taper, with wool

Small plug of **glass wool** near bottom of liner filters matrix

Splitless liners

Single taper with or without wool (alternately a frit)

Splitless has lower flows through liner

- Splitless liners are typically wider for a more snug fit
  - Ensures all available flow funnels through the liner, not around
- Do **not** do split injections on a splitless liner
  - Poor reproducibility, not enough room for flow

Ultra Inert liners enable excellent peak shapes for tricky analytes

- 5190-2293 is a recommended splitless liner – single taper with wool
- 5190-5112 is a recommended splitless liner – single taper with frit

In low carrier gas flow splitless analysis, a **bottom taper** helps focus analytes onto head of column

Small plug of **glass wool** near bottom of the liner filters the matrix

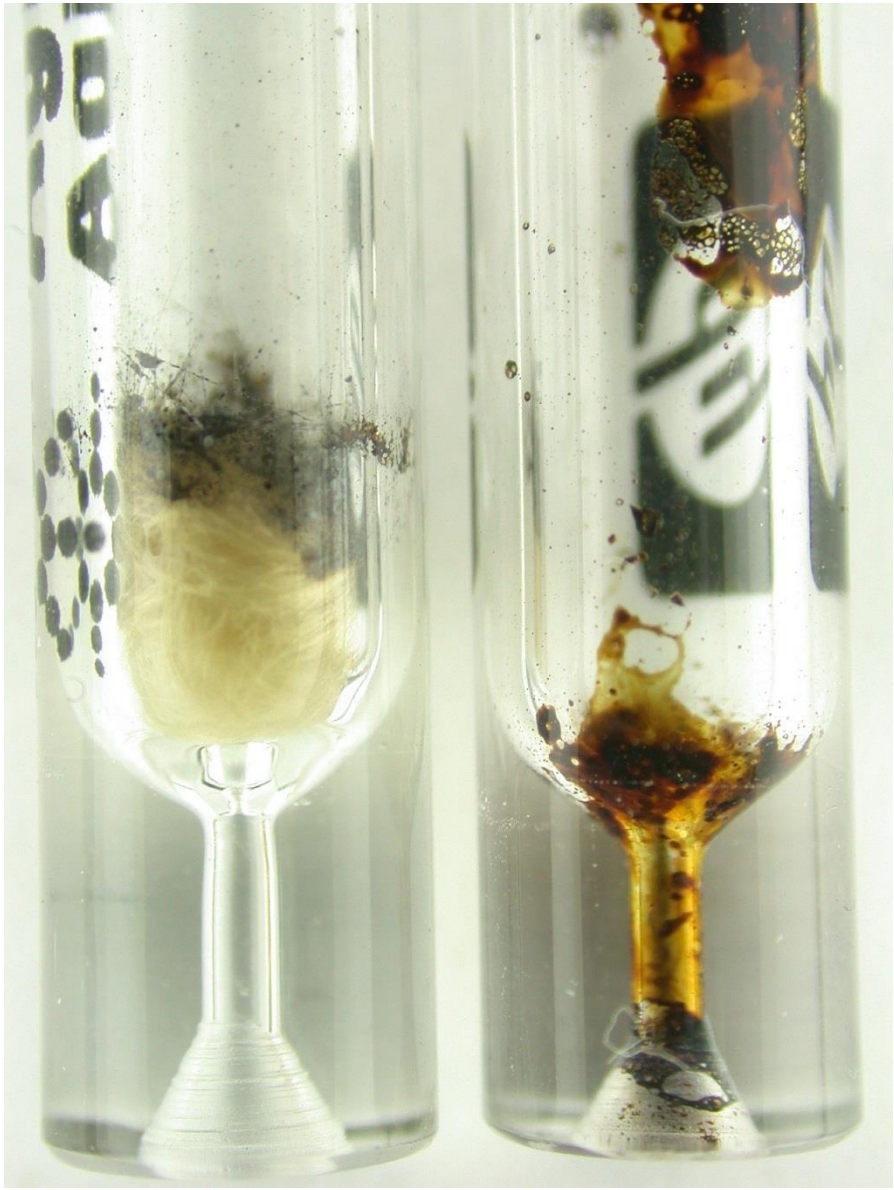
Small sintered **glass frit** near bottom of the liner filters the matrix

Splitless

Silylated glass wool

- Traps nonvolatile materials and mixes sample

- Peak shape and discrimination affected by amount, location, and packing density



Splitless liners – Specialty

Direct Connect

Directly connects capillary column to liner

Part No.	Glass Wool?	Taper Style?
5190-7011	No	Double taper
5190-7012	Yes	Single taper
5190-7013	No	Single taper
5190-7014	Yes	Single taper
5190-7020	No	Single taper

- Splitless injections
  - Nowhere else for the sample to go
  - Trace analytes
  - Longer residence time in liner → glass wool unnecessary
    - Longer residence time results in wider peaks → counteracted by Direct Connect liner
  - Better suited for wider bore columns (less back pressure/higher flow)
- Minimal sample loss during injection
  - Increases sensitivity
- Best for clean/low-matrix samples
  - Caveat – more matrix on the head of the column (use wool)

- Ideal for active analytes
  - Limits sample exposure to active sites within the inlet
- Why a hole?
  - Hole prevents pressure shutdown due to excessive backpressure
  - Direct Connect Liner with hole**

    necessary in models prior to 7890
  - EPC redesigned in 7890 models to better accommodate backpressure changes
- Top hole is better if….
  - Analytes elute later than solvent peak
- Bottom hole is better if…
  - Analytes elute close to solvent peak where long tailing solvent peak could merge with early eluting peaks
  - Bottom hole sweeps entire liner more efficiently

Splitless liners - Specialty

Dimpled liners

Typically Used with MMI injectors

- Cold splitless injection
  - Small internal volume → incompatible with most hot splitless injections

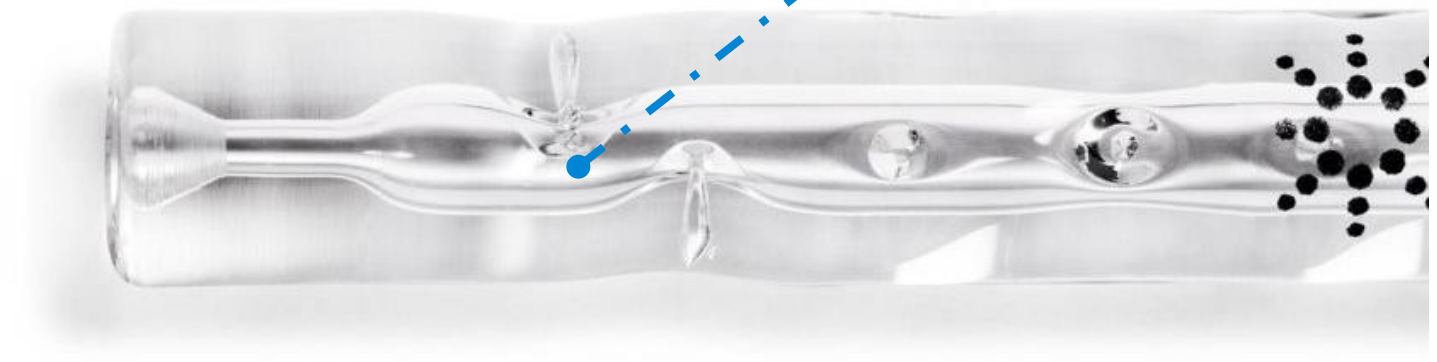
Ideal for high matrix samples

- Matrix spreads out on internal liner surface, bouncing from dimple to dimple
  - Filters matrix, prevents it from reaching the column
    - Longer lifetime
    - Reduces inlet/column maintenance
- Pesticides in food extracts
- Environmental extracts

No glass wool

- Ultra Inert is effective for very active compounds

Sample matrix spreads and collects on dimples, preventing it from reaching the column inlet

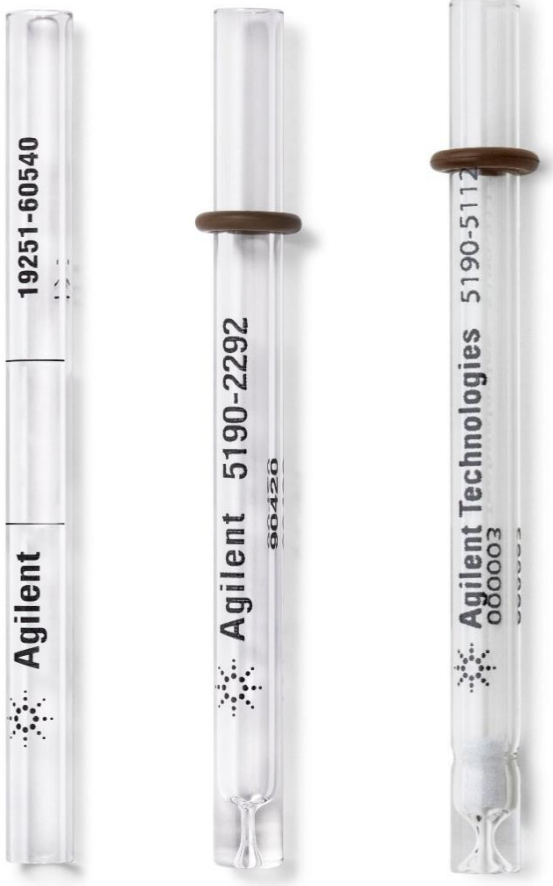


Ultra Inert dimple liner

Dimple liner

General Rule for Liner Installation

Liners must be inserted so that the Agilent logo and Spark are inserted first and face the bottom of the inlet, unless otherwise indicated by the liner print.



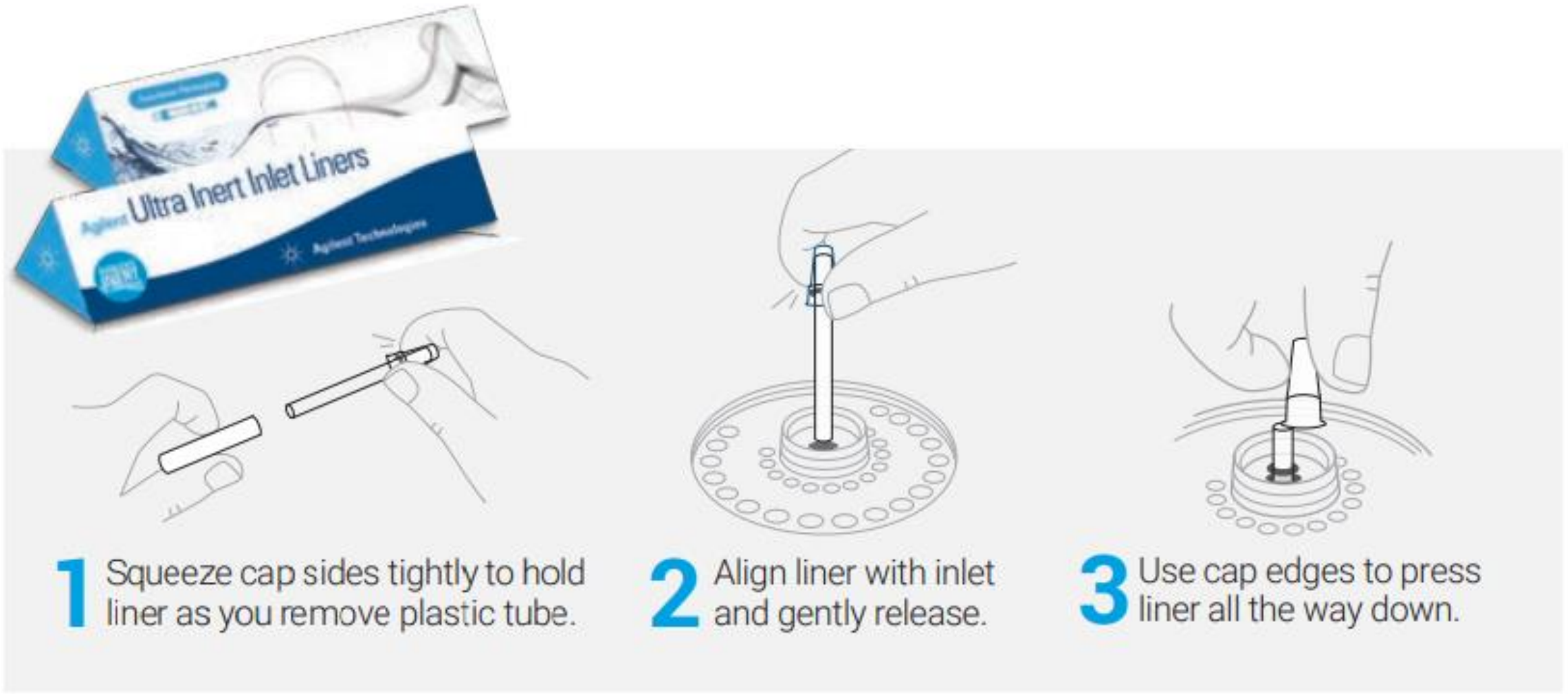
Top of inlet

Bottom of inlet

The exception

General Rule for Liner Installation

Touchless Packaging

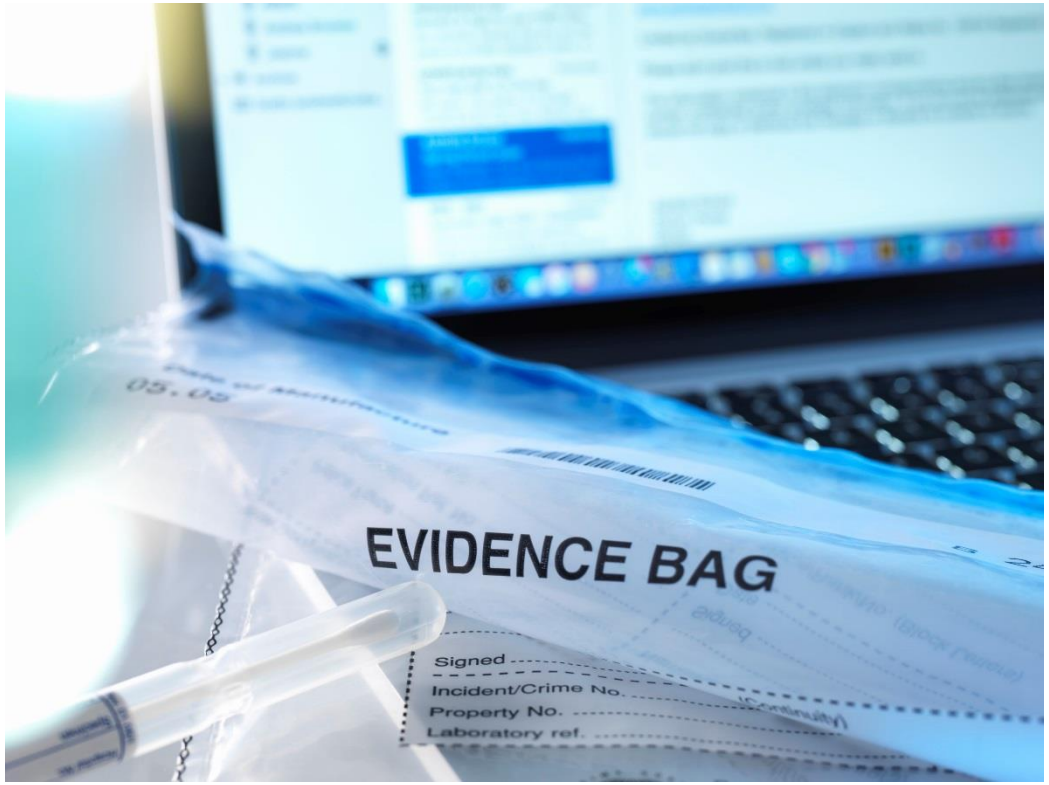


Inlet Liner Troubleshooting

- Many chromatographic problems are blamed on the column
- Often, a dirty liner is the culprit

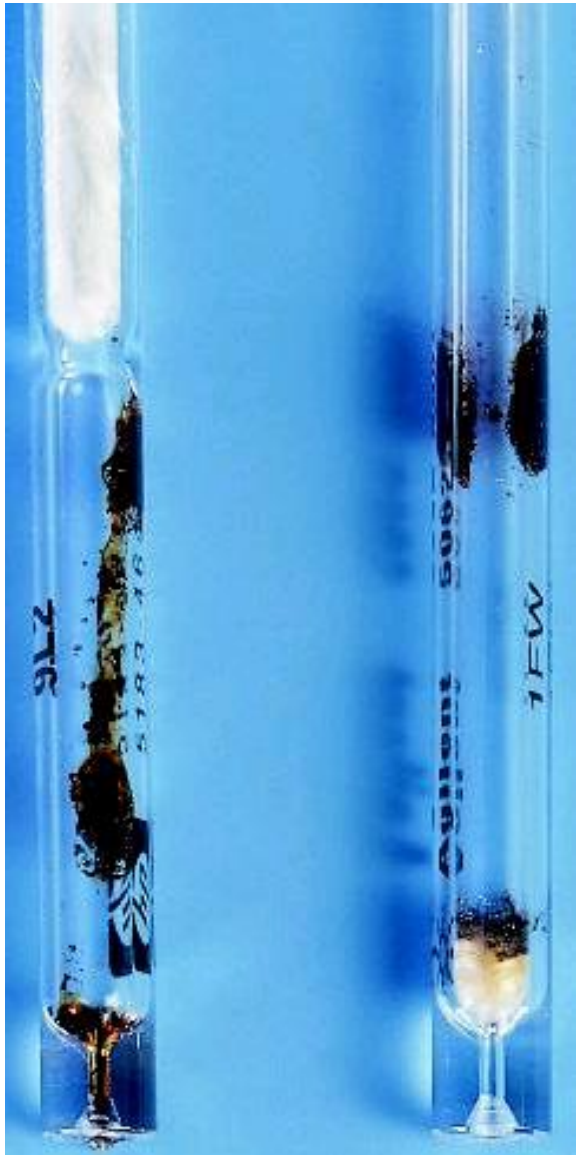
Evidence of a dirty liner:

- Poor peak shape
- Irregular baselines
- Poor resolution
- Poor response

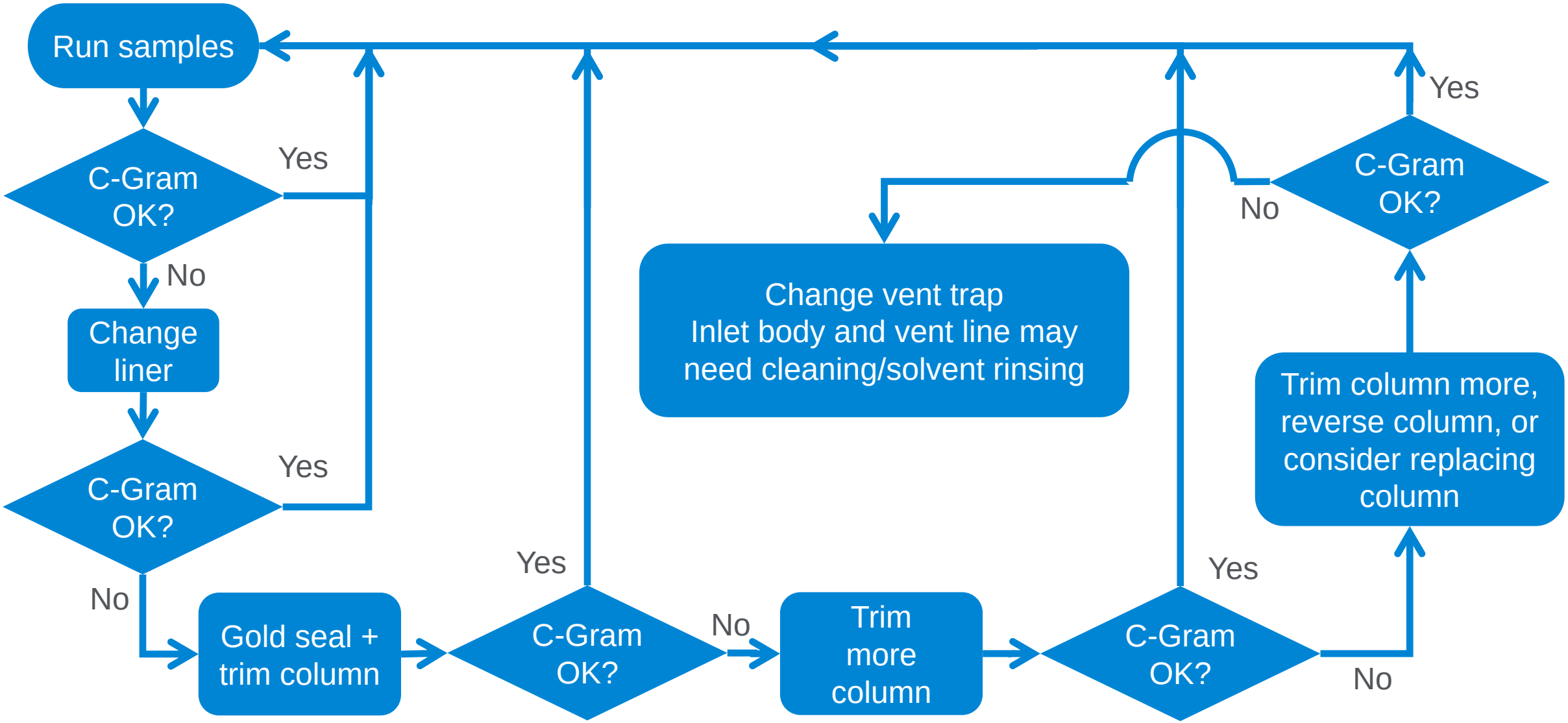


Liner Maintenance

- Liners become contaminated with use, collecting non-volatiles, salts, excess reagents, or become damaged/cracked
- Should inspect and replace liners often
- Handle with gloves and forceps
- Insert into or remove liners only from cool injection ports
- Replacing with a new liner is recommended, to ensure reproducibility



Inlet “Front-End” Maintenance Flow Chart



CrossLab Liners

Agilent CrossLab

Agilent Liners for Non-Agilent systems	
Shimadzu	https://www.agilent.com/en/products/gas-chromatography/gc-supplies/inlet-liners-sealing-systems/inlet-liners-for-shimadzu-gc-systems/models
Thermo	https://www.agilent.com/en/products/gas-chromatography/gc-supplies/inlet-liners-sealing-systems/inlet-liners-for-thermo-scientific-gc-systems/models
Varian/Bruker/Scion	https://www.agilent.com/en/products/gas-chromatography/gc-supplies/inlet-liners-sealing-systems/inlet-liners-for-varian-bruker-gc-systems/models
Perkin Elmer	https://www.agilent.com/en/products/gas-chromatography/gc-supplies/inlet-liners-sealing-systems/inlet-liners-for-perkinelmer-gc-systems/models

CrossLab Liners

The Varian/Bruker/Scion 1177 inlet uses the exact same dimension liner as Agilent X890 instruments with split/splitless inlet.

Agilent CrossLab

CrossLab Supplies for Bruker, Varian* GC Systems

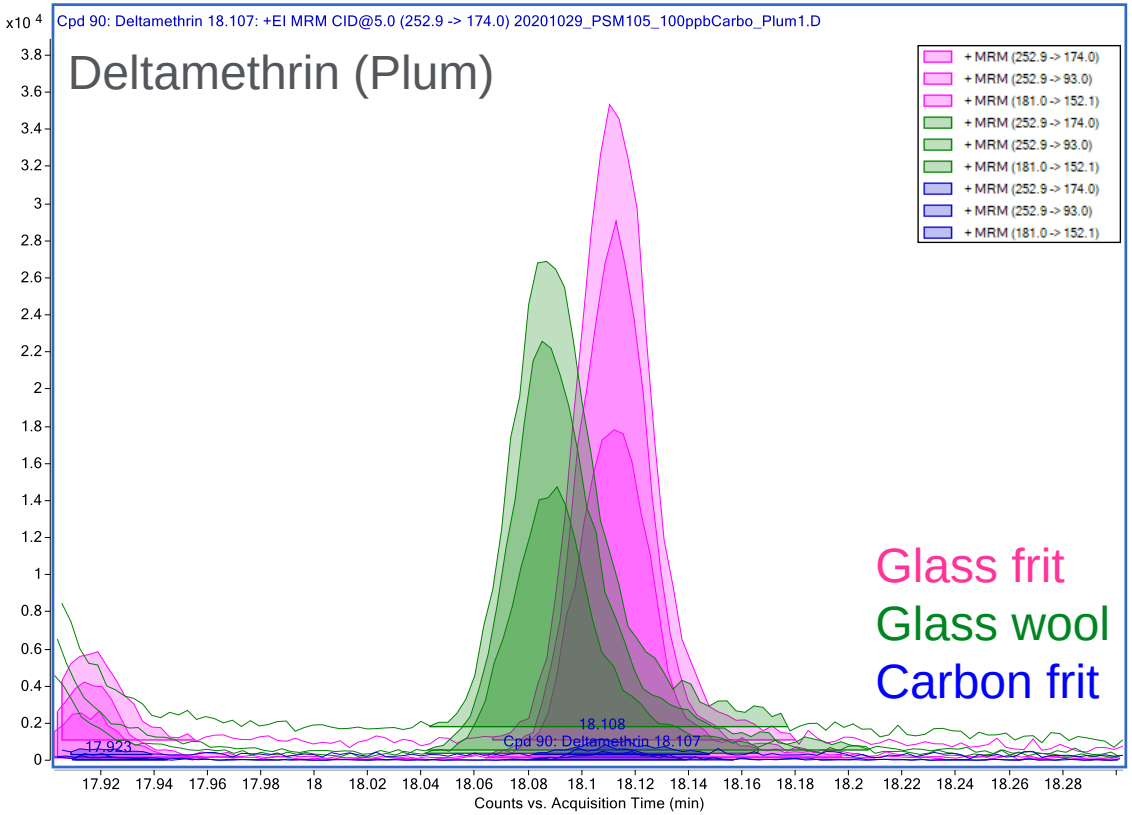
Products highlighted in gray are coming soon.

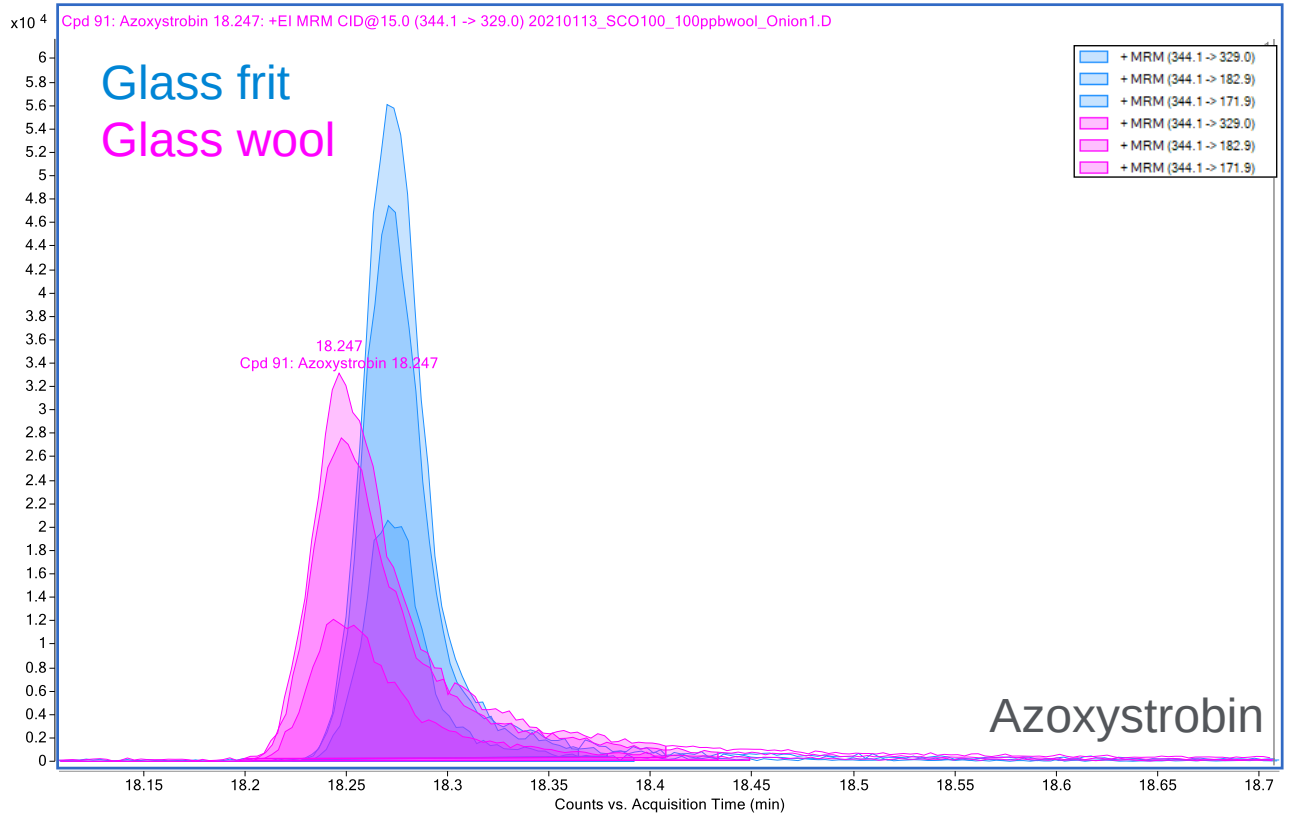
Liners for 1177 Split/Splitless Injector Ports

	Description	ID (mm)	OD (mm)	Length (mm)	Volume (µL)	Unit	Agilent Ultra Inert Deactivation	Agilent Original Deactivation
Split/Splitless Liners								
	Single taper	4.0	6.3	78.5	1000	5/pk	8004-0151	8004-0101
	Single taper, with wool	4.0	6.3	78.5	1000	5/pk	8004-0152	8004-0102
	Double taper	4.0	6.3	78.5	1000	5/pk	8004-0155	8004-0105
	Gooseneck, with wool	4.0	6.5	78.5	1000	5/pk	8004-0170	8004-0114
	Recessed gooseneck, with wool	4.0	6.3	78.5	1000	5/pk	8004-0153	8004-0103
	Gooseneck	2.0	6.5	78.5	250	5/pk	8004-0178	8004-0119
Splitless Liners								
	Straight, with wool	4.0	6.5	78.5	1000	5/pk	8004-0173	8004-0116
	Gooseneck	4.0	6.5	78.5	1000	5/pk	8004-0165	8004-0113
Split Liners								
	Straight-through	4.0	6.3	78.5	1000	5/pk	8004-0156	8004-0106
	Straight, with wool	4.0	6.3	78.5	1000	5/pk	8004-0154	8004-0104
	With frit, gooseneck	4.0	6.3	78.5	1000	5/pk	8004-0158	
Direct Liners								
	Straight-through	1.2	6.3	78.5	90	5/pk	8004-0157	8004-0107

Testing Wool and Fritted Liners: Sensitive Compounds

Pesticides in Food by GC-MS/MS





Higher Response with Glass Frit
Less Peak Tailing

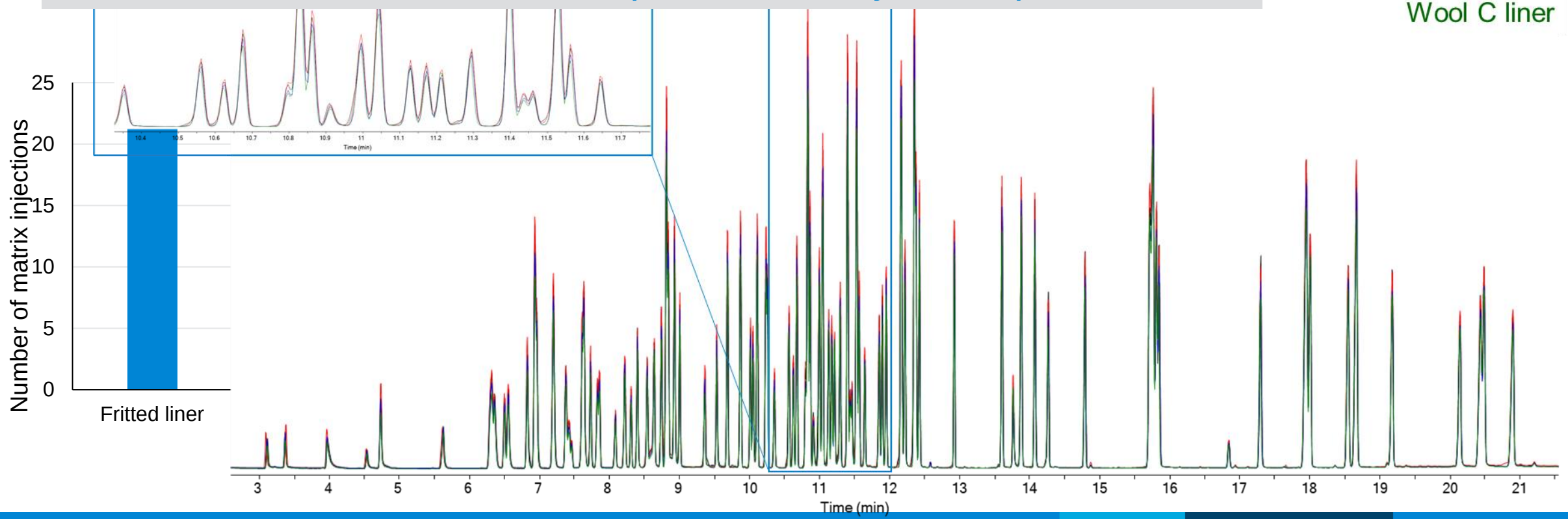
Less Peak Tailing with Fritted Liner

Testing Wool and Frit Liners

EPA 8270: Semivolatile organic compounds in soil matrix

Ultra Inert splitless fritted liner

Ultra Inert fritted liner: Matched responses to wool liners… double the lifetime (double the injections)



Liner selection – General/Summary

Liner Selection	Split	Splitless	HS / P&T	SPME
Inlet Flow	High	Low	High	Variable
Liner Features	Liner should have WOOL or other “feature” to aid in vaporization/ mixing	Much less important to have WOOL; use WOOL if you have dirty samples	Straight Sample is “pre-vaporized” so no need for wool or other similar features	Straight no solvent/diluent so no need for wool or other similar features
OD (mm)	6.3	6.5	6.3/6.5	6.3/6.5
ID (mm)	4	4	2 (straight)	0.75 – 1 (Straight)

Conclusions

- Proper sample introduction is critical
  - Liner plays a very big roll
  - Arguable as important as the column itself

- Generally choose the liner based on injection mode
  - **Split:** typically have wool/barrier/feature; aids in vaporization/mixing
  - **Splitless:** wool/barrier less critical, but use wool with dirty matrices
  - **HS/ P&T**: 2 mm straight
  - **SPME**: 0.75 – 1 mm straight

- Use Ultra Inert liners for optimal results when analyzing actives
  - UI Fritted liners for maximum liner life time; less tailing

- Use specialty liners when warranted
  - Direct connect, dimpled, Jenning's cup

- Match the liner to the method; do not mix and match!
  - Liner configuration needs to be consistent for consistent results

When in doubt, please contact us! (next slide)

Contact Agilent Chemistries and Supplies Technical Support

1-800-227-9770 option 3, option 3:

Option 1 for GC or GC/MS columns and supplies

Option 2 for LC or LC/MS columns and supplies

Option 3 for sample preparation, filtration and QuEChERS

Option 4 for spectroscopy supplies

Option 5 for Chemical Standards

Available in the U.S. 8-5 all time zones

gc-column-support@Agilent.com

lc-column-support@agilent.com

spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com

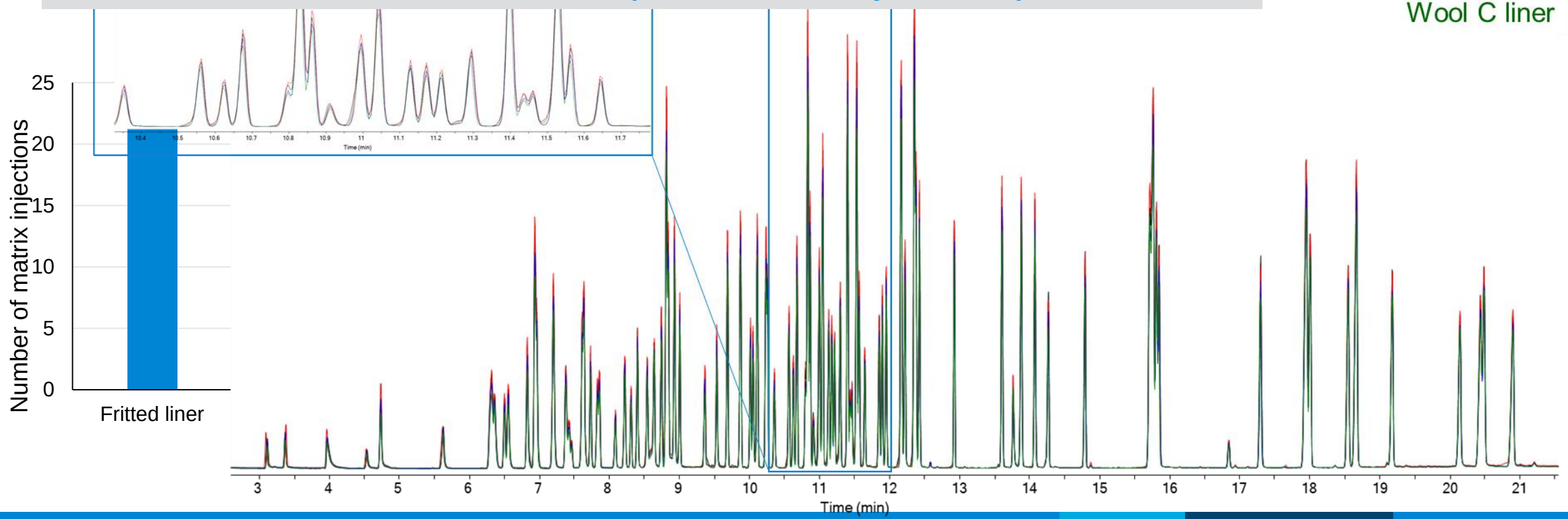
Appendix

Testing Wool and Frit Liners

EPA 8270: Semivolatile organic compounds in soil matrix

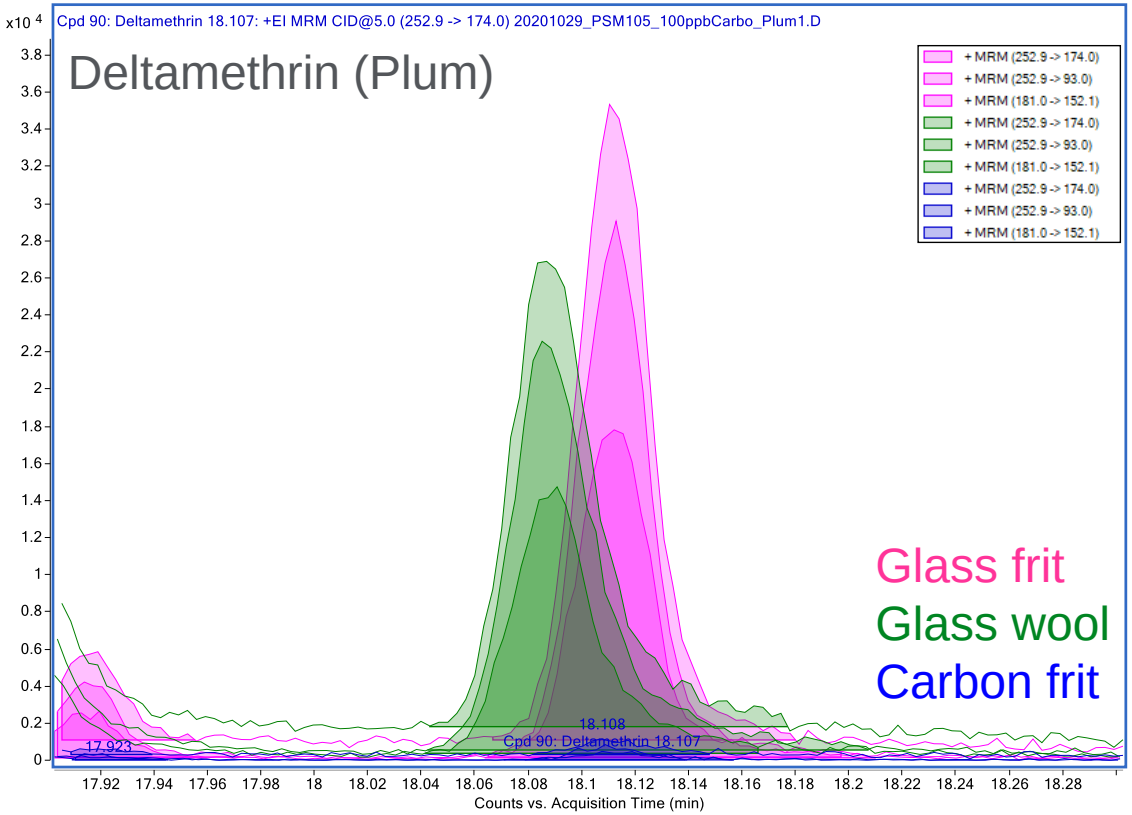
Ultra Inert splitless fritted liner

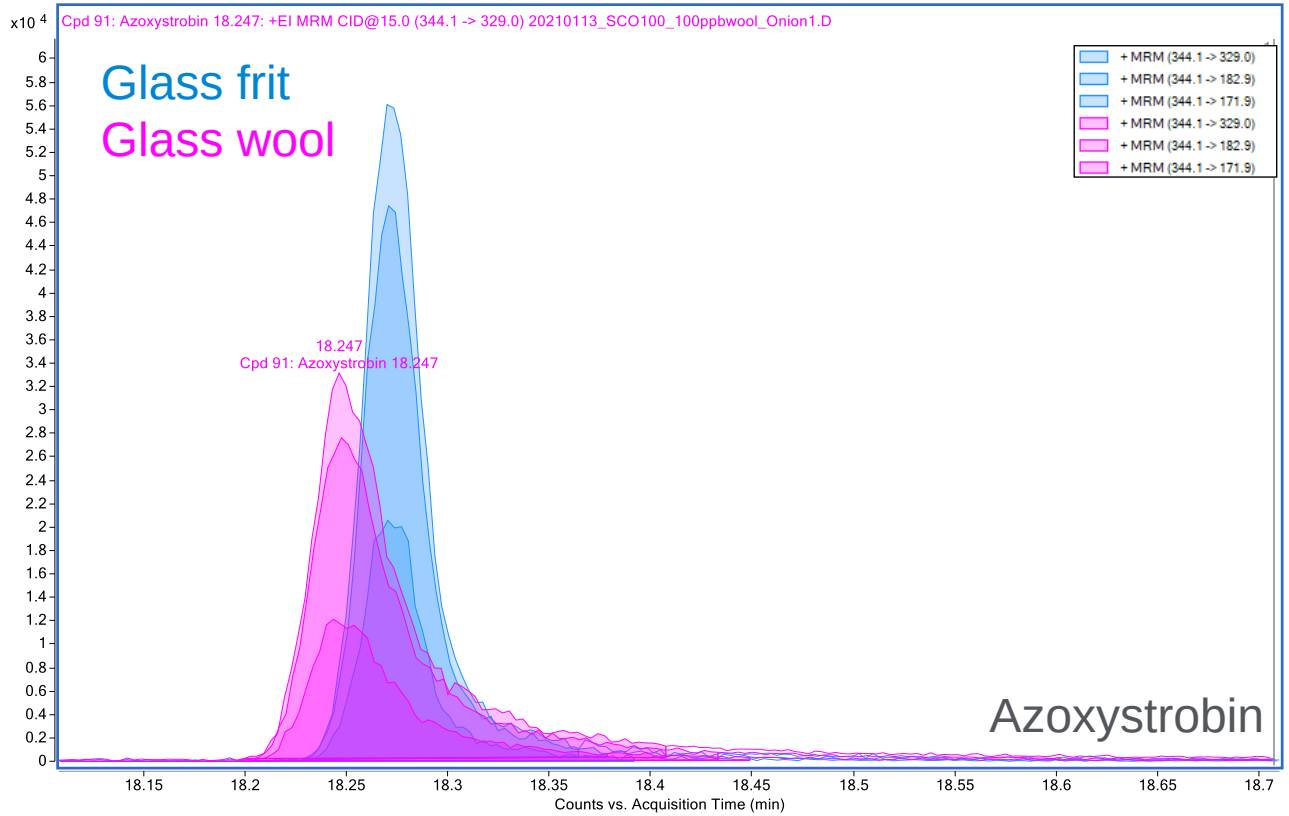
Ultra Inert fritted liner: Matched responses to wool liners… double the lifetime (double the injections)



Testing Wool and Fritted Liners: Sensitive Compounds

Pesticides in Food by GC-MS/MS

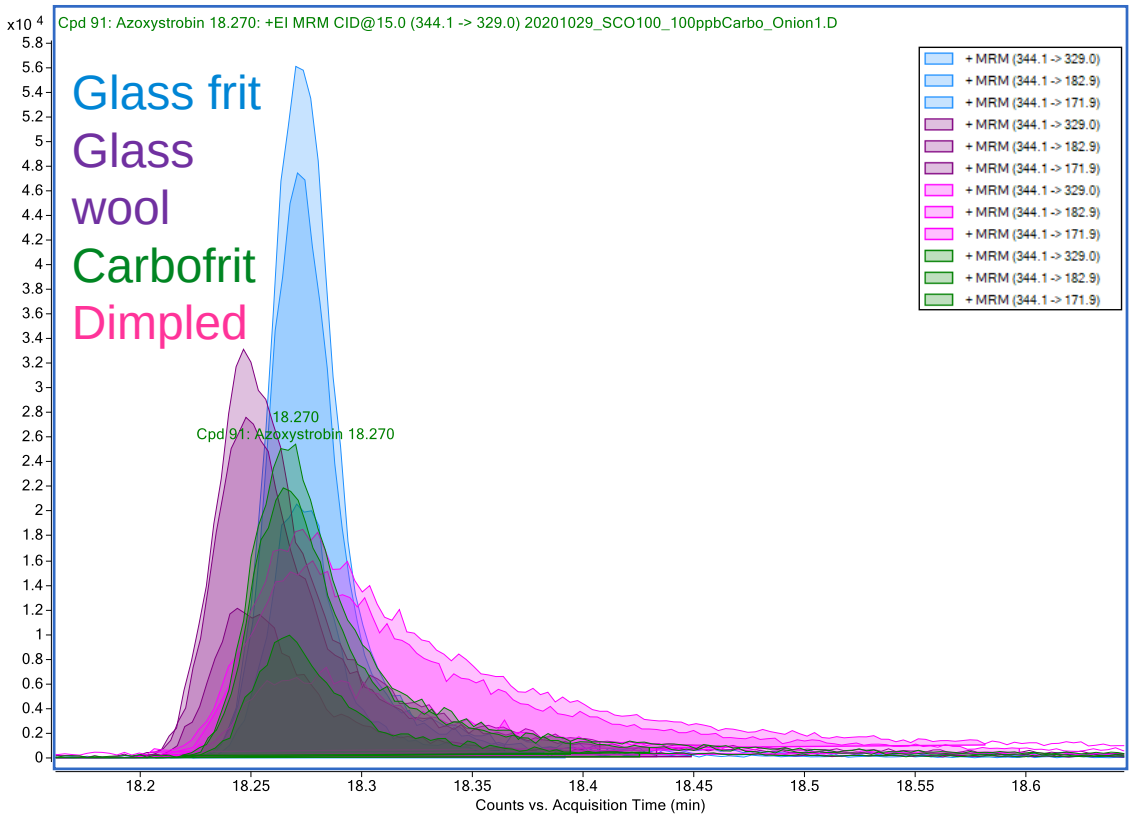


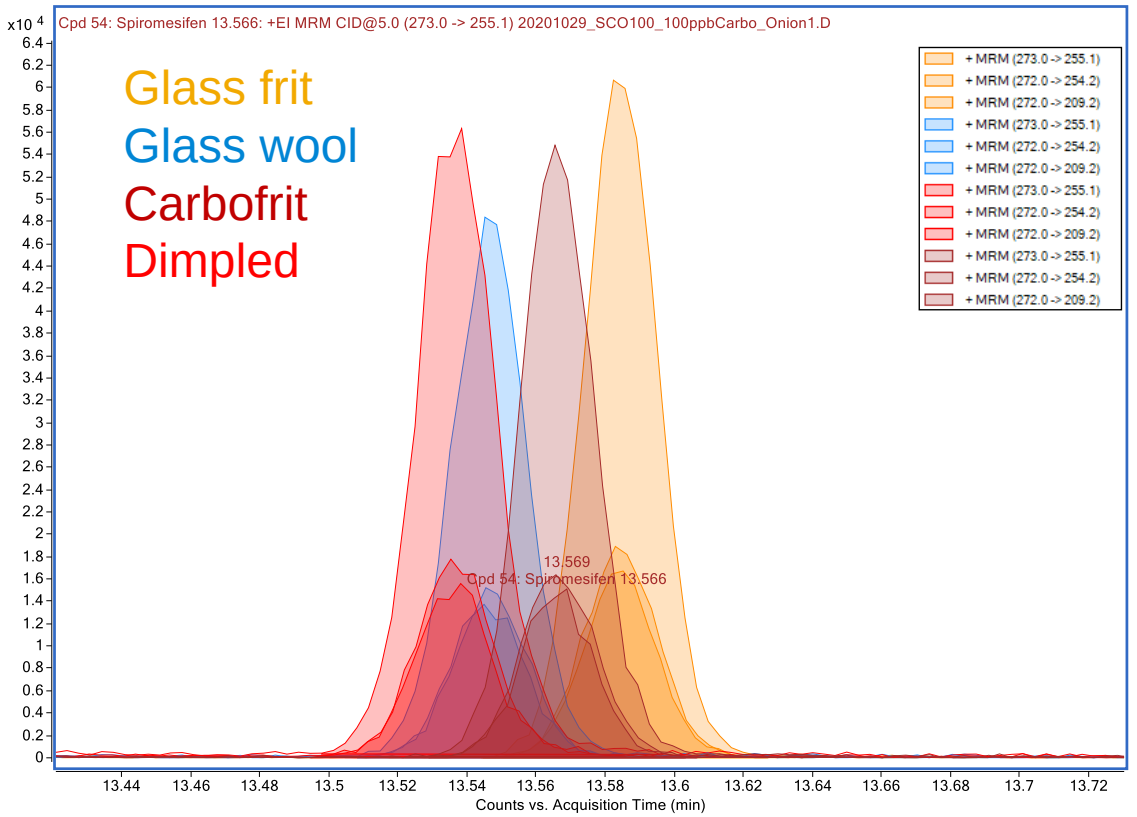


Higher Response with Glass Frit
Less Peak Tailing

Less Peak Tailing with Fritted Liner

Onion – Azoxystrobin and Spiromesifen

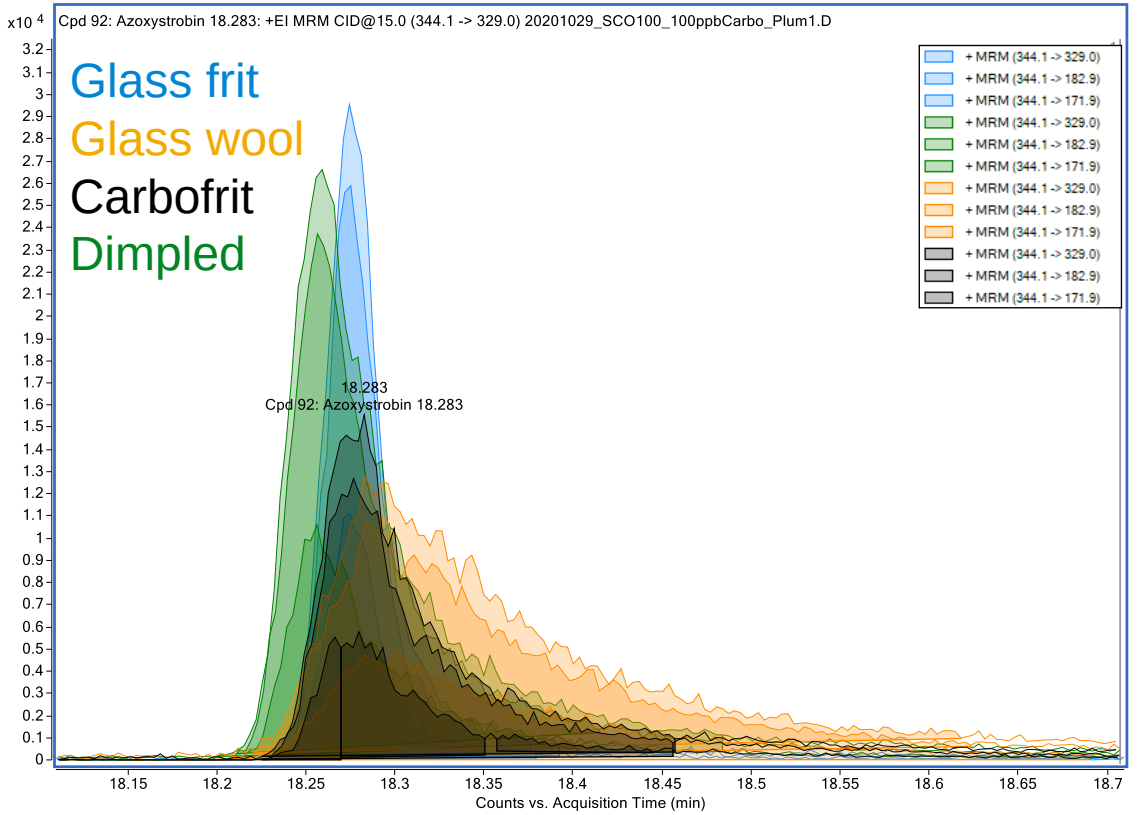




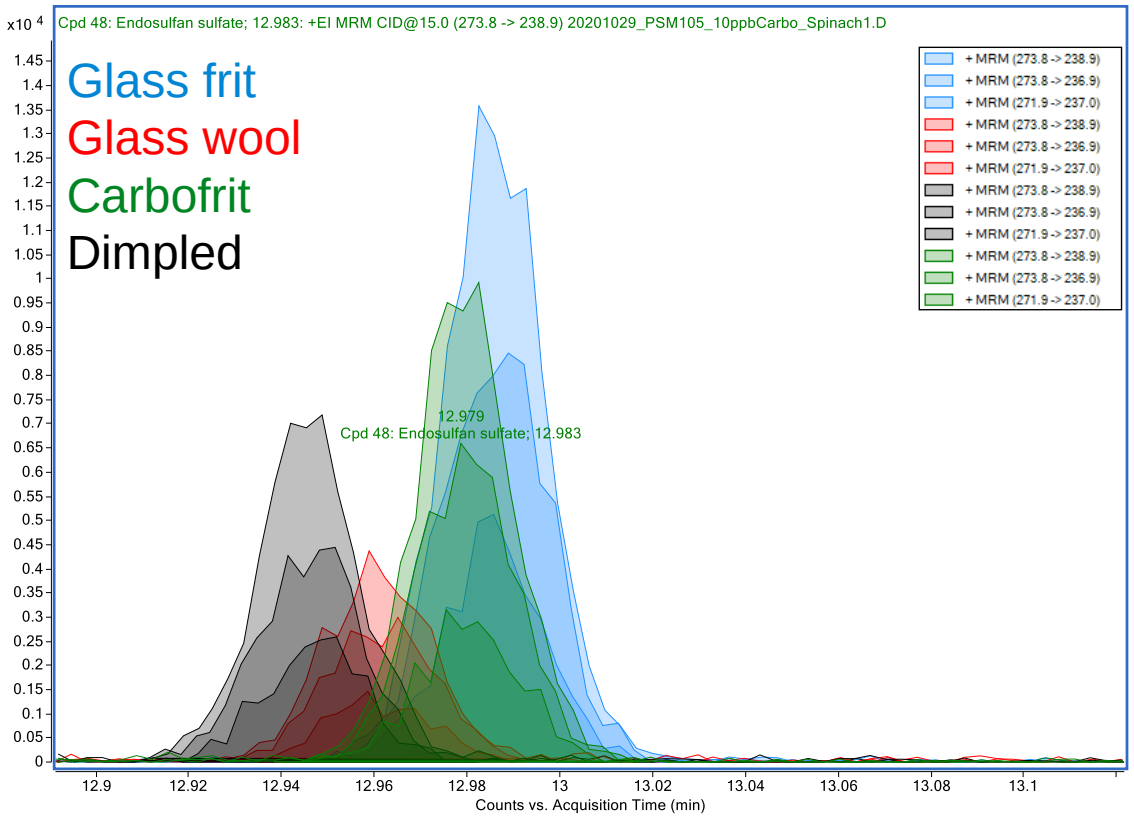
Azoxystrobin in Plum

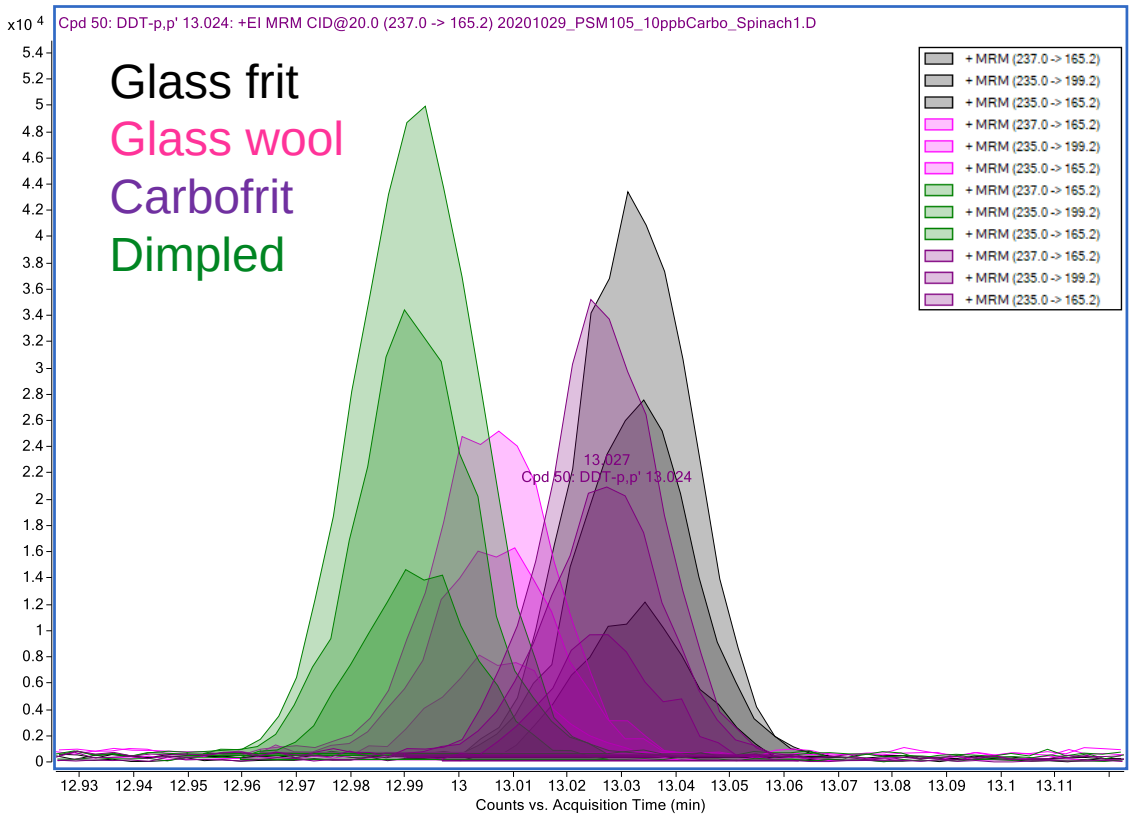
Glass wool and carbon frit liners = ugly lumps that tail

Frit and dimpled = better (and taller) peak shapes, less tailing



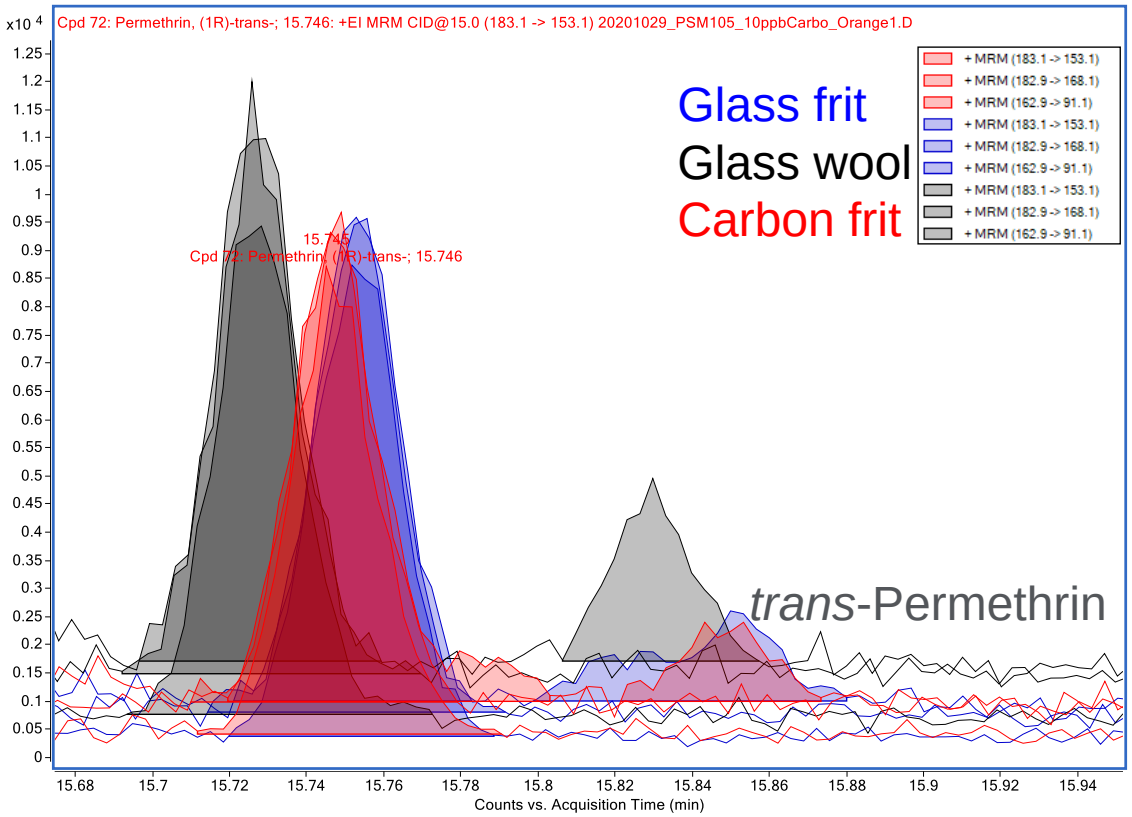
Endosulfan Sulfate and DDT in Spinach

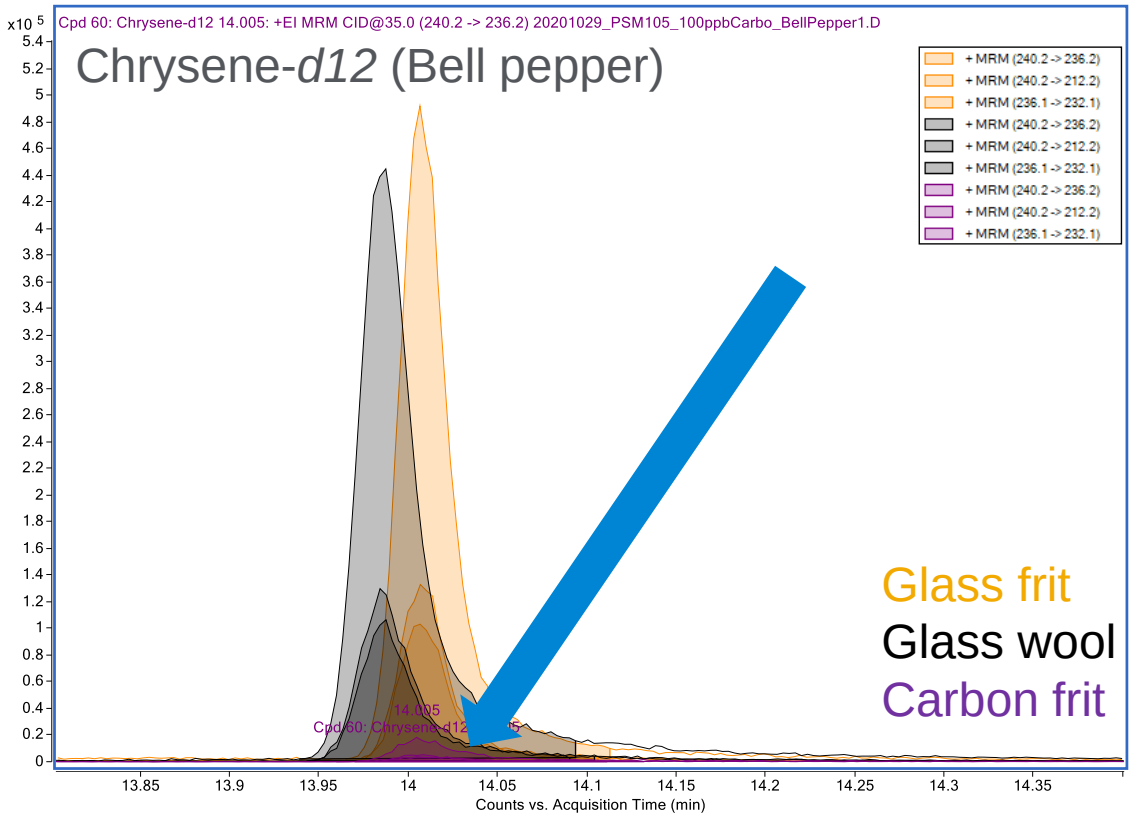




Testing Wool and Fritted Liners

Pesticides in food by GC/MS/MS

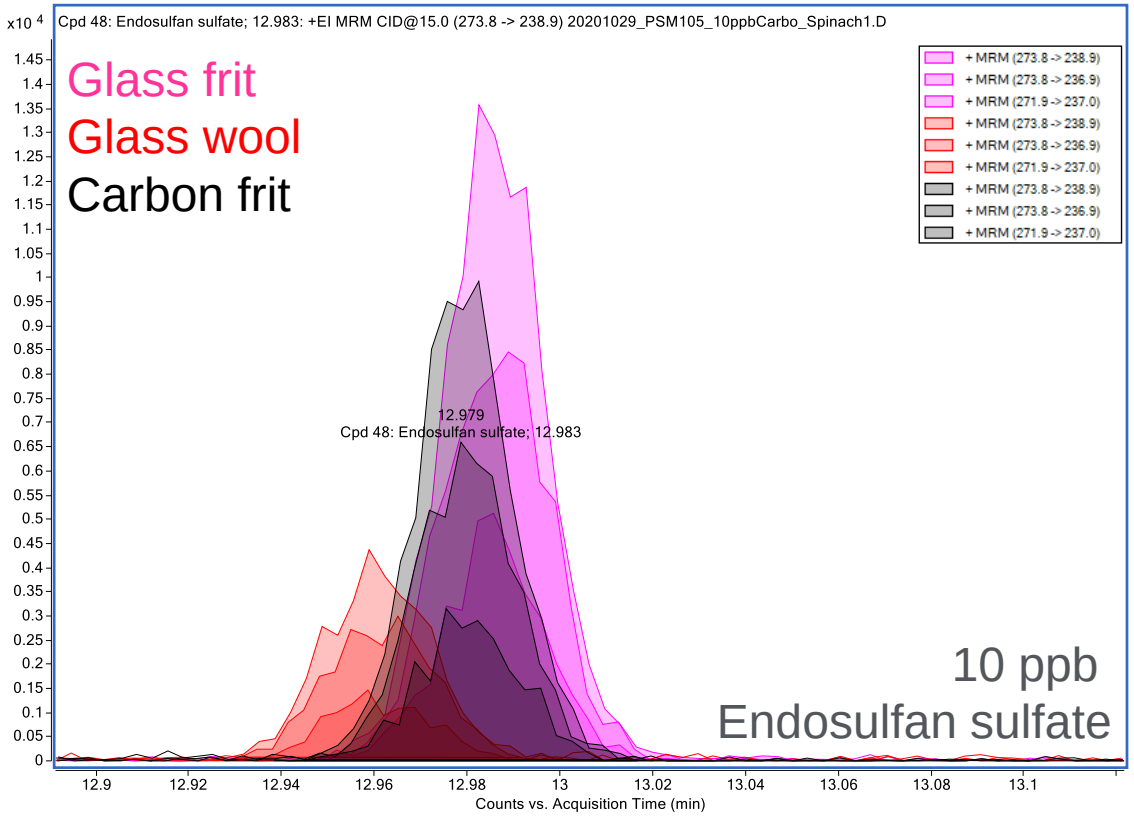


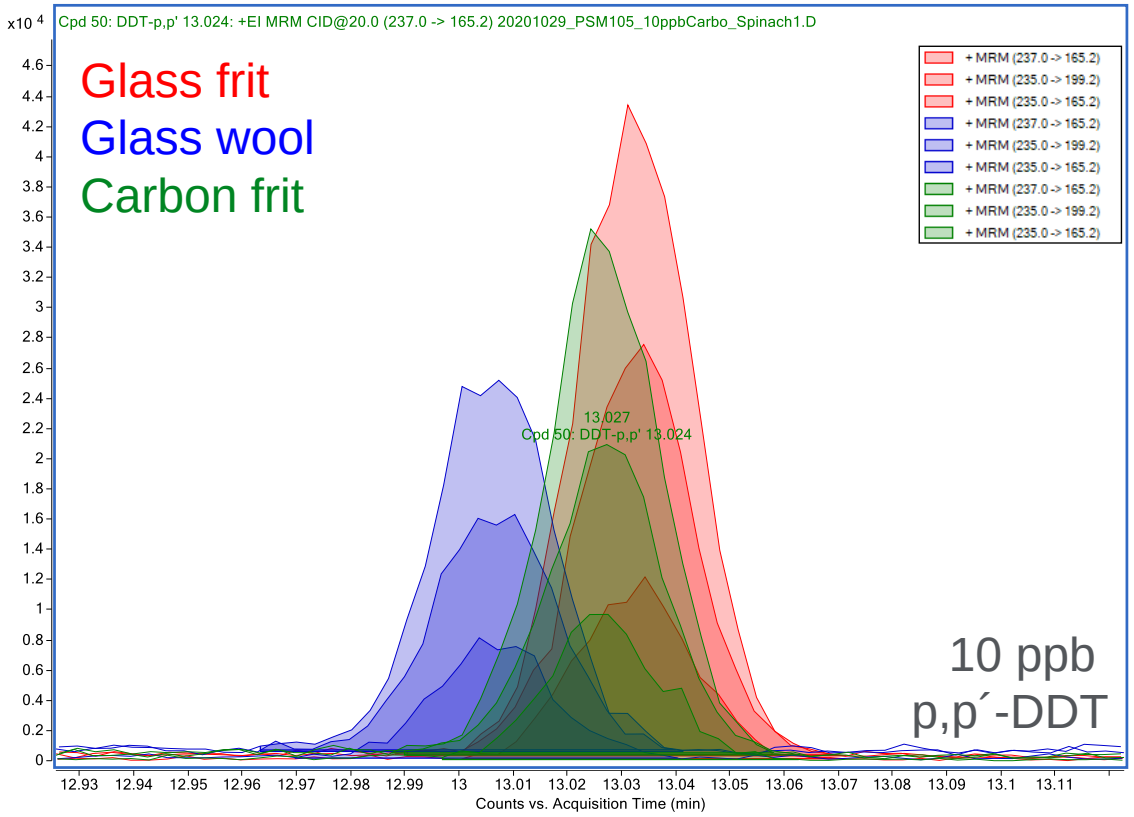


Be careful with carbon or nondeactivated liners and internal standards

Testing Wool and Frit Liners: Sensitive Compounds

Pesticides in food by GC/MS/MS





Higher responses with glass frit for sensitive compounds