Cut Down Your Column Cuts: Matching GC Liner Style to Matrices and Reduce Column Trimming Headaches

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Operational Issues Impacting the Environmental Laboratory Industry (Session 2)

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Trusted Answers

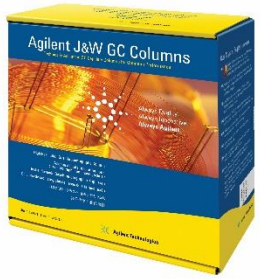
We want to reduce analyte loss in the flow path…

Deactivated Inlet Weldments and CFT devices

Ultra Inert Gold Seal

Ultra Inert Inlet Liner

Ultra Inert GC Column

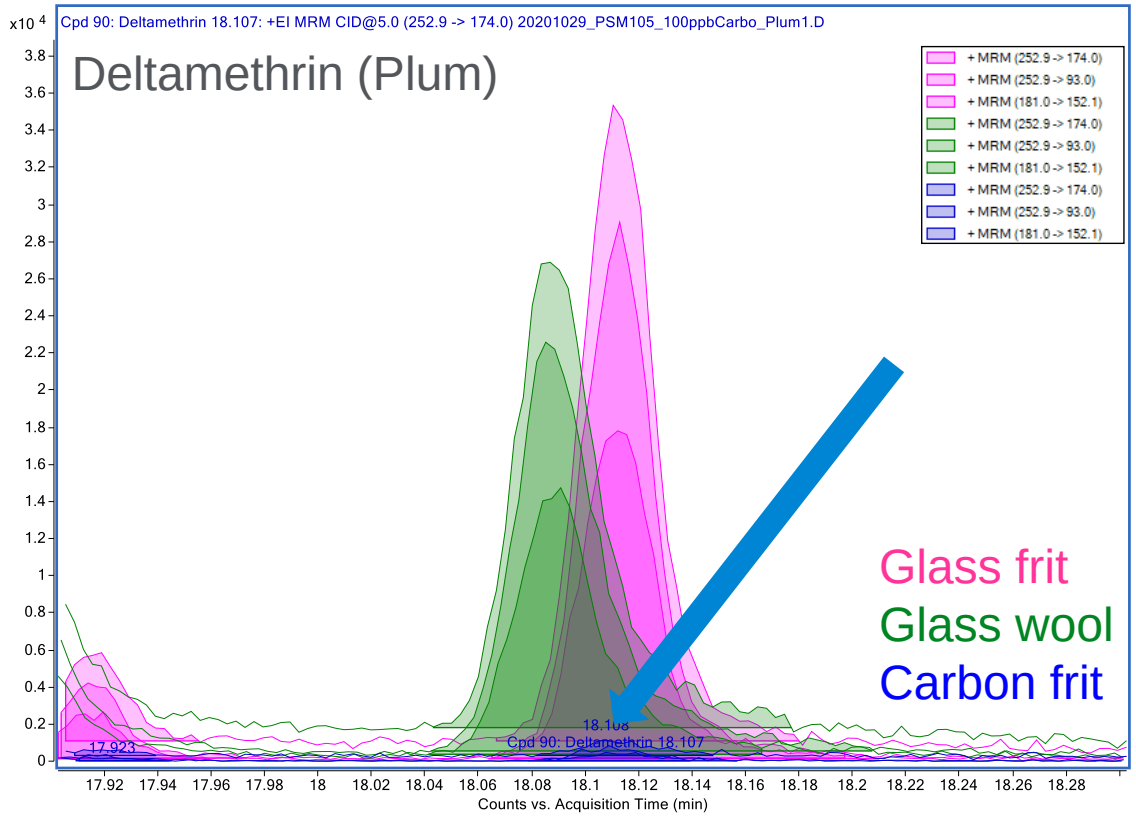


Utilize deactivated consumables

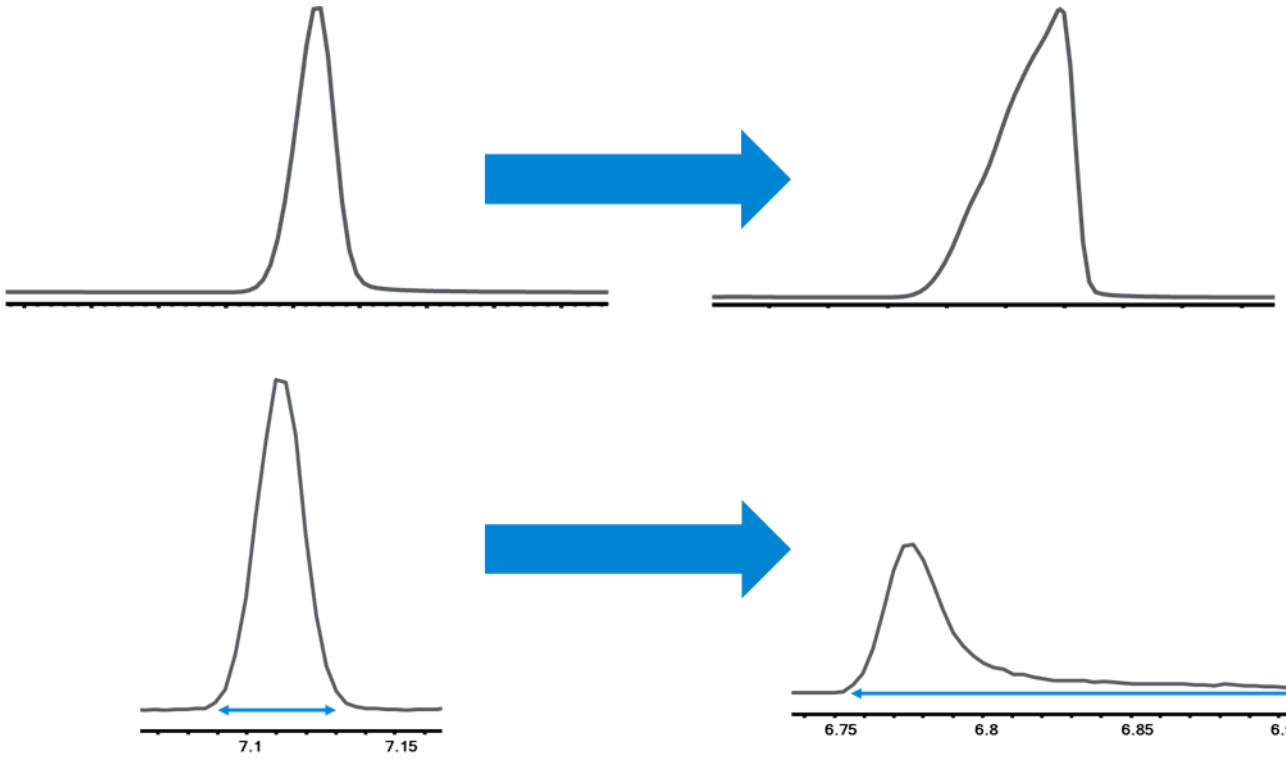
Gas Clean Gas Purifier

Ensure clean gas supply

Match the liner to the application



Match the liner to the application



Do I really need a barrier in my liner?

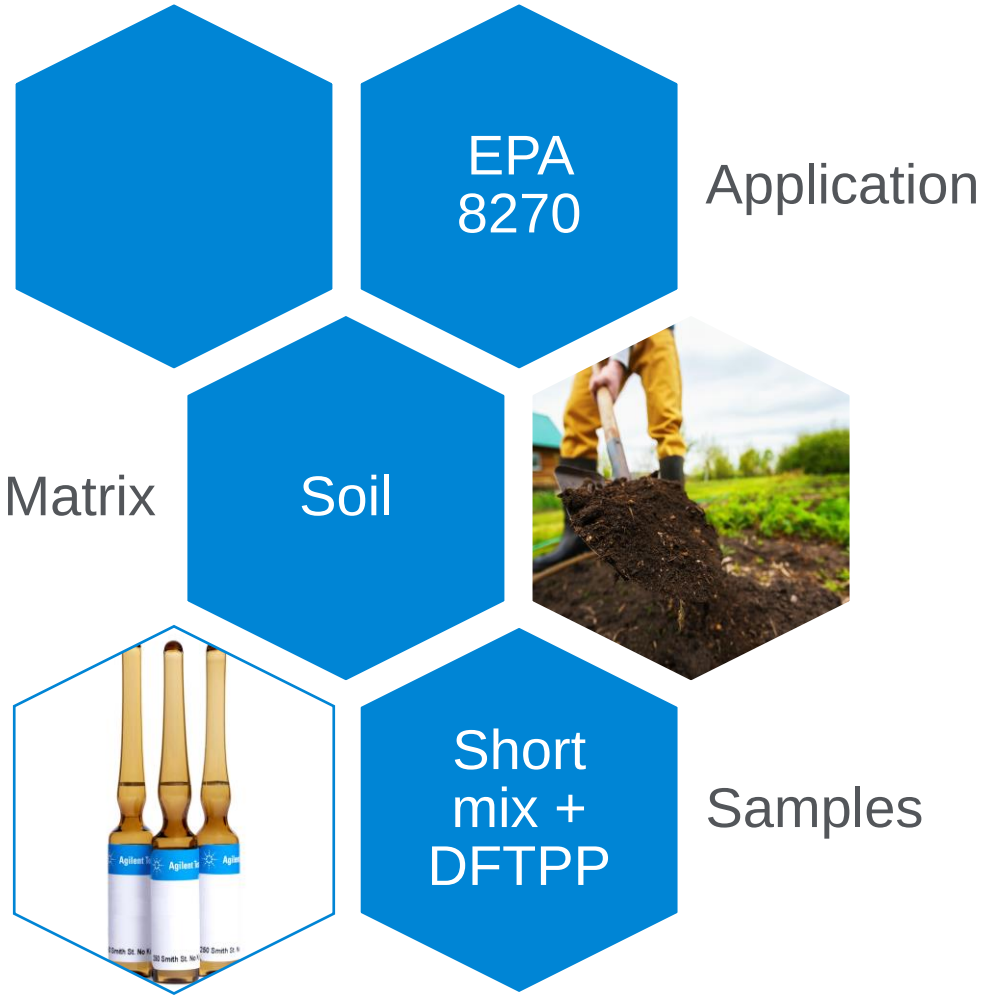
I can just trim my column and be fine….

How does liner style (barrier or not) affect my column maintenance?

Test plan for evaluating liner effect on column trimming frequency

3 Liner Styles

- Single taper liner
- Single taper with wool
- Single taper with glass frit

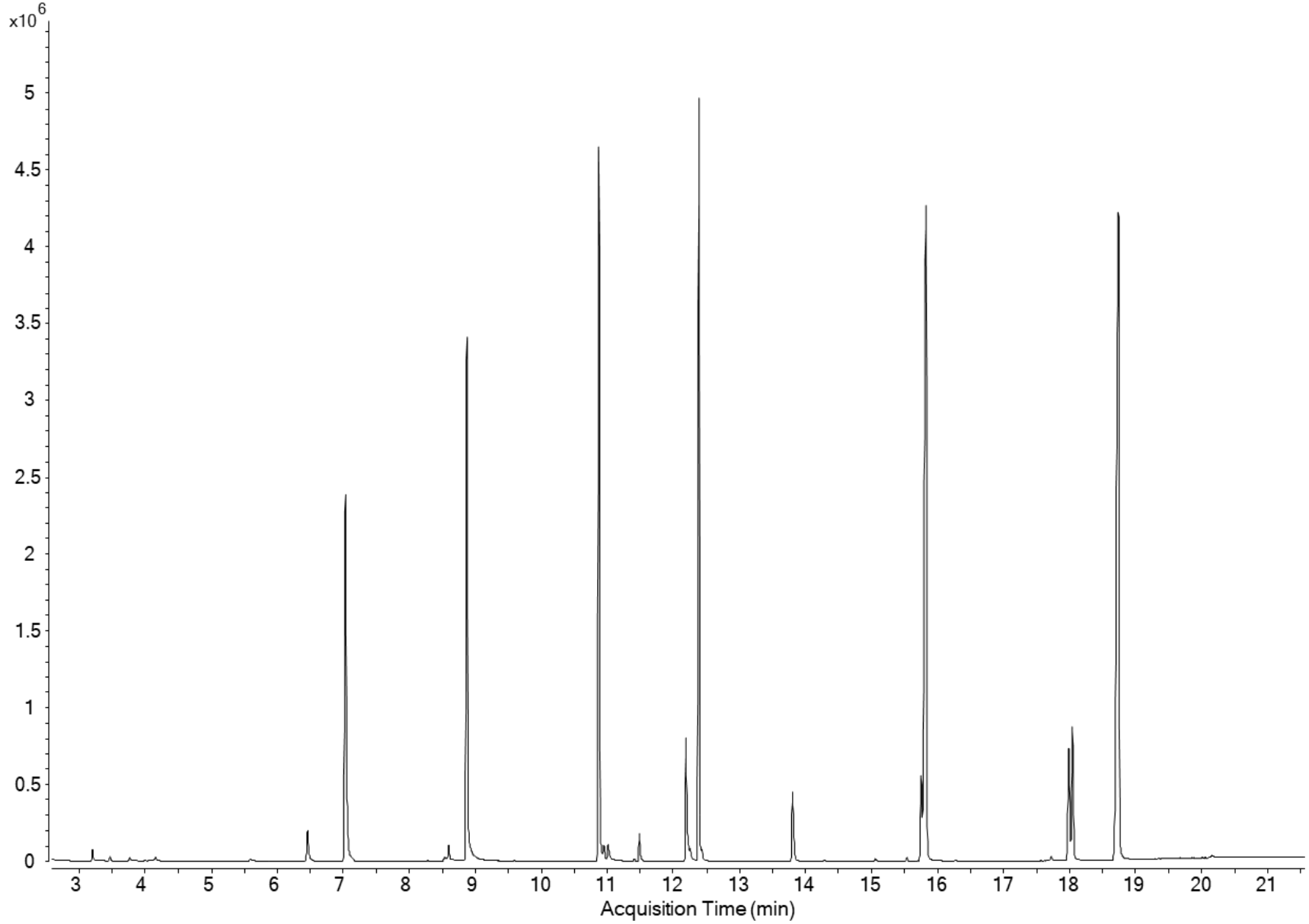


Evaluating methods with EPA 8270 short mix @ 5ppm

Why use the short mix?

- 12 challenging EPA 8270 compounds
  - 2 PAHs - critical pair resolution
  - Nitrophenols
  - NDMA, etc.
- 6 ISTDs (40ppm)

- Quick decisions on method changes



EPA short mix: p/n 5191-3905
Internal standards: p/n ISM-560

Liner + Column Trim Testing: GC/MS Method parameters

GC/MS Method Parameters	
Injection volume	1 µL
Inlet	Split/splitless 280 °C; **Pulsed splitless** 30 psi until 0.6 min; Purge 50 mL/min at 0.6 min; Switched septum purge 3 mL/min
Column	**Agilent J&W DB-5ms UI** (30 m x 0.25 mm x 0.25 µm, p/n 122-5532UI)
Column temperature program	40 °C (hold for 0.5 min), 10 °C/min to 100 °C, 25 °C/min to 260 °C, 5 °C/min to 280 °C, 15 °C/min to 320 °C (hold 2 min)
Carrier gas and flow rate	Helium at 1.30 mL/min, constant flow
Transfer line temperature	320 °C
Ion source temperature	300 °C
Quadrupole temperature	150 °C
Scan	m/z 35 to 500
Gain factor	0.4
Threshold	0
A/D samples	4

Liner + Column Trim Testing: Experimental Design

Run	Sample type	Name
1	DCM blank	DCM blank
2	DFTPP tuning mix 25ppm	QC check
3	EPA 8270 short mix 20ppm	Mini-CCV
4-13	**Soil matrix (10runs)**	**Soil matrix**
14	DCM blank	DCM blank
15	DFTPP tuning mix 25ppm	QC check
16	8270 short mix 20ppm	Mini-CCV
17-26	**Soil matrix (10runs)**	**Soil matrix**
27	DCM blank	DCM blank
28	DFTPP tuning mix 25ppm	QC check
29	8270 short mix 20ppm	Mini-CCV

Passing Criteria:

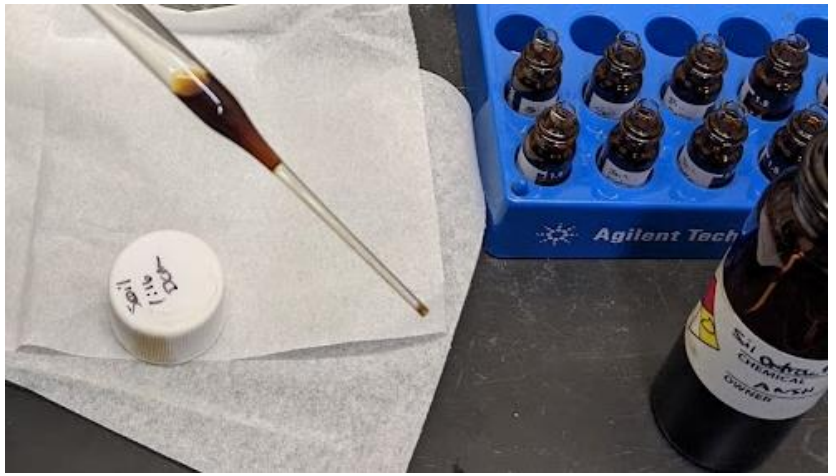
DDT % breakdown <20%

Pentachlorophenol, benzidine tailing factor <2.0

DFTPP Ion ratios pass

Mini-CCV: Cal drift within ±20% for ≥70% compounds

ISTD: Area of IS peak area drift within ±50%



How many liners and column trims occurred per liner style?

Liner Type	Average Lifetime (number of matrix injections)	Number of Liners Used Before Column Trim	Number of Column Trims Before New Column Required
Single taper	14	2.33	1
Wool	10	6.67	2
Fritted	12	7	2

How did the liners compare?

Data Set 1

	Number of liners before trim	Failure point
Single taper	3	DDT % breakdown (22.3%) and pentachlorophenol TF (2.0)
Wool	9	4 CCV compounds outside ±20%
Fritted	7	Pentachlorophenol TF (2.0)

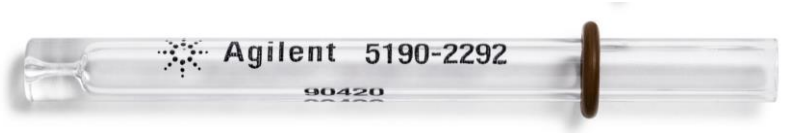
Data Set 2

	Number of liners before trim/change	Failure point
Single taper	2	6 CCV compounds outside ±20%
Wool	8	6 CCV compounds outside ±20%
Fritted	7	11 CCV compounds outside ±20%

Data Set 3

	Number of liners before trim	Failure point
Single taper	2	DDT %, pentachlorophenol TF, **and** 5 CC compounds outside ±20%
Wool	3	6 CCV compounds outside ±20%
Fritted	7	4 CCV compounds outside ±20%

Single taper liner: Detailed example



Data Set Number	Liner Number	Initial DDT Percent Breakdown	Initial CCV Failures	Notes
1	1	1.4	0	New column
	2	14.2	3	
	3	18.8	3	
	“4”	22.3	3	Trim
2	1	4.5	1	
	2	17.3	3	
	“3”	19.3	4	Trim column
		3.9	6	Replace column
3	1	2.9		New column
	2	13.3		
	“3”	35.3	5	Trim column
4	1	4.0	0	

- Not the best choice for heavy matrices
- Transfers more matrix onto column
  - Increased column trims and changes

Best choice for drinking water or other clean samples

Single taper with Wool liner: Detailed breakdown

Data set number	Liner Number	Initial DDT % breakdown	Initial CCV failures	Notes
1	1	2.1	0	New column
	2	1.6	1	
	3	0.9	0	
	4	0.8	3	
	5	1.5	0	
	6	1.1	1	
	7	0.9	3	
	8	1.7	0	
	9	1.2	2	
	“10”	1.1	4	Trim column
3*	1	0.7	0	New Column*
	2	1.3	0	
	3	2.2	0	
	4	1.9	1	
	5	2.9	0	
	6	1.9	1	
	7	2.3	1	
	8	2.3	1	
	“9”	2.7	6	Trim column
4	1	4.1	1	
	2	6.2	1	
	3	5.4	3	
	“4”	3.8	6	Trim column
5	1	3.3	1	

- Harder to connect DDT breakdown to column health
  - Variability related to inherent variability of wool
- Better to trim early than waiting for true CCV failure

Remains a good option for heavy matrices

Single taper with Glass Frit liner: Detailed breakdown

Data Set Number	Liner Number	Initial DDT % Breakdown	Initial CCV failures	Notes
1	1	0.3	0	New column
	2	0.4	0	
	3	0.7	0	
	4	2	0	
	5	2.5	1	
	6	2.6	1	
	7	2.4	1	
	“8”	2.4	1	TF = 2.0; Trim column
2	1	0.3	0	
	2	0.5	1	
	3	1.1	1	
	4	1.5	1	
	5	1.4	1	
	6	0.9	1	
	7	2.1	3	End at 11 CCV; Trim column
3	1	0.5	0	

- Track column health with DDT % breakdown
- Column trim frequency was very consistent

Remains a good option for heavy matrices

What about a low pressure drop liner?

Liner style	Run #1 Avg. % breakdown	% RSDs	Avg. Lifetime (over 20% at run…)	% RSDs	Avg. Starting Area Endrin | DDT	
Single taper	5.5%	8.5%	96*	18%*	6800	11000
Single taper with wool	5.1%	15%	74	34%	7000	12000
Single taper with glass frit	10.2%	14%	87*	17%*	6000	12000
Low pressure drop single taper with wool	7.6%	30%†	100*	27%*	**3150**	**4500**
Carbofrit	>20%					

*1 or more run/liner lasted longer than a complete sequence before reaching 20% breakdown; use 106 to generate an approximate average
†One (or more) result is outside the standard deviation range

- Previous GC-ECD work on liner lifetime (Application note: 5994-2111EN)
  - Tracked endrin and DDT breakdown during soil matrix injections
- Low pressure drop liner areas: ~**1/2 of splitless liner areas**
- If liner lifetime matters most, may be the best option

Summary

- Single taper (no barrier) liner – not the best choice for heavy matrix
  - More matrix transferred onto column
  - Still great for drinking water analyses
- Reduced column trims with wool or glass fritted liner
- Fritted and wool liner remain very similar in performance
  - Fritted liner more consistent in column trim frequency

Thank you!

Any Questions?