

SPLITLESS LINERS IN GAS CHROMATOGRAPHY



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TRAINING
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ALMERIA

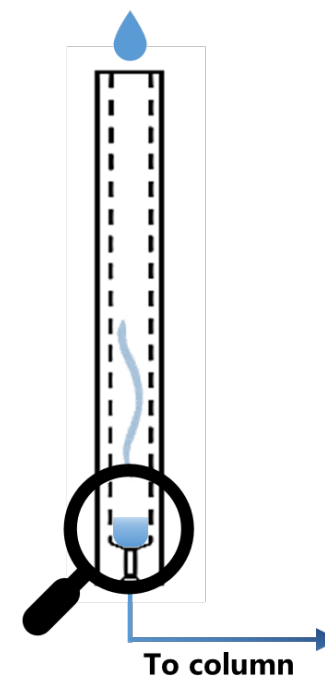
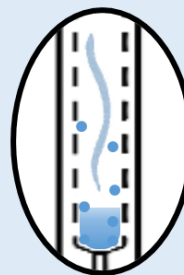
In gas chromatography, samples are typically injected in liquid state and turned into **gas state** in the injector

Liner

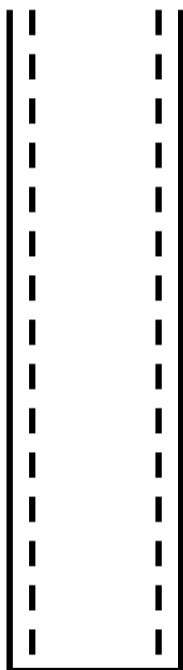


A large number of liner references with **different configurations** is offered by specialized companies

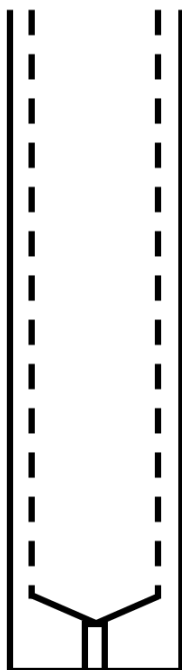
Liners possess **active sites** in which some sample components can be retained; each manufacturer employs a specific deactivation methodology



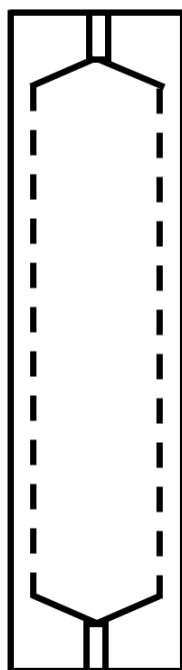
Geometry



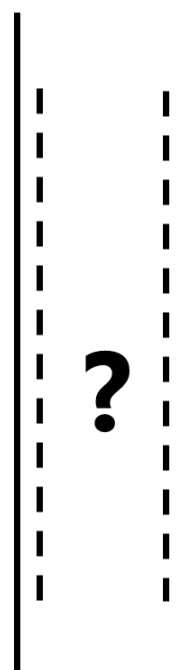
Straight



Single taper



Double taper

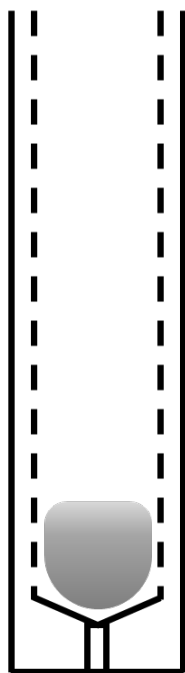


Especial
geometry

Tapers may help in

1. Minimizing the interactions of the sample components with the inlet
2. Focusing the analytes to the column
3. Reducing the loss of matrix components during the evaporation (double taper)

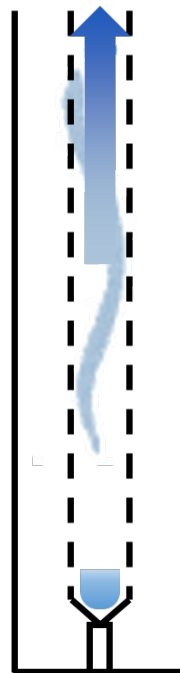
Packing



Glass wool or frit

- ✓ Provides a support for the sample during the evaporation
- ✓ Prevents the non-volatile components from reaching the column
- ✗ Might interact with some labile analytes
- ✗ Defective packing might result in loss of reproducibility

Internal diameter



Reduced internal diameter

- ✓ Helps transfer the sample to the column in a narrow band (increased gas velocity)
- ✗ The maximum capacity should be carefully considered

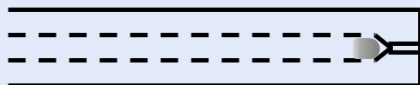
**Deactivation
Geometry
Packing
Internal diameter**

Liners # 1-2-3



Single taper, glass wool, three companies (deactivation processes)

Liner # 4



Reduced internal diameter

Liner # 5



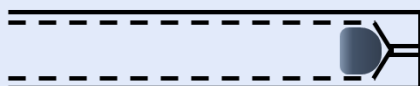
Special geometry (cyclo)

Liner # 6



No packing

Liner # 7



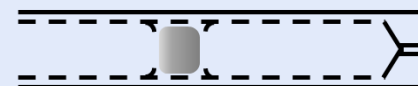
Glass frit

Liner # 8



Glass frit, medium position

Liner # 9



Tapered glass wool, medium position



Intuvo 9000 GC system (Agilent)

- Injection mode: Splitless
- Sample injection volume: 1 μL (EtAc)
- Inlet temperature:
 - 80 $^{\circ}\text{C}$ (for 0.1 min),
 - up to 300 $^{\circ}\text{C}$ at 600 $^{\circ}\text{C}/\text{min}$
- Oven T $^{\circ}$:
 - 60 $^{\circ}\text{C}$ for 0.5 min,
 - up to 170 $^{\circ}\text{C}$ (80 $^{\circ}\text{C}/\text{min}$),
 - up to 310 $^{\circ}\text{C}$ (20 $^{\circ}\text{C}/\text{min}$, hold for 3.5 min)
- Post Run: 2.1 min, 310 $^{\circ}\text{C}$

Estimated volume
after evaporation:
140 μL

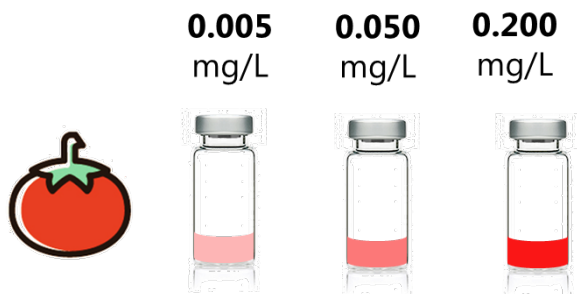
7410 triple quadrupole system (Agilent)

- Ionisation mode: electron impact ionization
- T $^{\circ}$ transfer line and ion source: 280 $^{\circ}\text{C}$
- Collision gas: nitrogen
- Solvent delay: 2.6 min

Signal intensity

Liner # 1 was selected as the **reference**. Each day, a new liner # 1 was placed in the inlet and three reference injections were performed

188 pesticide residues in ethyl acetate



Durability and peak shapes

0.005 Tomato (x4)
0.050 Tomato (x4)
0.200 Tomato (x4)



0.005
mg/L

0.050
mg/L

0.200
mg/L

0.005 Orange (x4)
0.050 Orange (x4)
0.200 Orange (x4)



0.005 Tomato (x4)
0.050 Tomato (x4)
0.200 Tomato (x4)



[...]

72 injections





SIGNAL INTENSITY

Liner # 1 was selected as the **reference**. Each day, a new liner # 1 was placed in the inlet and three reference injections were performed (**tomato matrix** 🍅)



SEQUENCE

Reference liner (new)

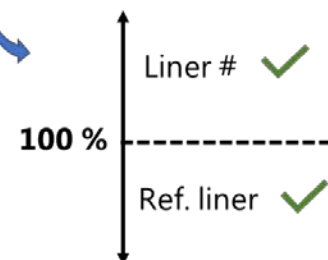
1. Tomato 0.005 mg/L
2. Tomato 0.050 mg/L
3. Tomato 0.200 mg/L

New liner

4. Tomato 0.005 mg/L
5. Tomato 0.050 mg/L
6. Tomato 0.200 mg/L
7. [...]

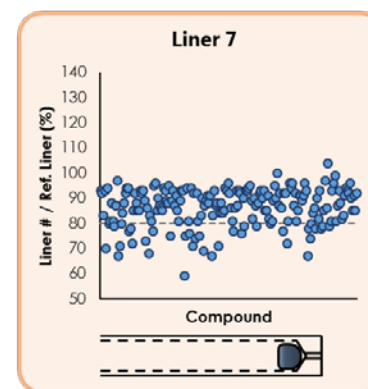
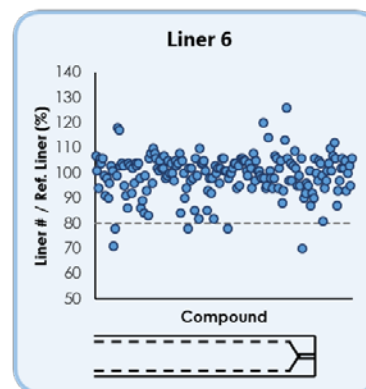
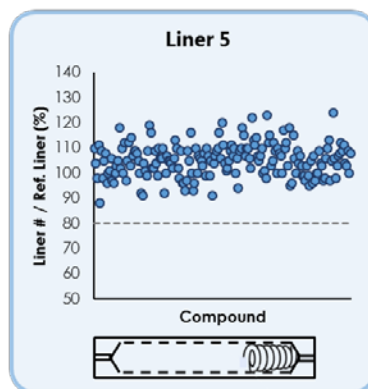
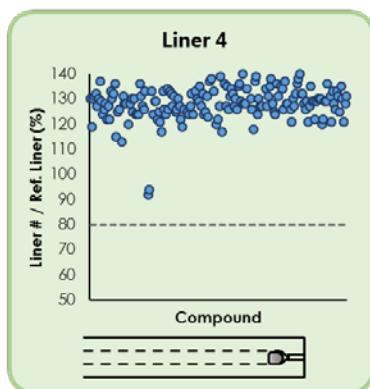
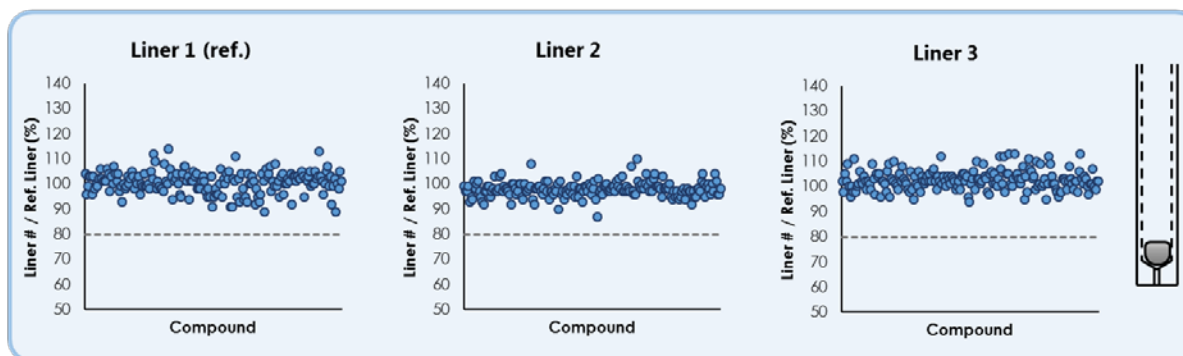
$$\frac{\text{Area liner \#}}{\text{Area ref. liner}} (\%)$$

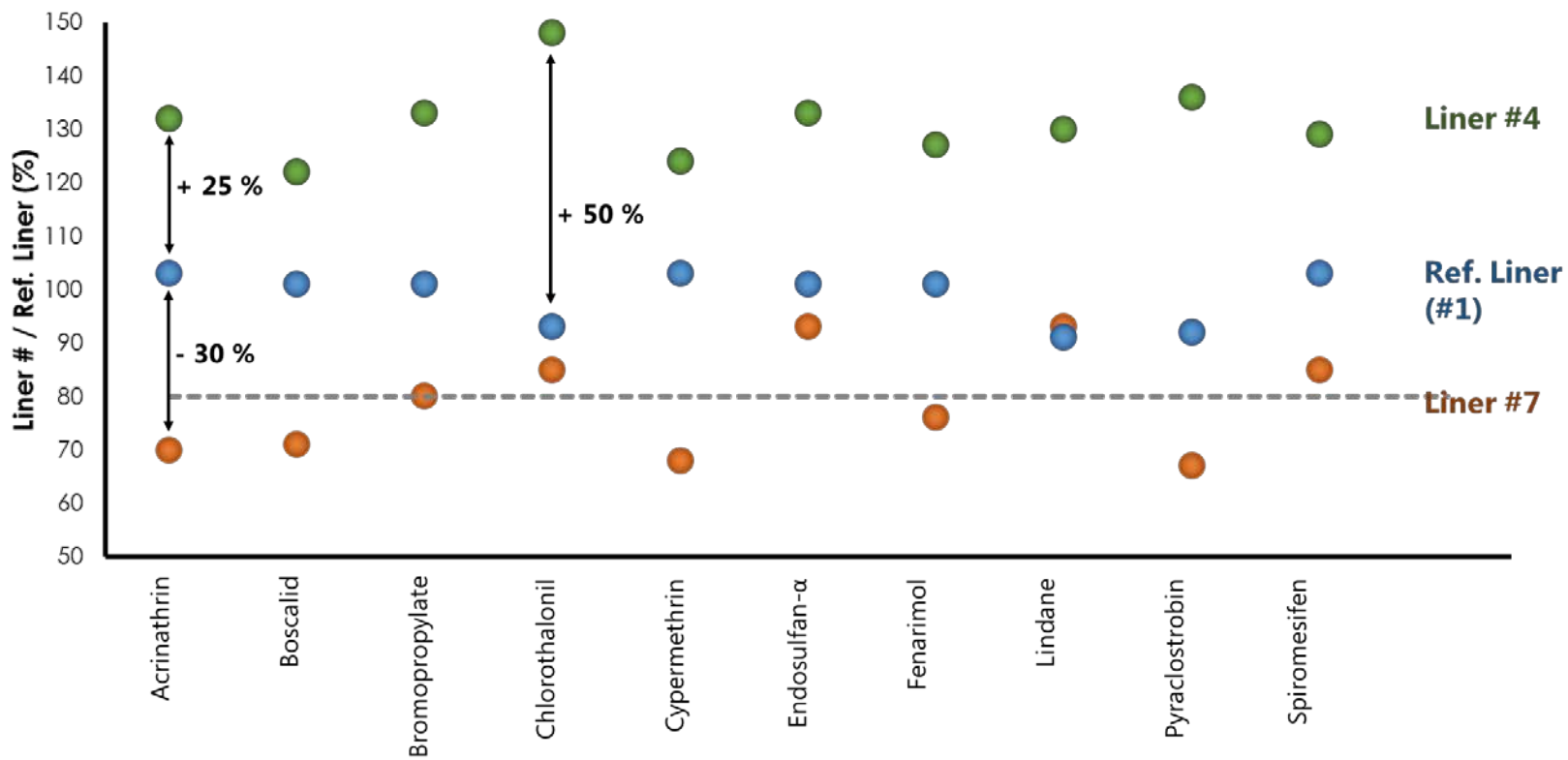
Average value for
3 concentrations



SIGNAL INTENSITY

$$\frac{\text{Area liner \#}}{\text{Area ref. liner}} (\%)$$





Signal intensity

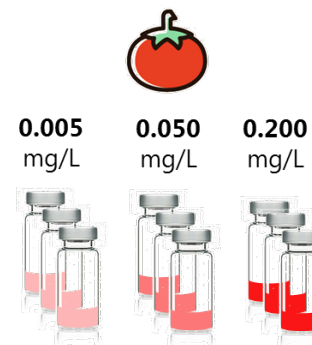
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188 pesticide residues in ethyl acetate

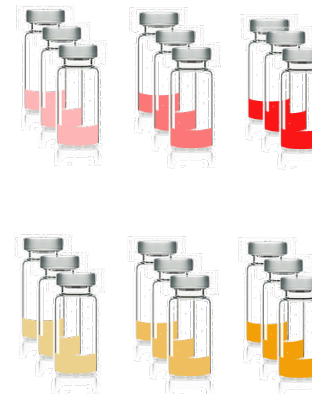


Durability and peak shapes

0.005 Tomato (x4)
0.050 Tomato (x4)
0.200 Tomato (x4)



0.005 Orange (x4)
0.050 Orange (x4)
0.200 Orange (x4)



0.005 Tomato (x4)
0.050 Tomato (x4)
0.200 Tomato (x4)

[...]

72 injections





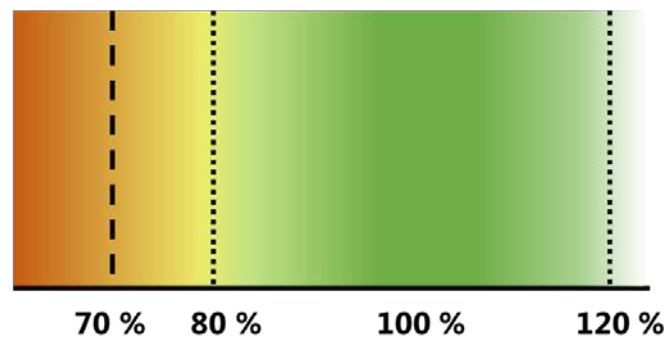
DURABILITY

$\frac{\text{Area after 51 injections}}{\text{Area first injection}}$ (%)

Difference between areas of the **1st** and **51th** injection

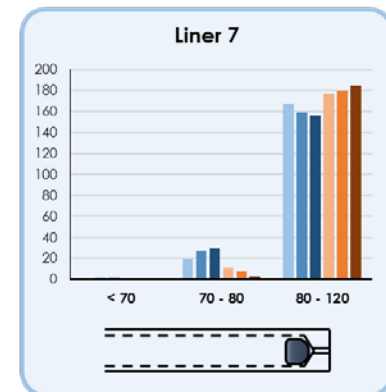
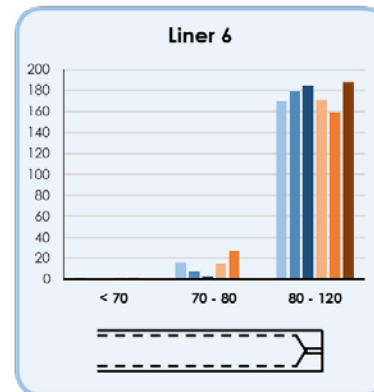
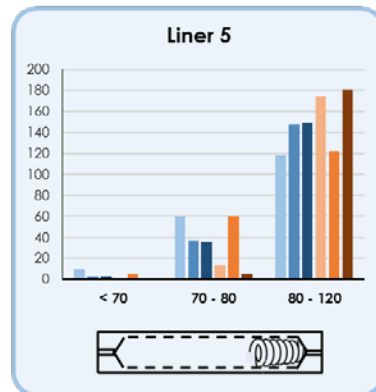
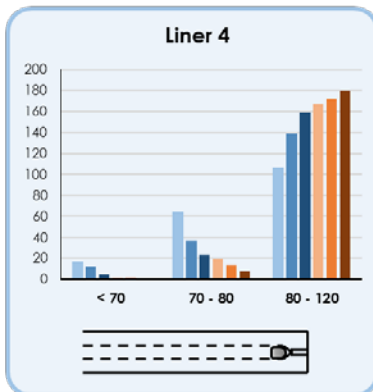
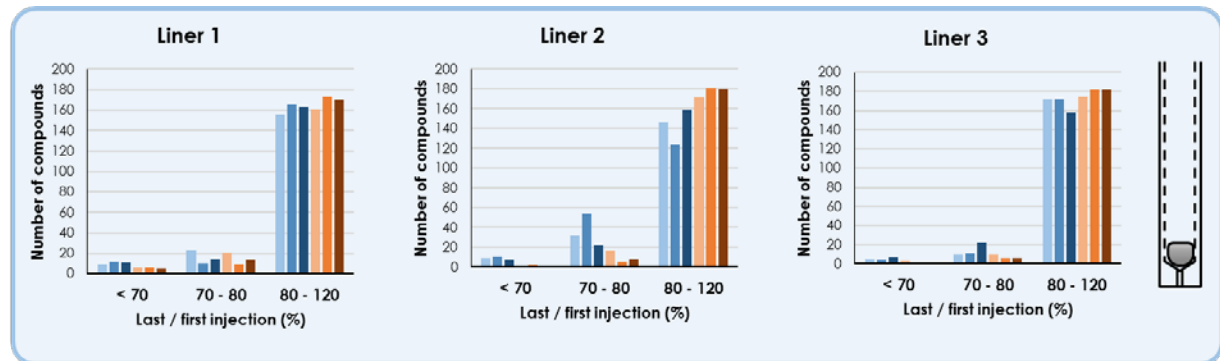
2 matrices (tomato, orange)

3 concentration levels
(0.005, 0.050, 0.200 mg/L)



Area after 51 injections
Area first injection

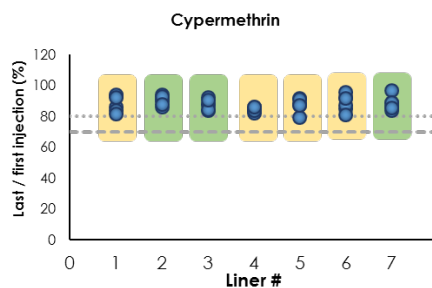
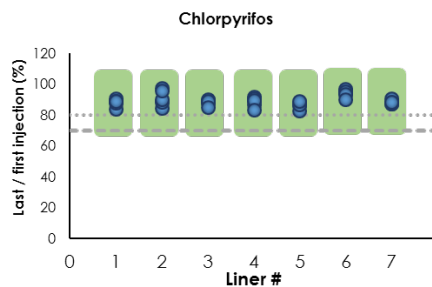
Worse	Tomato 0.005 mg/L
	Tomato 0.050 mg/L
Better	Tomato 0.200 mg/L
	Orange 0.005 mg/L
	Orange 0.050 mg/L
	Orange 0.200 mg/L



2 matrices * 3 conc. Levels = 6 points per liner

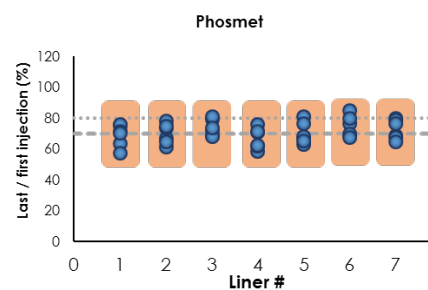
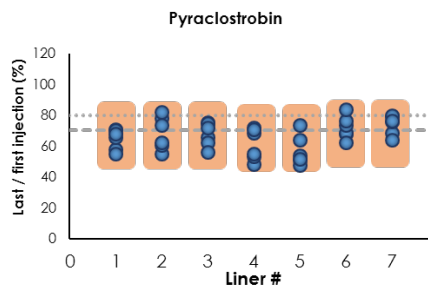
No liner-dependent

90 % compounds (169/188)



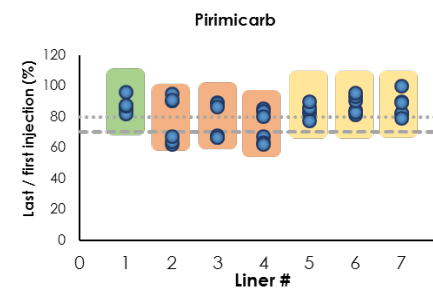
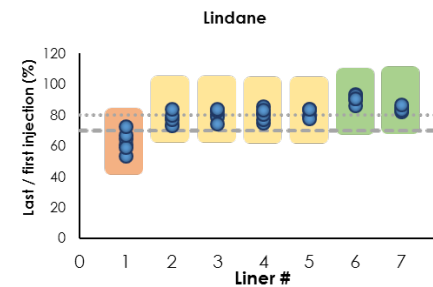
No liner-dependent

1 % compounds (2/188)



Liner-dependent

9 % compounds (17/188)

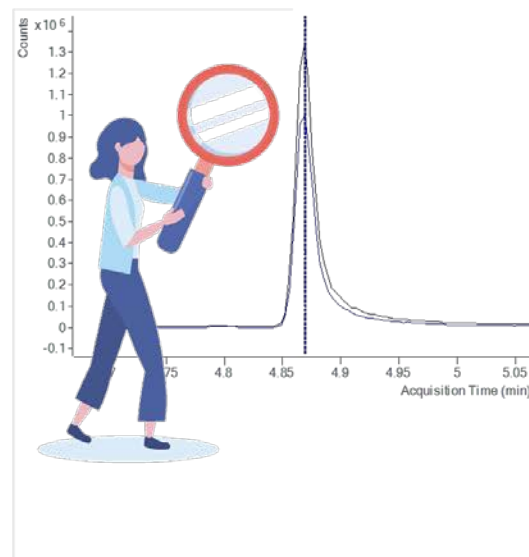




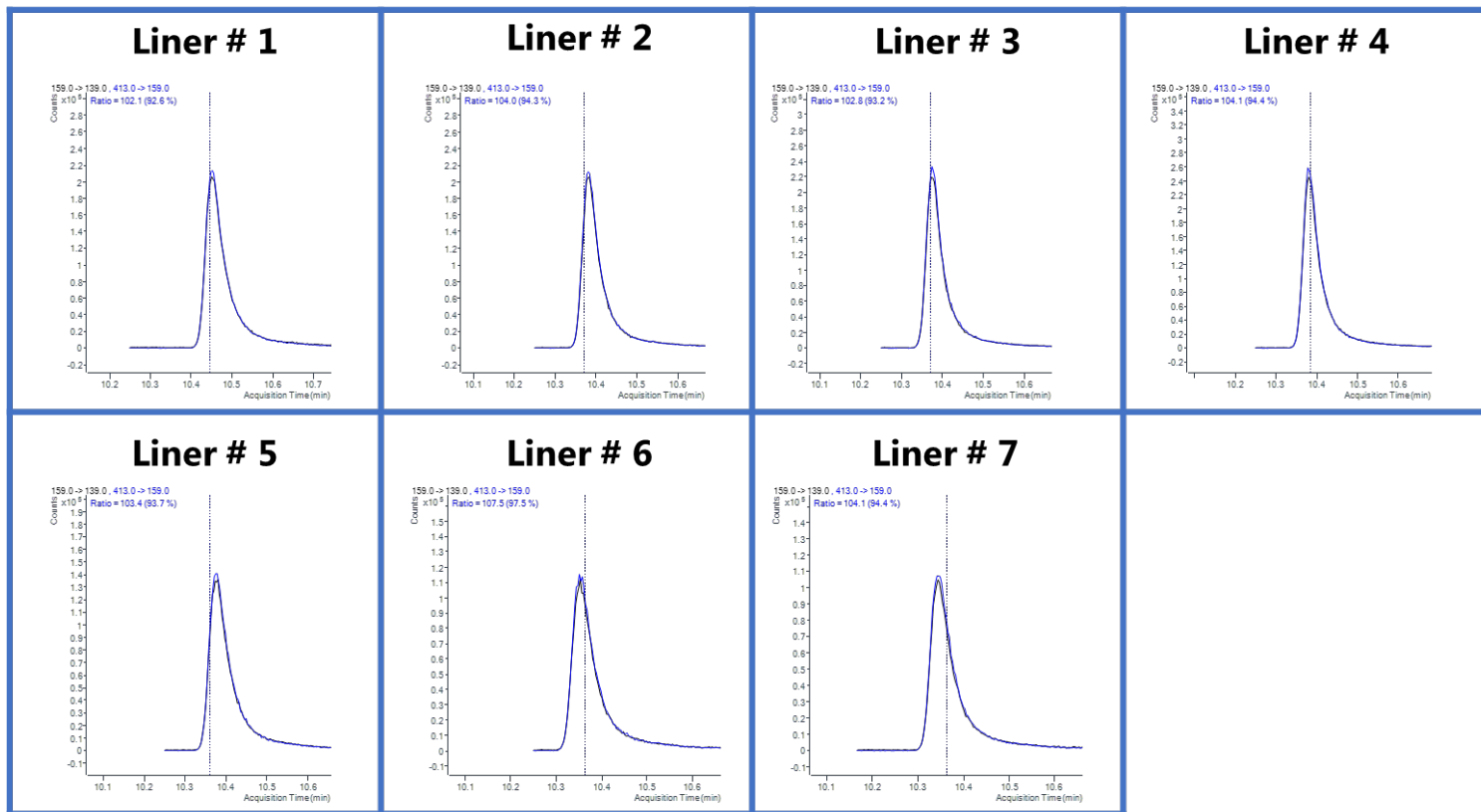
PEAK SHAPES

Manual evaluation of the peak shapes of 188 compounds to spot differences among the liners.

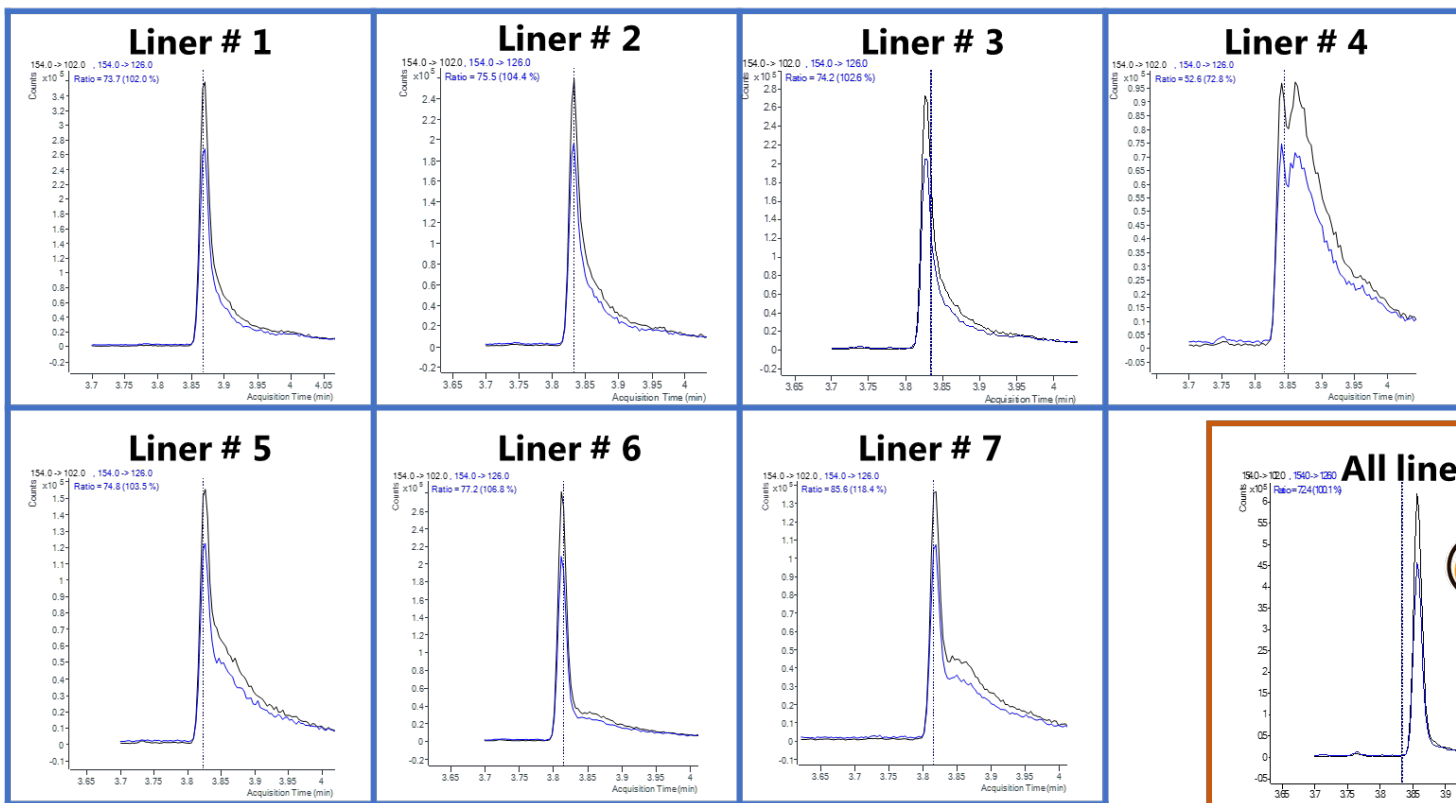
The liner configuration **did not affect** the peak shapes of the compounds, with just one exception.



Bixafen in tomato



Biphenyl in tomato





LINERS # 8 AND 9

They contained glass frit/woll in **middle position**

Their **maximum capacity** was in both cases higher than the sample volume after evaporation

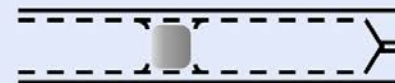
Suitable for **splitless** injection ?

Liner # 8



Glass frit, **medium position**

Liner # 9



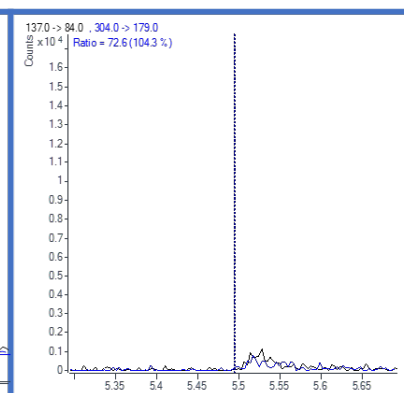
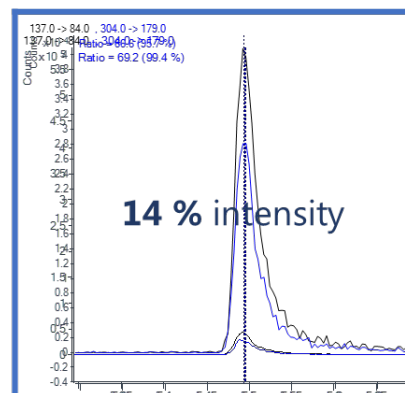
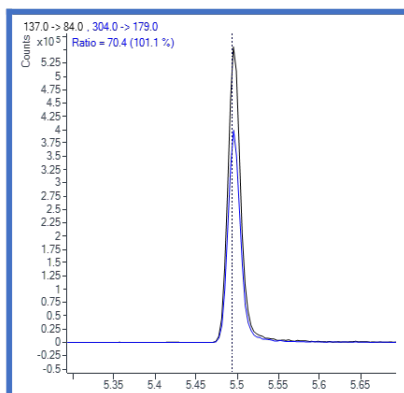
Tapered glass wool, **medium position**

Liner # 1

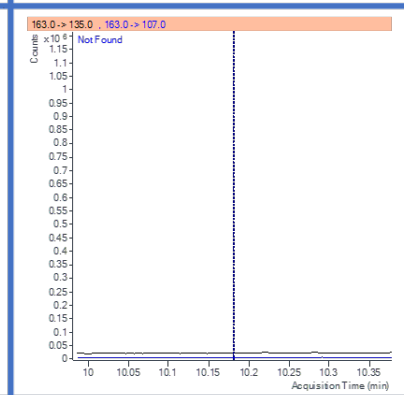
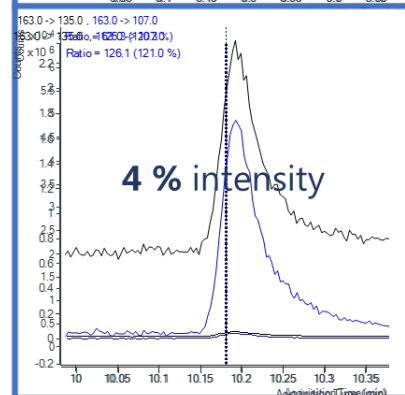
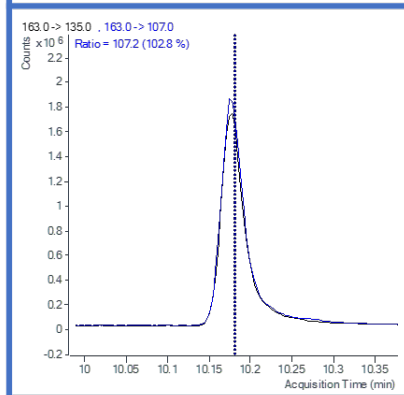
Liner # 8

Liner # 9

Diazinon



Etofenprox

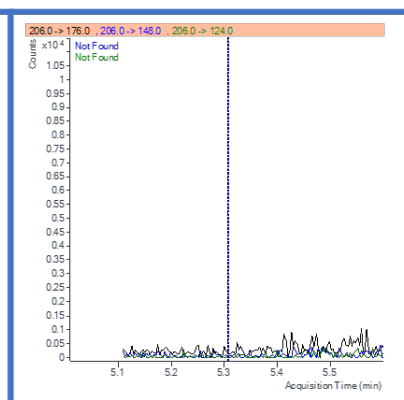
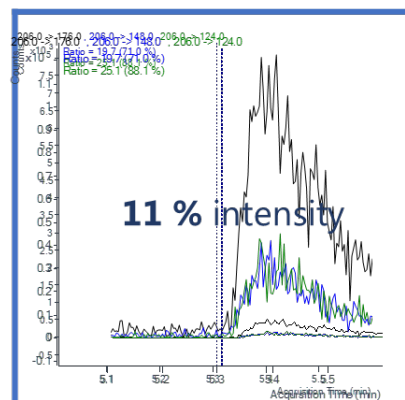
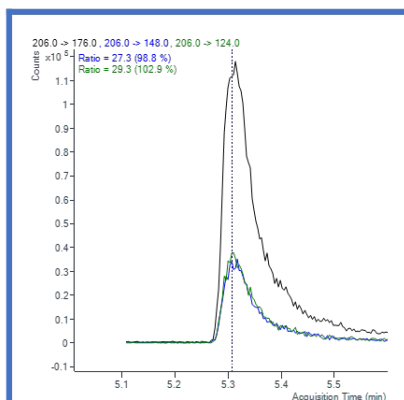


Liner # 1

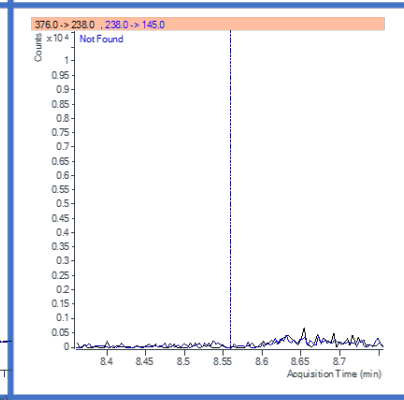
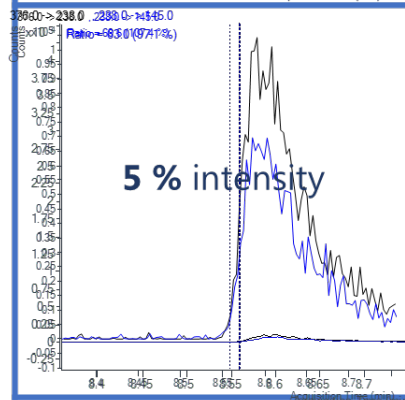
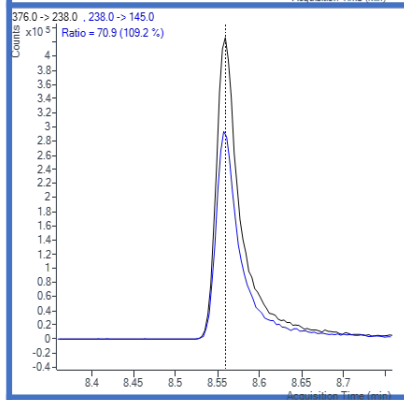
Liner # 8

Liner # 9

Dicloran



Picolinafen



- ▶▶▶▶ The **deactivation process** may affect the liner durability, especially for the analysis of clean matrices (more susceptible to the presence of active sites).

- ▶▶▶▶ A **reduced internal diameter** does not have an intense effect on peak shapes, but it provides an increase in their sensitivity

- ▶▶▶▶ Liners with **packing in middle position** might result in an intense loss of sensitivity in splitless injection.

- ▶▶▶▶ The presence of a **complex matrix** in the sample could help maximize the lifetime of any type of liner (dual layer injection).





Thank You for Your Attention



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LABORATORY