

Take the Trouble Out of Troubleshooting

Gas chromatography

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Online Application Engineer
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“Everything Was Just Fine... and Then This Happened”

“How do I troubleshoot?”

Track your actions/keep a logbook of events:

- Changed column, liner, septum, or syringe
- Injected samples, or used another method
- Carried out maintenance, cut column, or inlet flush



Logic
=
**Something changed
(slowly or suddenly)**
=
Something is different

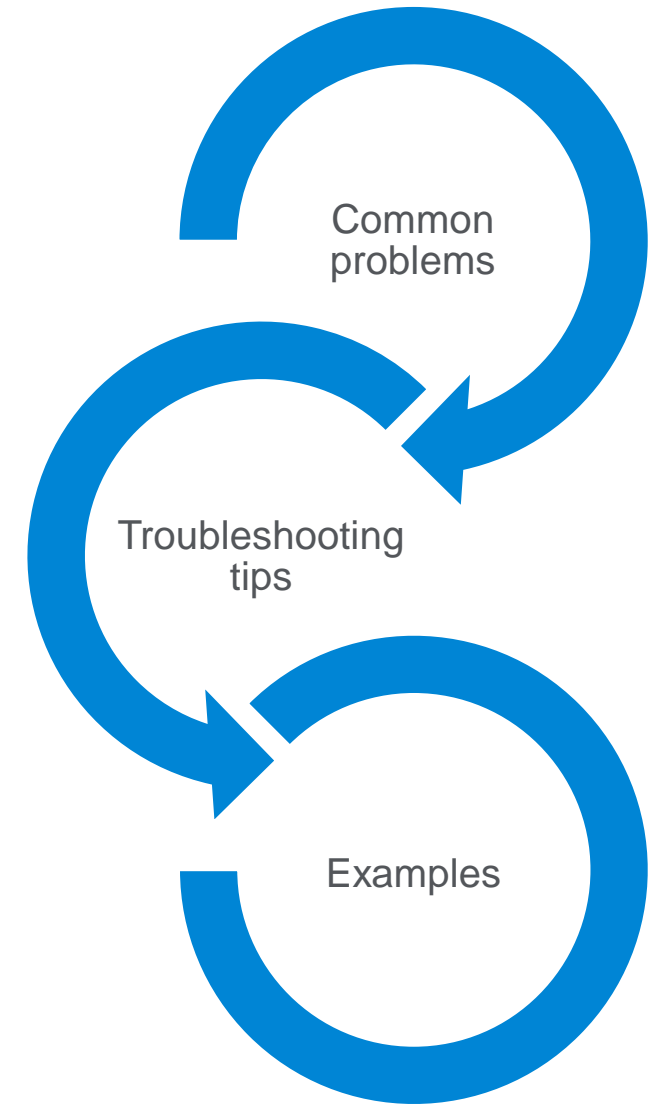
Logical Troubleshooting

Troubleshooting starts with isolating the problem

- There are five basic areas that problems can arise from:
 - Injector
 - Flow
 - Column
 - Detector
 - Electronics
- Or...
- A combination of all of these

Knowing what can and cannot cause the symptom is key, and most importantly **don't panic**

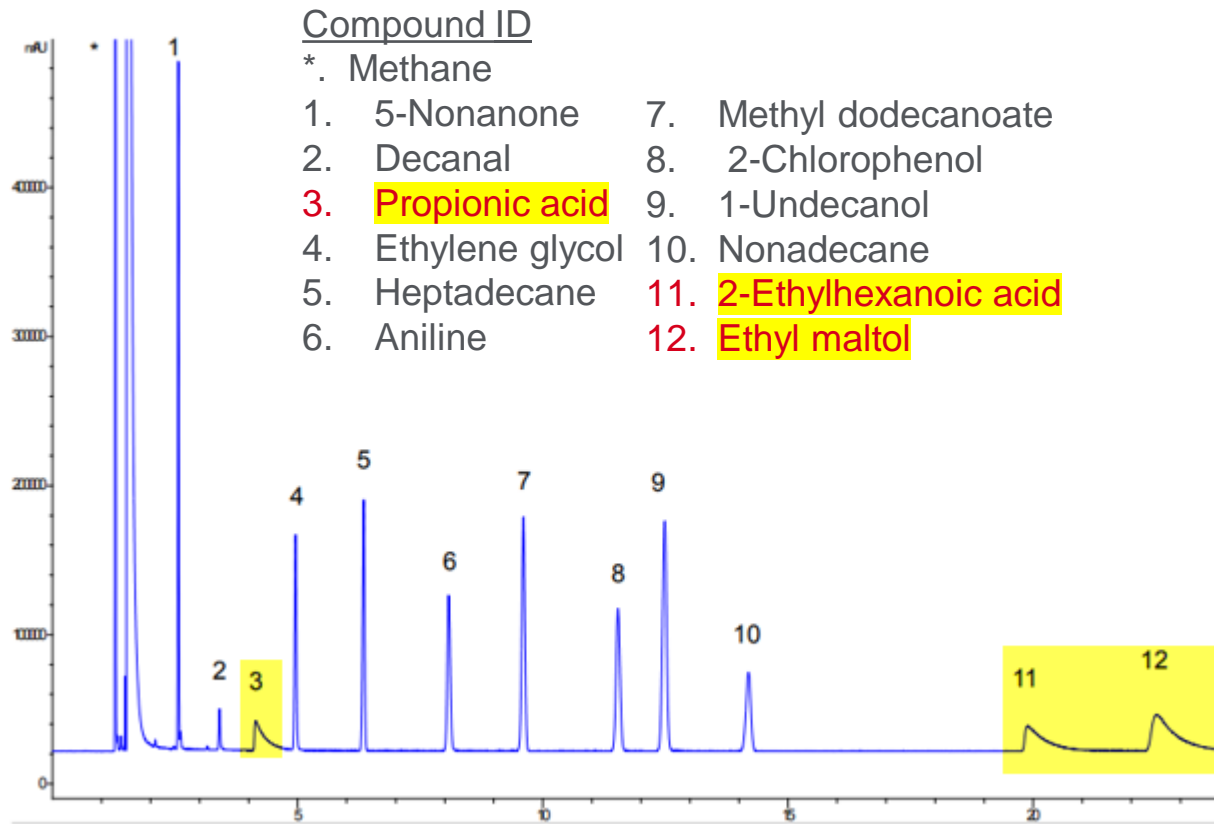
Agenda



Common Peak Shape Issues

- **Peak tailing** – flow path or activity
- **Bonus peaks** – in sample or backflash (carryover)
- **Split peaks** – injector problems, mixed solvent
- **No peaks** – sample wasn't introduced, wasn't detected
- **Response changes** – activity, injector discrimination, detector problem
- **Peak fronting** – overload or solubility mismatch, injector problems
- **Shifting retention** – leaks, column aging, contamination, or damage
- **Loss of resolution** – separation decreasing, peak broadening
- **Baseline disturbances** – column bleed, contamination, electronics issues
- **Noisy or spiking baseline** – electronics or contaminated detector
- **Quantitation problems** – activity, injector, or detector problems
- **Other**

Peak Tailing



Injector or column is active

- Reversible adsorption of active compounds (-OH, -NH, -SH)

Flow problem

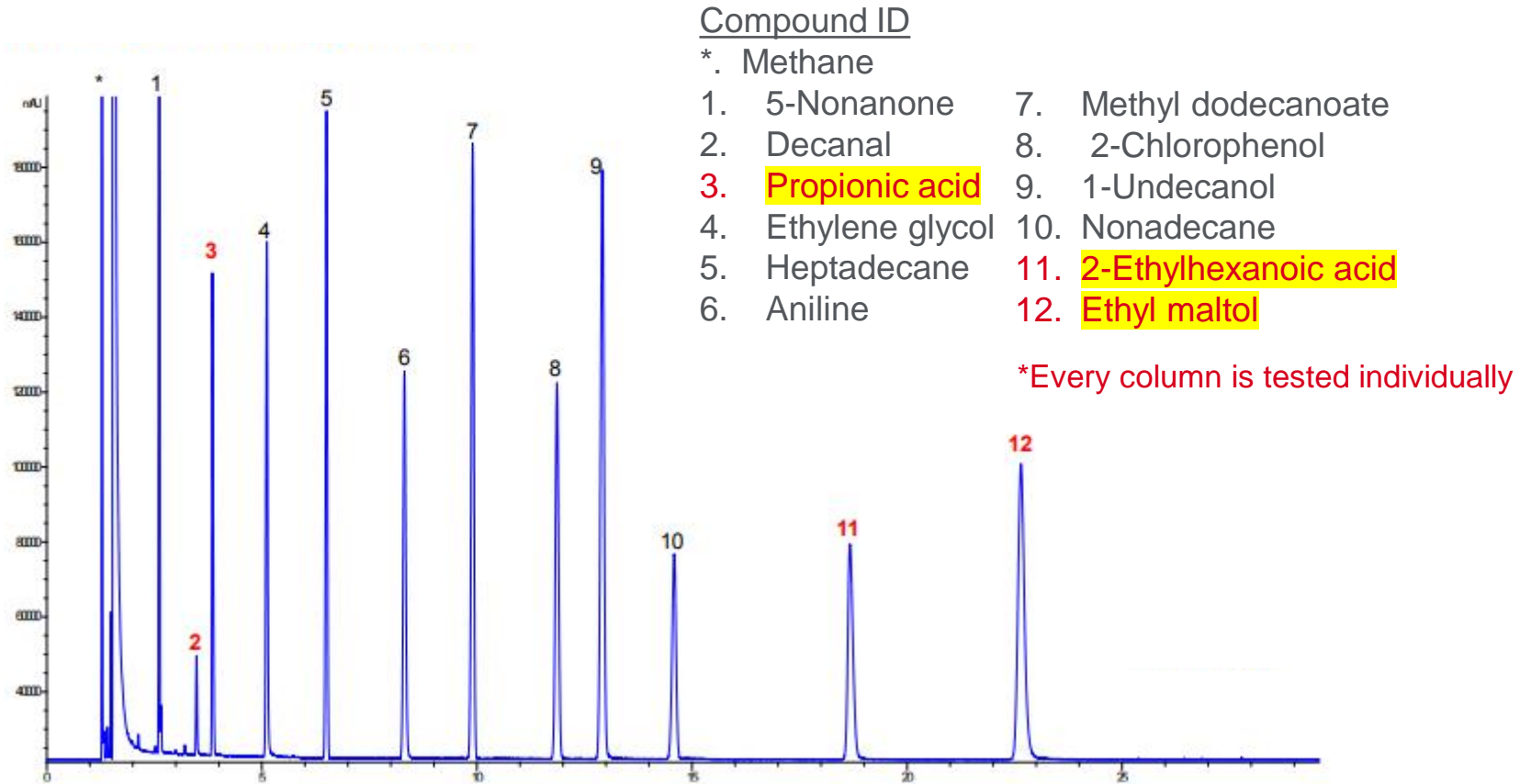
- Dead volume, obstruction, poor installation, or severe column contamination

Miscellaneous issues – overloading of PLOT columns, coelution, polarity mismatch between phase, solute or solvent, some compounds always tail

***Tip:** Inject a light hydrocarbon; this should not tail unless there is a flow path problem.

Agilent Inert Flow Solution

Modified Agilent J&W DB-WAX UI mix on DB-WAX UI, 122-7032UI



Brochure: 5991-6709EN

Agilent Inert Flow Solution

Agilent UltiMetal Plus inlet weldment, shell and transfer lines



Agilent Ultra Inert inlet liner



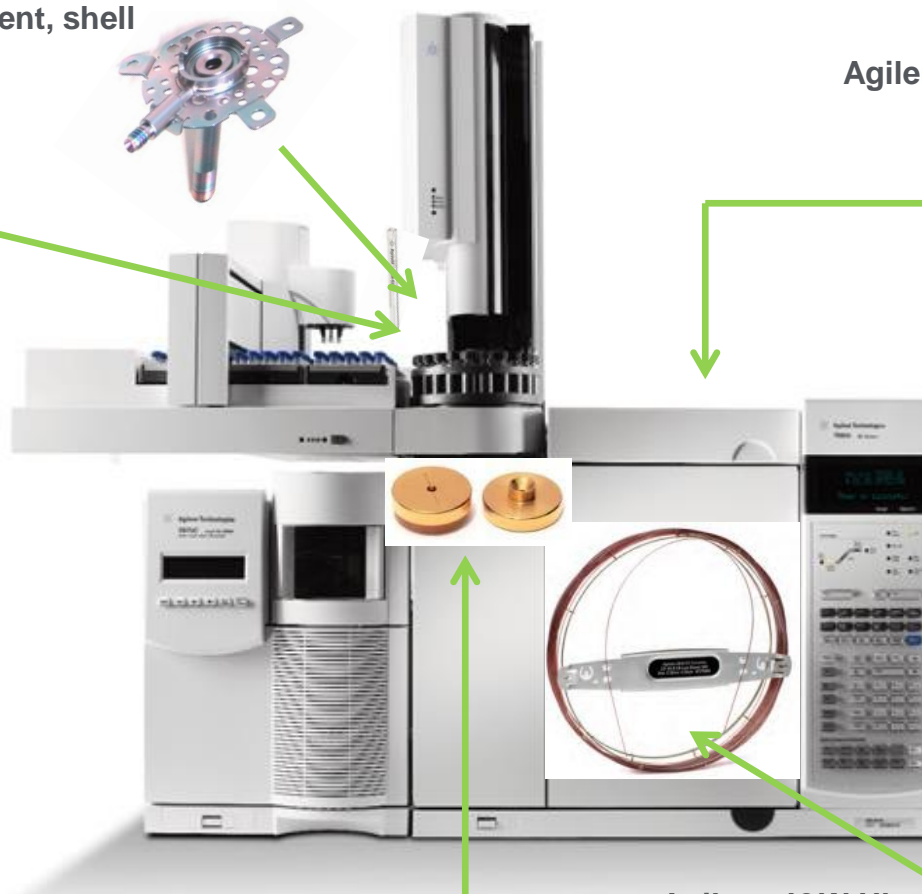
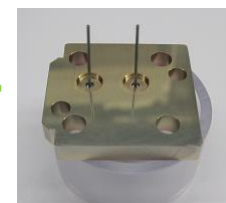
Agilent UltiMetal Plus ferrules



Agilent UltiMetal Capillary Flow Technology devices, Ultimate union



Agilent UltiMetal Plus- TCD, FPD, NPD/FID jets



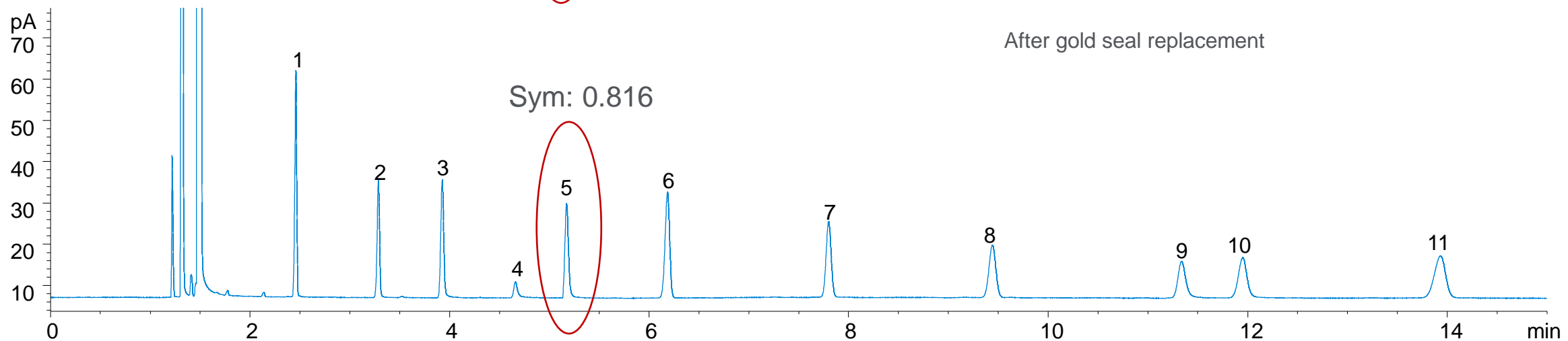
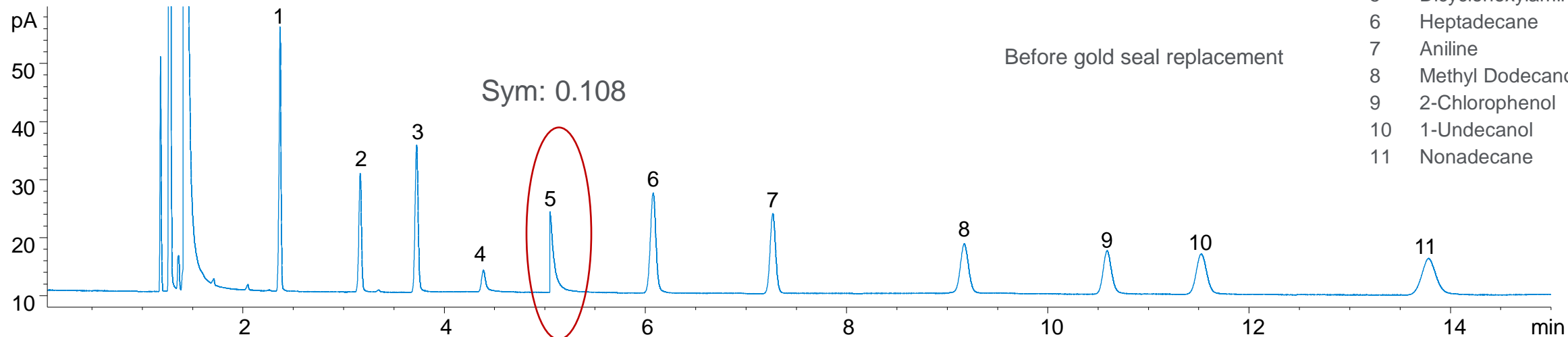
Agilent Ultra Inert gold seal

Agilent J&W Ultra Inert GC column

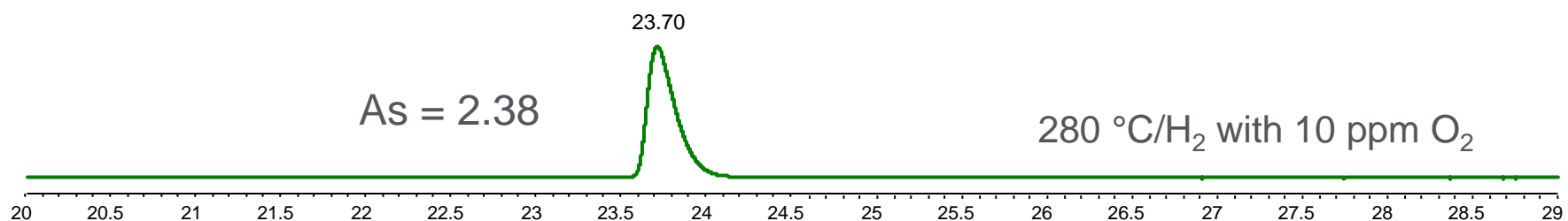
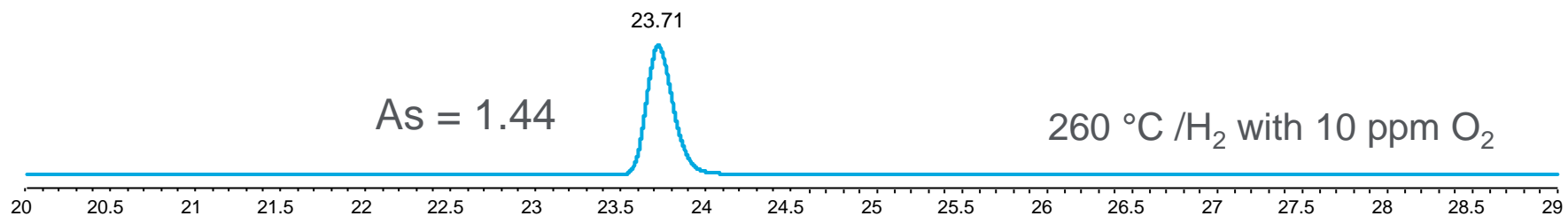
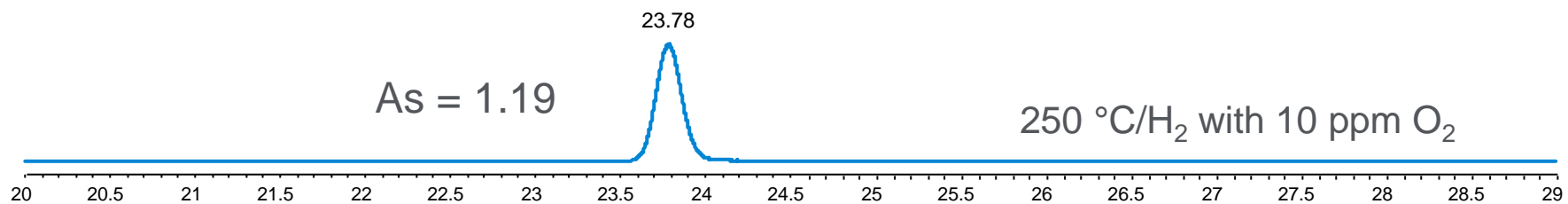
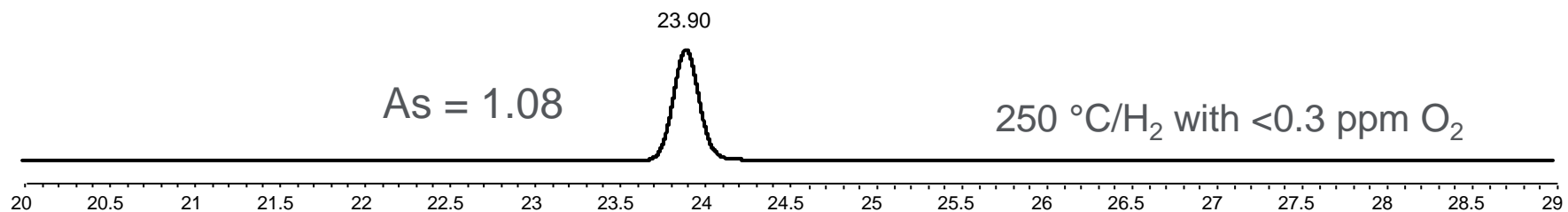
Brochure: 5990-8532EN

Peak Tailing from Contaminated Consumables

Peak	
0	Methane
1	2-Nonanone
2	Decanal
3	2,3-Butanediol
4	Ethylene Glycol
5	Dicyclohexylamine
6	Heptadecane
7	Aniline
8	Methyl Dodecanoate
9	2-Chlorophenol
10	1-Undecanol
11	Nonadecane



Effect of Oxygen on Peak Shape of 2-ethylhexanoic Acid



Self Tightening Nuts: No Leaks, No Downtime, No Frustration



- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- No tools needed
- Works only with graphite/vespel ferrules

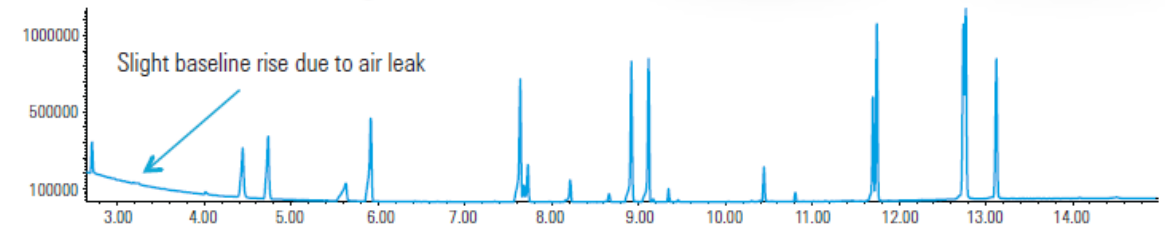
Part Number	Description
G3440-81013	Column Nut, Collared Self-Tightening MSD
G3440-81011	Column nut, Collared Self Tightening Inlet/Detect
G3440-81012	Collar for Self Tightening Nut

<https://www.agilent.com/en/video/gc-supplies-innovation>

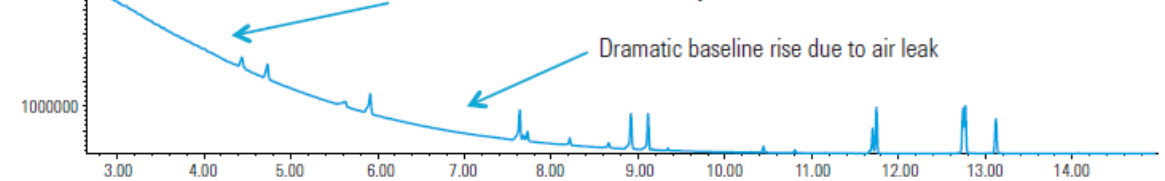
<https://www.agilent.com/en/video/stcn-inlet-detector>

<https://www.agilent.com/en/video/stcn-mass-spec>

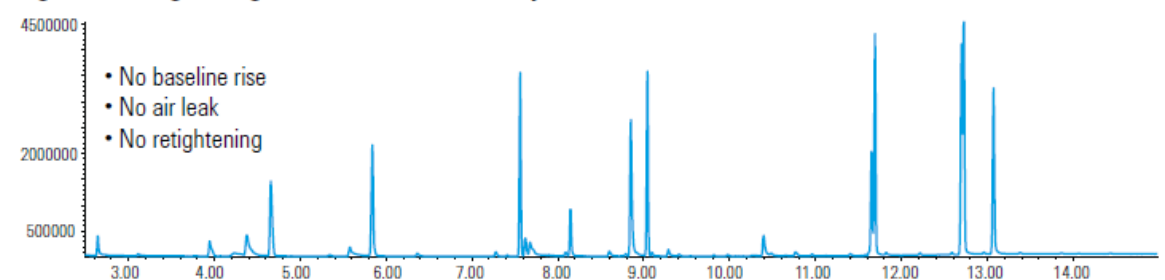
Standard column nuts new fitting



Standard column nuts after 25 injections

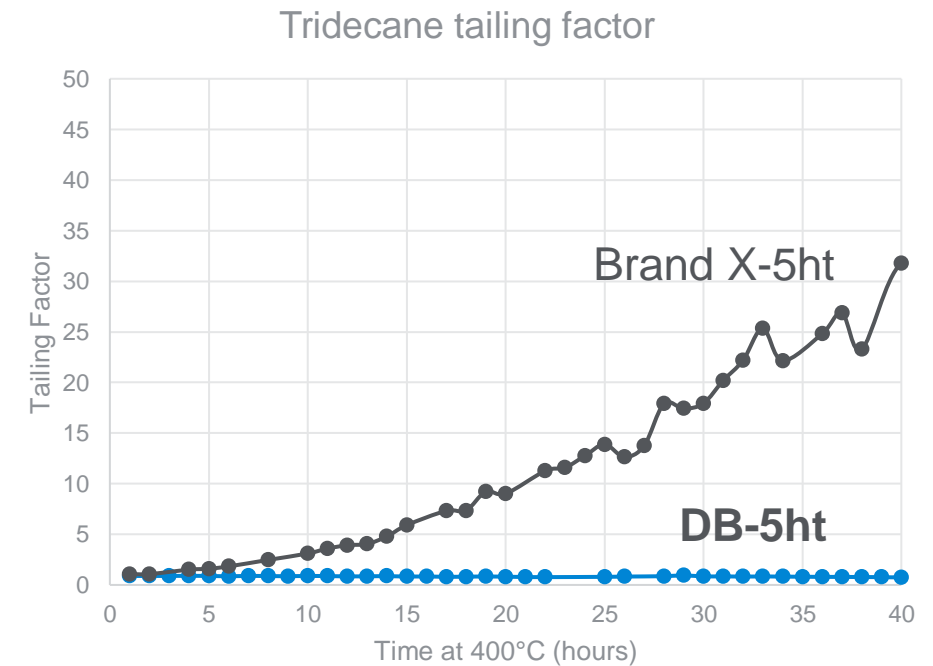
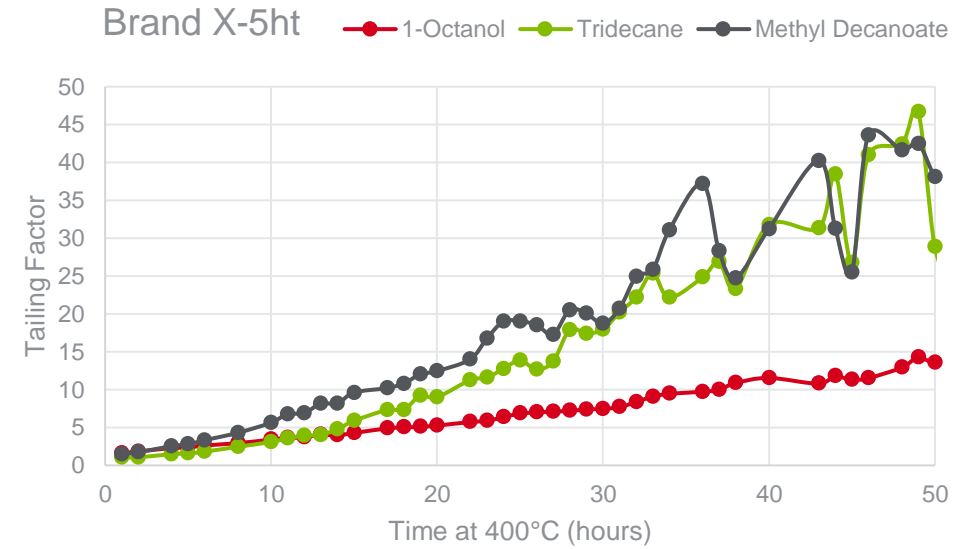
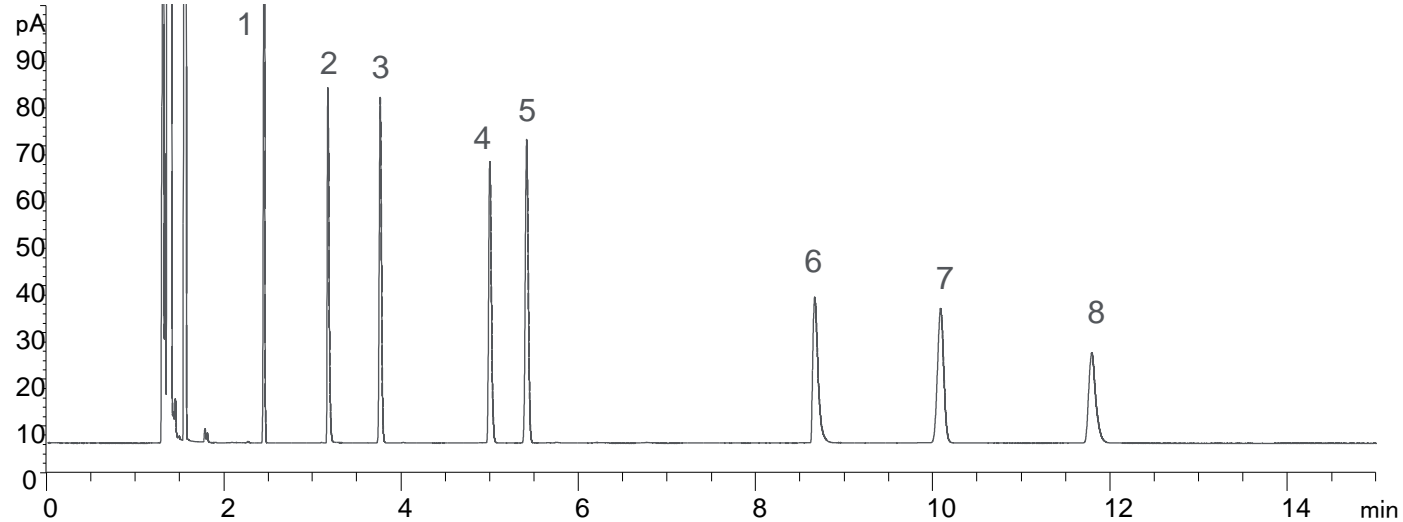
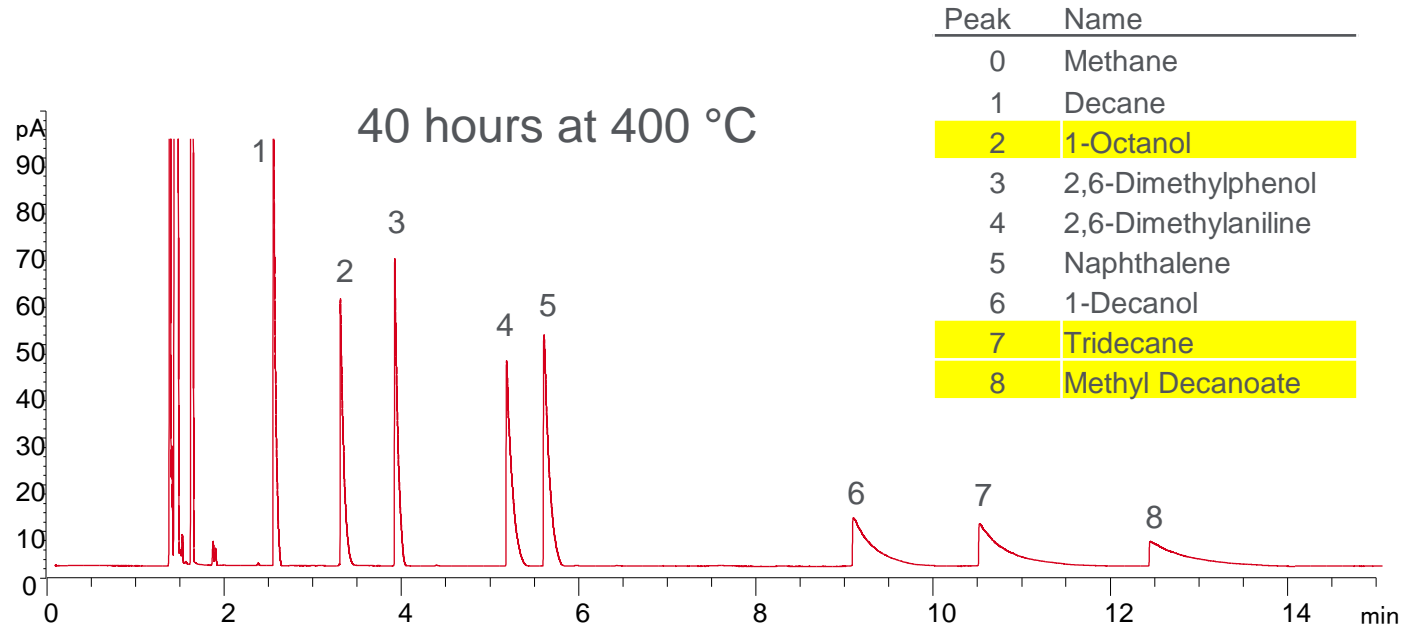


Agilent Self Tightening Column Nuts after 400 injections

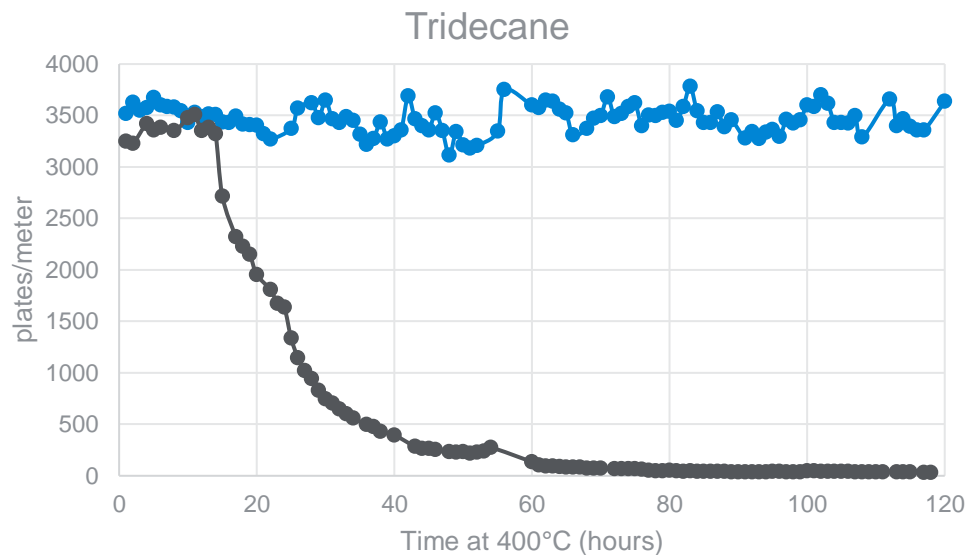


400 injections

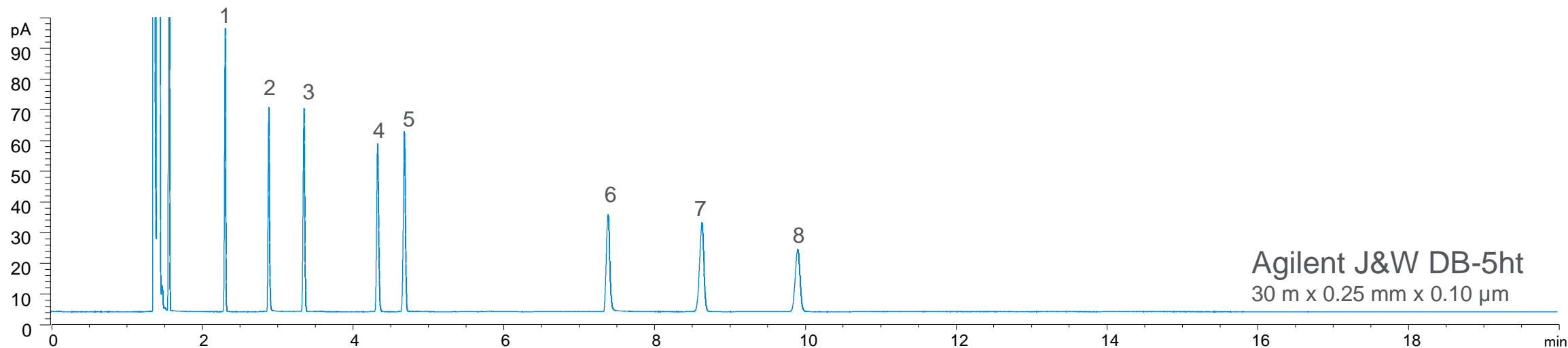
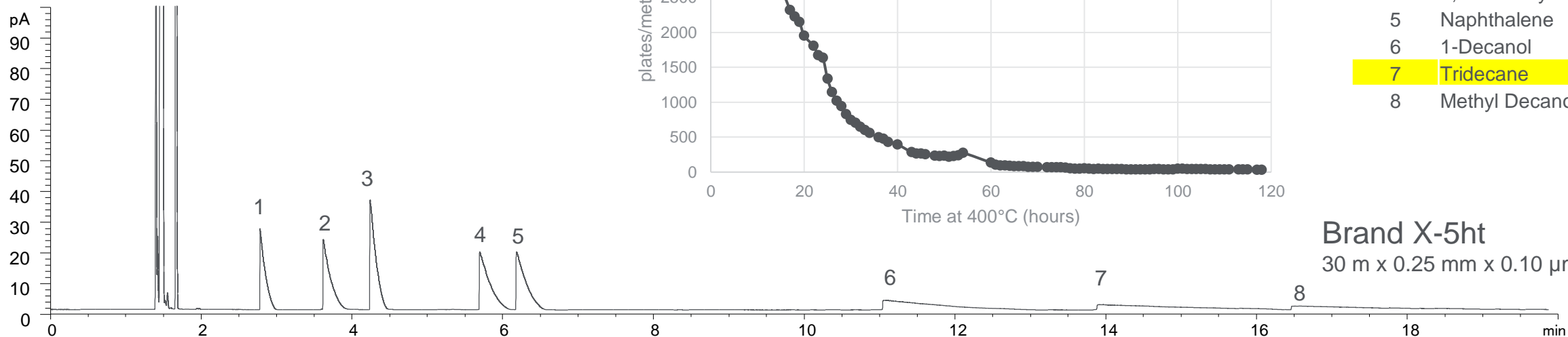
Peak Tailing from Thermal Degradation



Column Efficiency Over 120 Hours at 400 °C

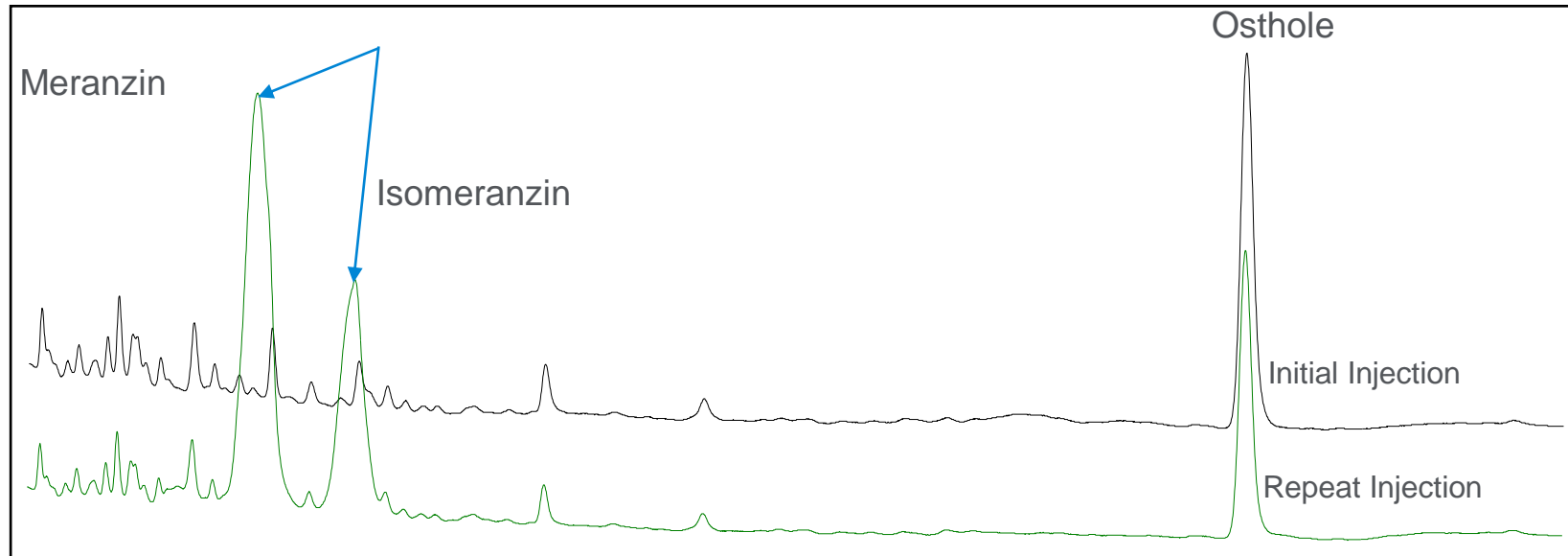


Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate



Agilent publication: 5994-1013EN

Bonus or Ghost Peaks



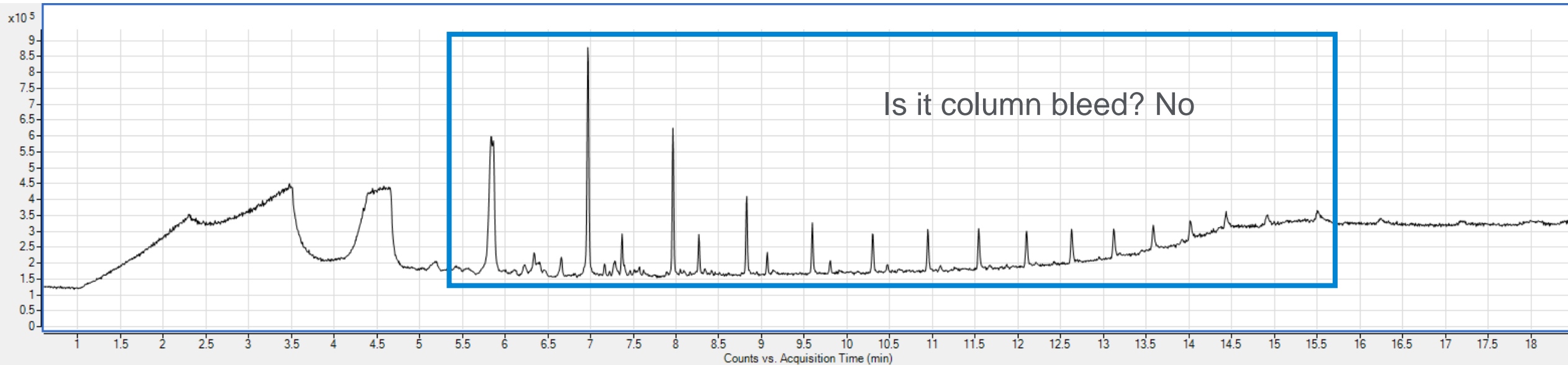
5991-9078EN

Contamination in injector, column, or flow
(carrier gas)

- Carryover from a backflash or previous sample
- Bad tank of gas, or traps have expired
- Septum bleed

Tip: Start a blank run...it should be blank

What Are These Repeating Peaks?



Common ions for siloxane molecules:

73

147

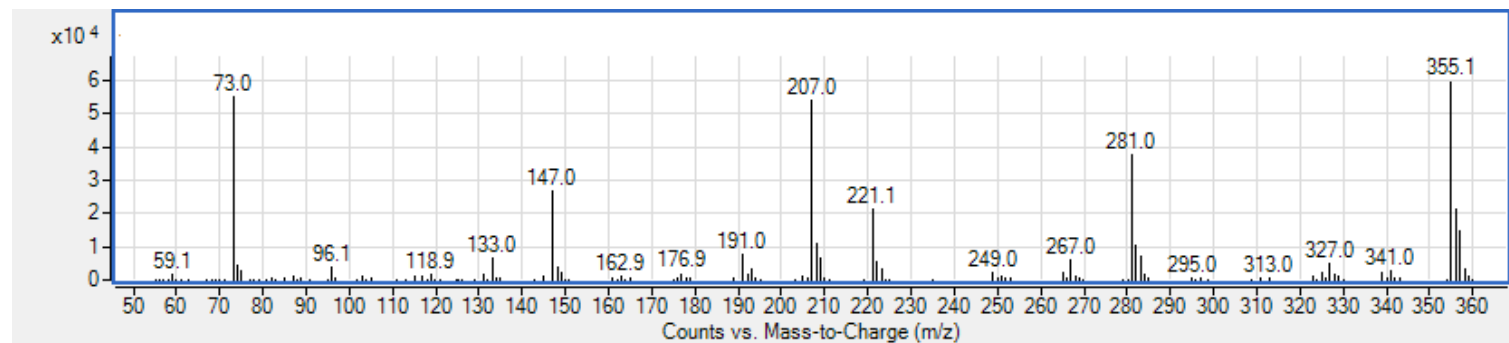
207

281

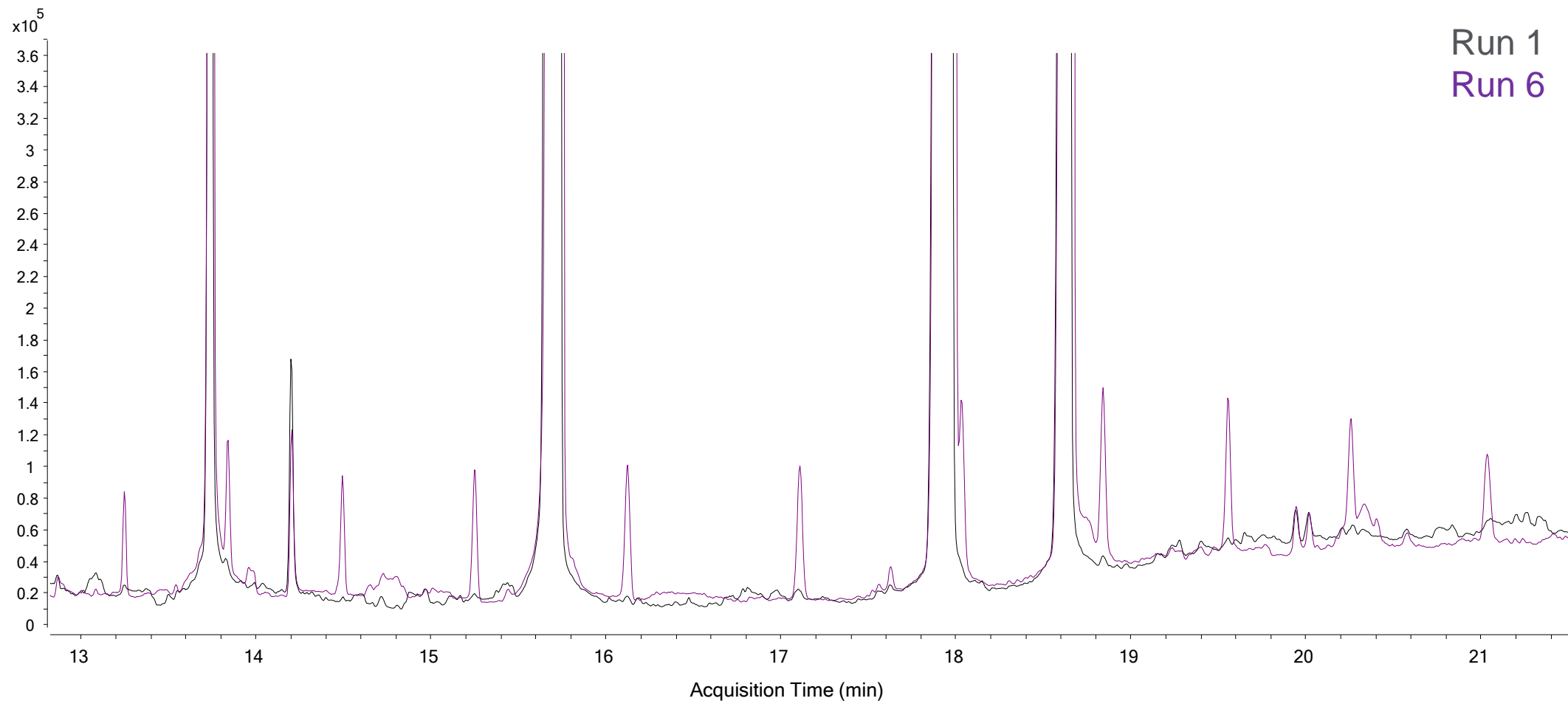
355

Septa contamination in wash vials or inlet liners can be diagnosed by looking for siloxane polymers in your total ion chromatogram. Each peak in the chromatogram corresponds to a cyclized (ring structure) siloxane molecule. These molecules fragment with similar patterns.

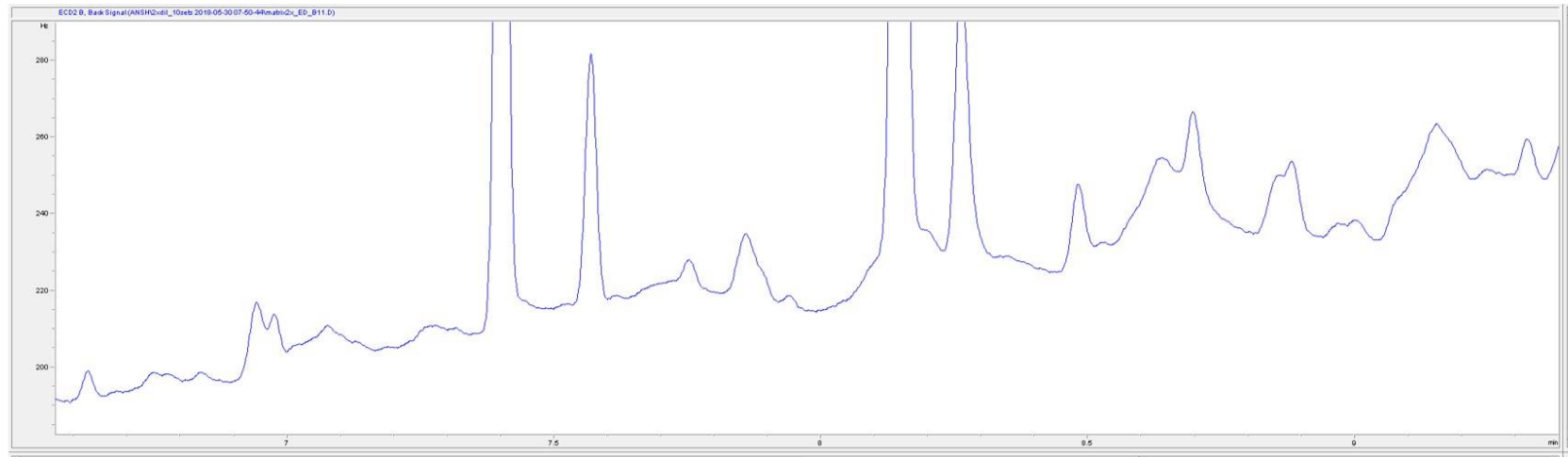
Example spectrum:



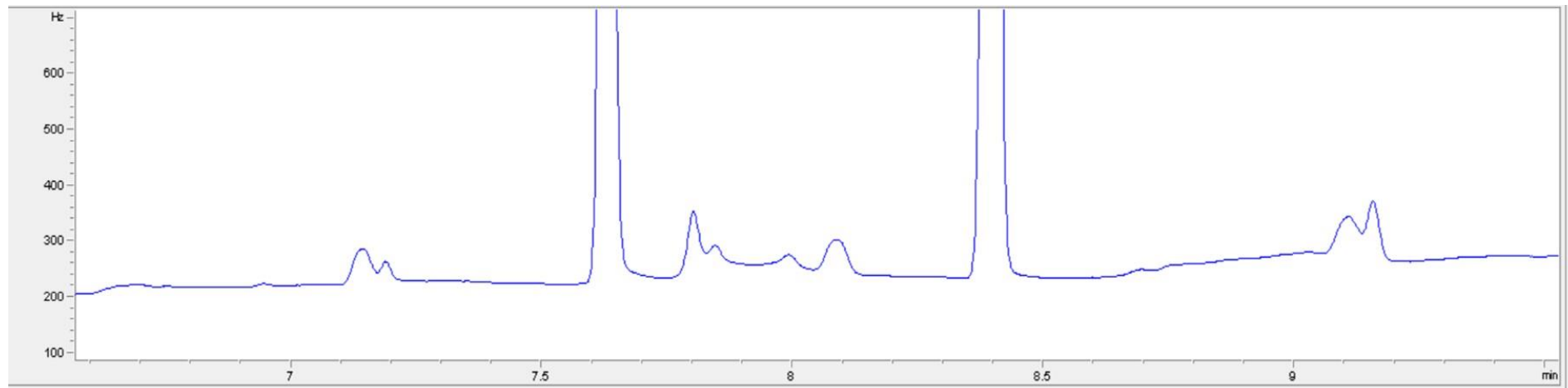
Multiple Injections from the Same Vial: Siloxanes



Does Your Baseline Look Like This? Do You See Extra Peaks?

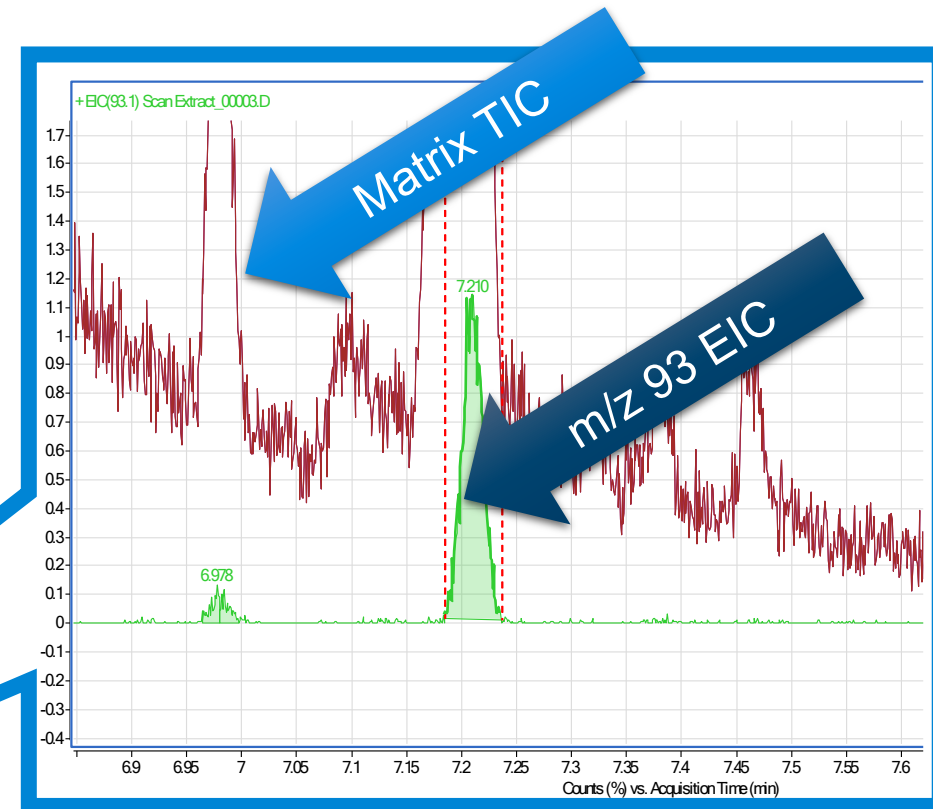
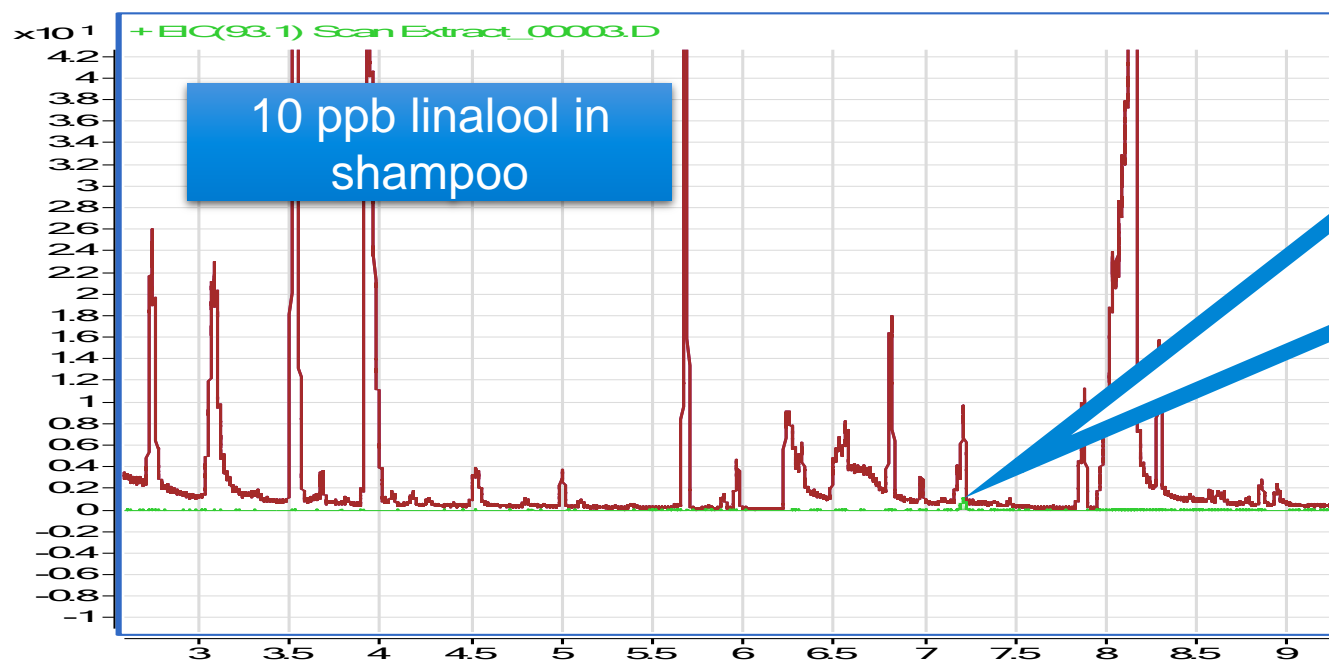


When it *should* look like...



The Matrix

If your target ions are buried beneath matrix peaks, it might be time to trim the column or do sample cleanup

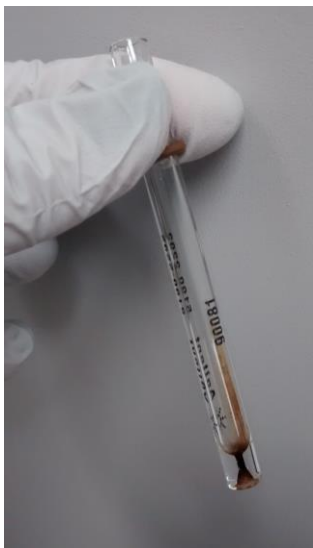


...or improve your sample cleanup

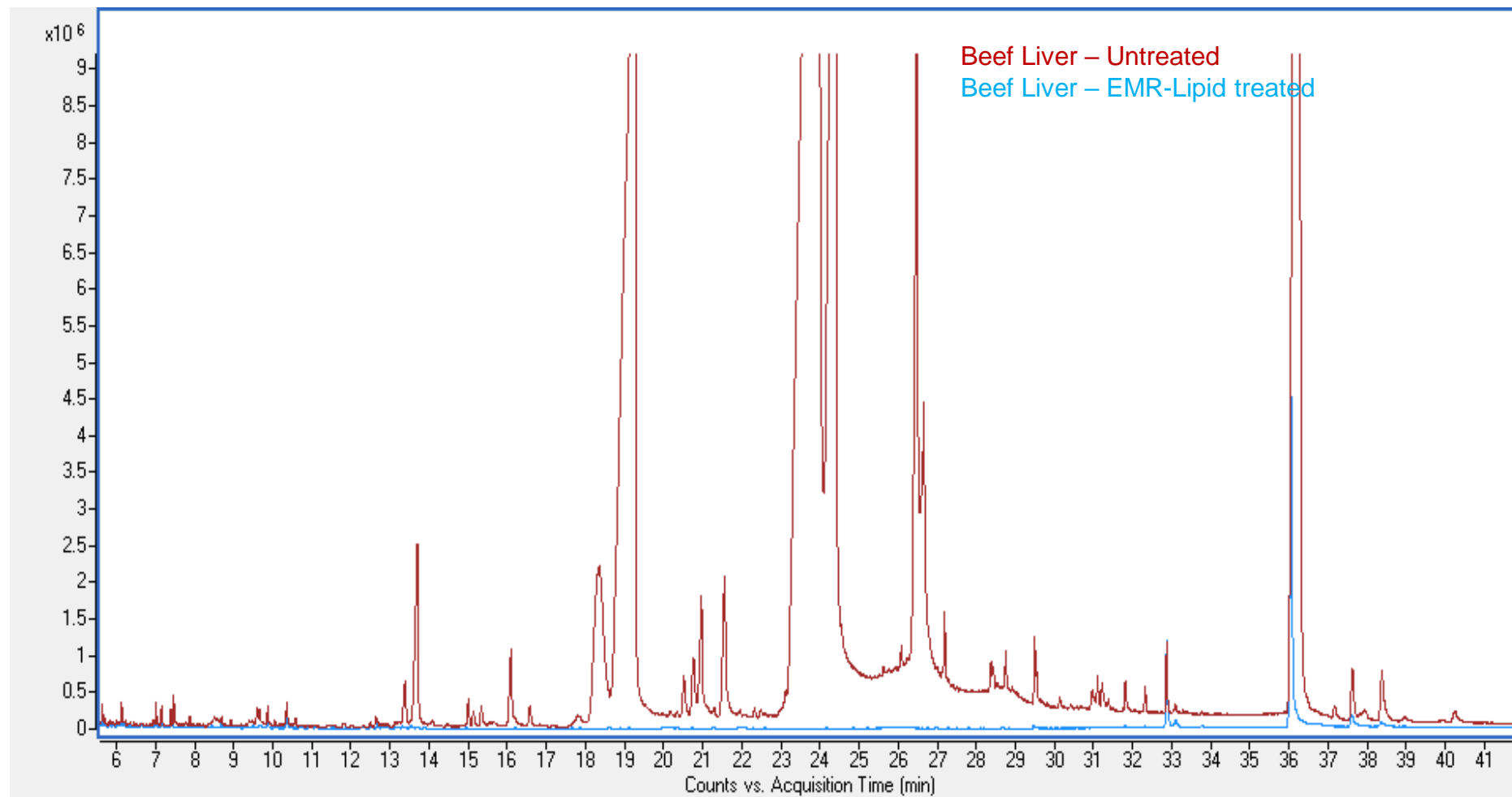
The Importance of Sample Cleanup



50 samples
with cleanup

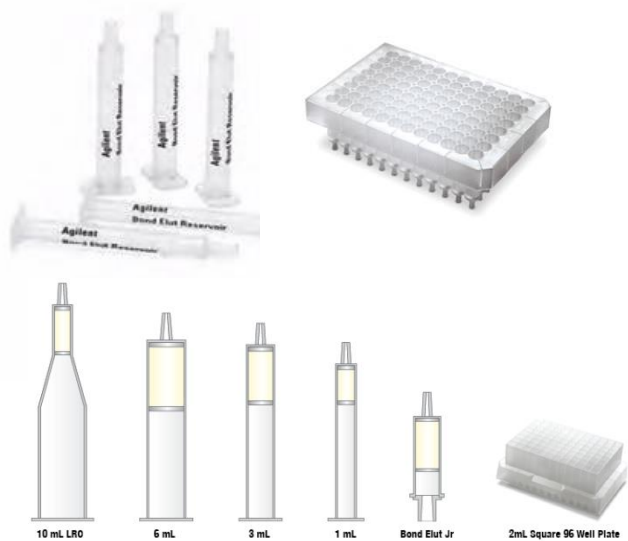


50 samples
without cleanup



For sample cleanup help, please contact us at spp-support@agilent.com.

Offline Options for Sample Matrix Removal



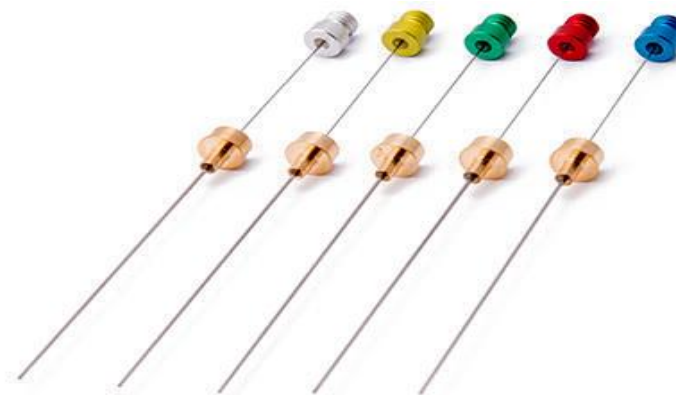
Bond Elut Solid Phase Extraction cartridges and plates



Filter vials



QuEChERS



SPME



Captiva EMR-Lipid filtration cartridges and plates

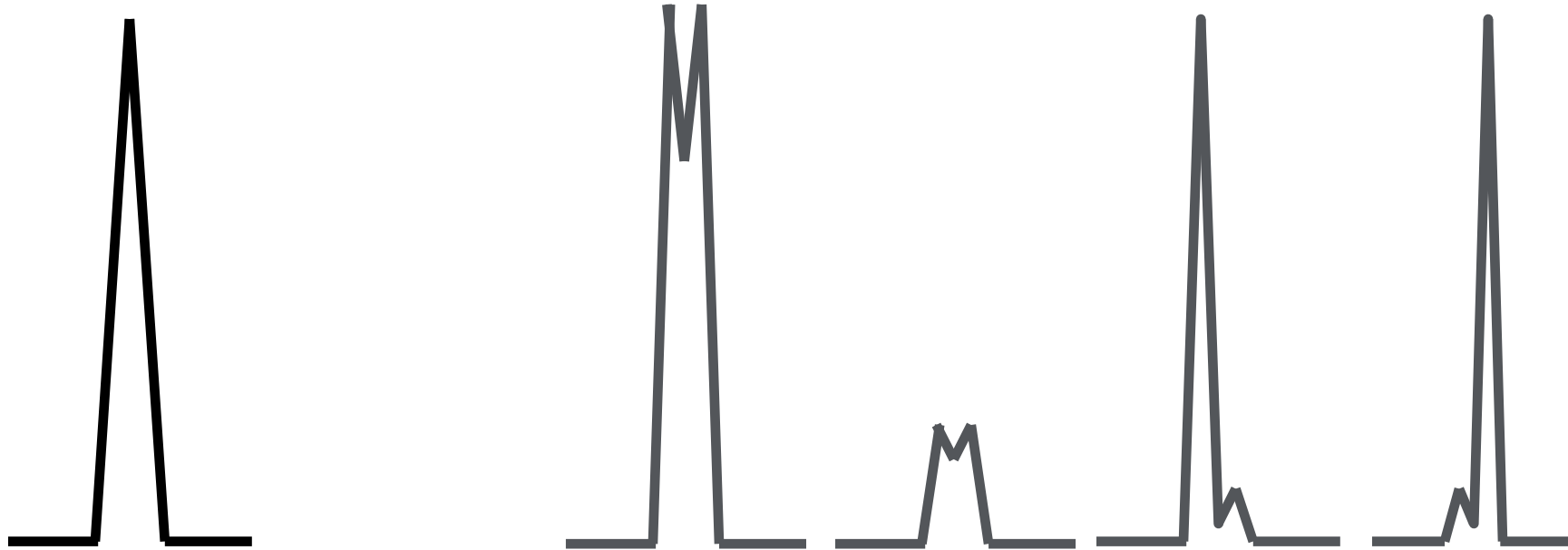


Chem Elut S



Captiva syringe filters

Split Peaks



Injector (poor sample introduction)

- Injecting the sample twice (somehow?)
- Mixed sample solvent (polarity difference)
- Sample in syringe needle (manual inject)

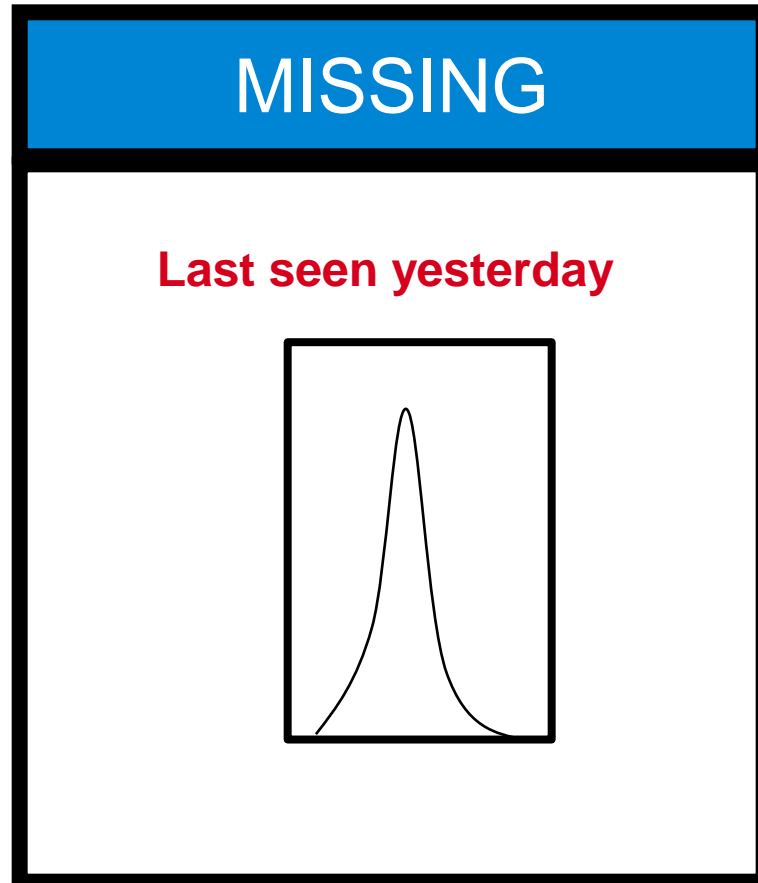
Injector (activity)

- Breakdown (not really a split peak, two peaks)
- Sample degradation in injector

Volatility

- High boilers dropping out on cold spots
- Transfer line temperatures
- Unions or fittings not tracking column temperature

No Peaks



Detector (not on, or not operational)

Injector (not working)

Plugged syringe/plunger not moving

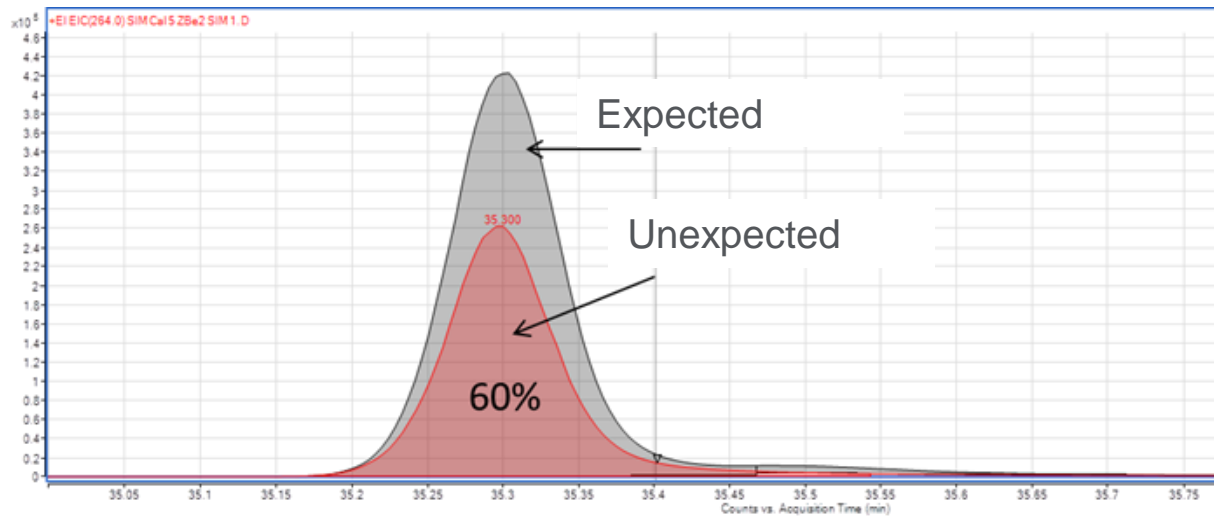
- Wrong injector (or detector)
- Huge leak (older systems)
- No carrier gas flow

Not the column unless...

- Broken column or no column

Peak Response

All change in size



Injector

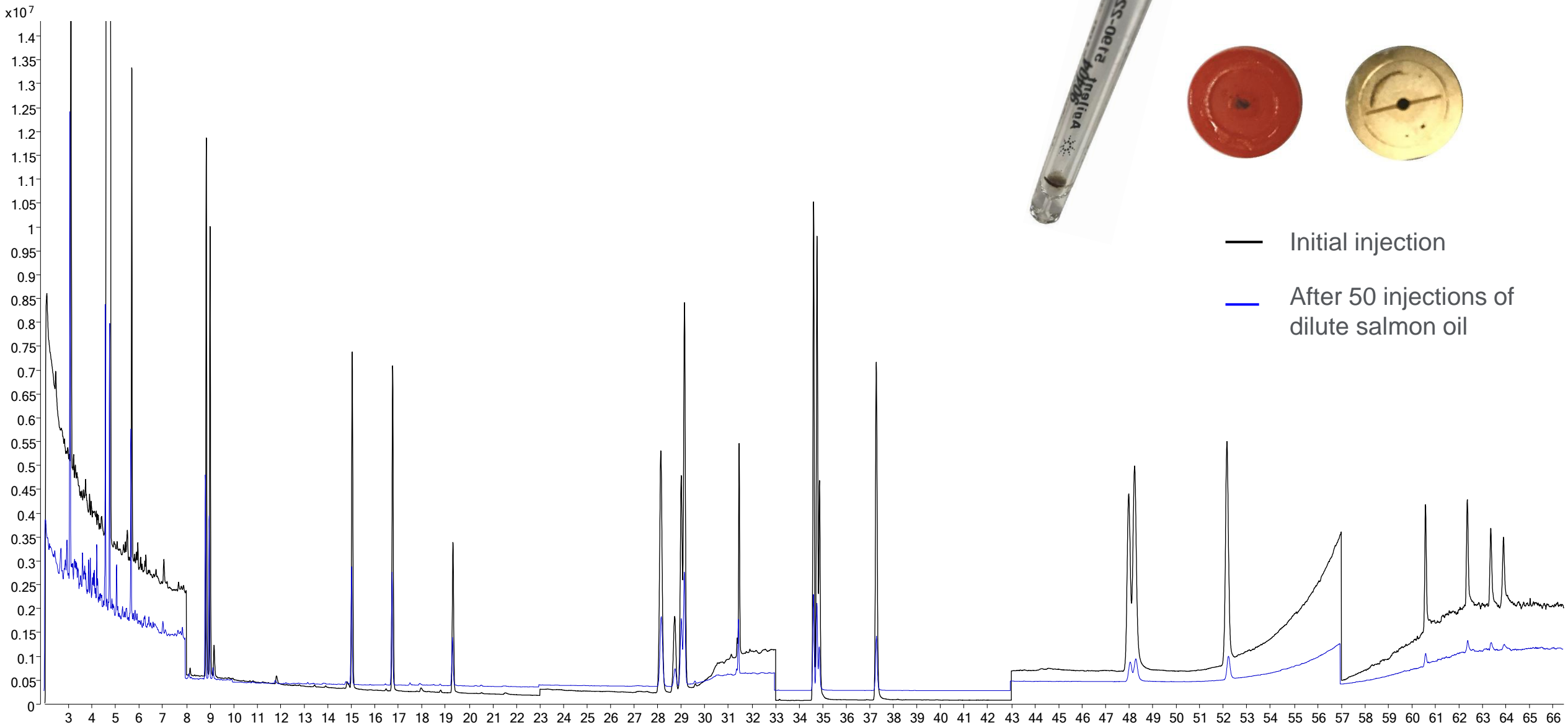
- Leaky syringe
- Split ratio set incorrectly
- Wrong purge activation time
- Septum purge flow too high
- Injector temperature too low*

Detector (response problem)

- Settings or flows have been changed
- Electronics failing

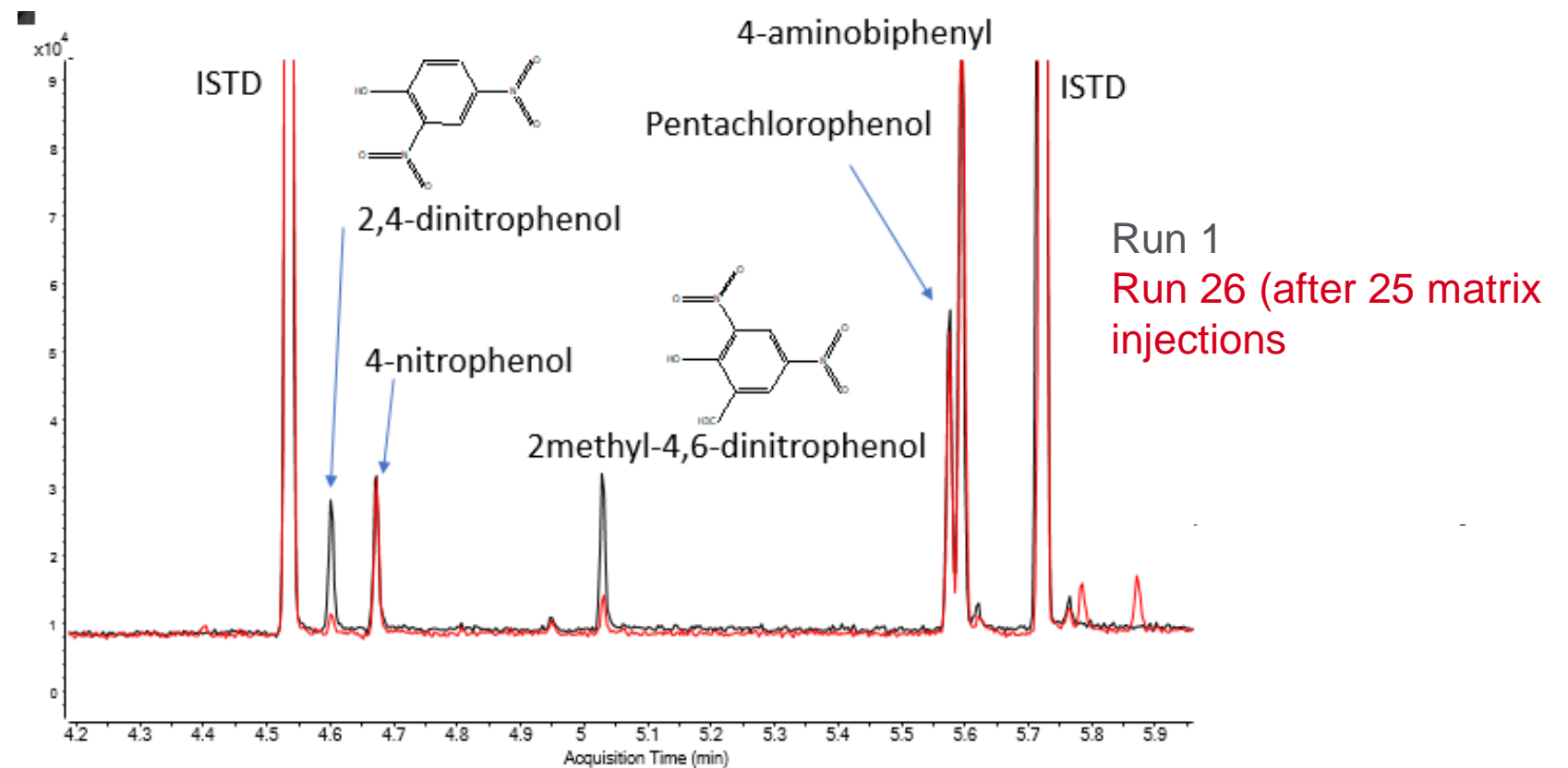
***Tip:** Ask, is it all of them or some of them? If it's all then it's a problem with the injector or detector.

50 ng/mL Before 50 Injections of Salmon Oil



Peak Response

Some change in size



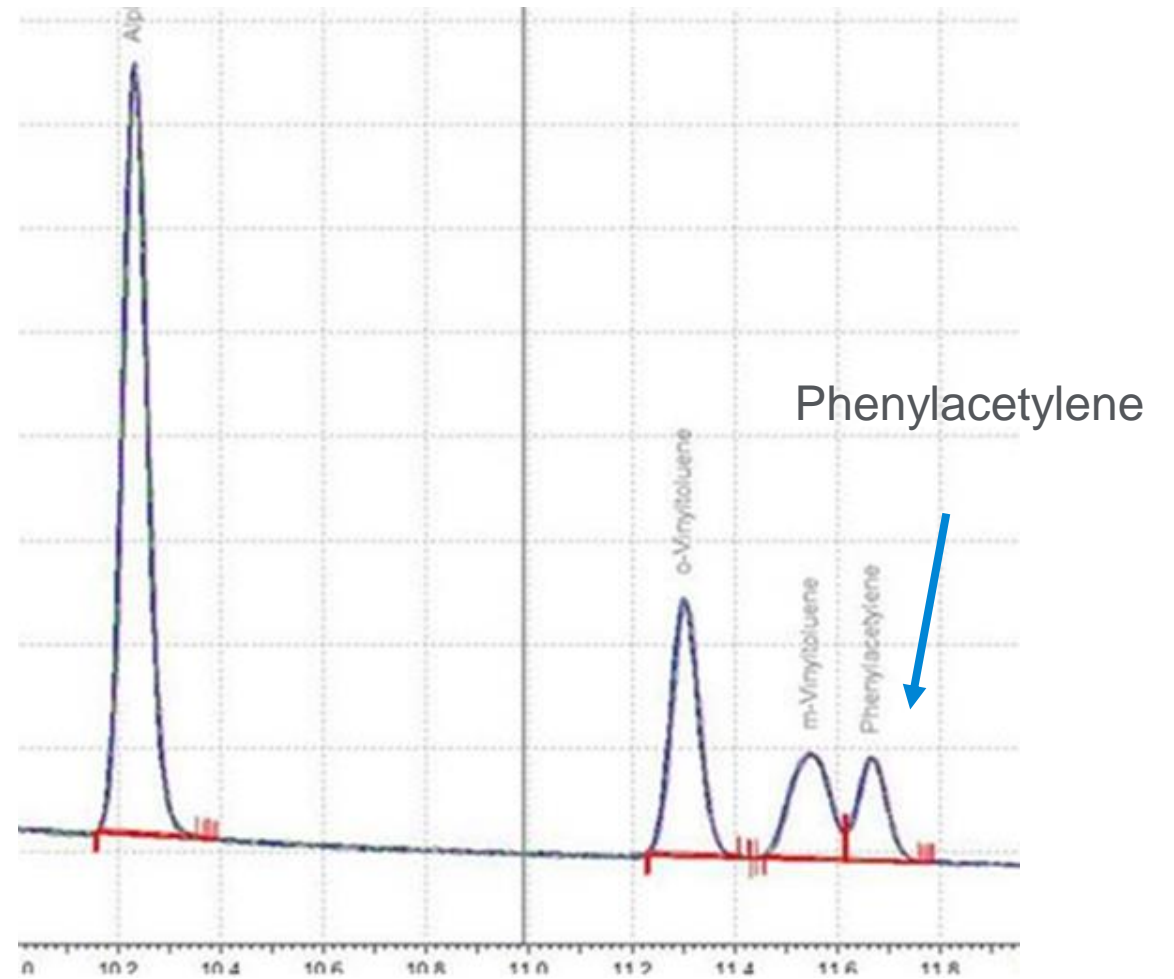
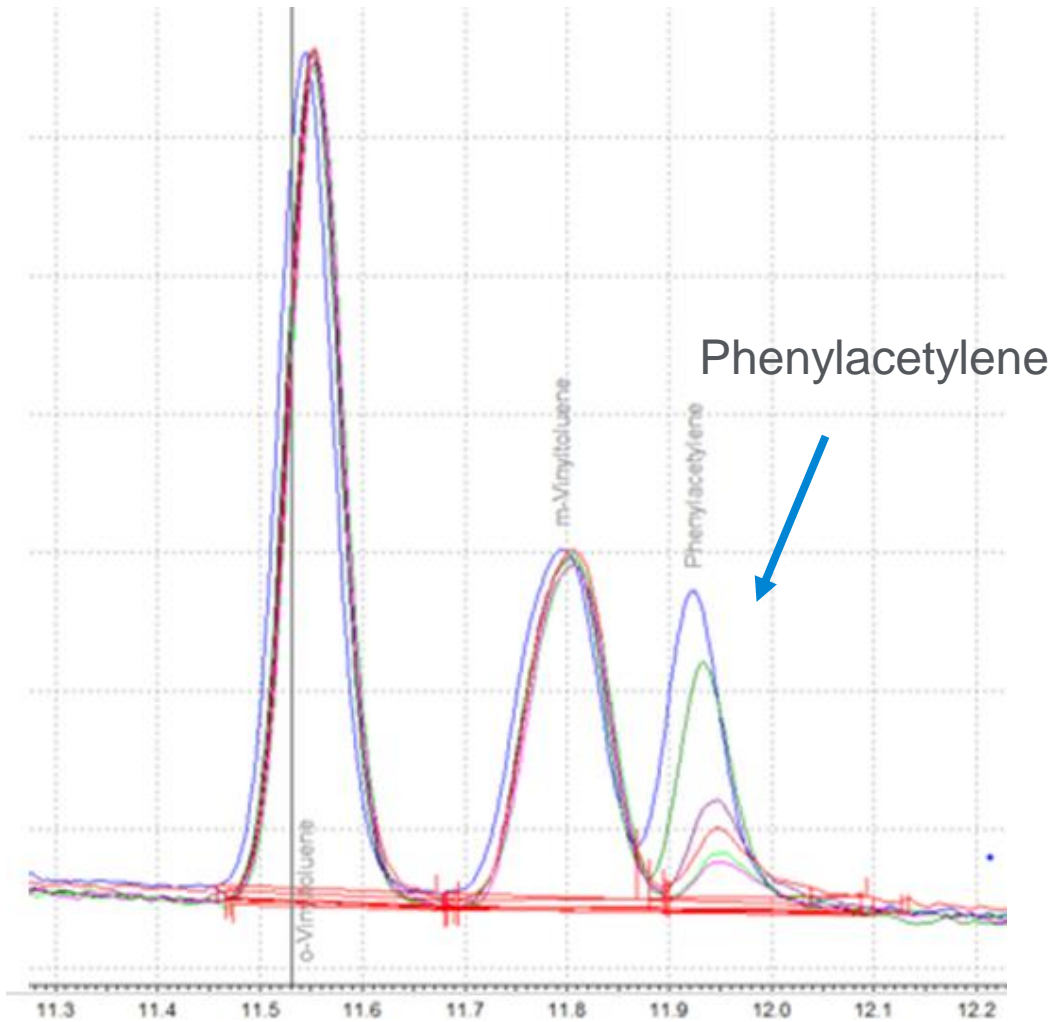
Injector or column is active/contaminated

- Irreversible adsorption of active compounds (-OH, -NH, -SH)

Decomposition of sample

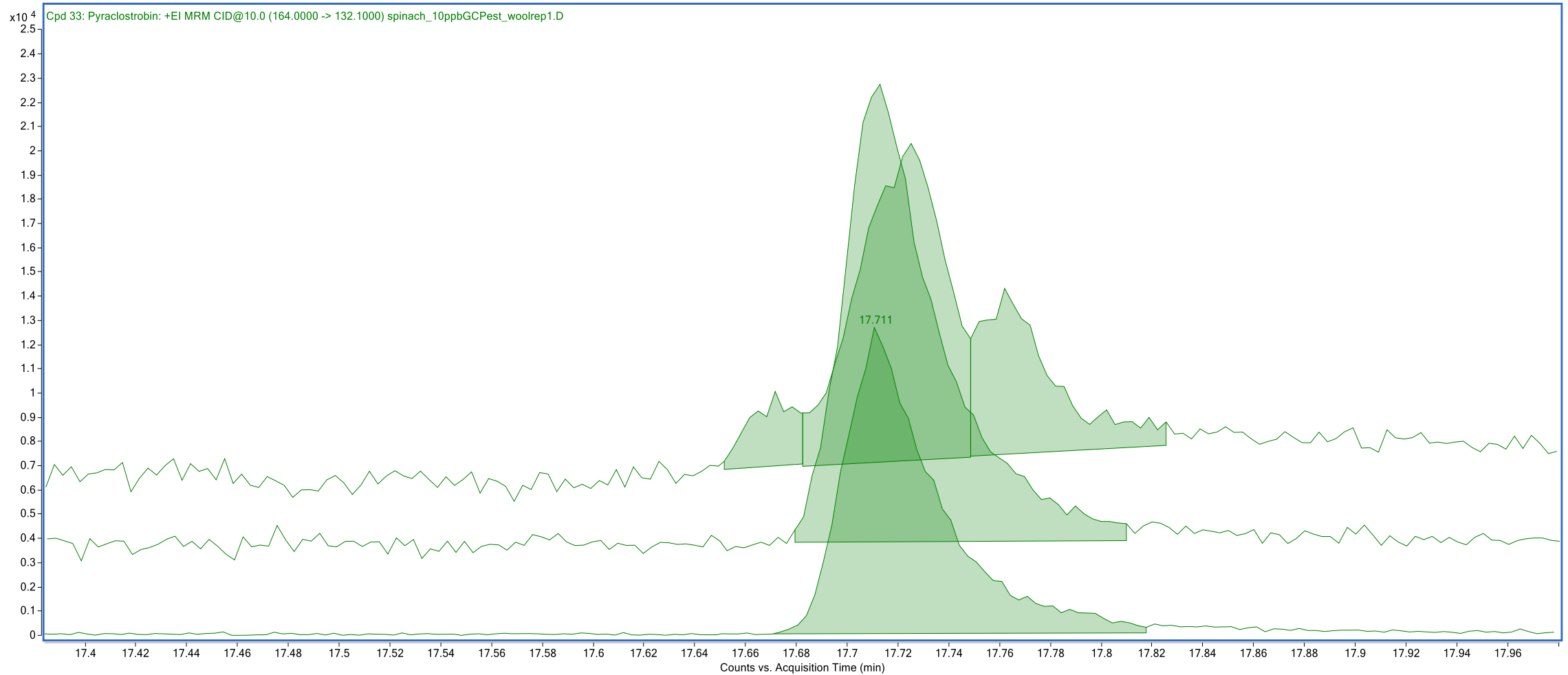
- Temperature change – discrimination
- Evaporation from sample

Example of Reduction in Response for One Peak



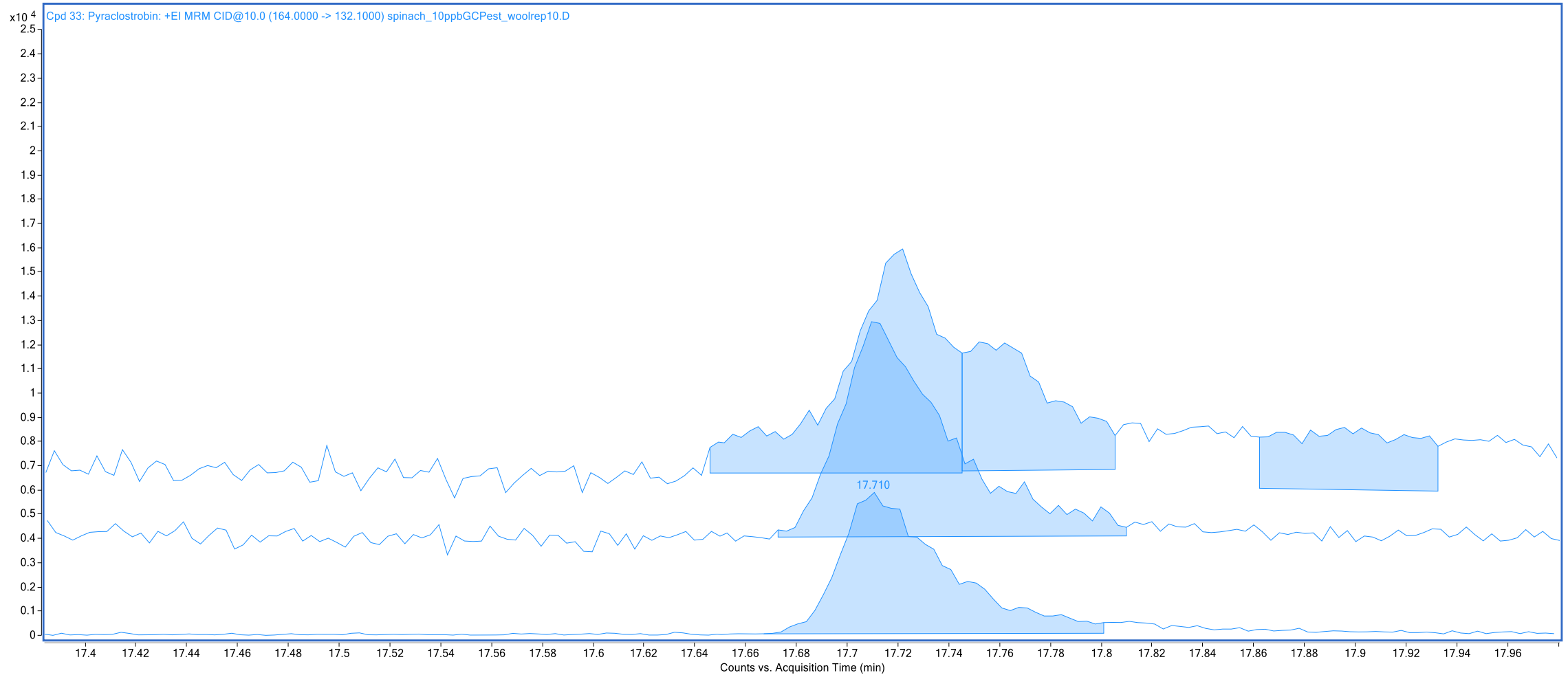
Change in Response: Pyraclostrobin in Spinach on Run 1

Run 1 can be seen in green



Change in Response: Pyraclostrobin in Spinach on Run 65

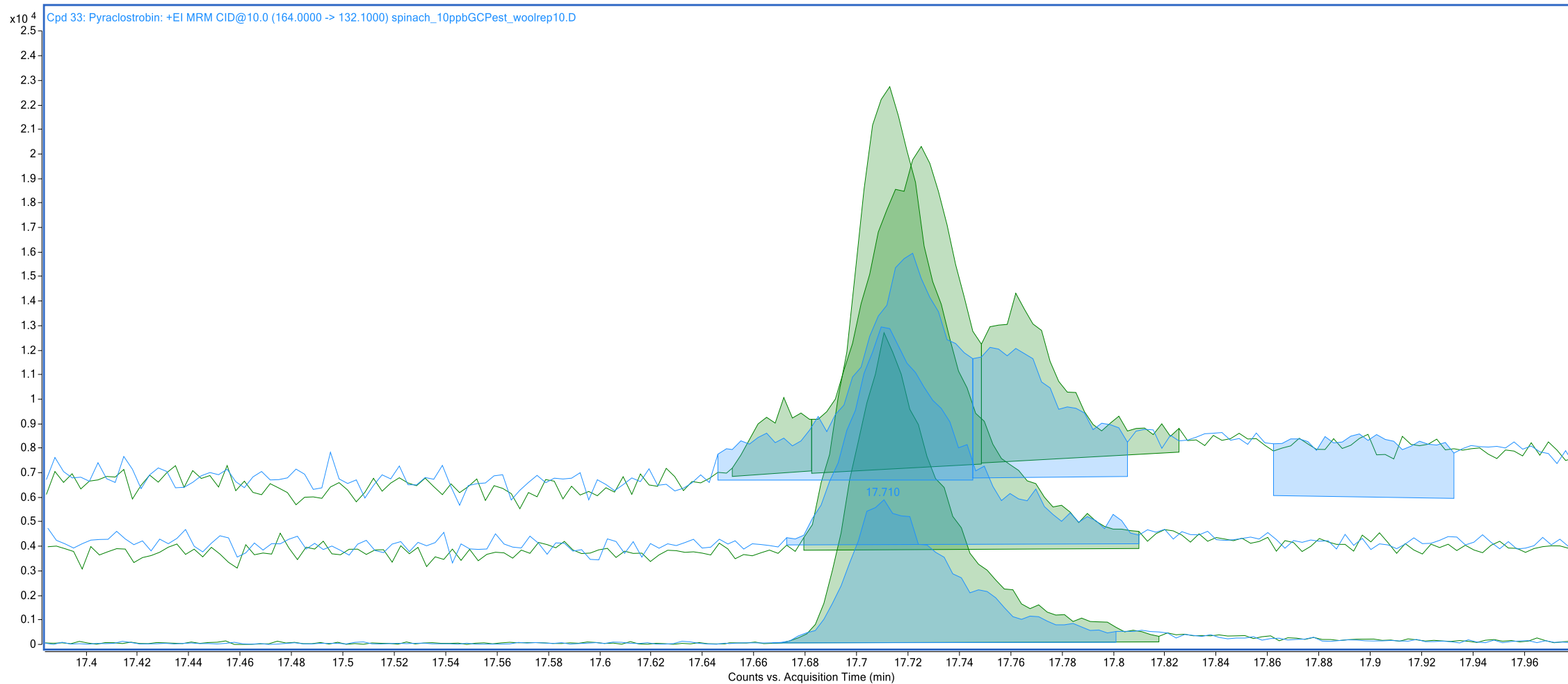
Run 65 can be seen in blue



Change in Response: Pyraclostrobin in Spinach on Run 1 vs Run 65

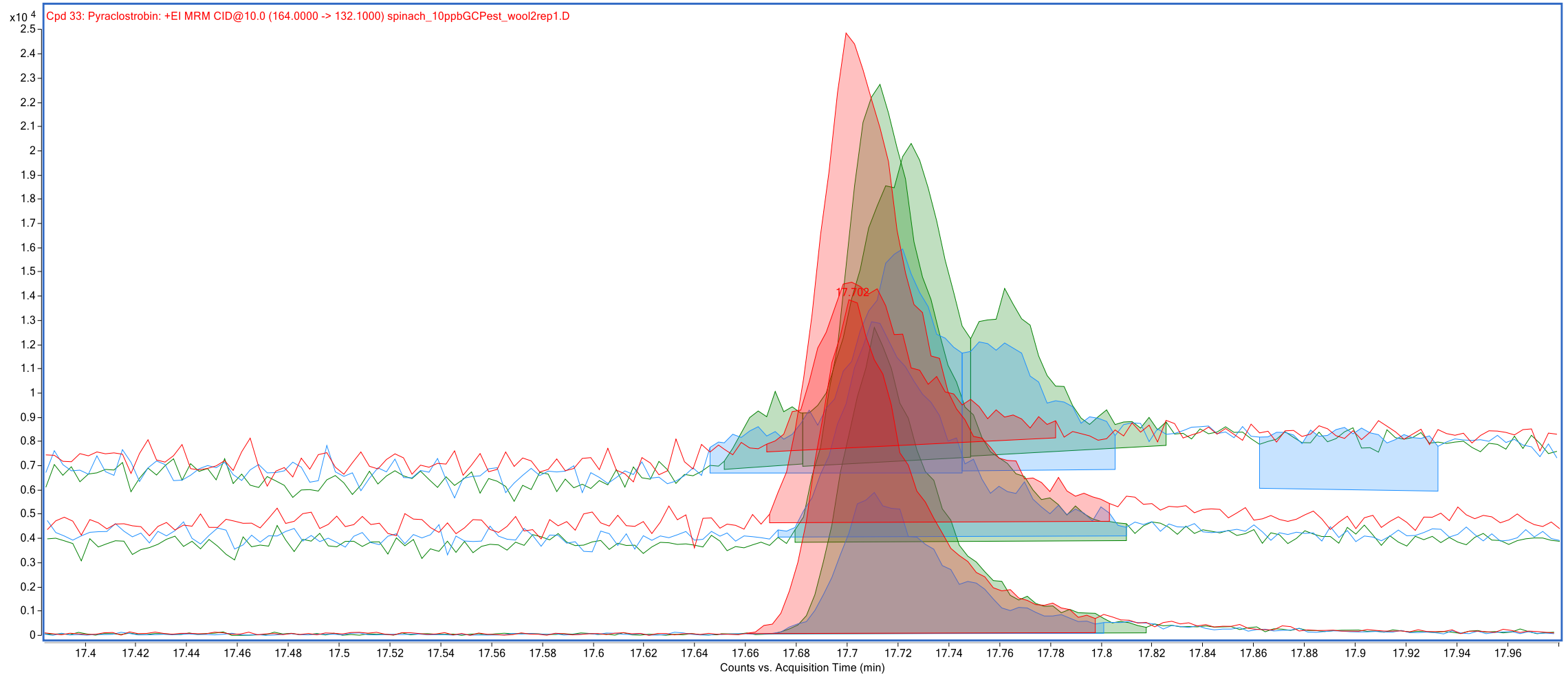
Run 1 can be seen in green

Run 65 can be seen in blue



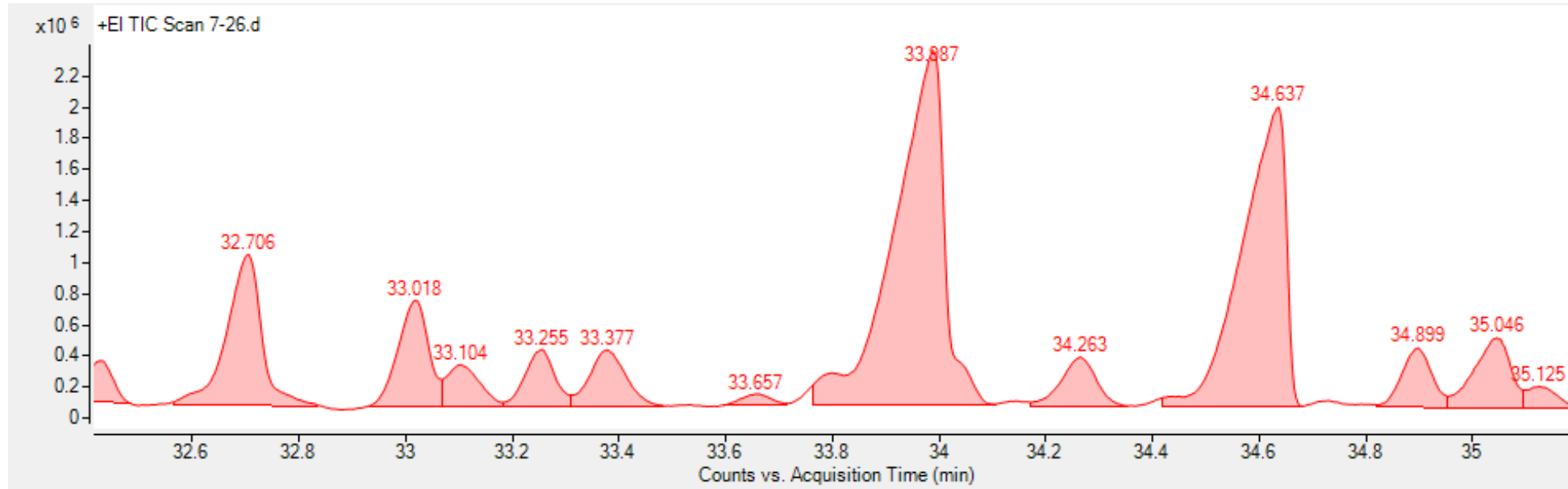
Change in Response: Pyraclostrobin in Spinach with New Liner

The new liner can be seen in **red**



Peak Fronting

Shark fin-shaped



Column (contaminated)

- Overload (more pronounced with large solute and phase polarity differences)

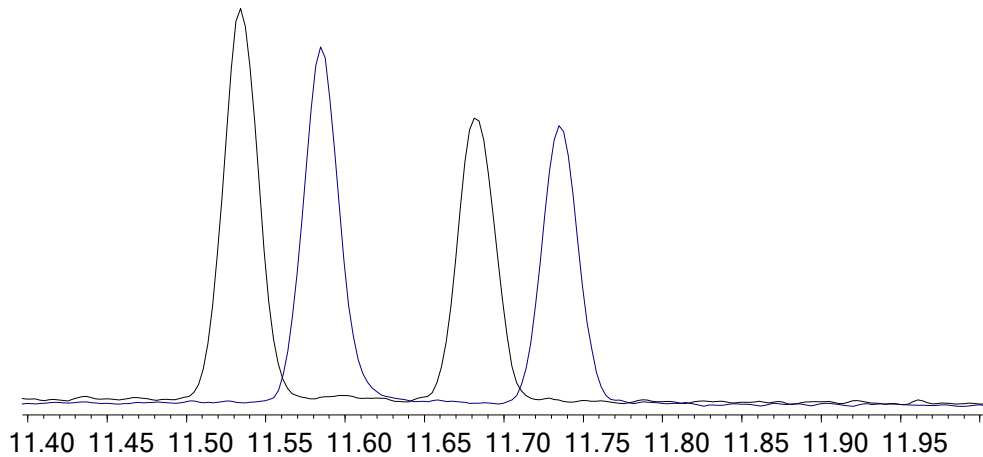
Injector

- Compound soluble in injection solvent (need retention gap)
- Mixed sample solvent

Other

- Coelution
- Breakdown

Retention Time Shift



Injector

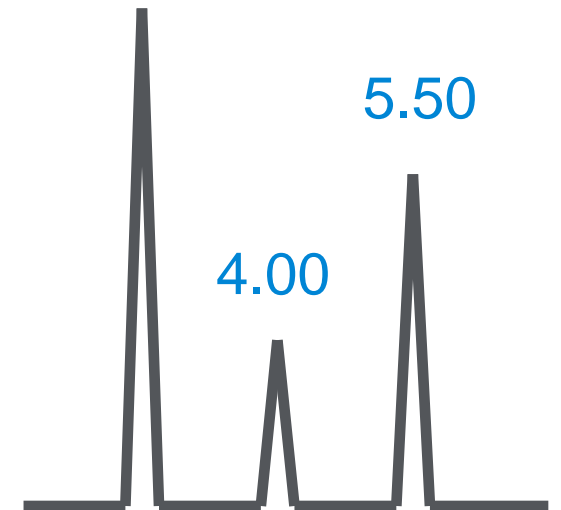
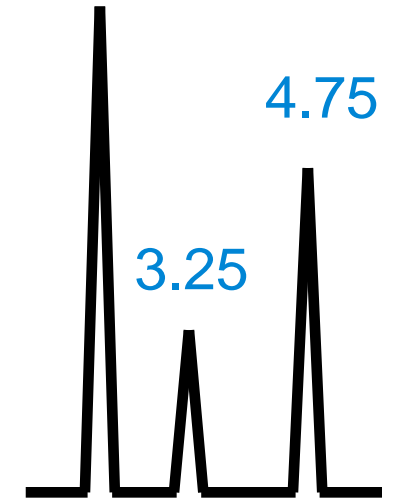
- Leak in the septum
- Change in injection solvent
- Large change in sample concentration

Flow

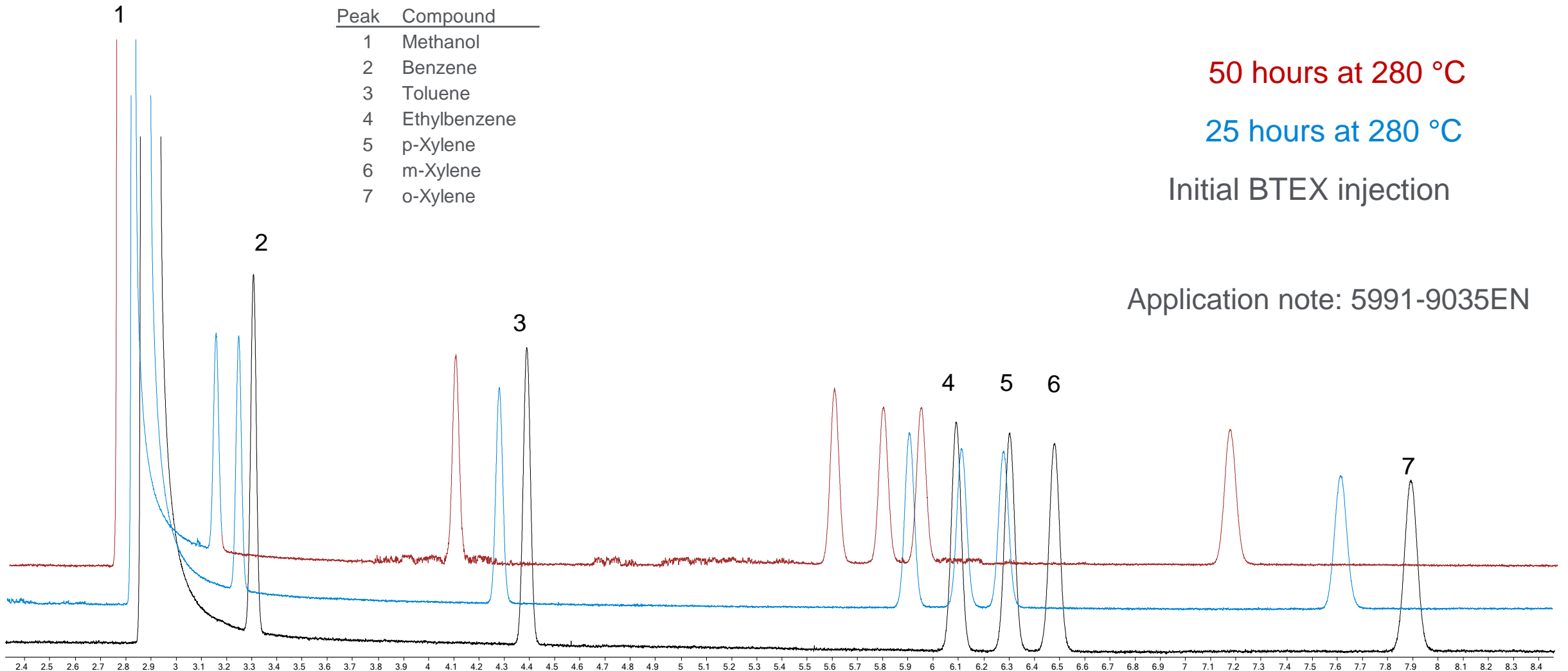
- Change in gas velocity

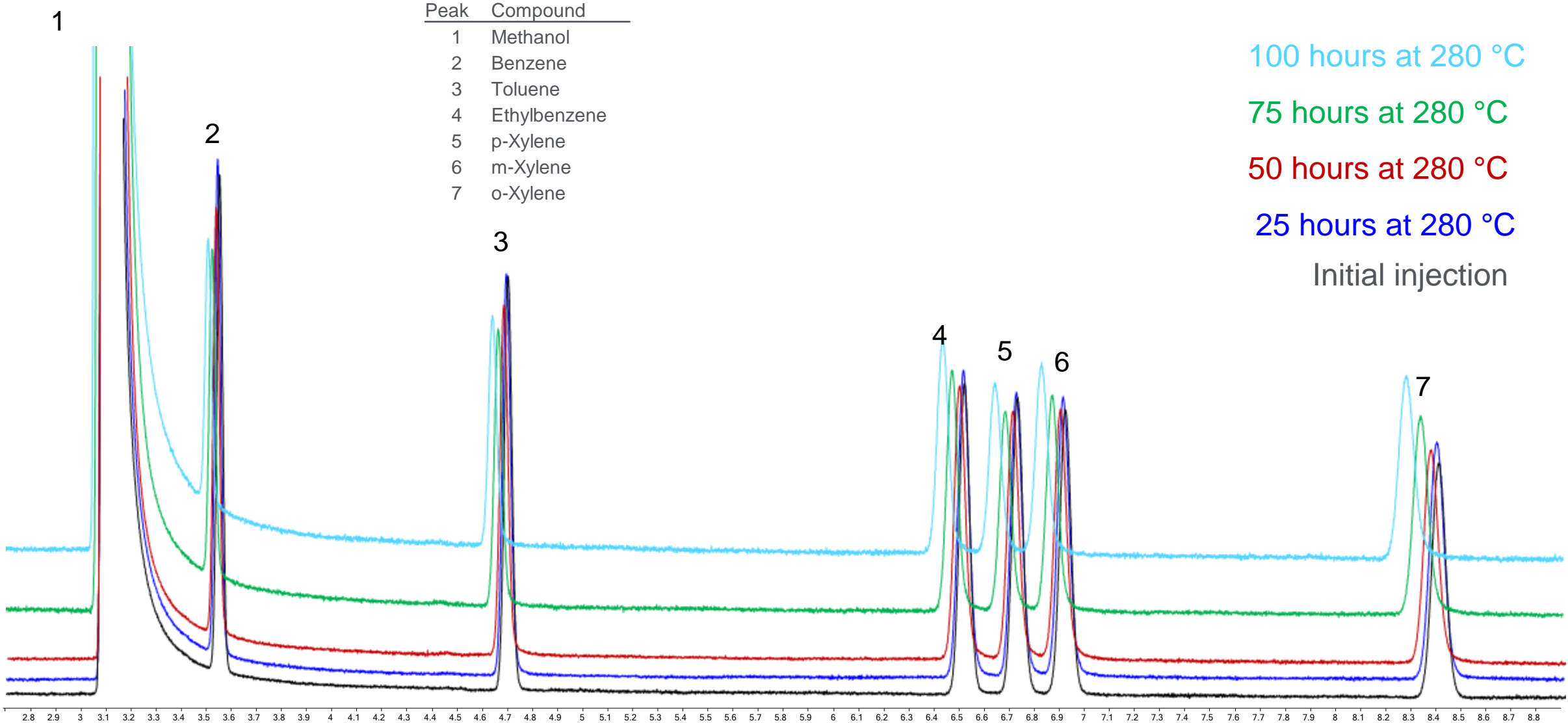
Column

- Contamination
- Damaged stationary phase
- Loss of stationary phase
- Change in temperature

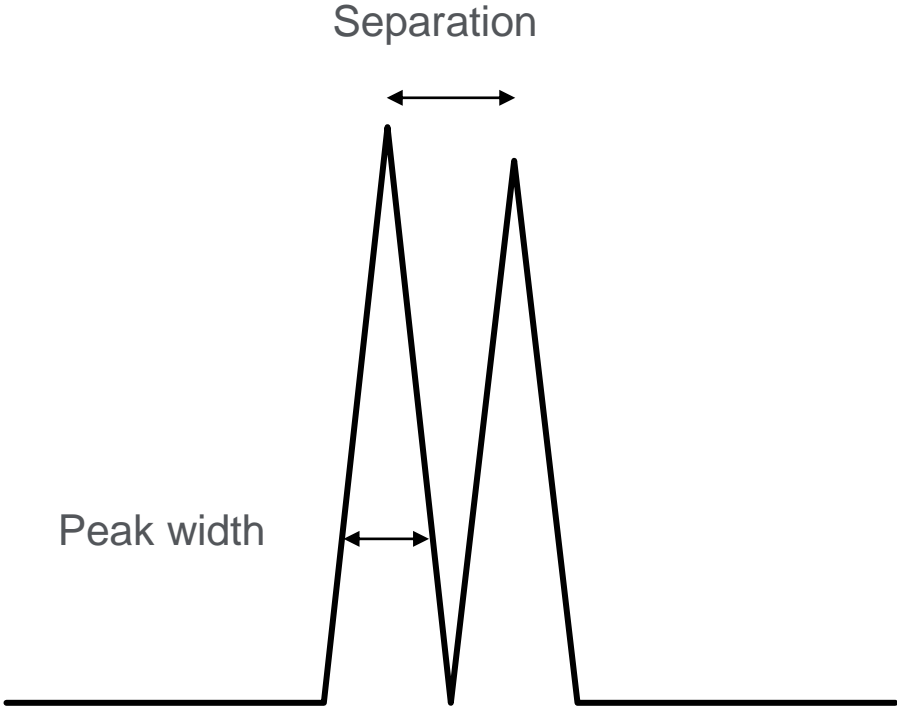


Thermal Stability and Retention Time Shifting on Standard WAX Column





Loss of Resolution



Resolution is a function of separation and peak width

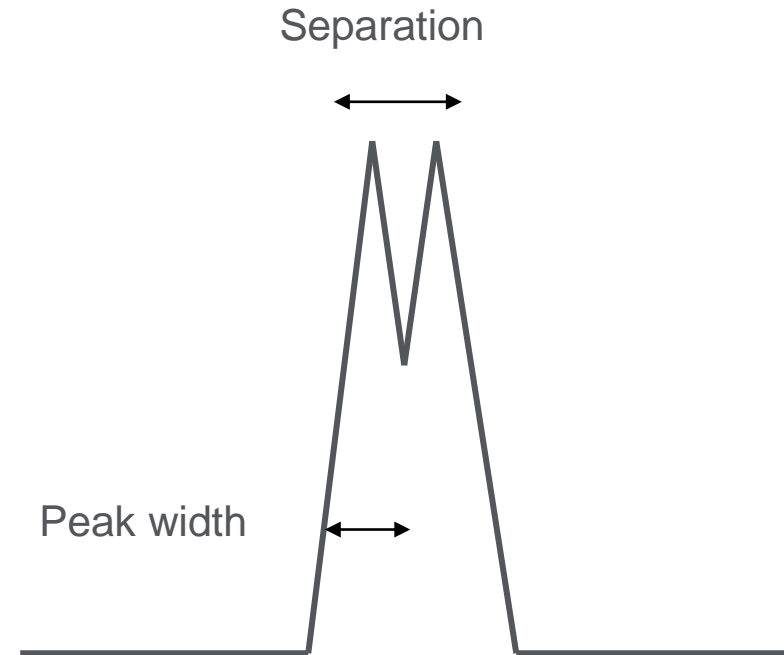
Loss of Resolution – Separation Decrease (Retention Times Changed)

Column

- Different column temperature
- Contamination (more phases?)
- Matrix components coeluting

Flow

- Change in velocity?



Loss of Resolution - Peak Broadening (Retention Times Unchanged)

Flow

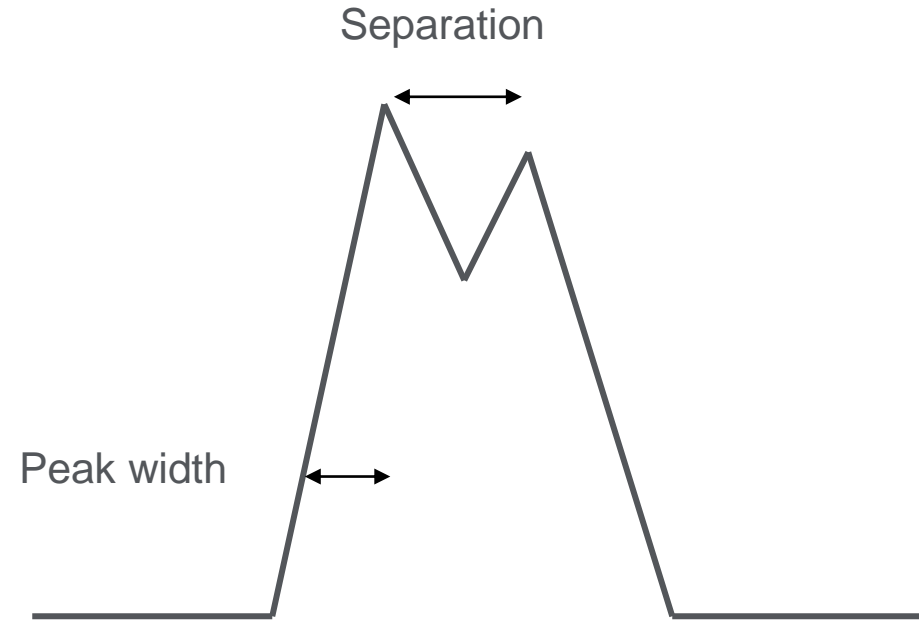
- Makeup gas

Column

- Contamination
- Phase degradation

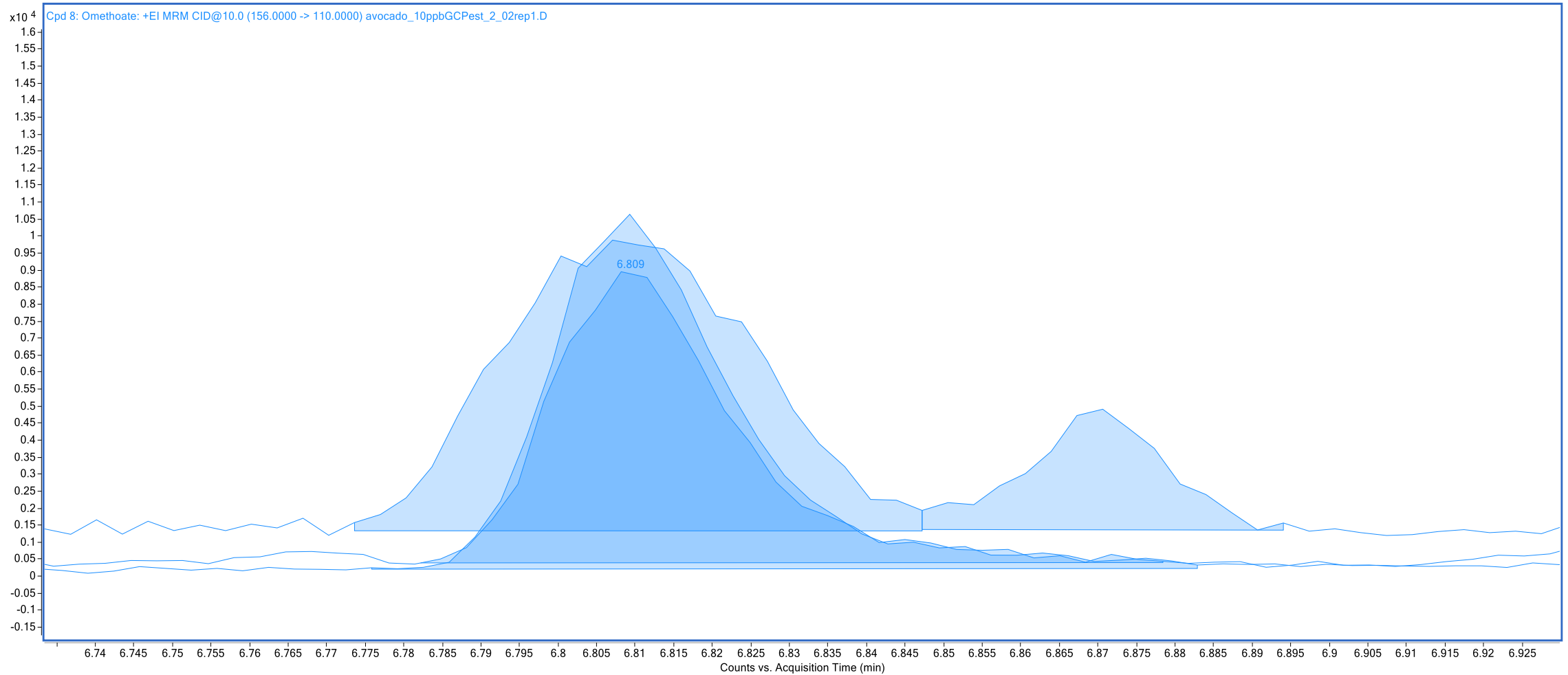
Injector (efficiency)

- Settings, liner, installation



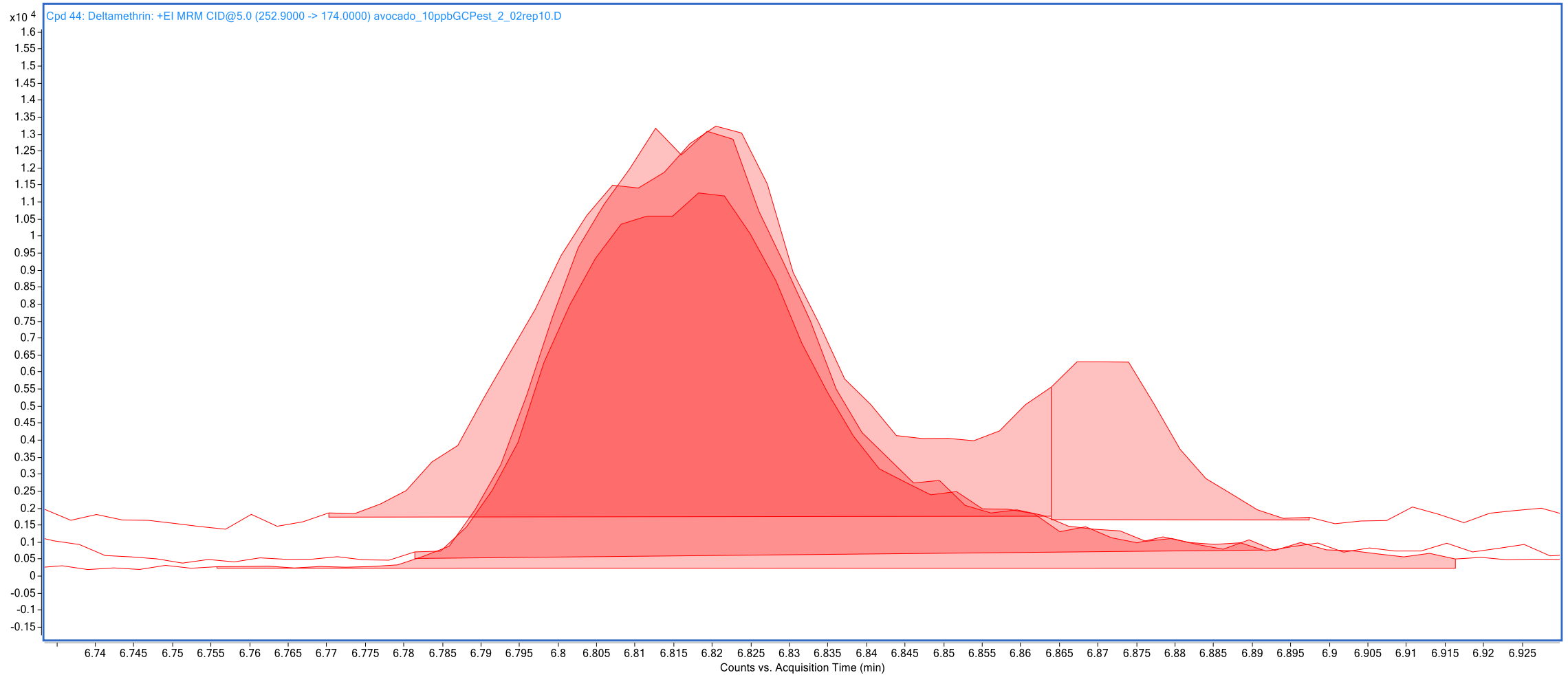
Peak Broadening: Omethoate in Avocado in Run 1

Run 1 can be seen in blue



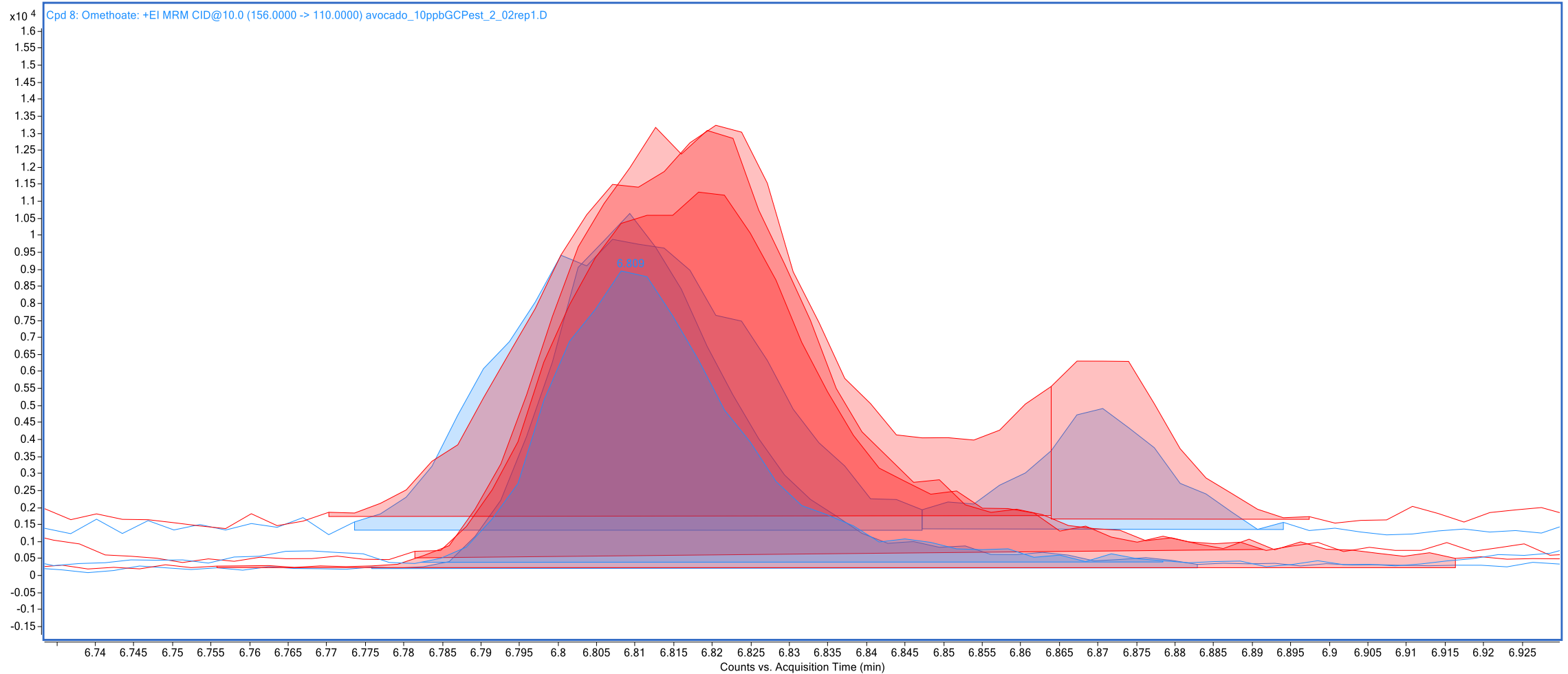
Peak Broadening: Omethoate in Avocado in Run 65

Run 65 can be seen in red



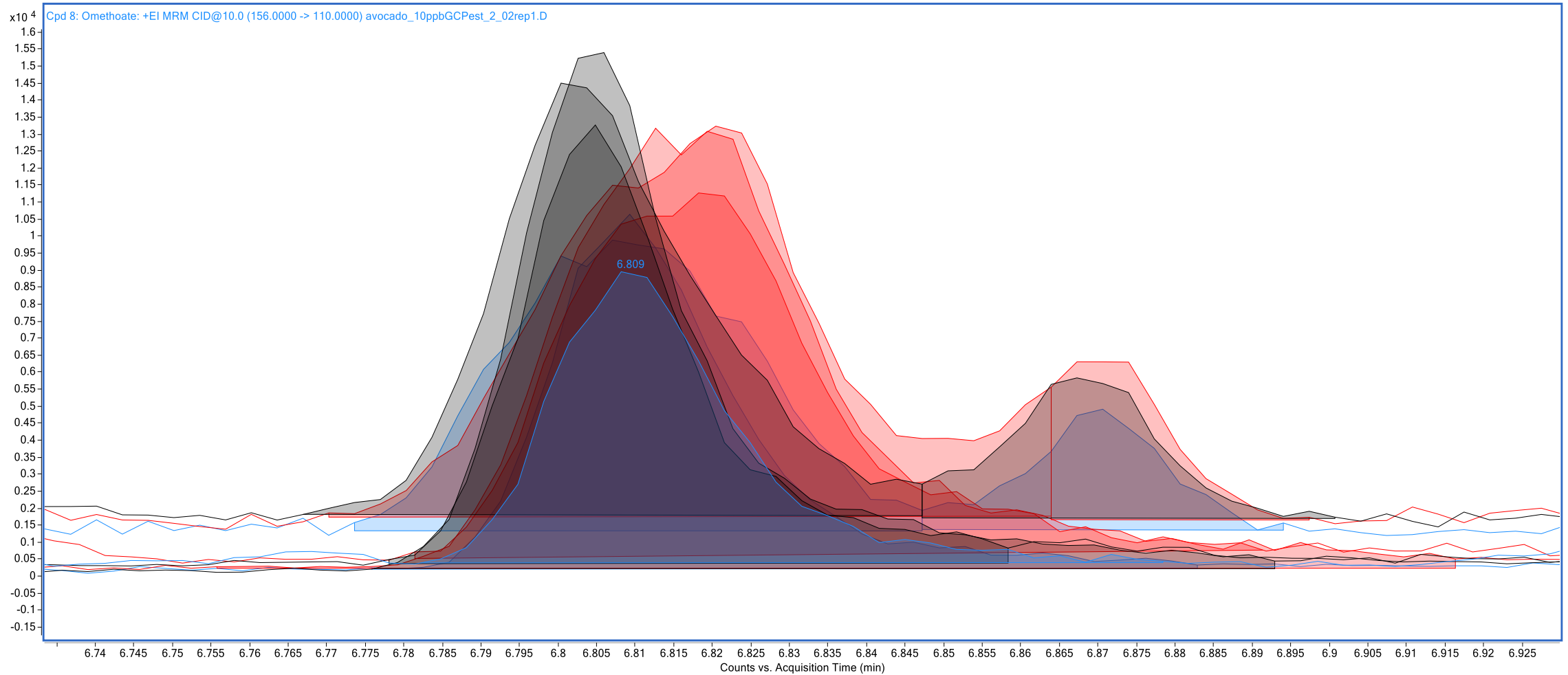
Peak Broadening: Omethoate in Avocado in Run 1 versus Run 65

Run 1 can be seen in blue
Run 65 can be seen in red

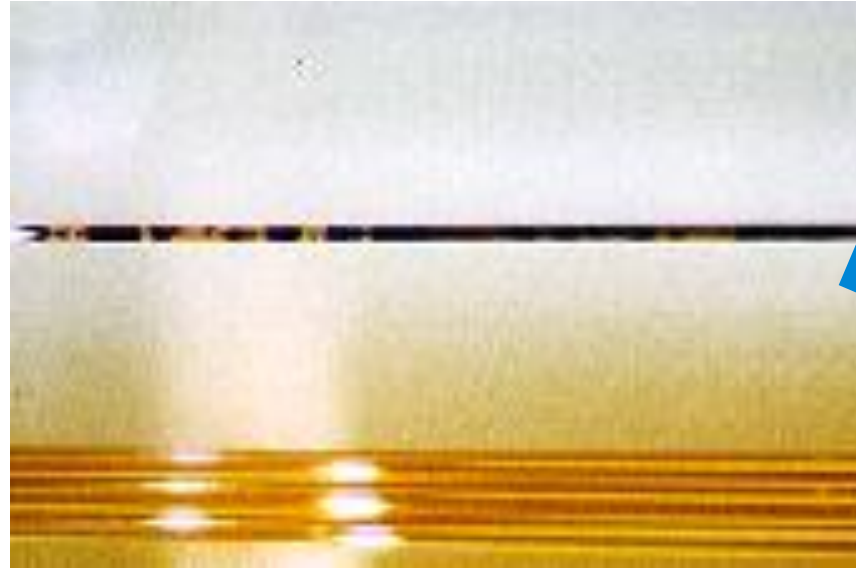
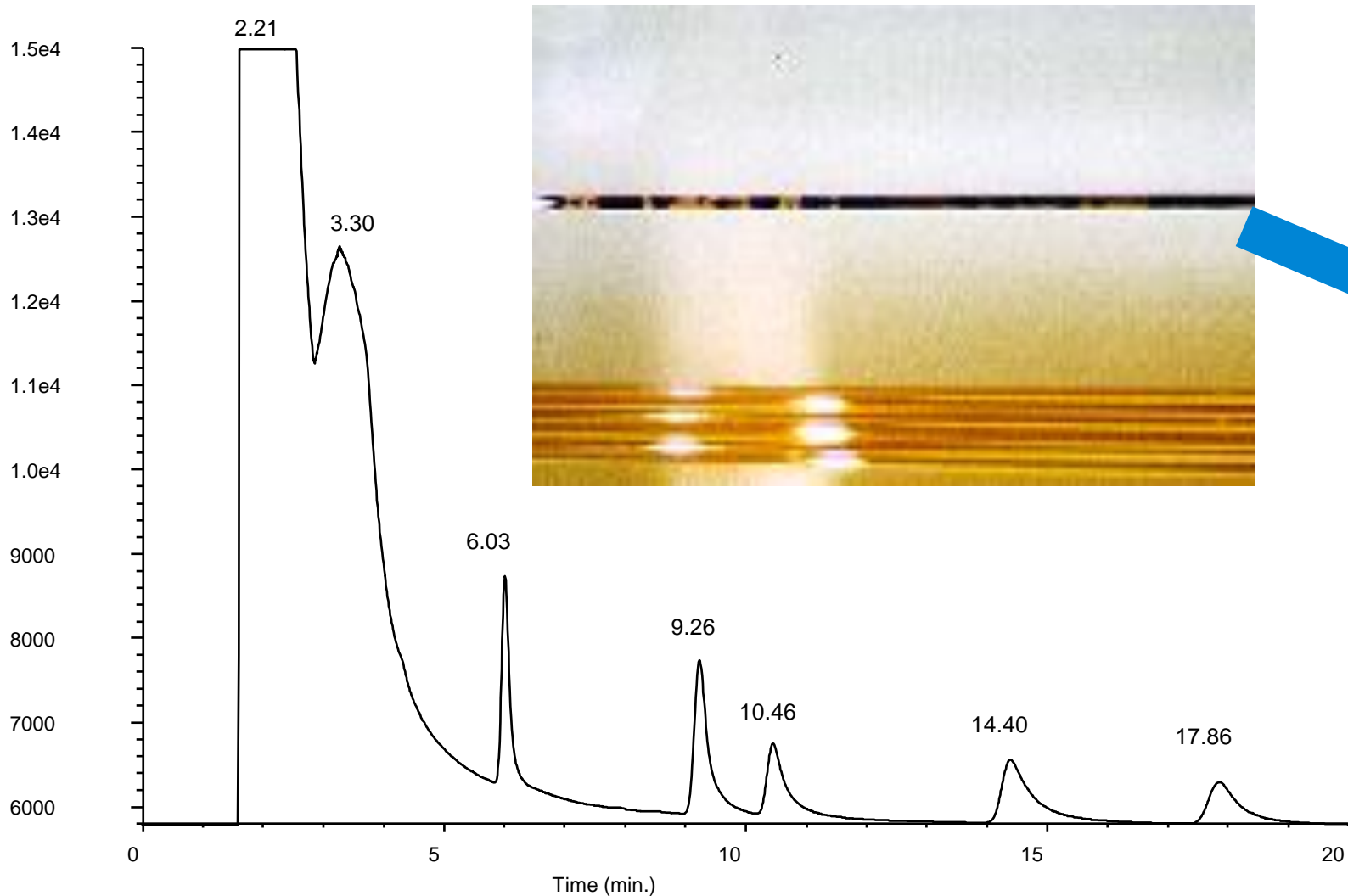


Peak Broadening: Recover Peak Shape with New Liner

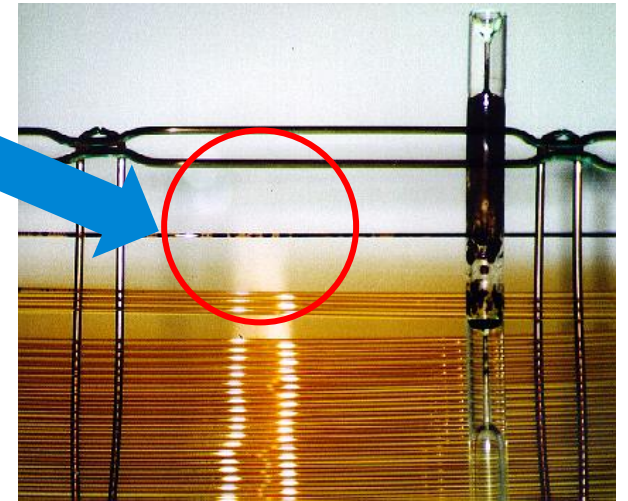
The new liner can be seen in black



Example of Column Contamination and Broad Peaks

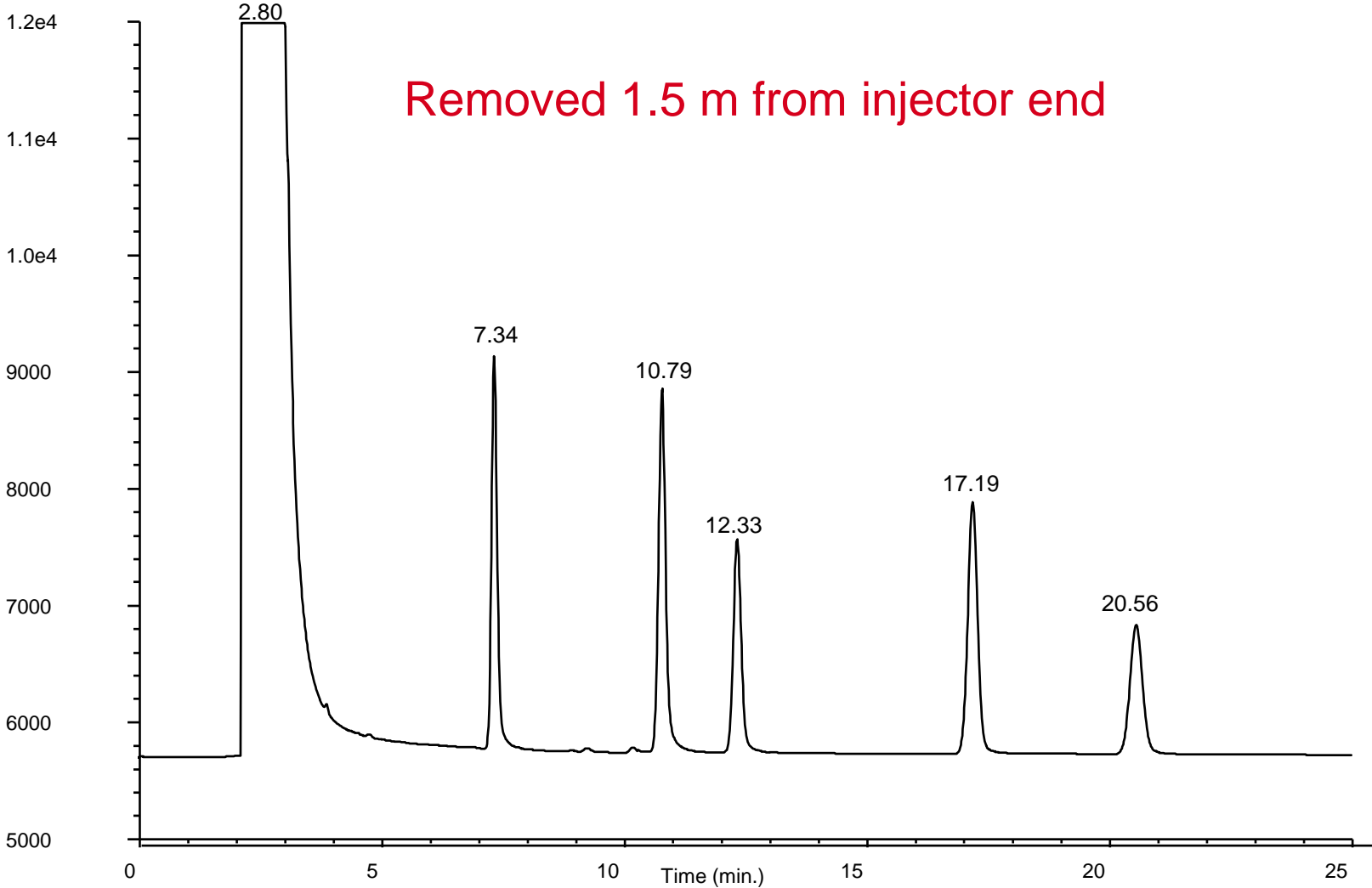


Agilent J&W DB-624 QC test mix
After 75 injections of oily sample



*Temperature program // 35 °C hold 1.50 min // 30 °C/min to 65 °C, hold 10 min

Example of Column Contamination

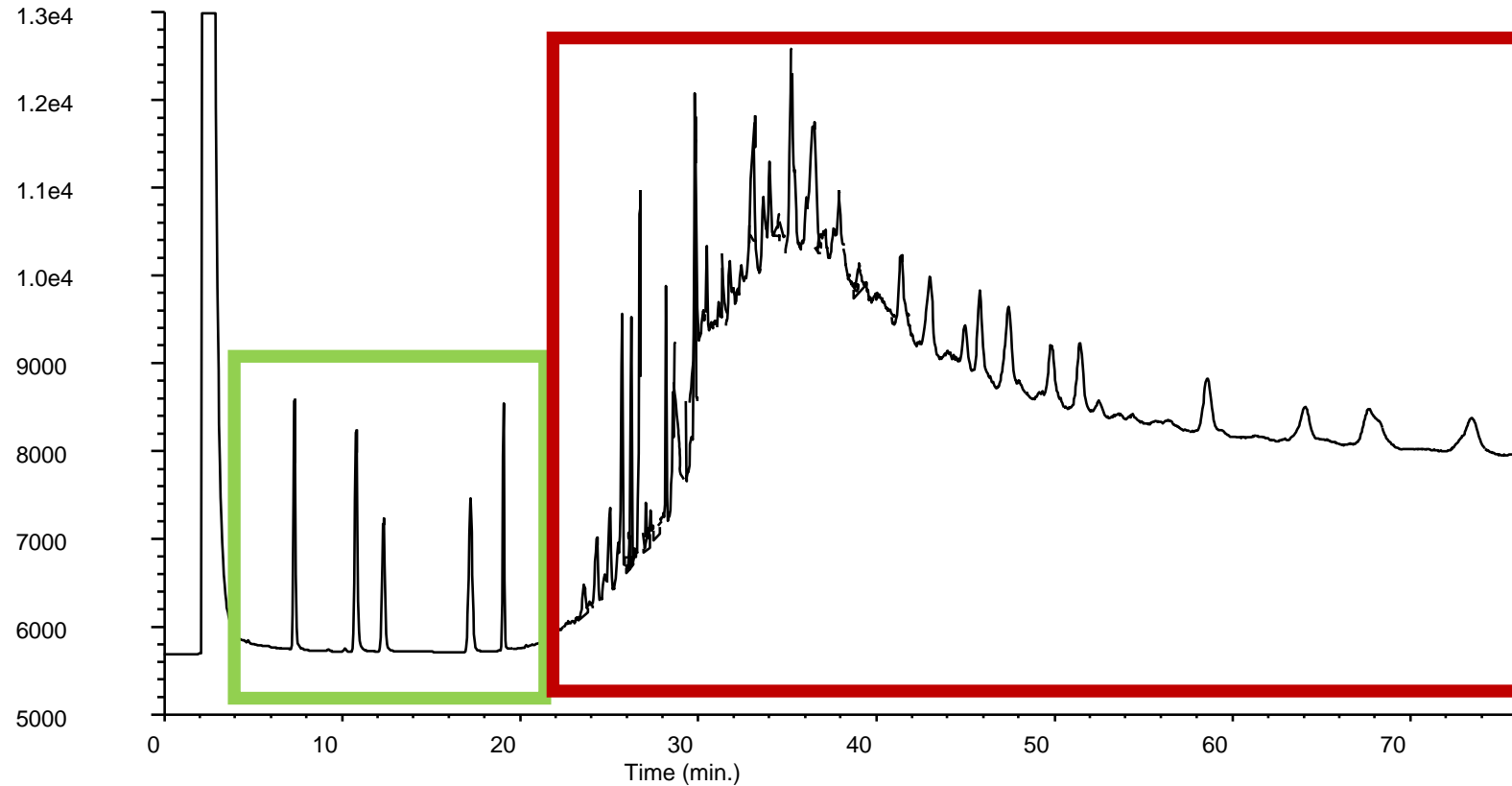


*Before column rinse and bake
Temperature program // 35 °C hold 1.50 min // 30° C/min to 65 °C, hold 10 min

Example of Column Contamination

1.5 m removed*

QC test mix to upper temperature limit



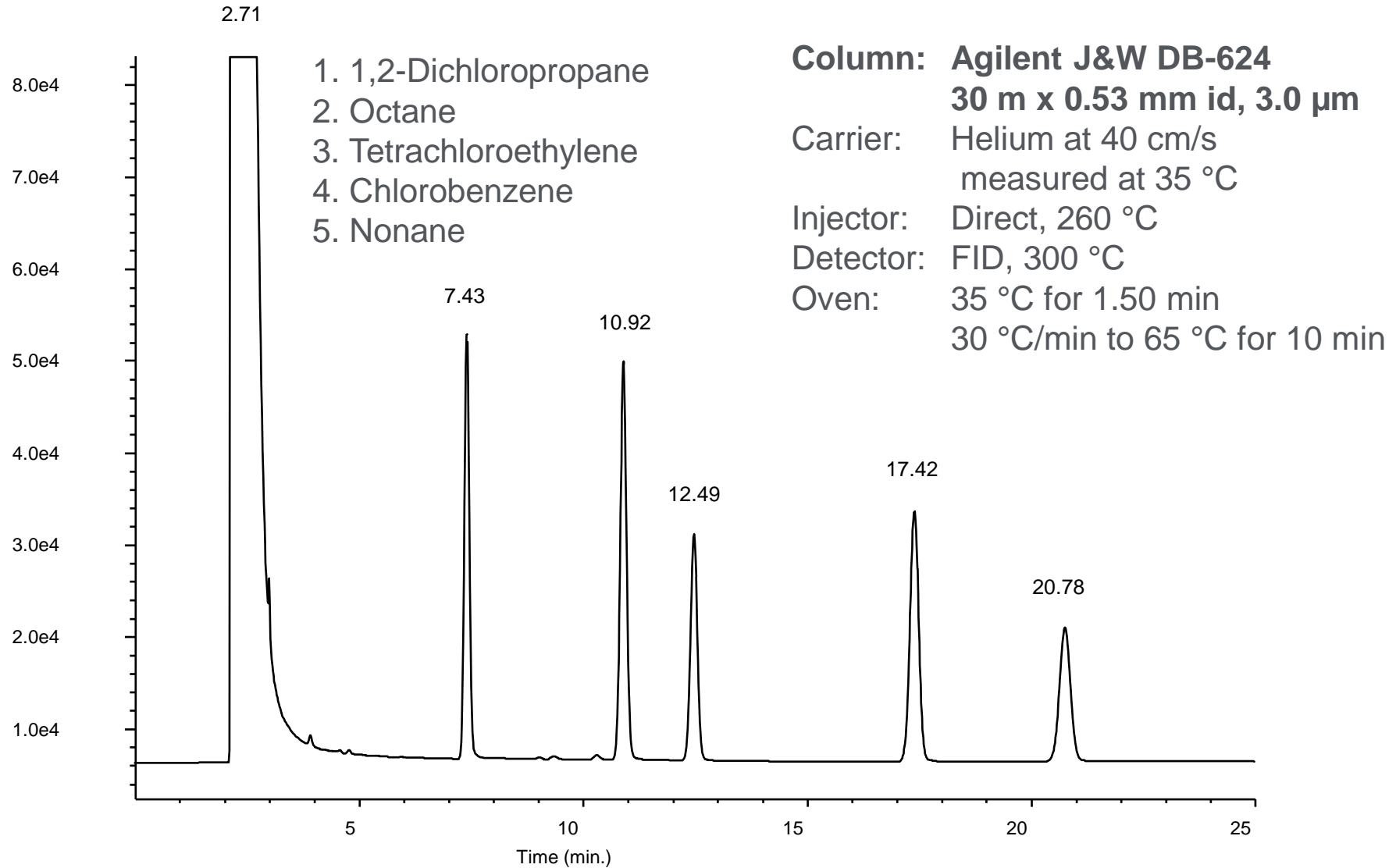
We have more semivolatile contamination

*Before column bake

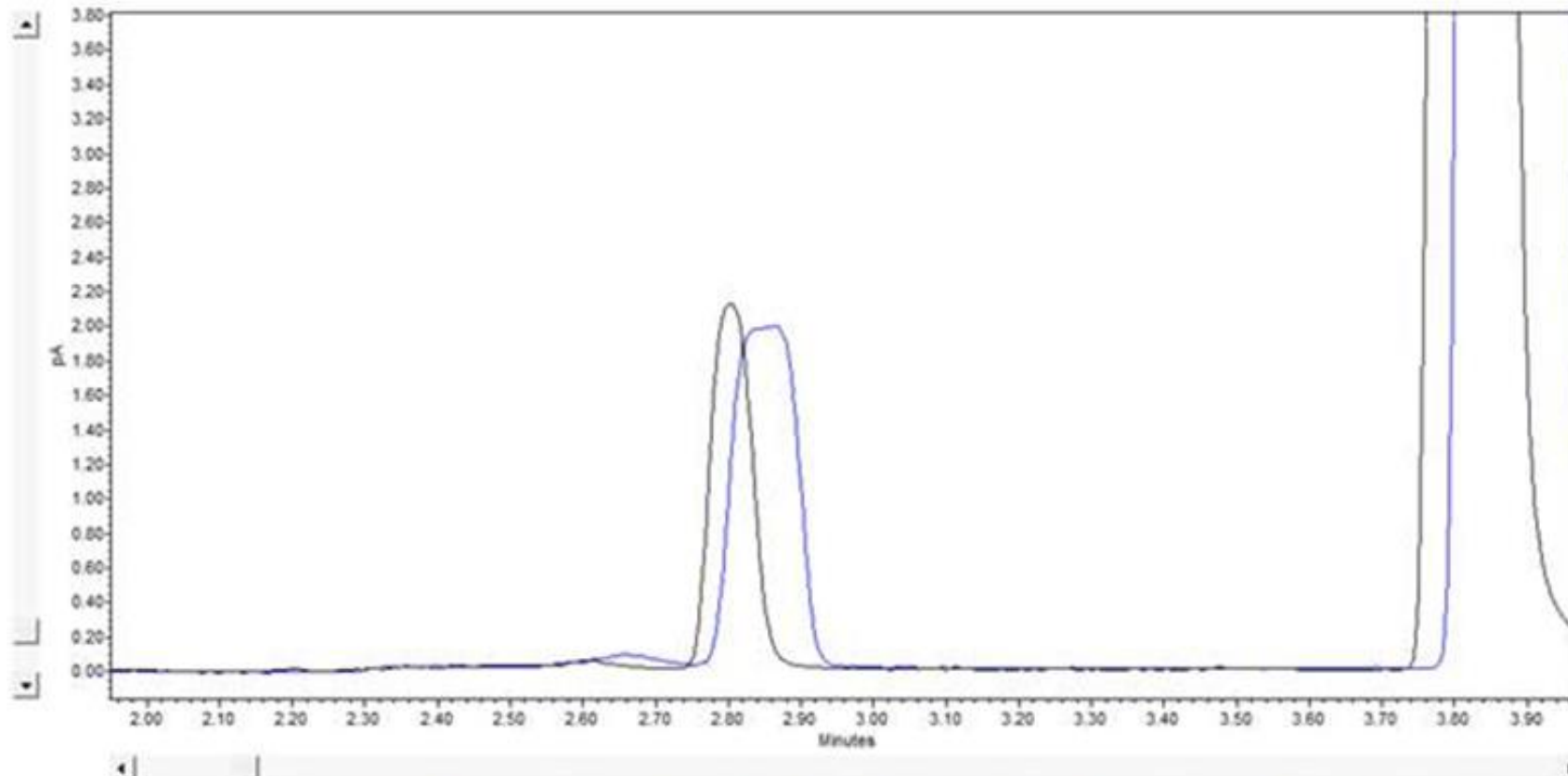
Temperature program // 35 °C, hold 1.50 min // 30 °C/min to 65 °C, hold 15 min // 20 °C/min to 260 °C, hold 50 min

Agilent J&W DB-624 Column

QC test mix



Changing to a Higher Split Ratio Improves Peak Sharpness



5:1 split ratio

10:1 split ratio

Baseline Disturbances

Sudden changes, wandering, or drifting

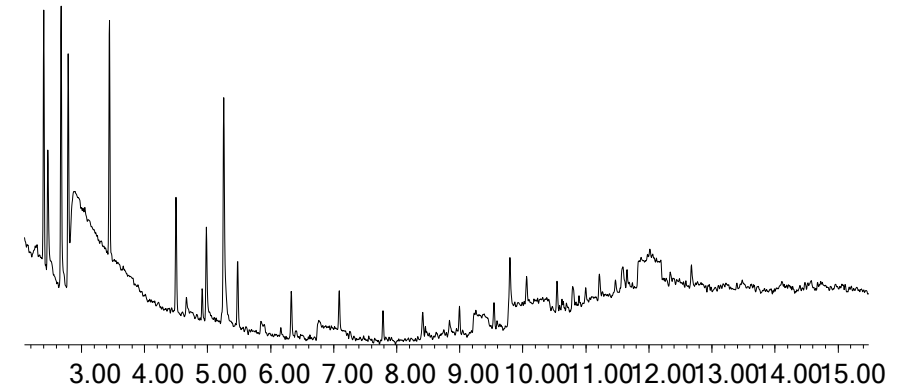
Drifting/wandering/unusual disturbances

Column or detector

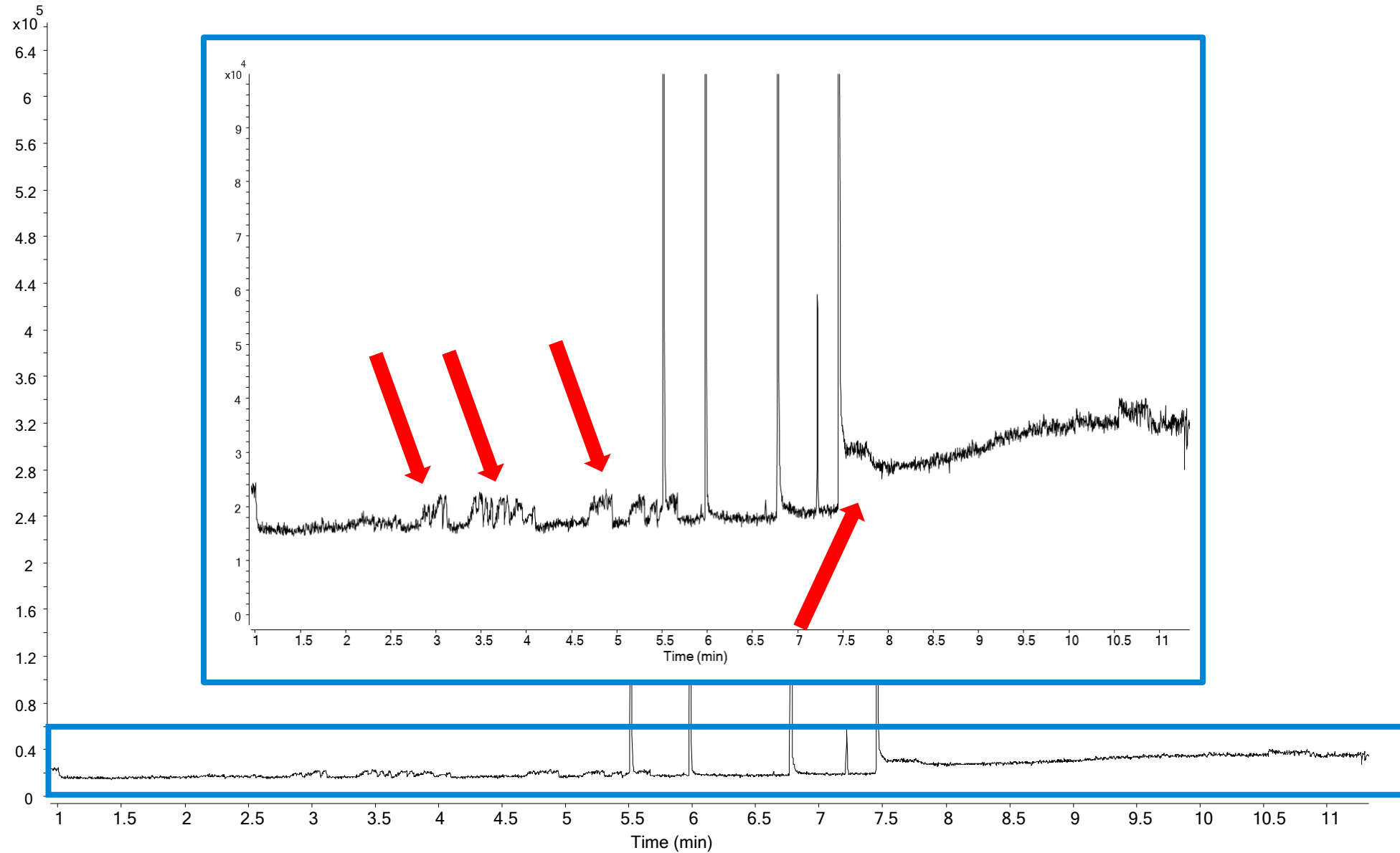
- Not fully conditioned or stabilized (electronics)
- Contamination

Flow

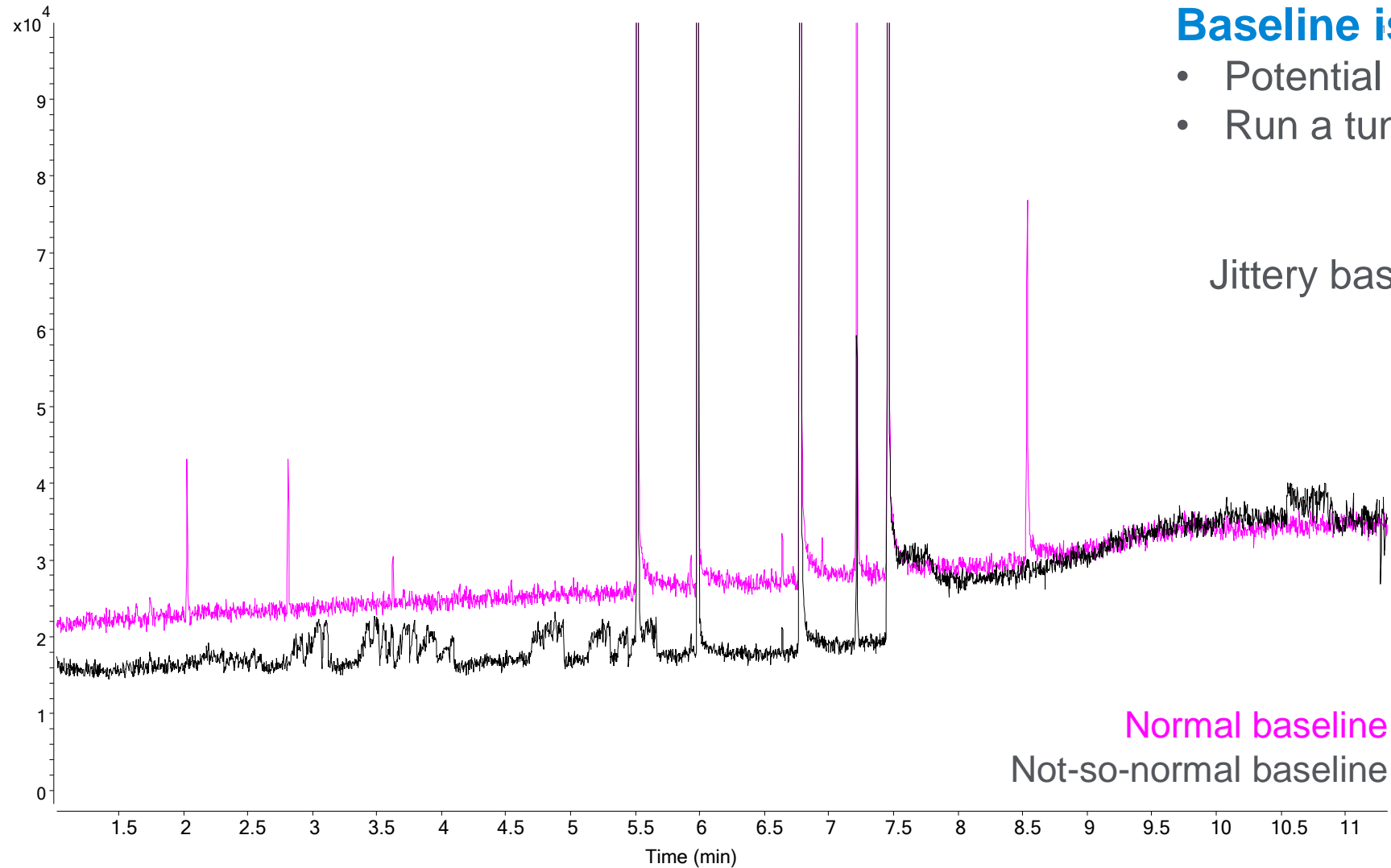
- Changes in carrier or detector gas flows
- Valves switching, leaks



Jittery Baseline Example



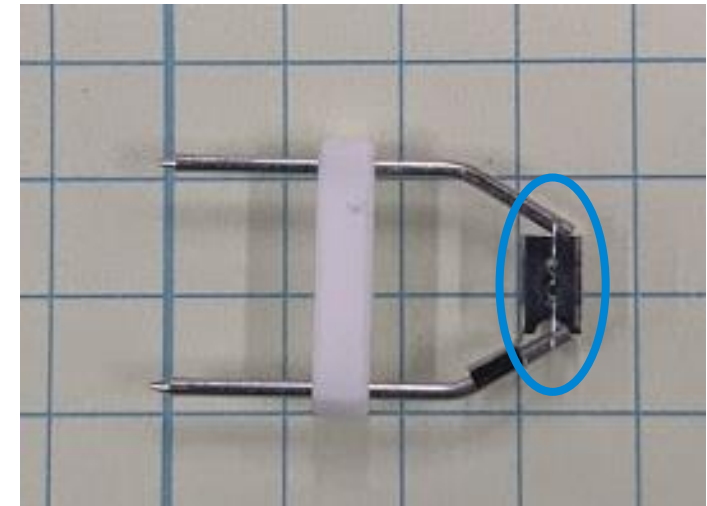
Jittery Baseline Example



Baseline is not normal

- Potential issues with filaments
- Run a tune evaluation

Jittery baseline + failed tune = check your filaments



Noisy Baseline

Mild



Severe



Flow

- Contaminated gas
- Incorrect detector settings

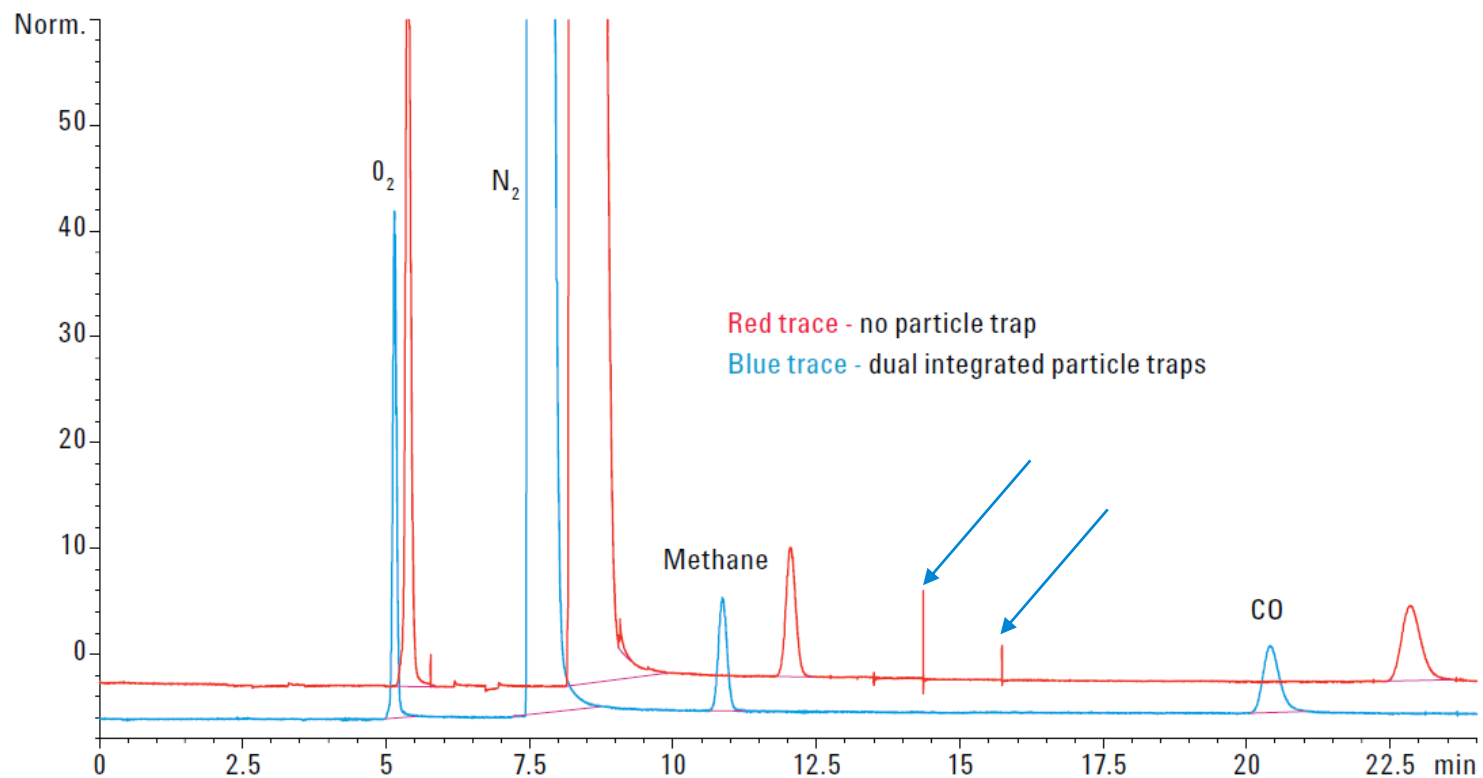
Detector

- Air leak - ECD, TCD
- Electronics malfunction

Column

- Bleed if at high temperature
- In detector flame (poor installation)

Spiking Baseline



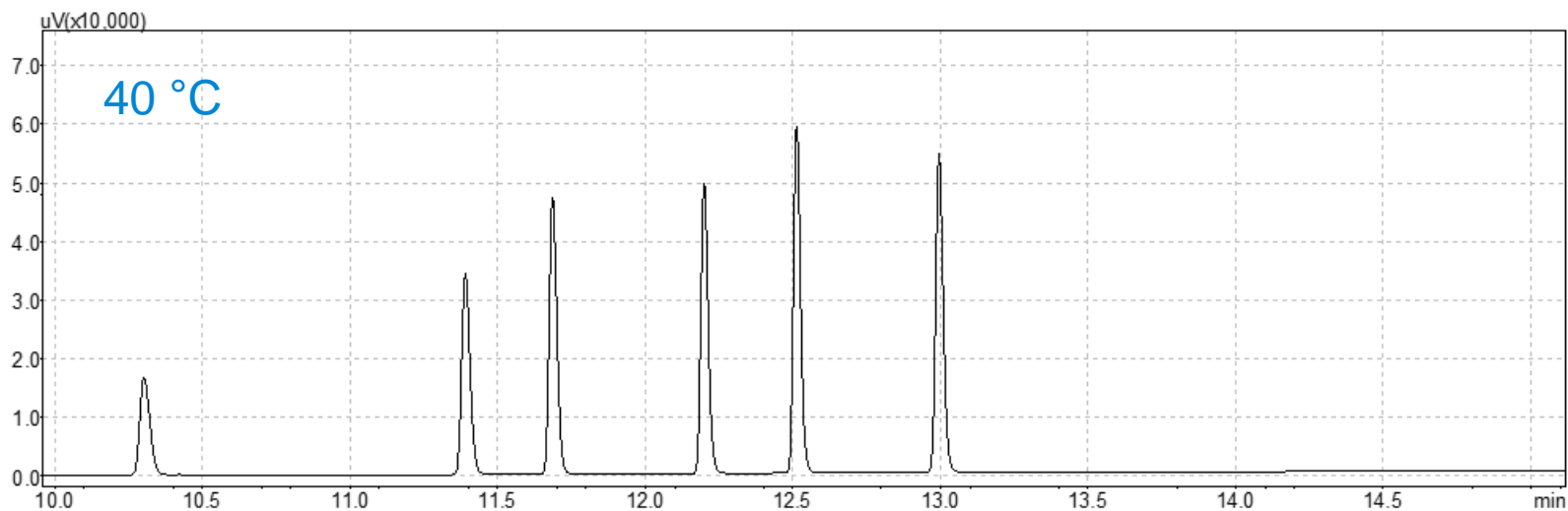
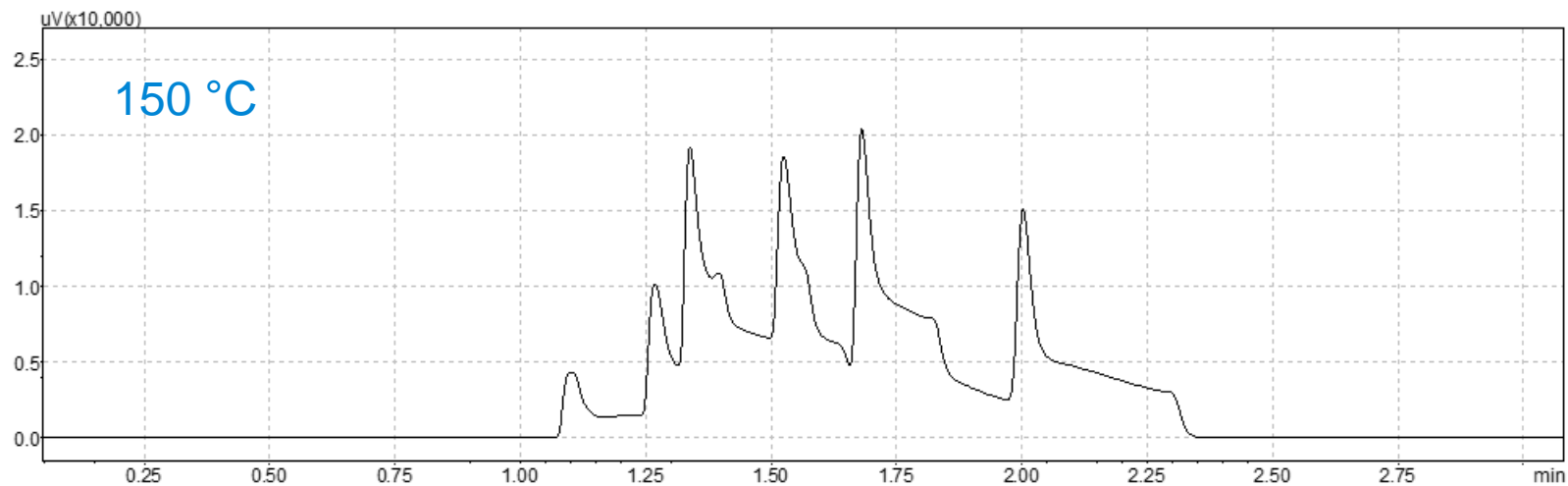
Detector

- Particles entering the detector
- Random: poor connection
- Regular: nearby "cycling" equipment (electronics)

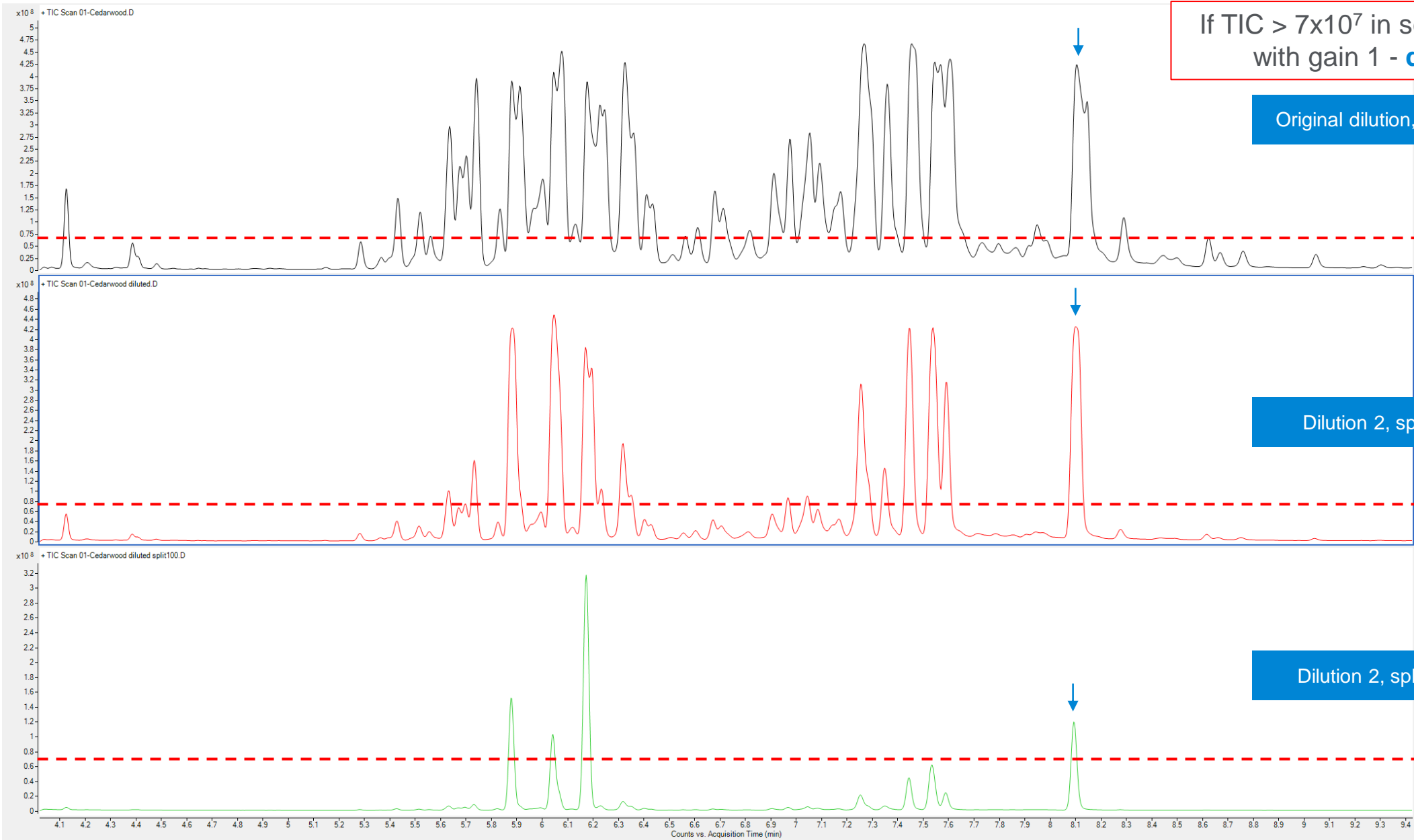
Application note: 5991-2975EN

Strange Peak Shape Due to Lack of Analyte Refocusing

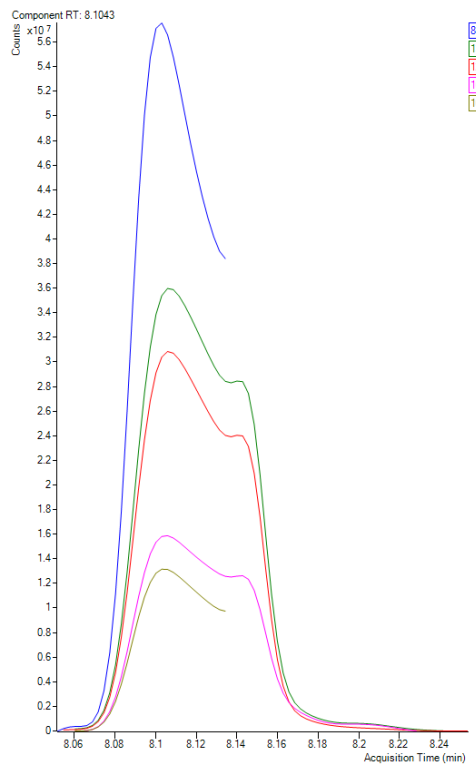
Free fatty acids in water on DB-FATWAX UI



Distorted Peak Shape Example – Total Ion Chromatograms

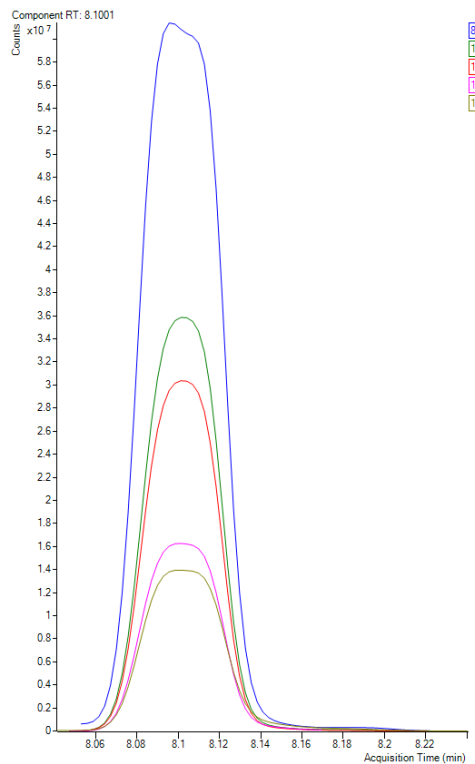


Distorted Peak Shape Example – Deconvoluted Extracted Ion Chromatograms



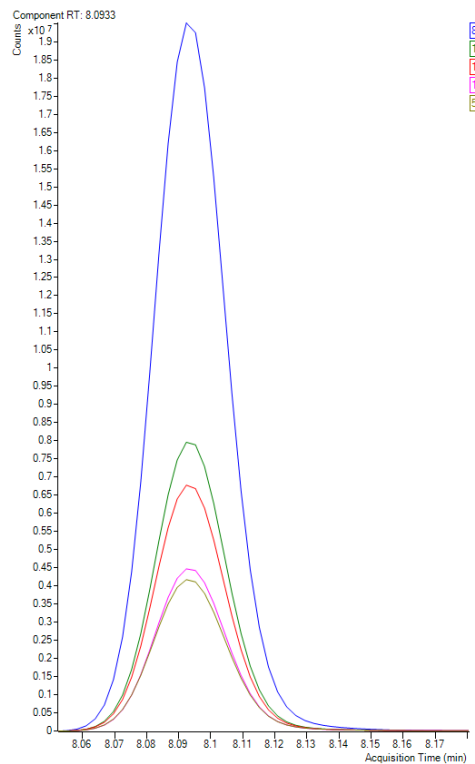
Original dilution, split 10:1

- EIC gaussian shape is lost and deconvolution is struggling to detect end of signal
- Highest abundance in overlay is 5×10^7 – **too high.**



Dilution 2, split 10:1

- Peak shape is broad and “rounded” at the top of peaks.
- Better, but still **too high.**



Dilution 2, split 100:1

- EIC gaussian peak shape is acceptable
- EIC abundance is acceptable

Quantitation Problems

Detector

- Poor stability (electronics) or baseline disturbances (contamination)
- Outside detector's linear range or wrong settings
- Integration parameters

Activity (adsorption) in injector or column

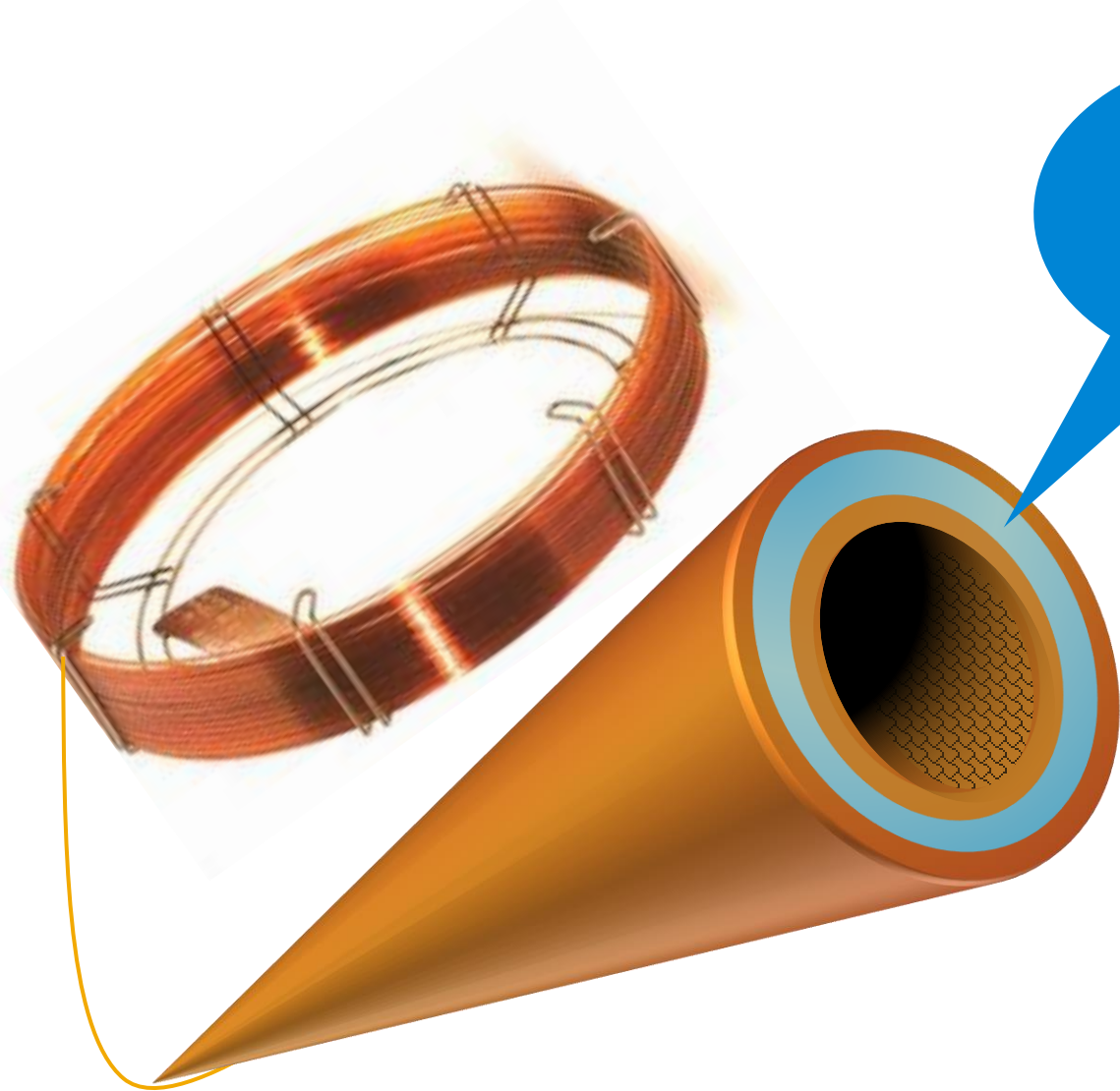
Injector

- Technique, settings, conditions
- Syringe worn

Other

- Coelution
- Matrix effects
- Sample evaporation – leaky vials
- Sample decomposition

What is Not Caused by a Column?



Not responsible

- Peaks
 - Any reproducible sharp chromatographed peak
- Siloxanes (even though it looks like bleed, spectrally)
- Degradation product peaks: endrin aldehyde, endrin ketone, DDE, DDD
- Carryover of sample compounds
- Splitting of peaks

Troubleshooting Techniques



Troubleshooting Tools

Bleed profile (noninjection): *baseline problems*

Inject a nonretained peak: *peak shape problems*

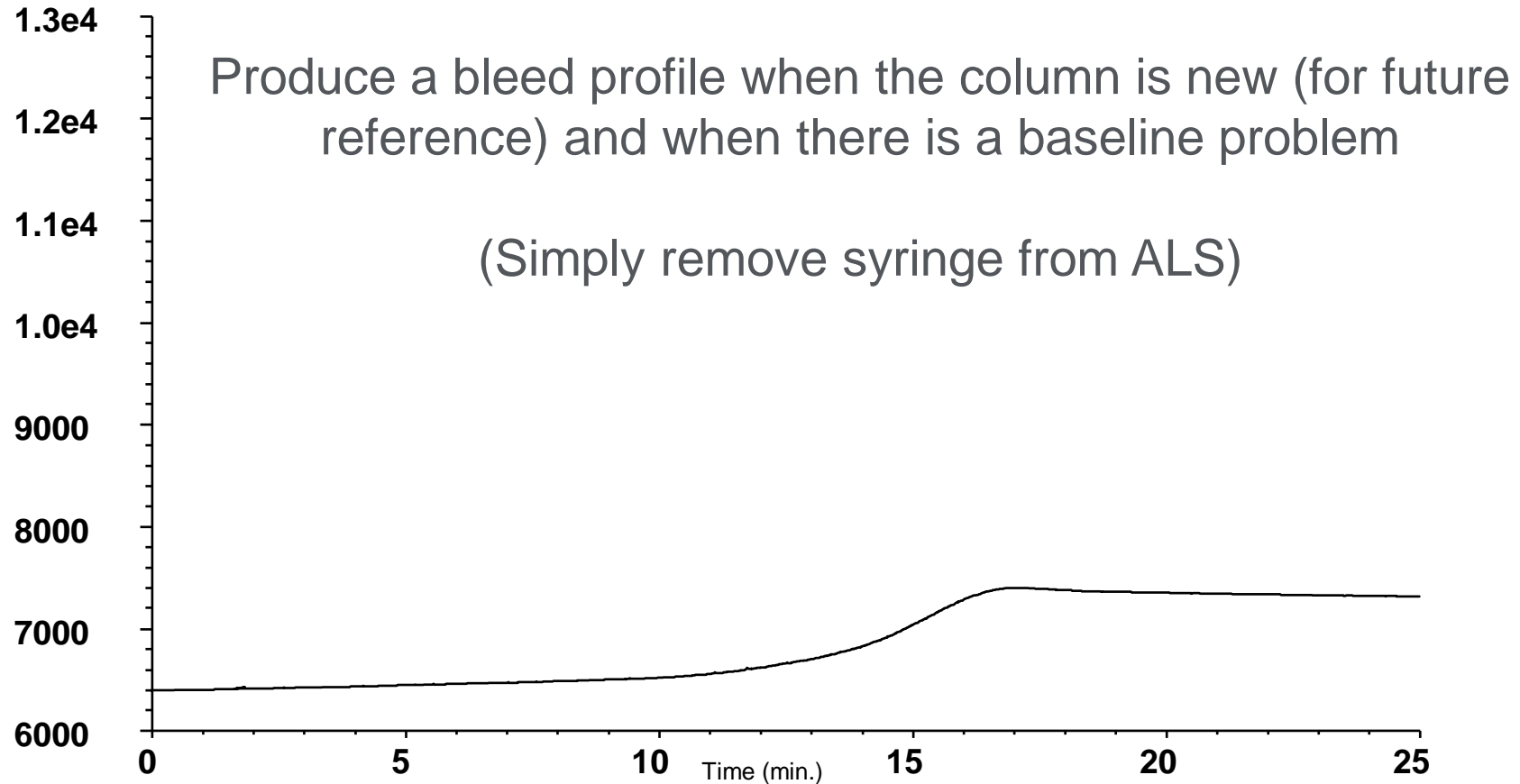
Test mix: *all problems*

Isolate the components: *all problems*

Condensation test: *baseline problems*

Jumper tube test: *baseline problems*

Generating a Bleed Profile

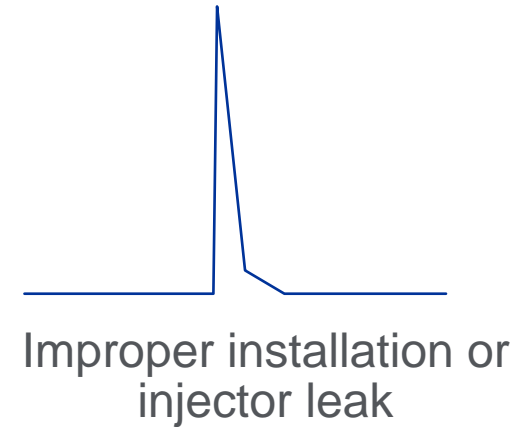
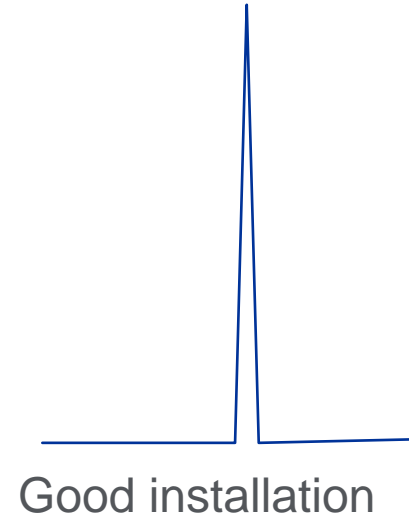


Agilent J&W DB-1, 30 m x 0.32 mm id, 0.25 μ m

Temperature program // 40 °C, hold 1 min // 20 °C/min to 320 °C, hold 10 min

Inject a Nonretained Compound to Check Flow Path

Used to check
flow path



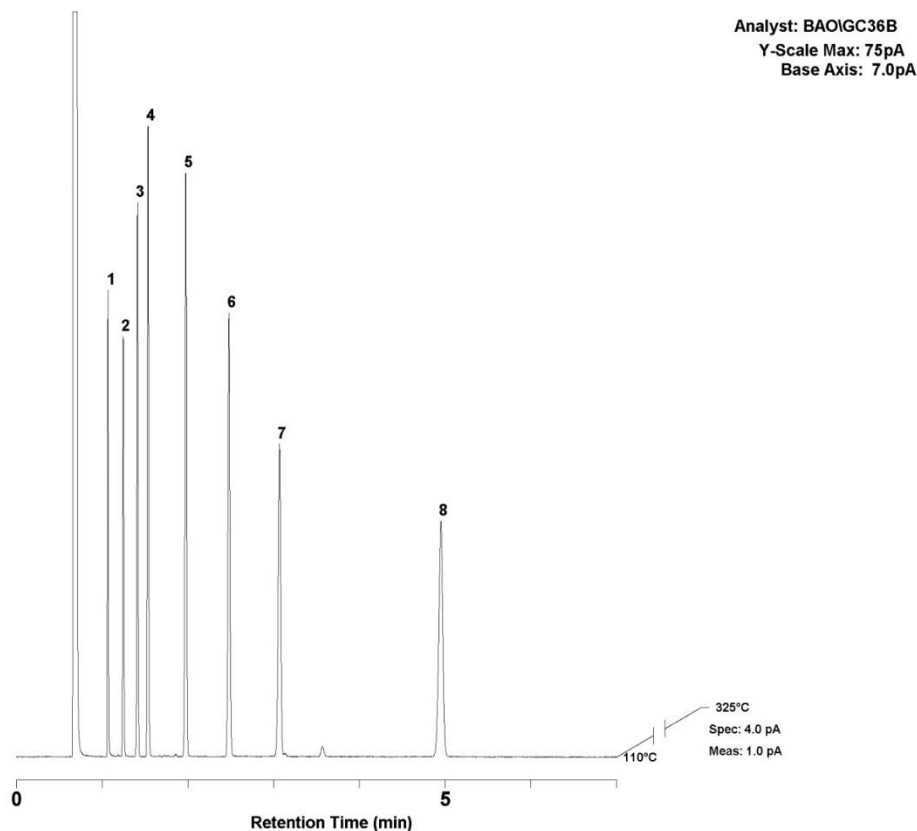
Potential explanations:

- Injector or septum leak
- Too low of a split ratio
- Liner problem
 - (Broken, leaking, misplaced)
- Column position in injector and detector

Test Mix – Make Your Own

A test mix is used to determine how “good” the column is, or whether the problem is related to the chemical properties of the analytes.

It is simplest to use your own standard.



Compound	Purpose
Hydrocarbons	Efficiency Retention
Alcohols	Activity
FAMEs, PAHs	Retention
Acids	Acidic character activity
Bases	Basic character activity

Test Conditions	
Inlet:	Split (250 °C)
Detector:	FID (320 °C)
Flow:	37.3 cm/s (1.8 mL/min)
Carrier gas:	Hydrogen
Holdup compound:	Methane (0.671 min)
Temperature program:	Isothermal (110 °C)

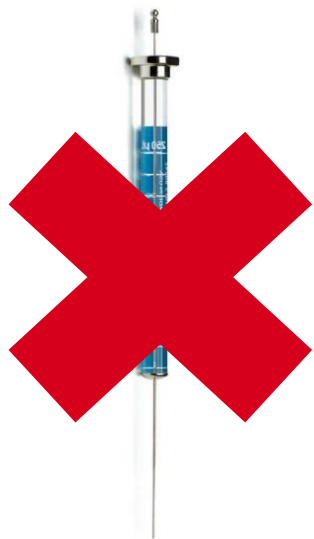
Standards Selection

Agilent ULTRA Chemical Standards have:

- Best in class online search, compare, and ordering capabilities
- Rapid shipping: 99.9% of orders are dispatched within 24 to 48 hours (continental U.S. only, as of now)
- Custom standard solutions including our online custom quoting tool, enabling customers to upload recipe formulations and to modify the recipe before submitting it
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 - Discover more at www.agilent.com/en/product/chemical-standards
- Rigorously tested and manufactured under ISO 9001, ISO 17025, and ISO 17034 accreditation
- Sample preparation materials, columns, supplies, instrumentation, and reference materials from a single source



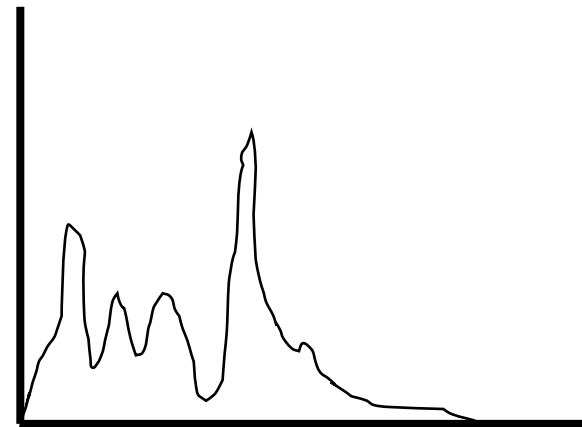
Perform a Noninjection “Blank”



Remove syringe
from autosampler



Run your program



If you see peaks, it is likely
that inlet contamination
exists

Condensation Test

A condensation test is used to isolate the cause of:

- Erratic baselines
- Ghost peaks or carryover

For use when problems are worse after periods of GC nonuse

Condensation Test

Procedure

- Leave GC at 40 to 50 °C for >8 hours
- Begin a blank run
- Do another blank run immediately after the first blank run is complete
- Compare the two blank runs

Condensation Test

Results

- First blank run is worse: contaminants (from injector, lines, traps, or carrier gas) carried into the column.
- Blank runs are the same: **contaminants are not strongly focused on the front of the column**

Jumper Tube Test

Purpose

- Helps to locate the source of contamination or noise
- Isolates GC components

Jumper Tube Test

Isolate the detector

- Remove column from the detector
- Cap detector and turn on
- Do a blank run

Jumper Tube Test

Isolation of detector – results:



Detector OK



Detector is the problem



Jumper Tube Test

Isolate the injector

- Connect the injector and detector
 - 1 to 2 m of deactivated fused silica tubing
- Turn on carrier gas
- Do a blank run

Jumper Tube Test

Isolate the injector – results:



Injector OK



Injector, lines, or carrier gas contaminated



Jumper Tube Test

Isolate the column

- Reinstall the column
- Set up as before
- Blank run

Jumper Tube Test

Isolate the column – results:

- If the problem persists, it's the column
- If the problem is gone, a previous leak, solid debris, or installation is the issue

Have a Good Troubleshooting Story? Let Us Know

Please call or email us today to share a troubleshooting success story or if you need help with troubleshooting



Agilent University

Why training? What can we help with?

Agilent University

- Trained over 38,000 students in 2019
- 98% customer recommended
- 4.6 out of 5 customer satisfaction
- 94% excellent and very good

Labs who want faster and more efficient learning options to help overcome training challenges

Overtasked staff

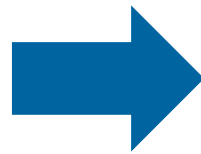
Staff turnover

Pressure to improve quality and productivity

Daily consistency with output and results

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Virtual training



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Troubleshooting Tips

1. Isolate the problem

(Do a blank run, inject an unretained compound, do a jumper tube test)

2. Change only one variable at a time

3. Compare before **and** after chromatograms

(Peak shape, response, retention, baseline rise, background, look for trends)

4. **Use** technical support

Remember

Complete system = carrier gas + injector +
column + detector + data system

- Multiple causes and effects
- Do not change too many variables at once



Contact Agilent Chemistries and Supplies Technical Support



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

[Option 1 for GC and GC/MS columns and supplies](#)

Option 2 for LC and 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. and Canada 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