

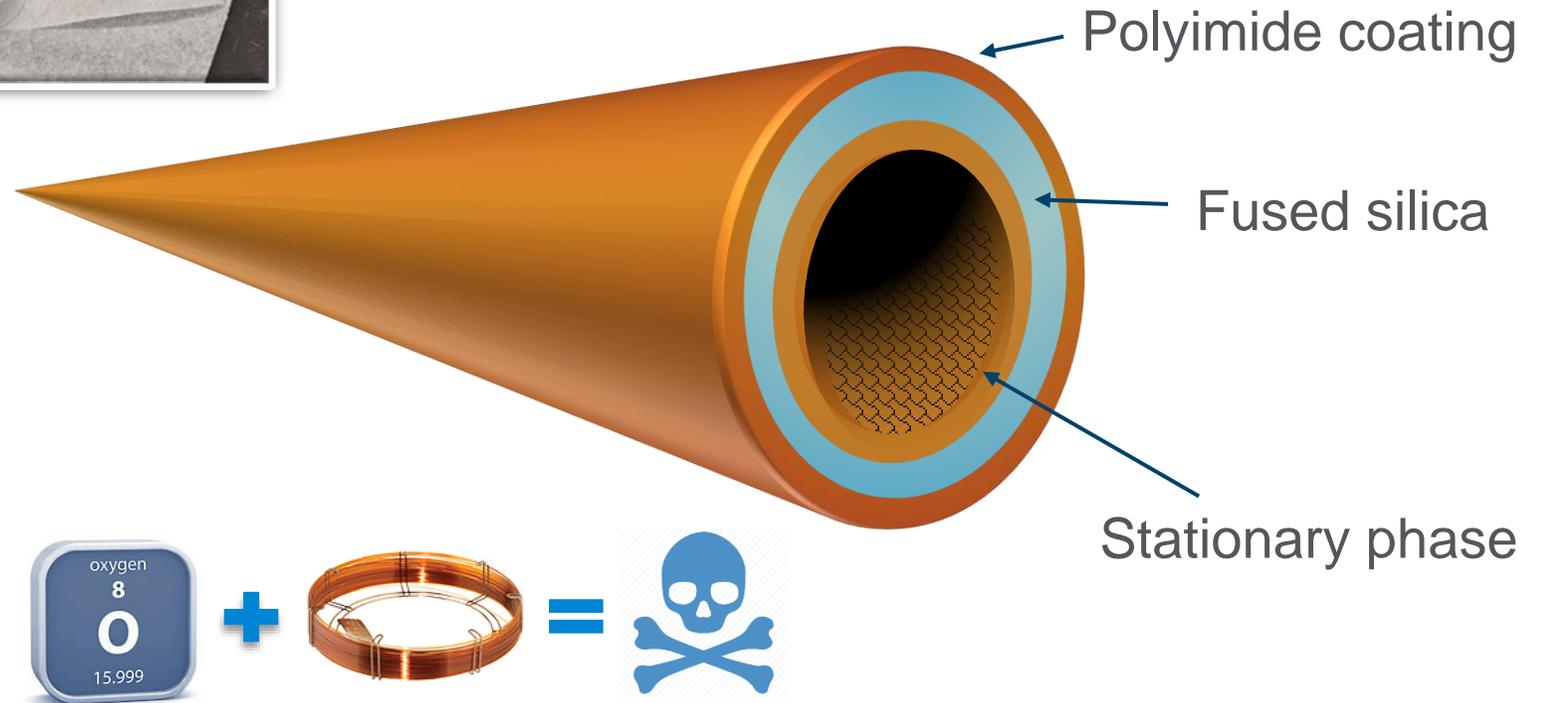
# Ask the Agilent Experts

GC and GC/MS scientists discuss and answer your most frequently asked questions

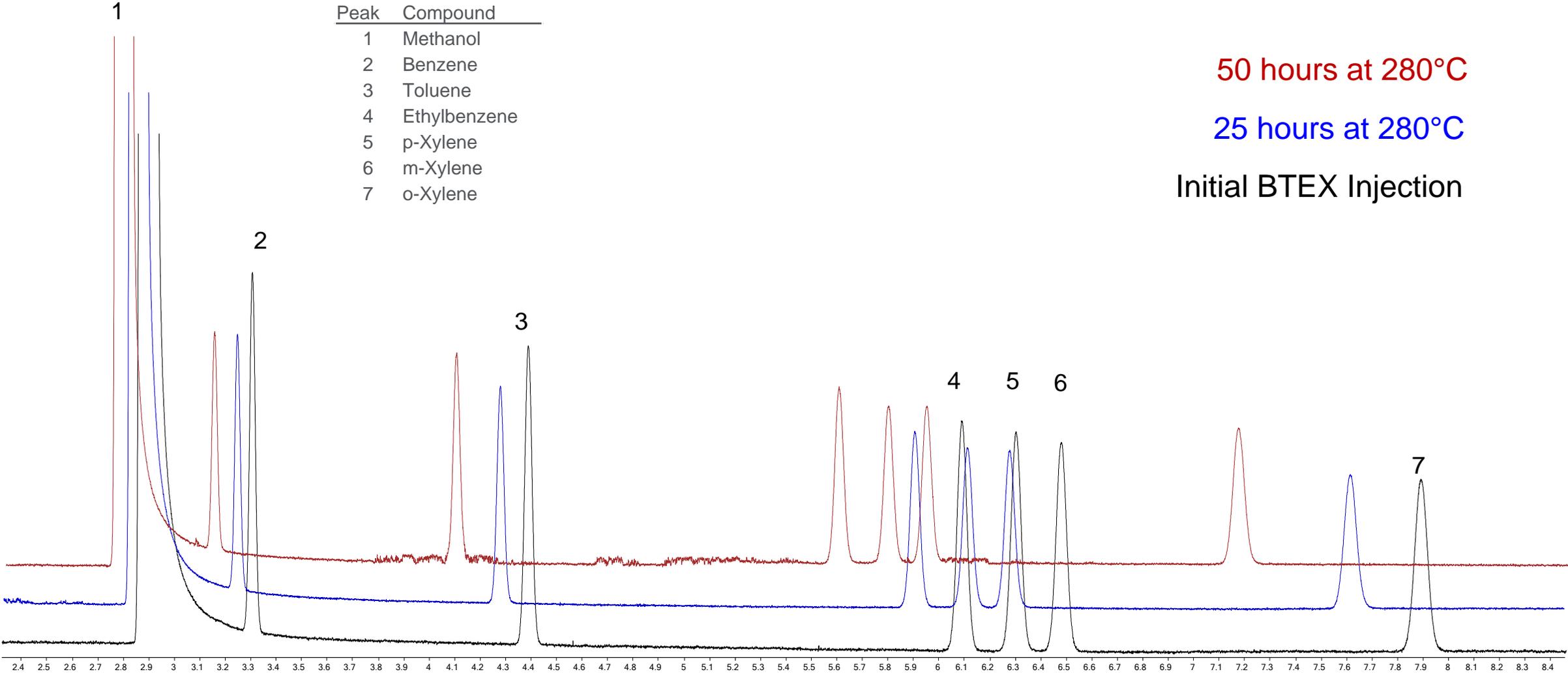
Angela Smith Henry  
Vanessa Abercrombie

# How Long Should my Column Last?

If you never install it, turn on gas flow, turn on heat, or inject anything...

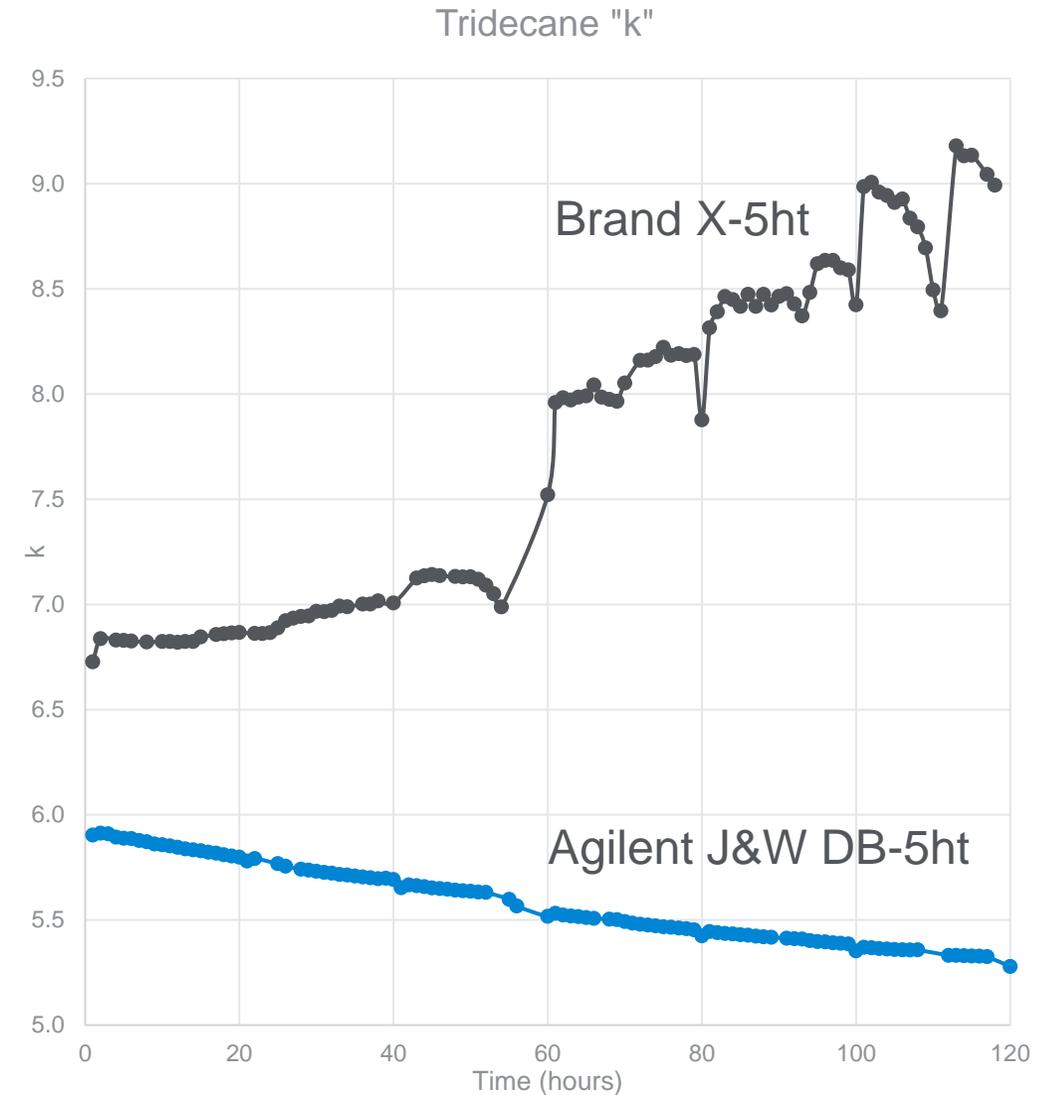
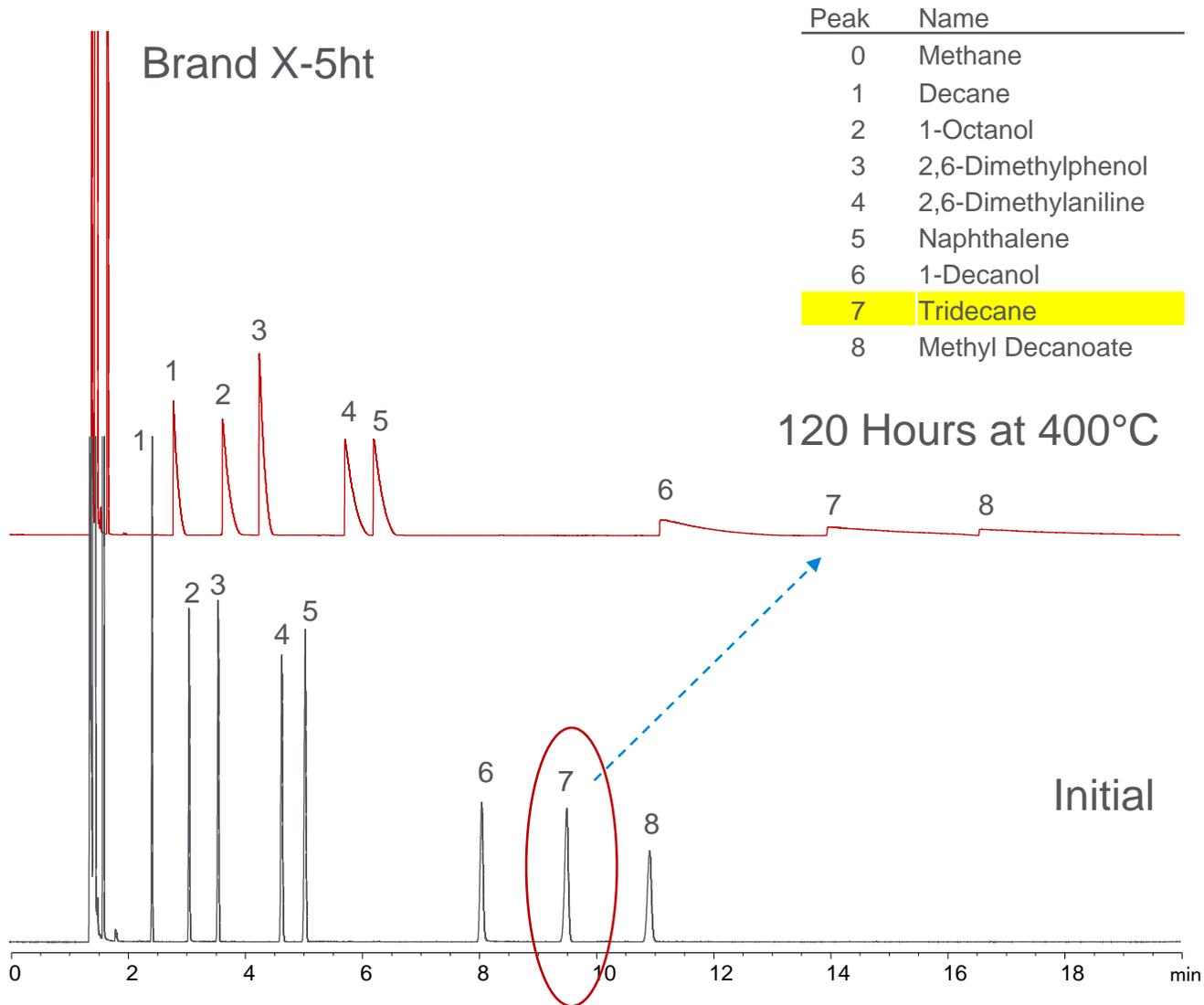


# Thermal Stability and Retention Time Shifting



# Phase Degradation and Increases Retention

$$k = \frac{t_{\text{compound}} - t_{\text{methane}}}{t_{\text{methane}}}$$

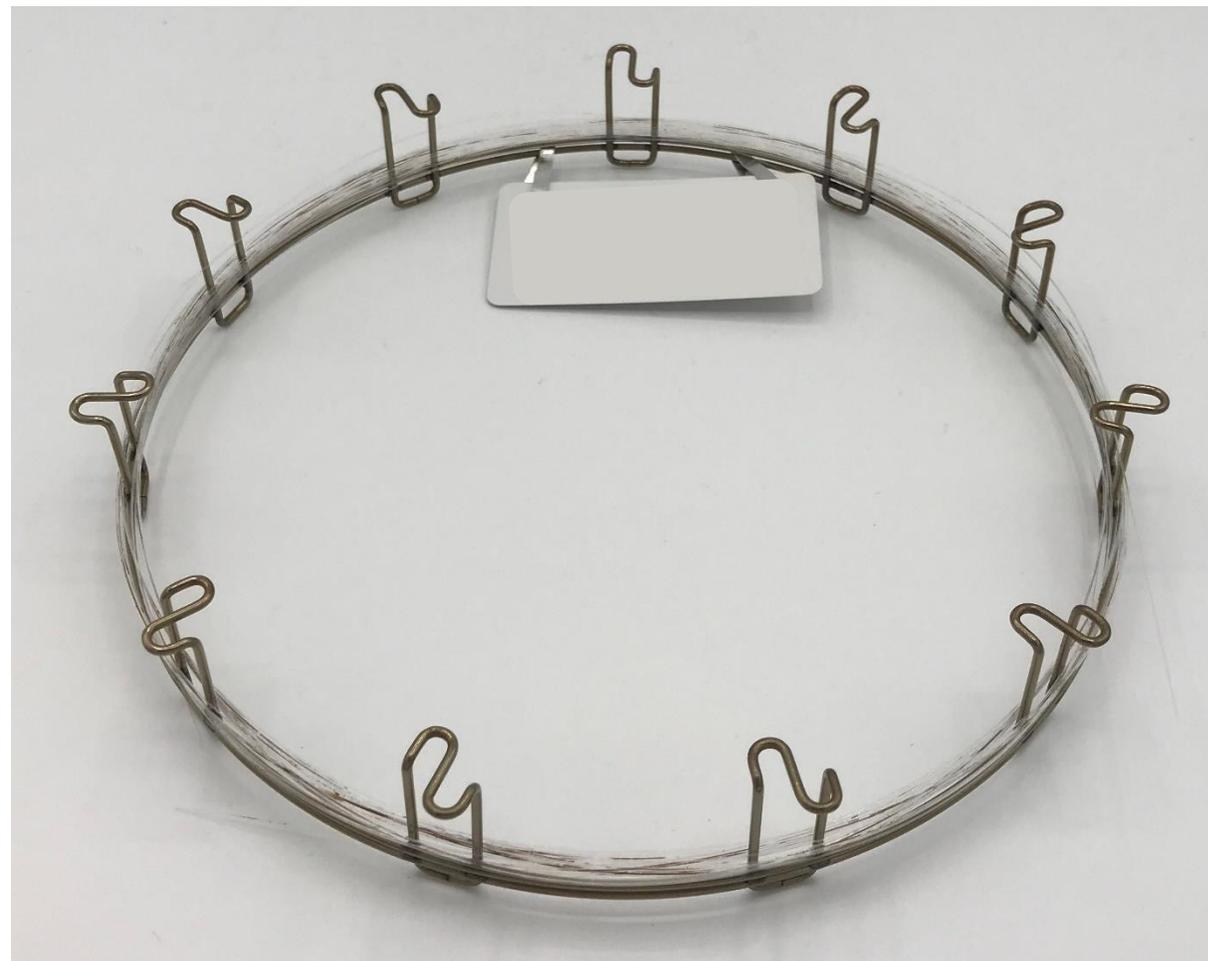


# Competitor Fused Silica doesn't always "take the heat"

Just because a company says their column can run at or above 400°C, doesn't mean it can.....



Brand Y  
After 20 hours at 400°C

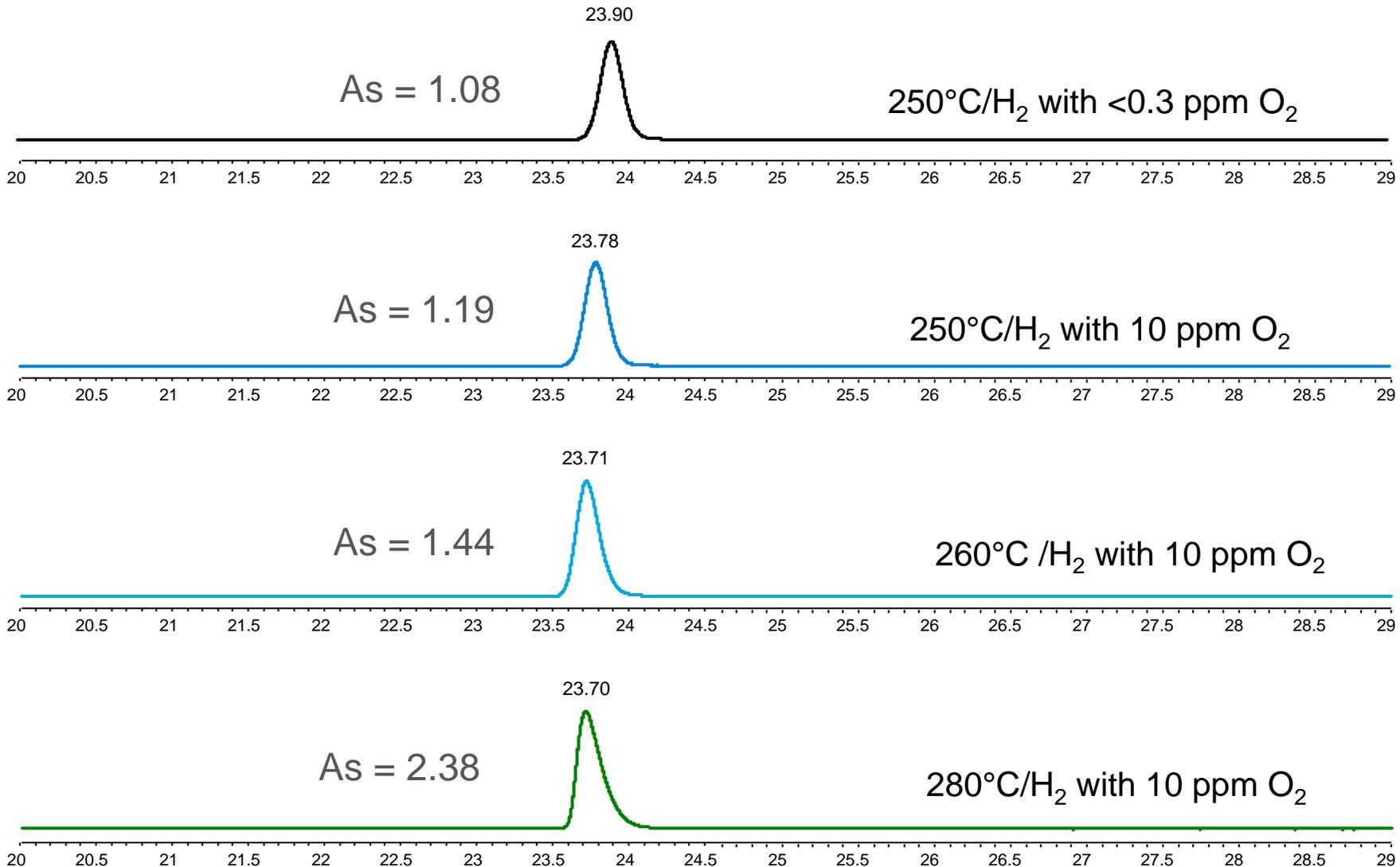


Brand Y  
After 25 hours at 400°C  
Agilent Publication 5994-1013EN



# What Happens if There's a Leak in my Gas Lines?

# Effect of Oxygen on Peak Shape of 2-ethylhexanoic Acid



Inlet or GC detector: P/n G3440-81011  
MS transfer line: P/n G3440-81013

Publication number: 5991-9032EN



# What other problems can a leak cause?

- Reduced peak response
- Elevated background

## GC/MS

- Impaired electron multiplier function
- Shorter filament and liner lifetime
- Excessive source maintenance

But how do I leak  
check everything?

Use the new Agilent CrossLab CS  
Leak Detector!



# Agilent CrossLab Cartridge System (CS)

## Leak detector and Flow Meter



- Exchangeable cartridge with ADM Flow Meter
- USB connects to web interface for added functionality and firmware updates
- Large OLED screen
- Able to detect N<sub>2</sub> leaks

Gas	Minimum Detectable Leak Rate (mL/min)
Hydrogen	0.0025
Helium	0.003
Methane	0.014
Nitrogen	0.4
Argon	0.03
Carbon dioxide	0.03

<https://www.agilent.com/en/product/gas-purification-gas-management/gas-management/gas-leak-detector>



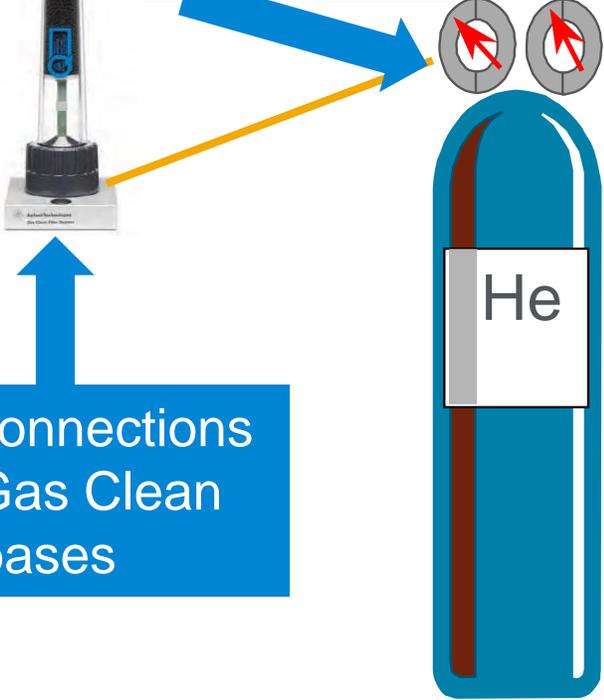
ALL\* fittings in the back of the GC



Gas source



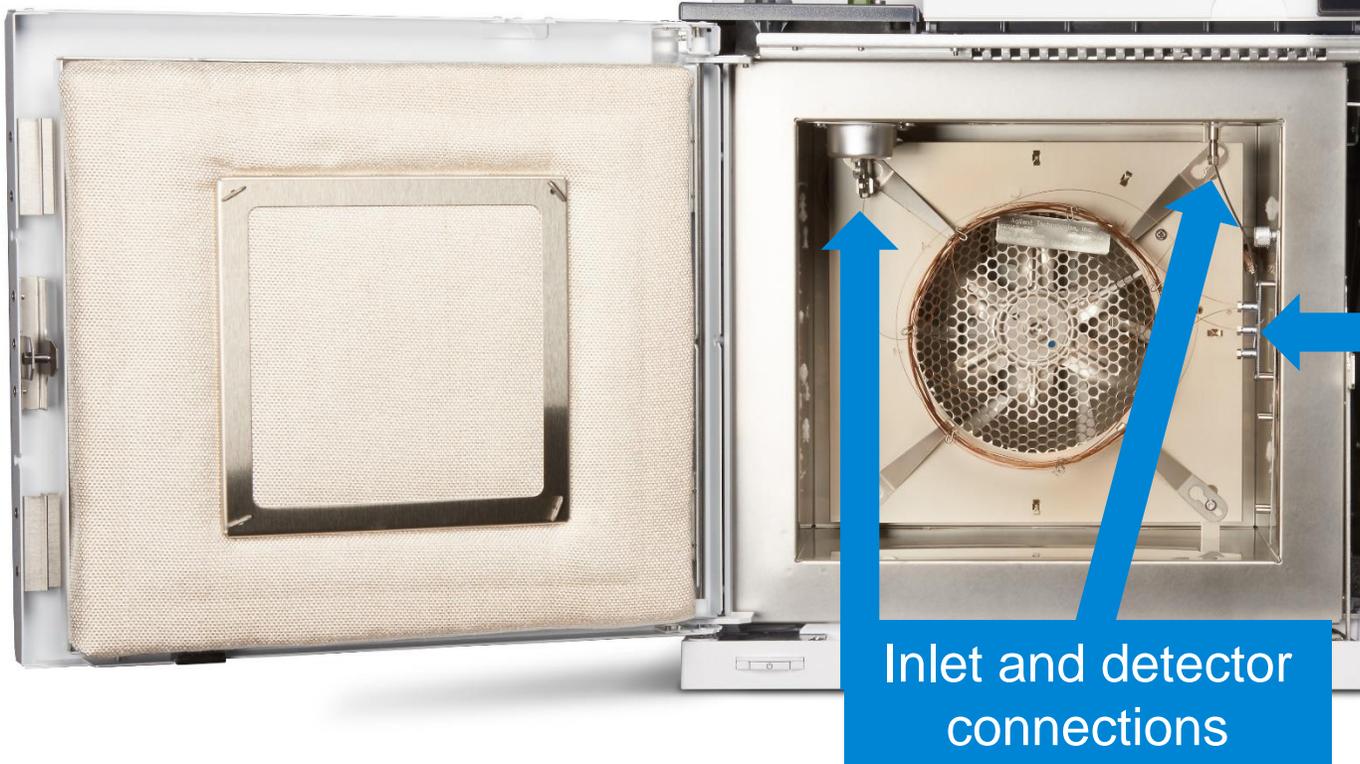
Filter connections and Gas Clean bases





MS transfer line

Septum nut and turn-top



Inlet and detector connections

CFT connections

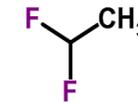
# How do we check for leaks on the MSD?

What to check?

- Vent valve
- MSD transfer line nut
- Analyzer side door

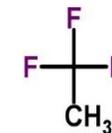


## Typical Electronic Duster Components and Ions



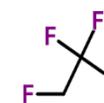
1,1-  
difluoroethane

*m/z*  
51,65



1,1,1-  
trifluoroethane

*m/z*  
69



1,1,1,2-  
tetrafluoroethane

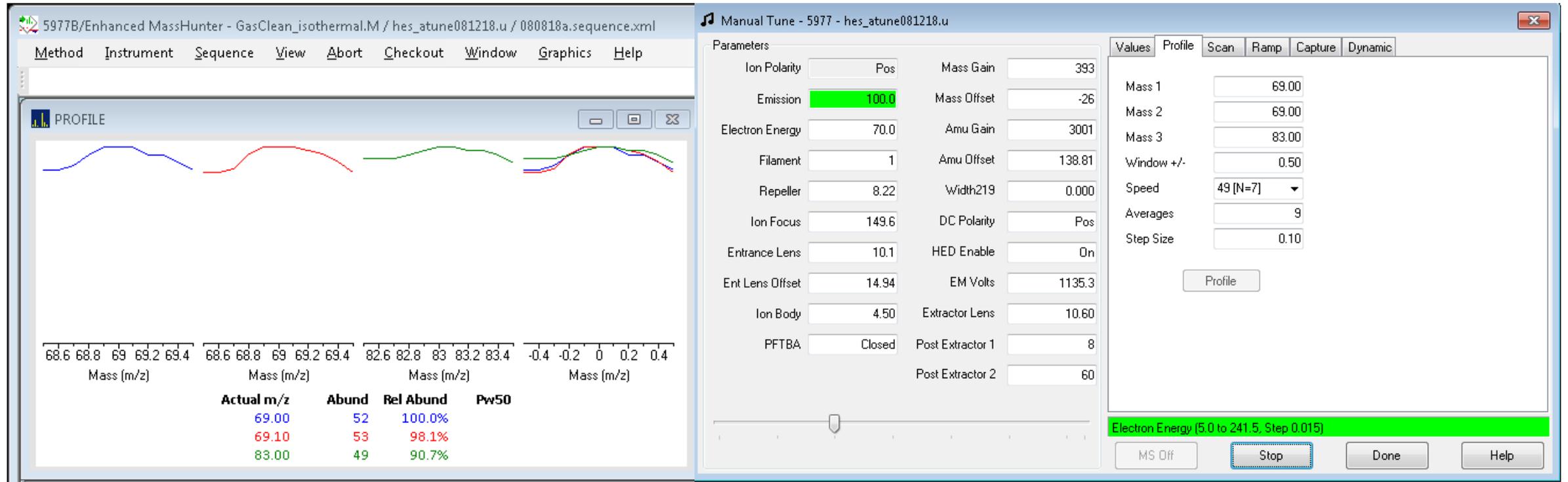
*m/z*  
69,83

## Use electronics duster

- Hold can upright (don't spray liquid!)
- Spray short bursts around possible leak points
- “Live” tune profiling for ions to pinpoint leak

# Leak checking the Mass Spectrometer

## Using electronics duster to find system leaks with Manual Tune



Navigate to MSD Manual Tune in the Data Acquisition

- Instrument > Edit Tune Parameters

Use Profile tab to watch the main ions (69 and 83 m/z for my electronics duster)

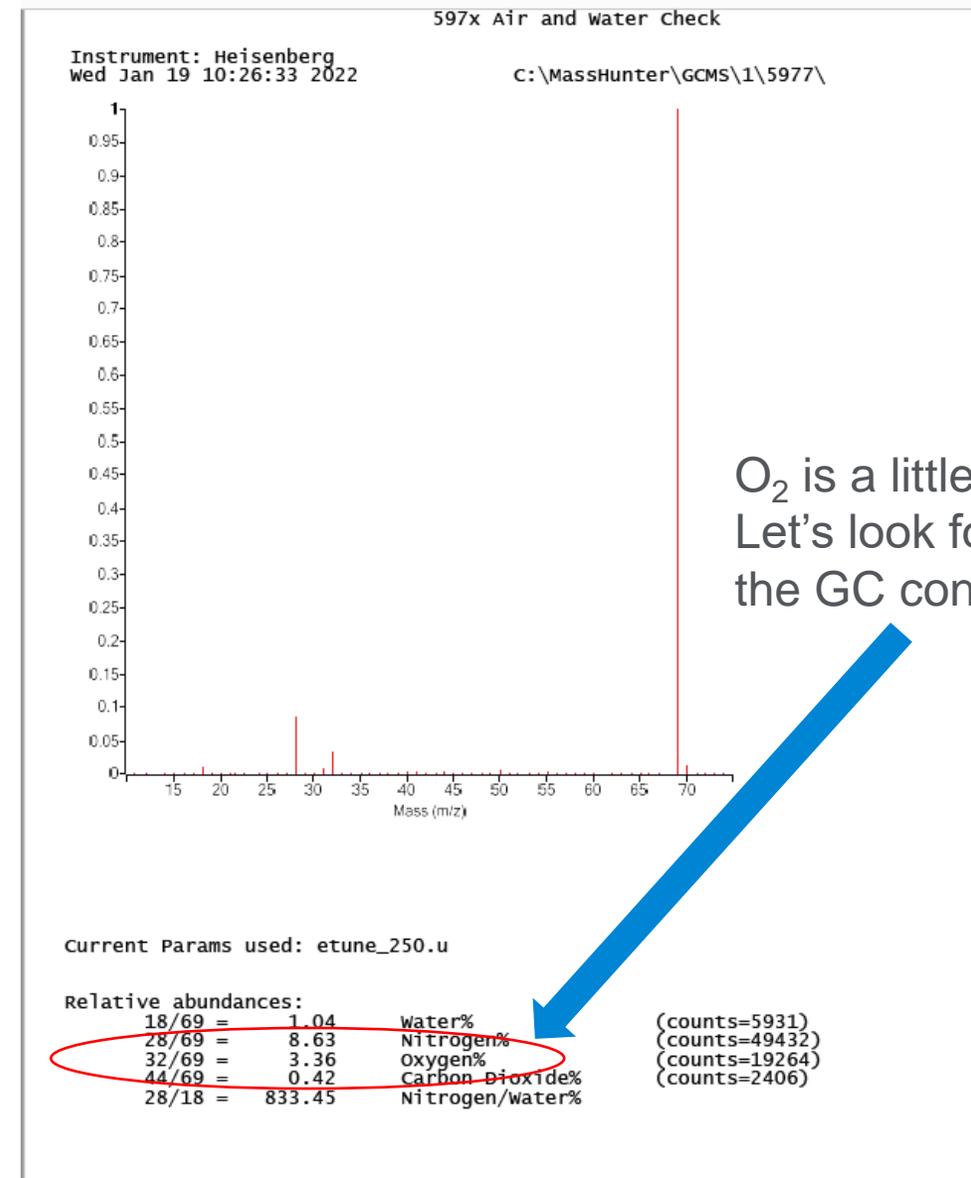
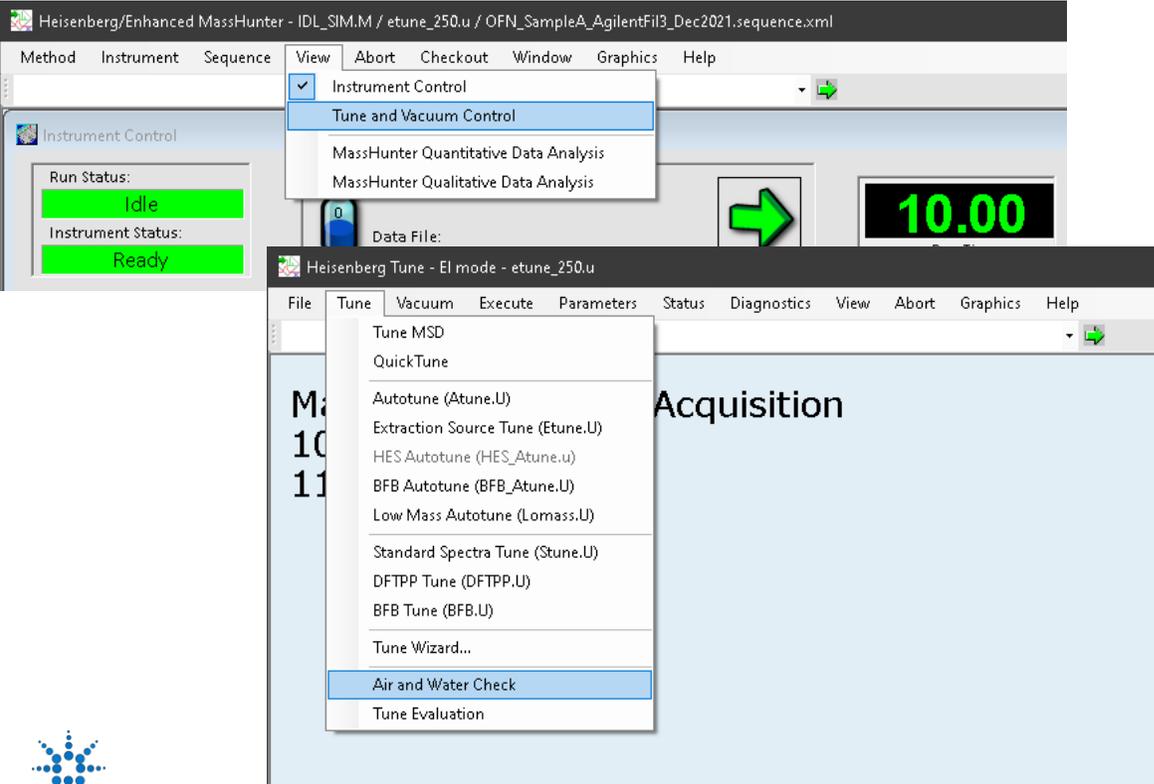
- Spray short bursts at vent valve, transfer line, and side door

# But I don't have an electronics duster...

## What now?

### Run an air water check!

- View > Tune Vacuum Control
  - Tune > Air and Water Check

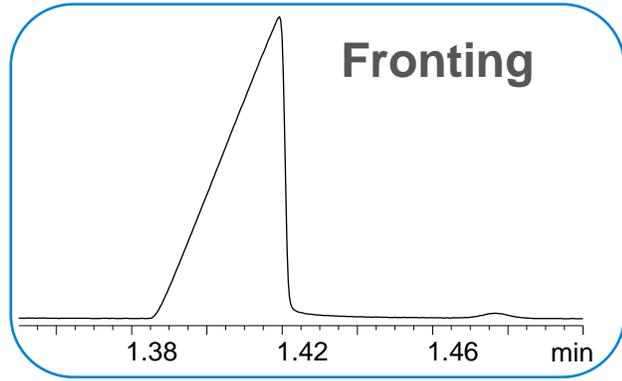


# Why do peaks tail?

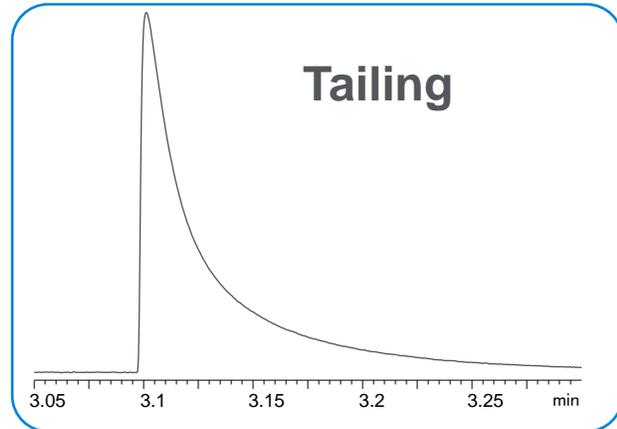


# What causes tailing?

Tailing can be caused by many different things



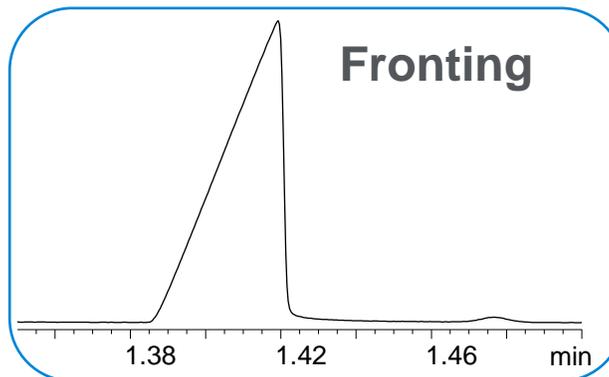
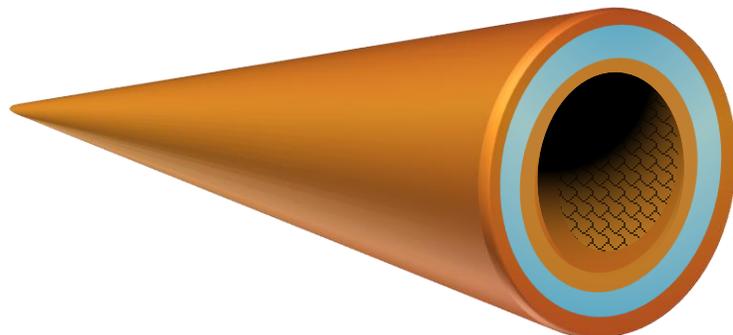
Fronting → Overloading the column



Tailing → Activity



# Peak Fronting and Column Capacity



Fronting → Overloading the column

Film Thickness ( $d_f$ ) $\mu\text{m}$	Column ID (mm)				
	0.1	0.18	0.25	0.32	0.53
0.1	250	450	625	800	1325
0.18	139	250	347	444	736
0.25	100	180	250	320	530
0.5		90	125	160	265
1.0			63	80	133
1.8			35	44	74
3.0			21	27	44
5.0			13	16	27

Increase Retention

$$\beta = \frac{ID}{4d_f}$$

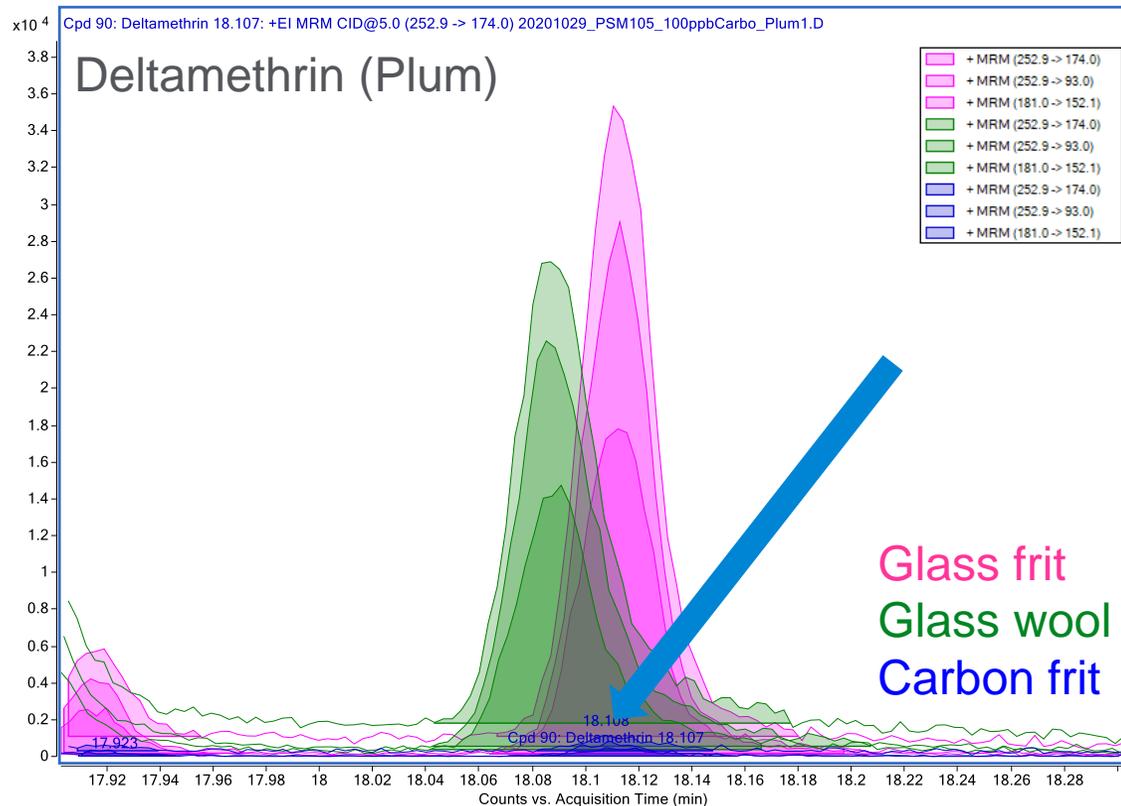
Increase Capacity

Be aware of column capacity

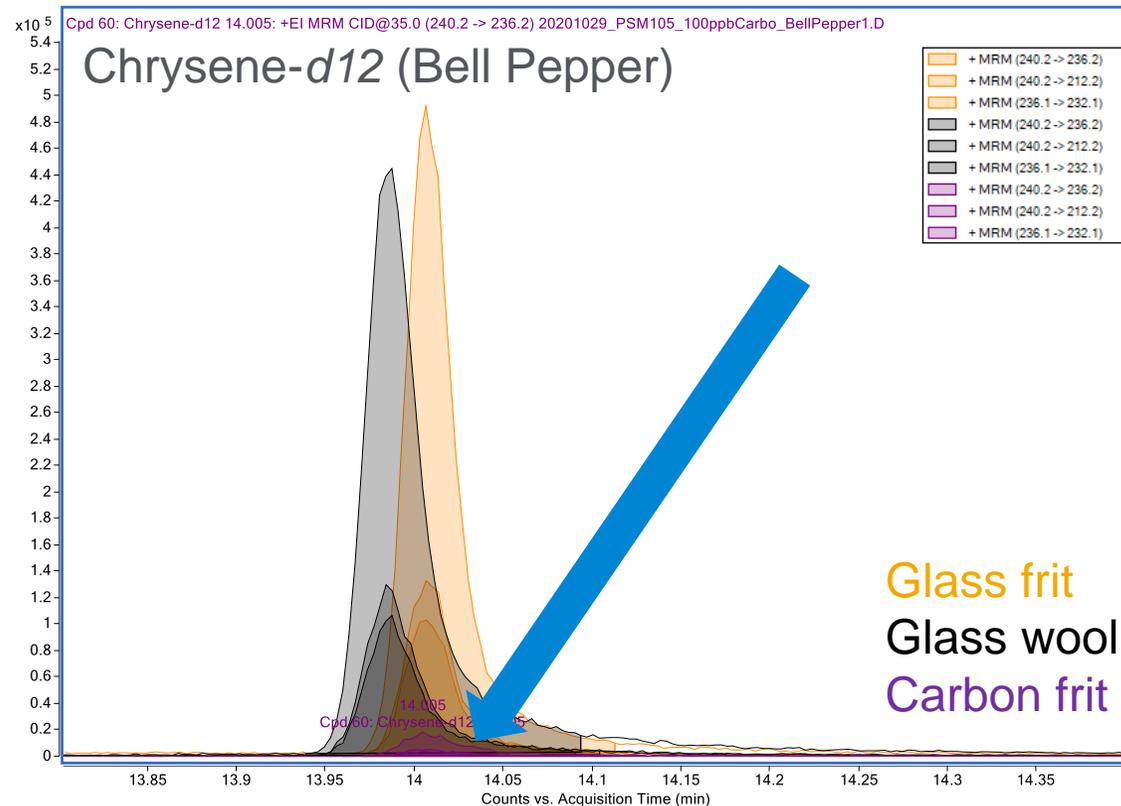
I.D. (mm)	Capacity (ng)
0.05	1-2
0.10	6-13
0.18	25-55
0.20	35-70
0.25	80-160
0.32	110-220
0.45	600-800
0.53	1000-2000

# Does it really matter if the liner is deactivated?

Higher Response with Glass Frit  
Less Peak Tailing



Be careful with carbon or non-deactivated liners and internal standards!

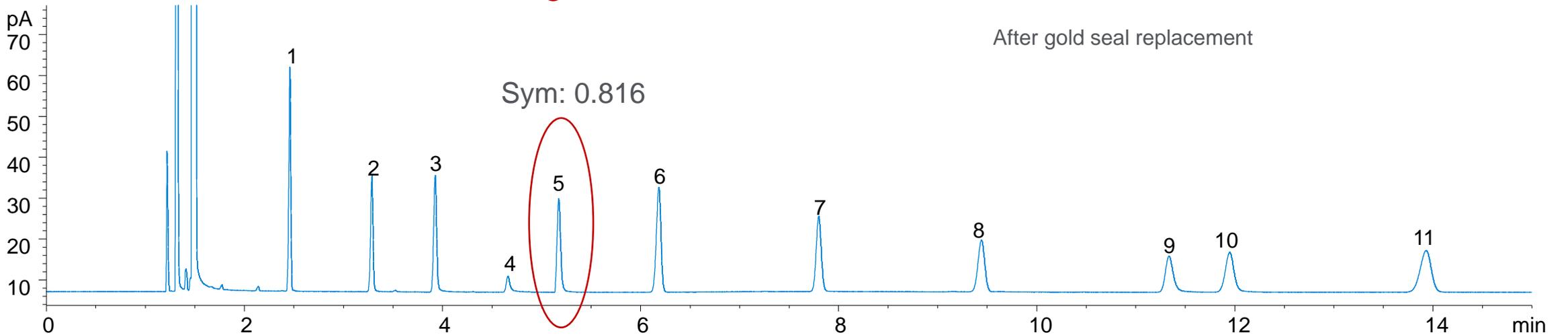
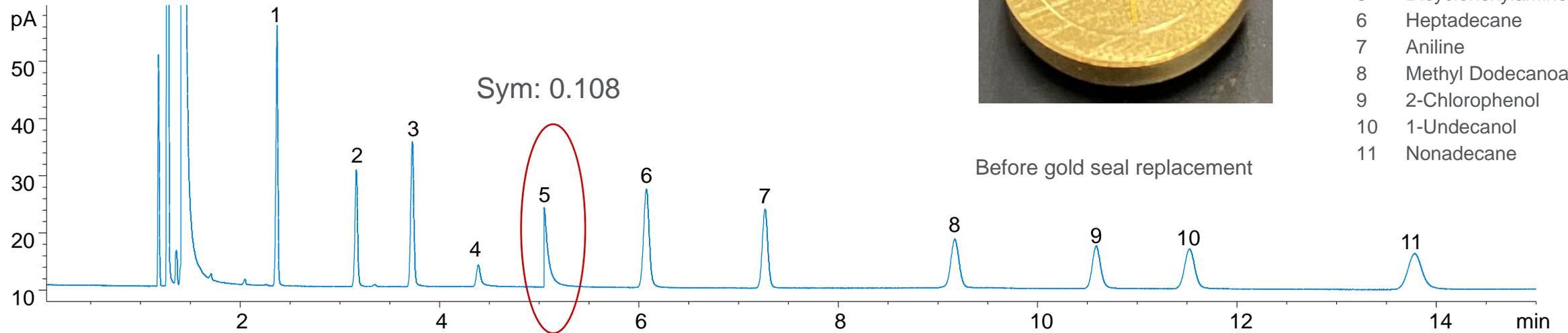


Ultra Inert Deactivation results in better peak shapes for tricky analytes

# Your gold seal can cause tailing too...

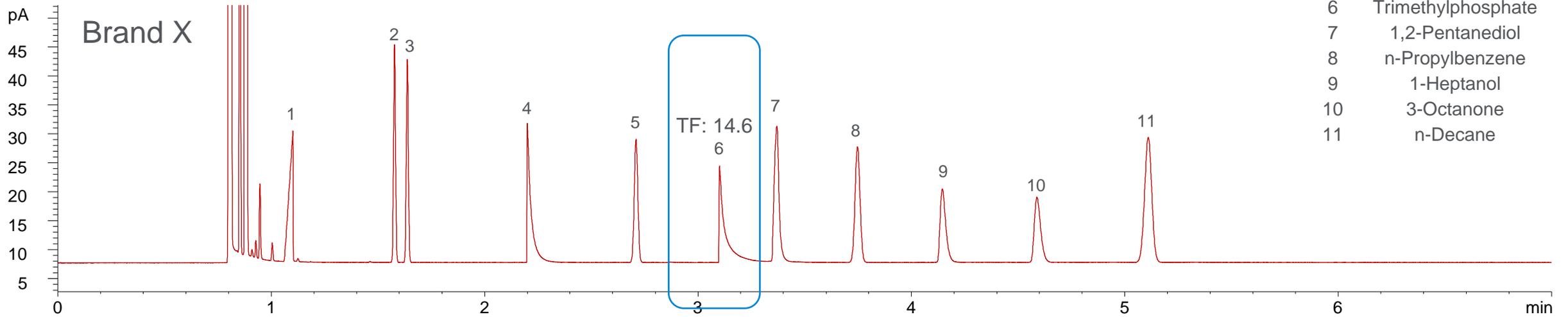


Peak	
0	Methane
1	2-Nonanone
2	Decanal
3	2,3-Butanediol
4	Ethylene Glycol
5	Dicyclohexylamine
6	Heptadecane
7	Aniline
8	Methyl Dodecanoate
9	2-Chlorophenol
10	1-Undecanol
11	Nonadecane

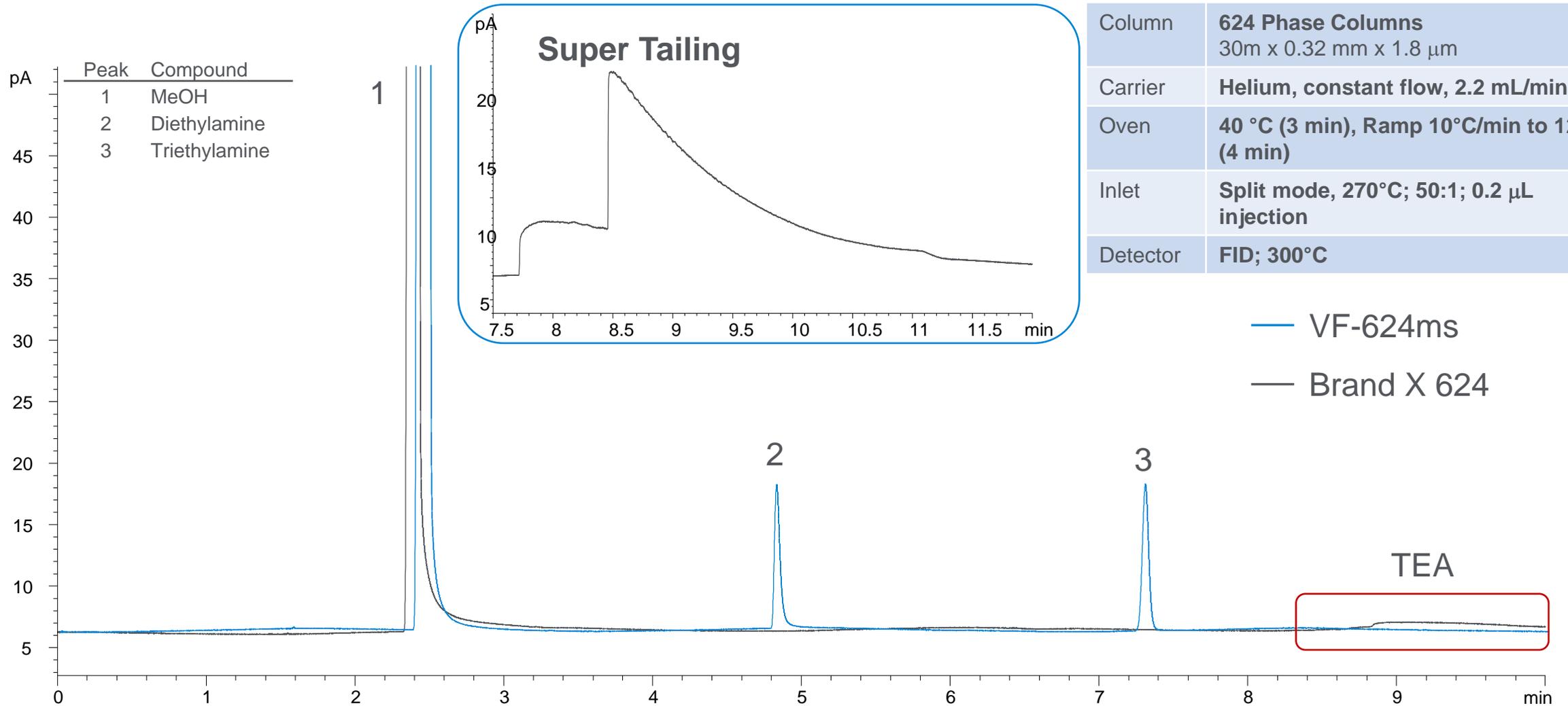


# Agilent Ultra Inert Column have Better Peak Shape for Acids and Bases

Peak	Compound
1	Propionic Acid
2	1-Octene
3	n-Octane
4	4-Methylpyridine
5	n-Nonane
6	Trimethylphosphate
7	1,2-Pentanediol
8	n-Propylbenzene
9	1-Heptanol
10	3-Octanone
11	n-Decane



# Did your peaks disappear or are you using the wrong deactivation?

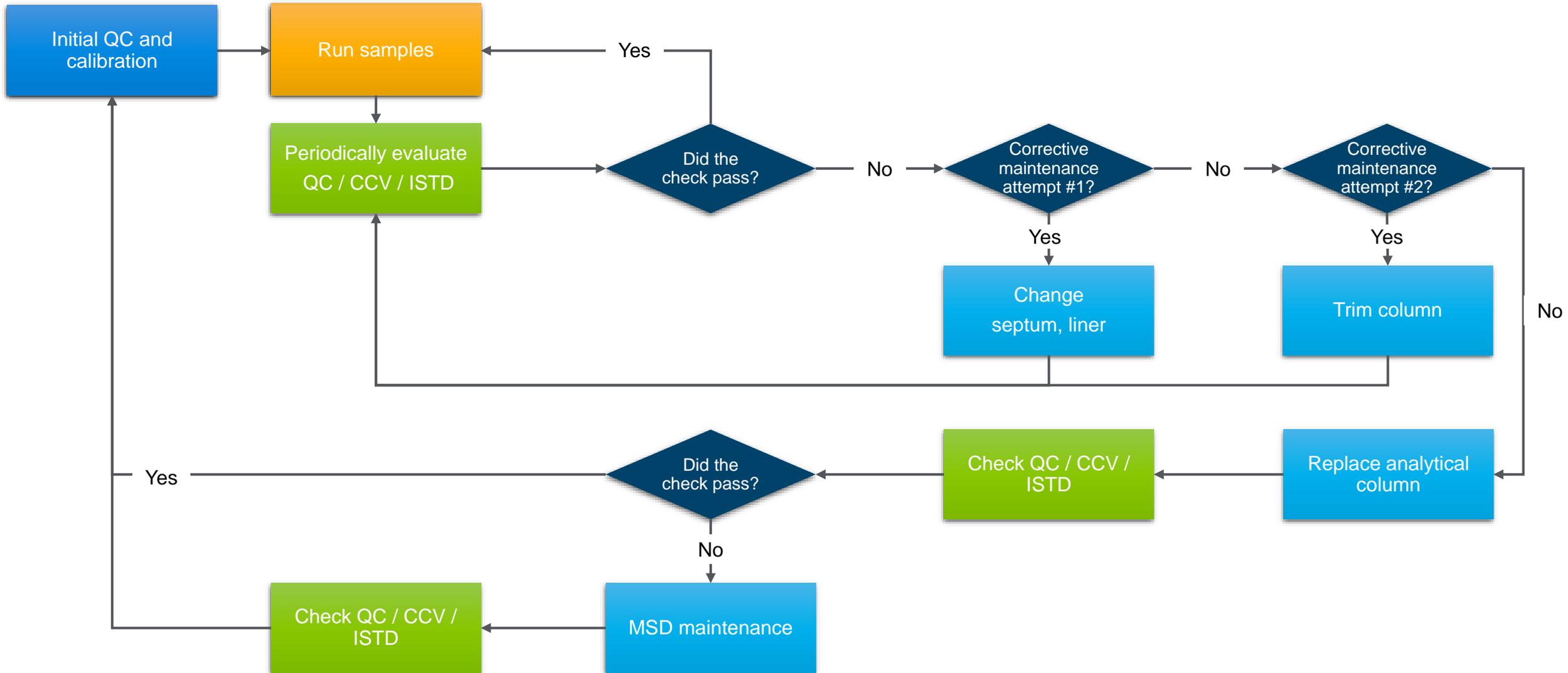


Column	<b>624 Phase Columns</b> 30m x 0.32 mm x 1.8 $\mu$ m
Carrier	<b>Helium, constant flow, 2.2 mL/min</b>
Oven	<b>40 °C (3 min), Ramp 10°C/min to 120 °C (4 min)</b>
Inlet	<b>Split mode, 270°C; 50:1; 0.2 <math>\mu</math>L injection</b>
Detector	<b>FID; 300°C</b>

# How Often Should I Clean my MS Source?

# Determining When to Complete Maintenance

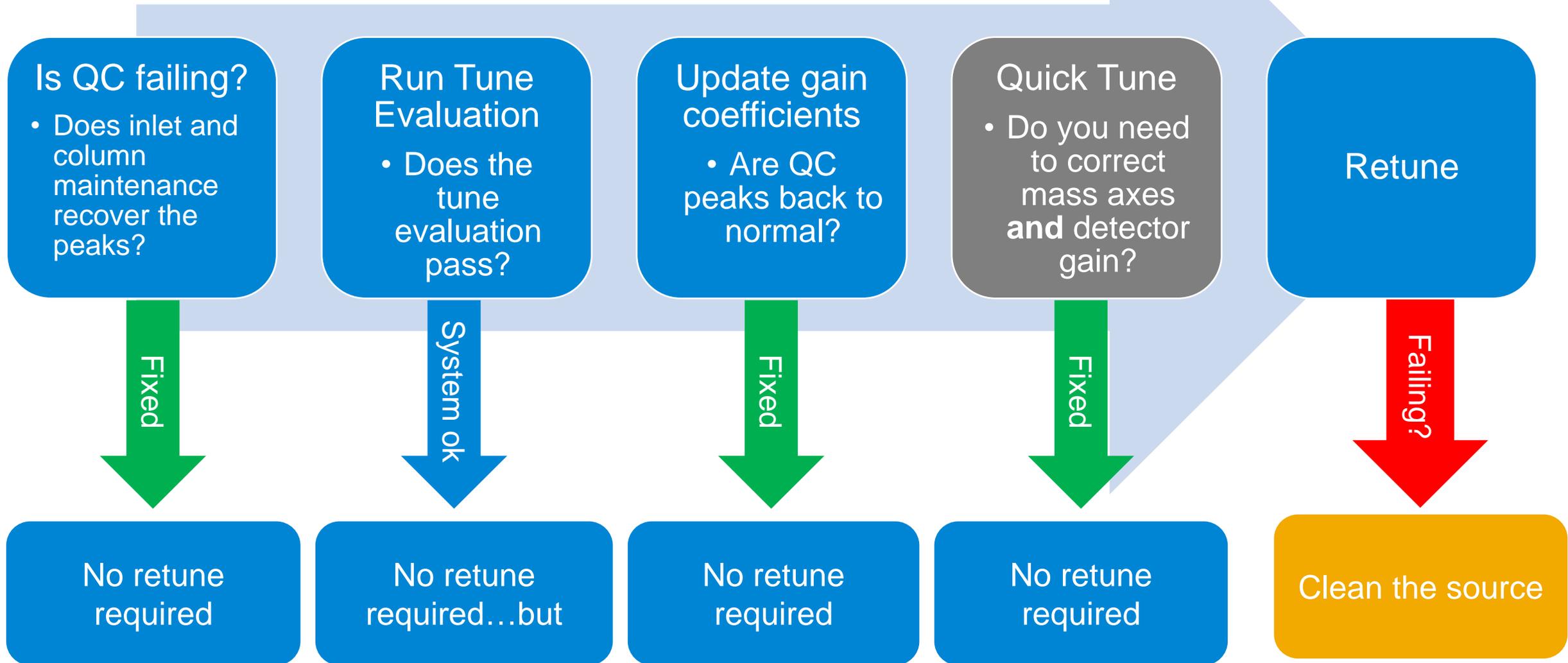
Use application-specific QC standards to evaluate chromatographic health





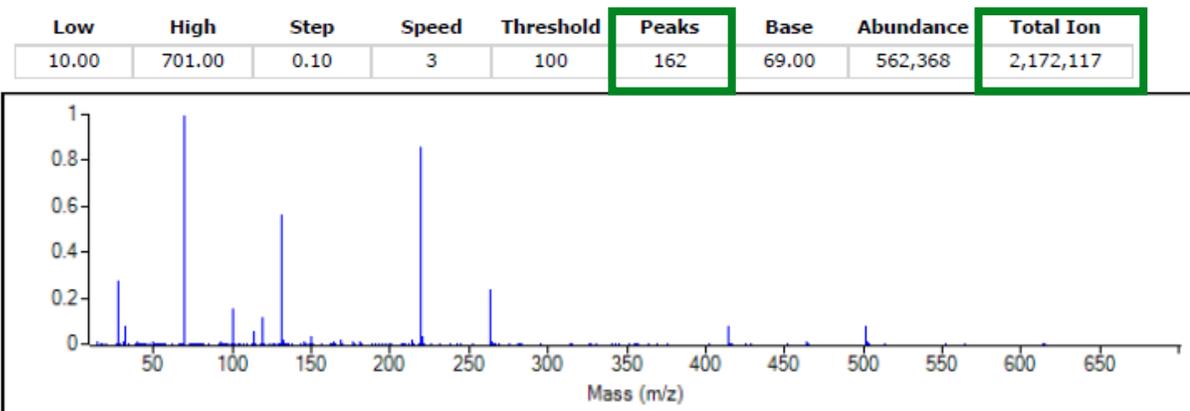
# How Do I Determine that I Need to Tune...or Clean?

## Peak response dropping



# Hints about a dirty source... in your tune report

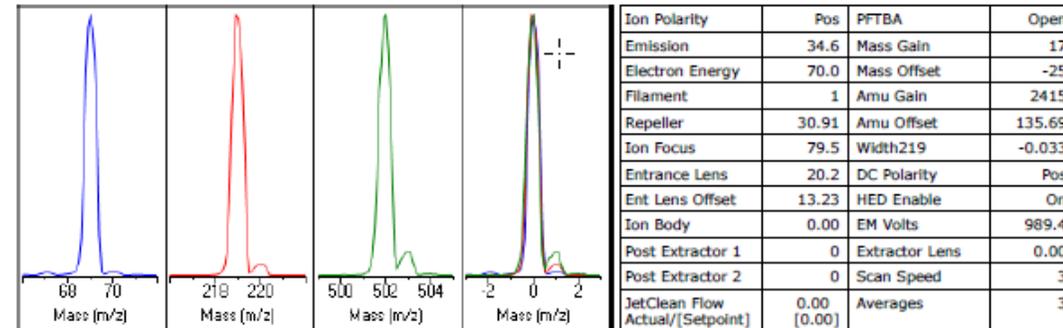
What are the peaks and total ion counts?



Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	562,368	100.0%	70.00	6,281	1.1%
219.00	219.00	481,152	85.6%	220.00	21,056	4.4%
502.00	502.00	46,664	8.3%	503.00	4,612	9.9%

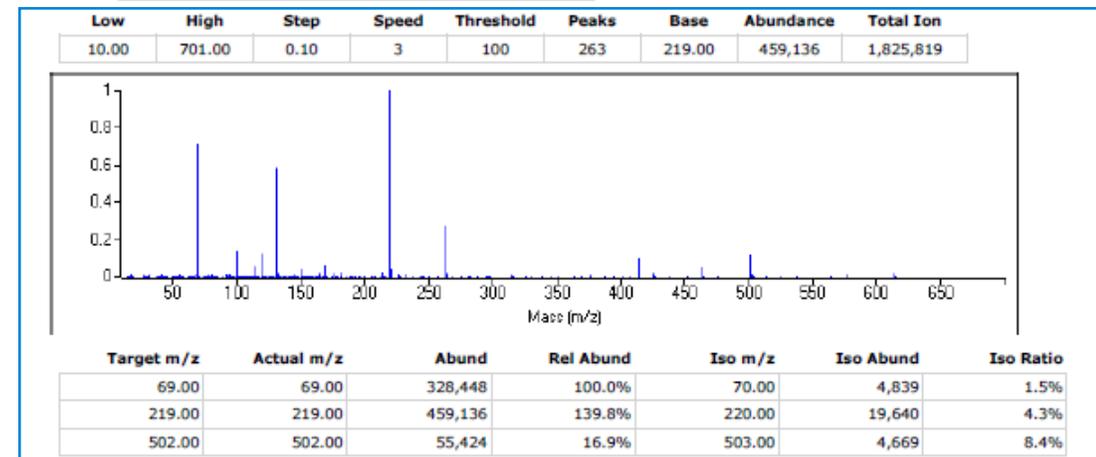
100-250 peaks = **Normal**

High number of peaks (>600) = **Detector noise or contamination**



Actual m/z	Abund	Rel Abund	Pw50
69.00	343,938	100.0%	0.60
218.90	467,811	136.0%	0.62
502.00	57,672	16.8%	0.62

Temperatures and Pressures		
MS Source	230 Turbo Speed	100.0
MS Quad	150 Hi Vac	N/C



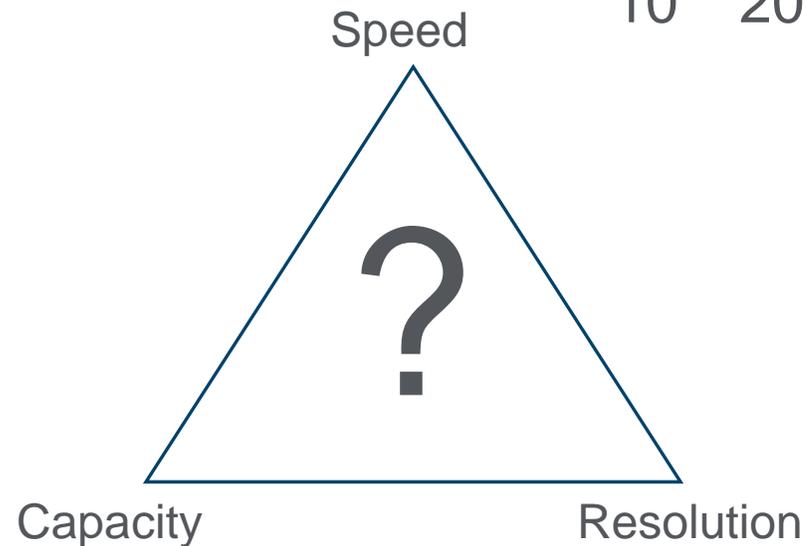
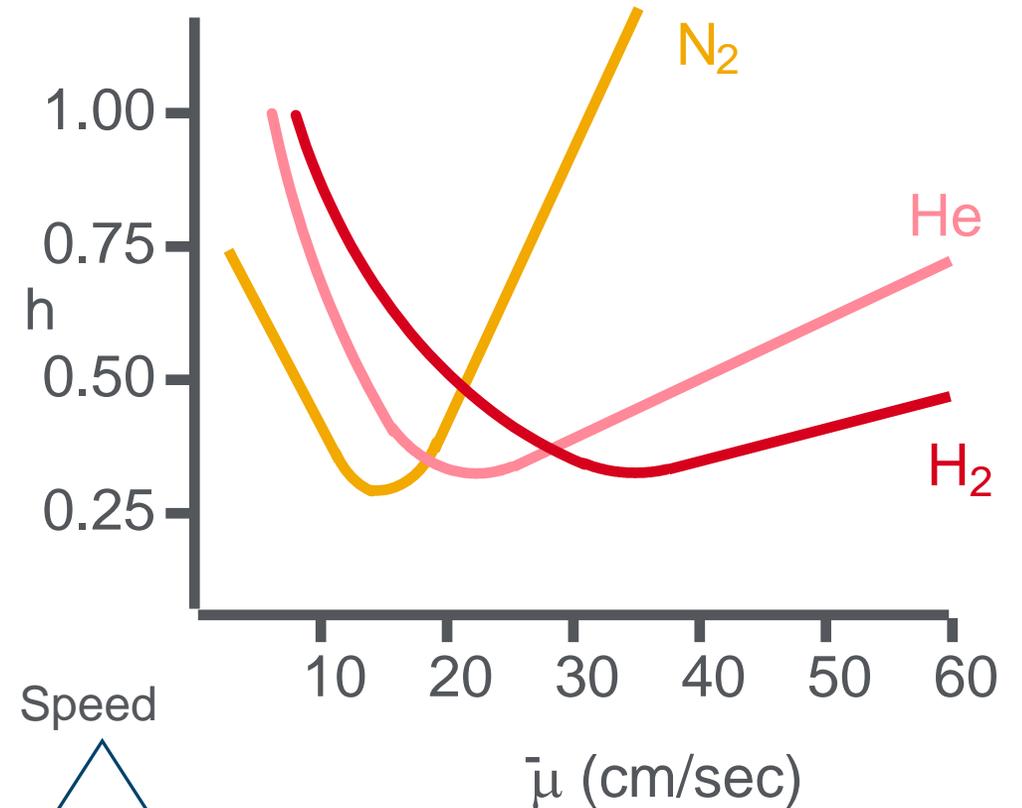
Air/Water Check: H2O ~1.3% N2 ~1.4% O2 ~0.2% CO2 ~0.5% N2/H2O ~108.0%  
 Column(1) Flow: 1.20 Column(2): 0.00 ml/min Interface Temp: 250  
 Ramp Criteria:  
 Ion Focus maximum 90 volts using ion 502; Electron Multiplier Gain 100464.862  
 Repeller maximum 35 volts using ion 219; Gain Factor 1.0046  
 Mass Gain Values(Scan Speed): 23(3) 34(2) 41(1) 66(0) 118(FS1) 126(FS2)

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset	135.7	135.7	135.7	135.7	135.7	135.7	135.7
Entrance Lens Offset	13.2	13.2	13.2	13.2	13.2	13.2	13.2

# How Can I Make My Analysis Go Faster?

# Variables for Speeding Up Analysis

- Carrier gas: Type and linear velocity
- Liquid phase
- Temperature programming
- Shorten column length
- Decrease film thickness
- Decrease internal diameter
- Use backflush to remove “gunk”



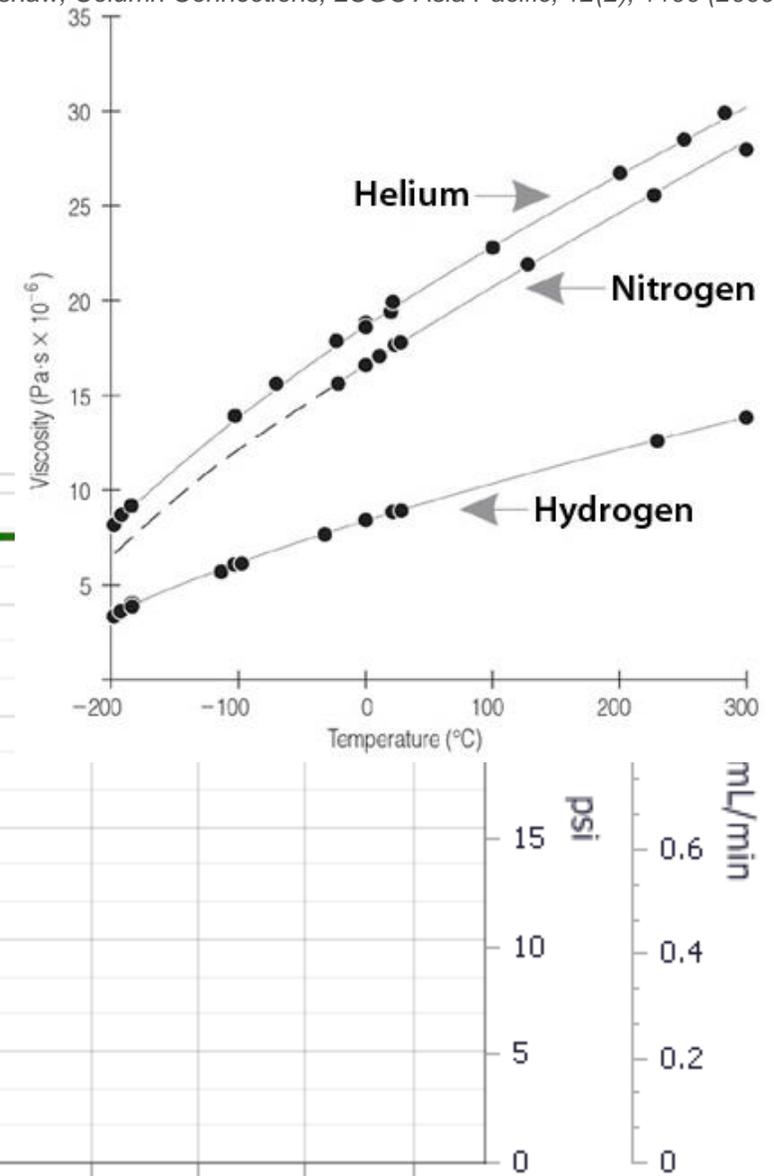
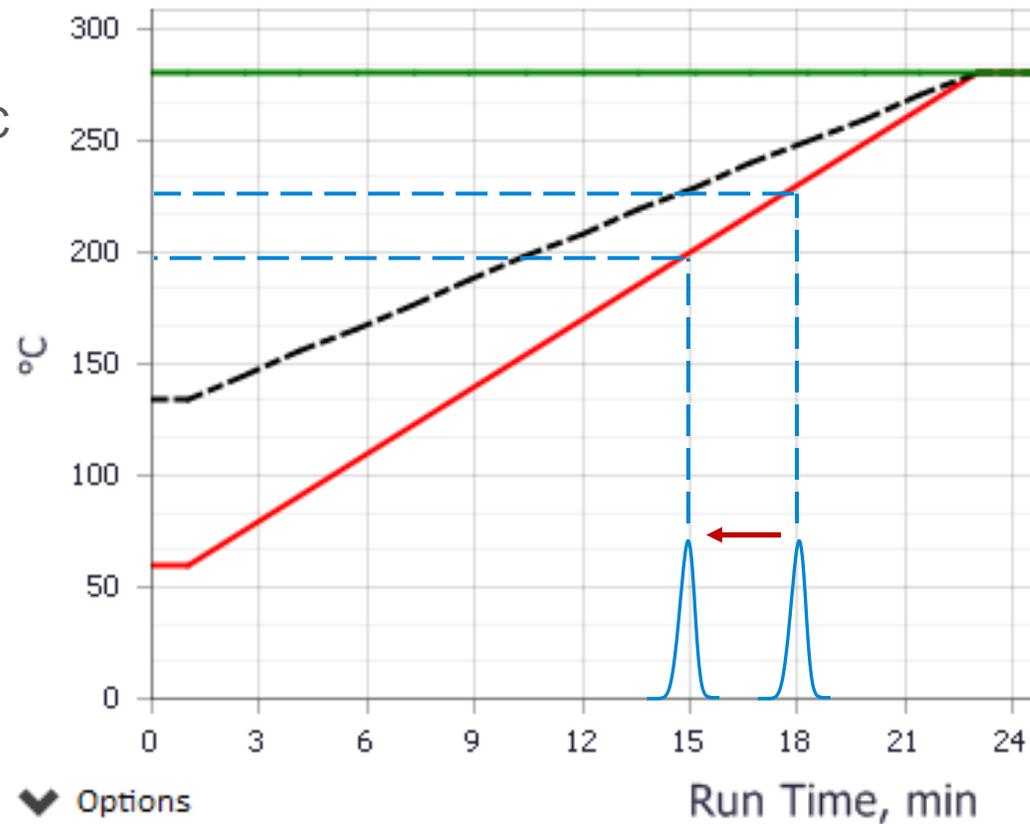
# Temperature and Selectivity

The retention time isn't the only thing that changes.

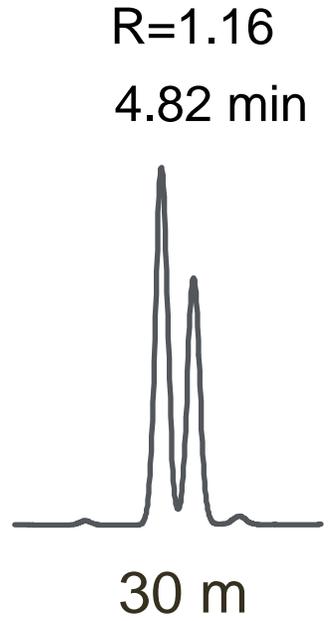
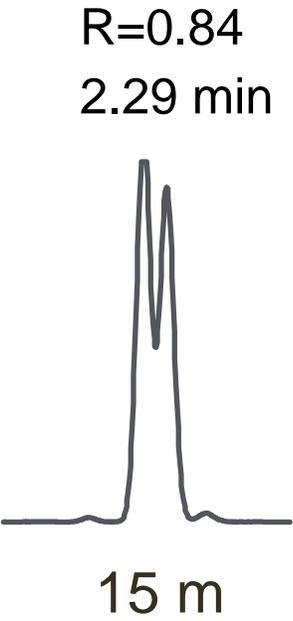
Eluting at 200 °C versus 225 °C

Decrease in gas viscosity

Change in thermodynamics



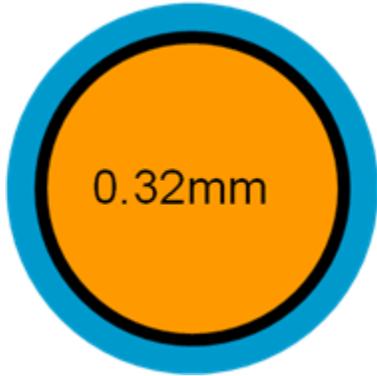
# Length Versus Resolution and Retention



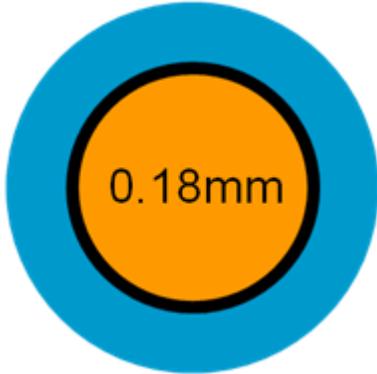
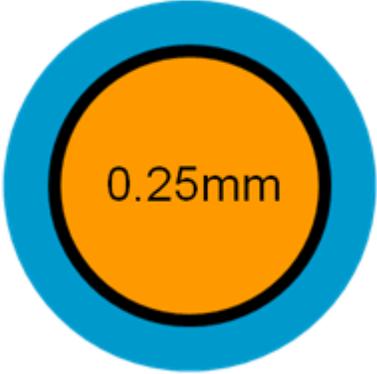
# High Efficiency Columns

High efficiency  
Fast run times

Improved separation  
More analyses/instrument

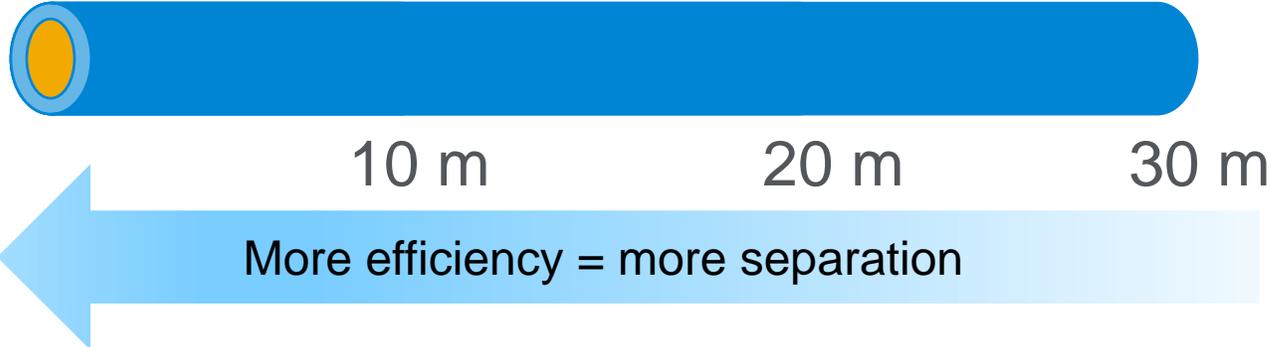


Regular internal diameters

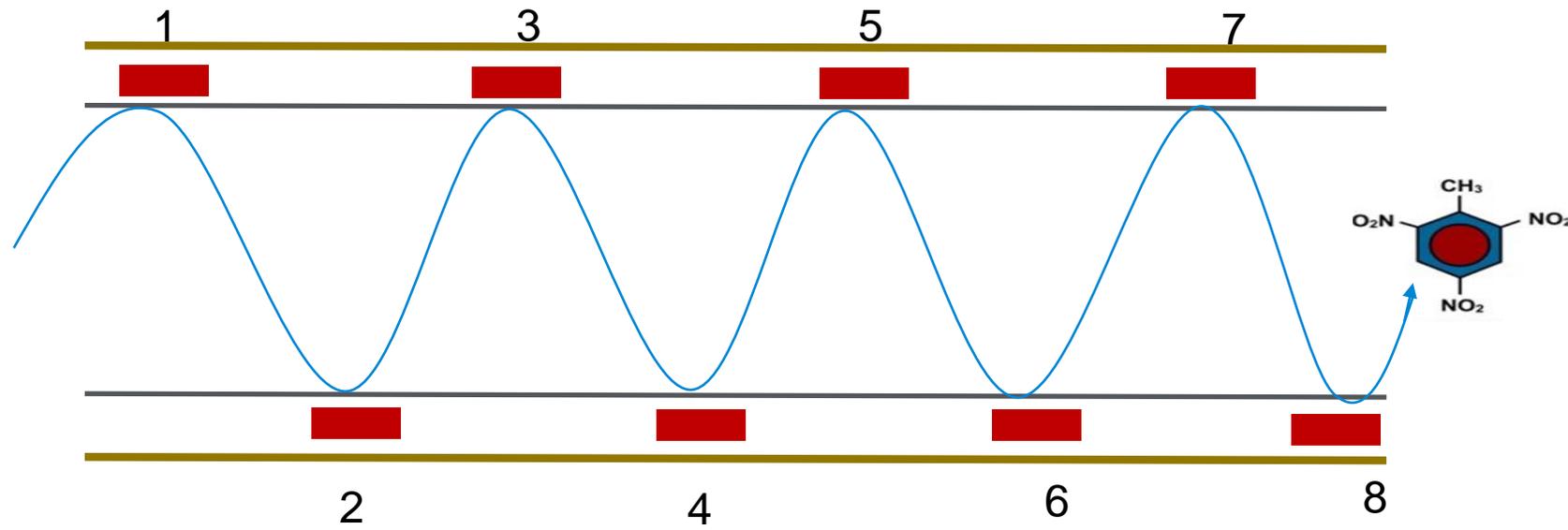


Agilent high efficiency

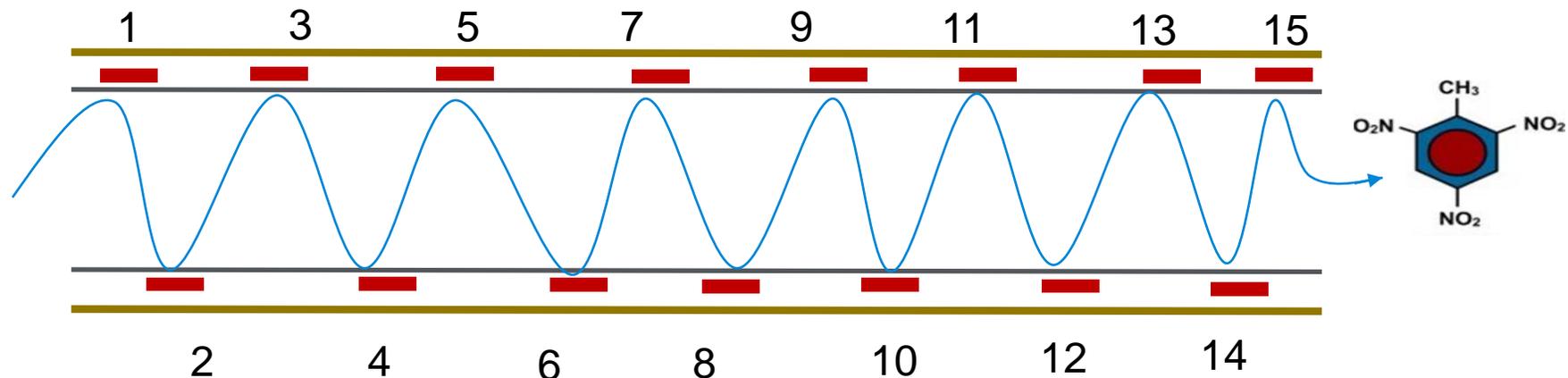
0.18 mm internal diameter  
Short 10/20 m column length



# Decrease Column id and Increase Column Interactions



Smaller I.D. columns provide more resolution for the same column length

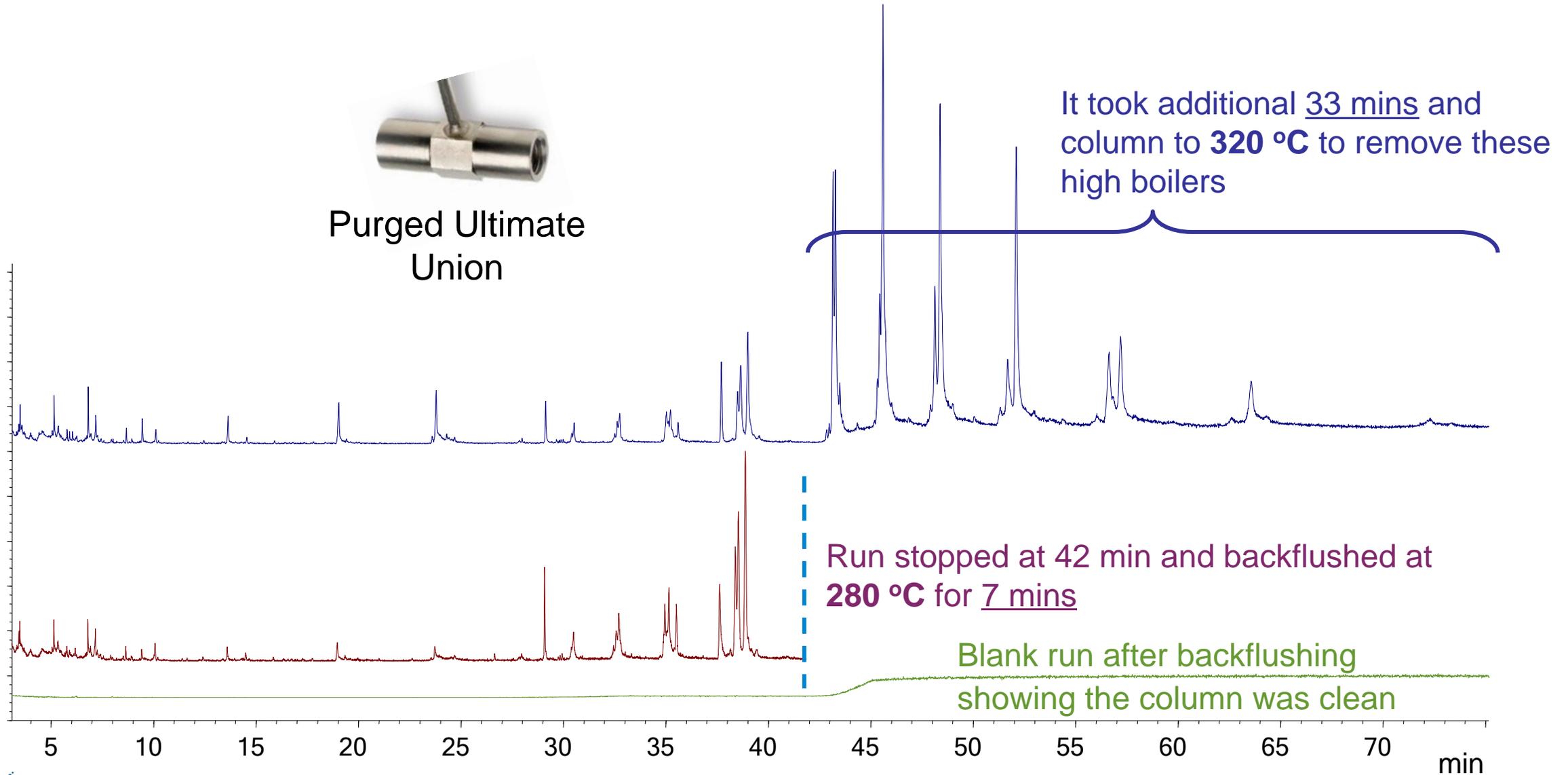




# Why Use Backflush?



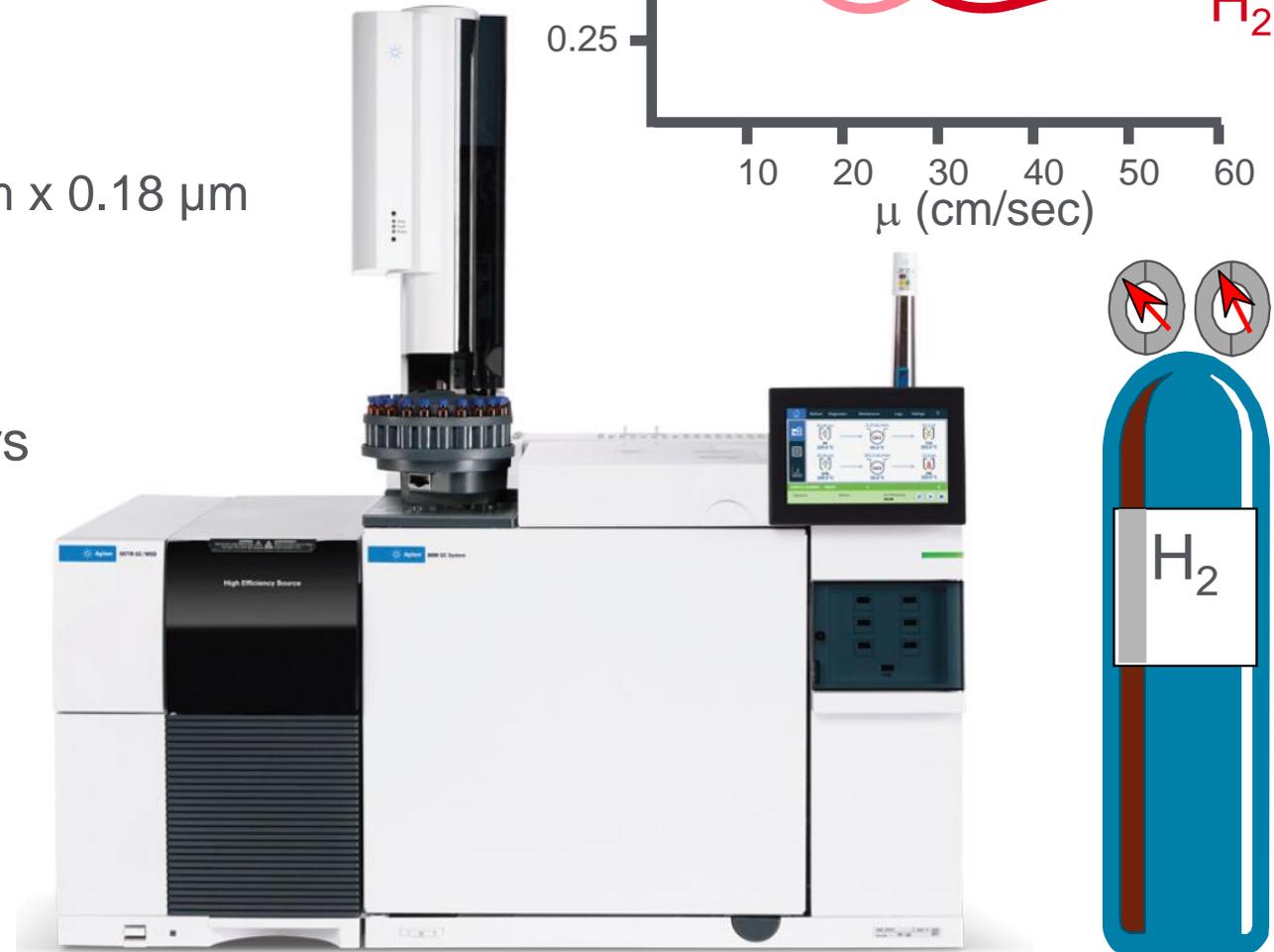
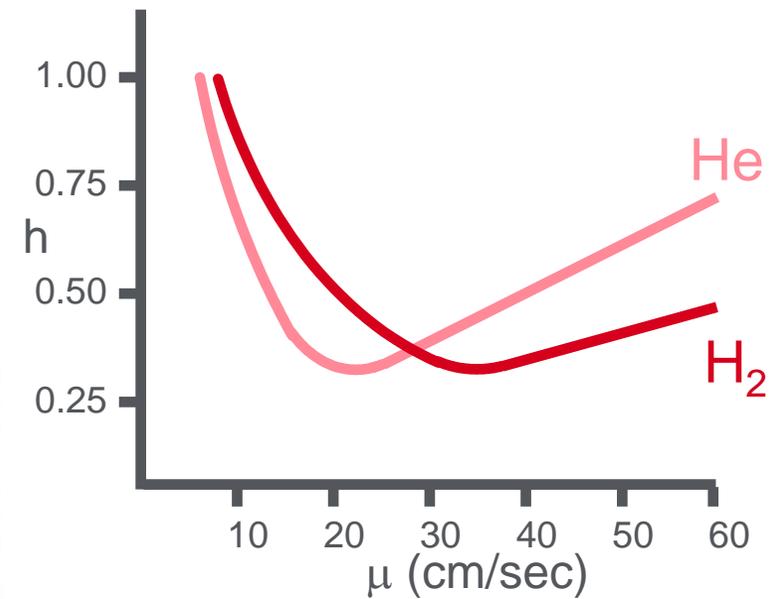
Purged Ultimate Union



# Should you Use Helium or Hydrogen as a Carrier Gas with Mass Spec?

# Why would I want to use H<sub>2</sub>?

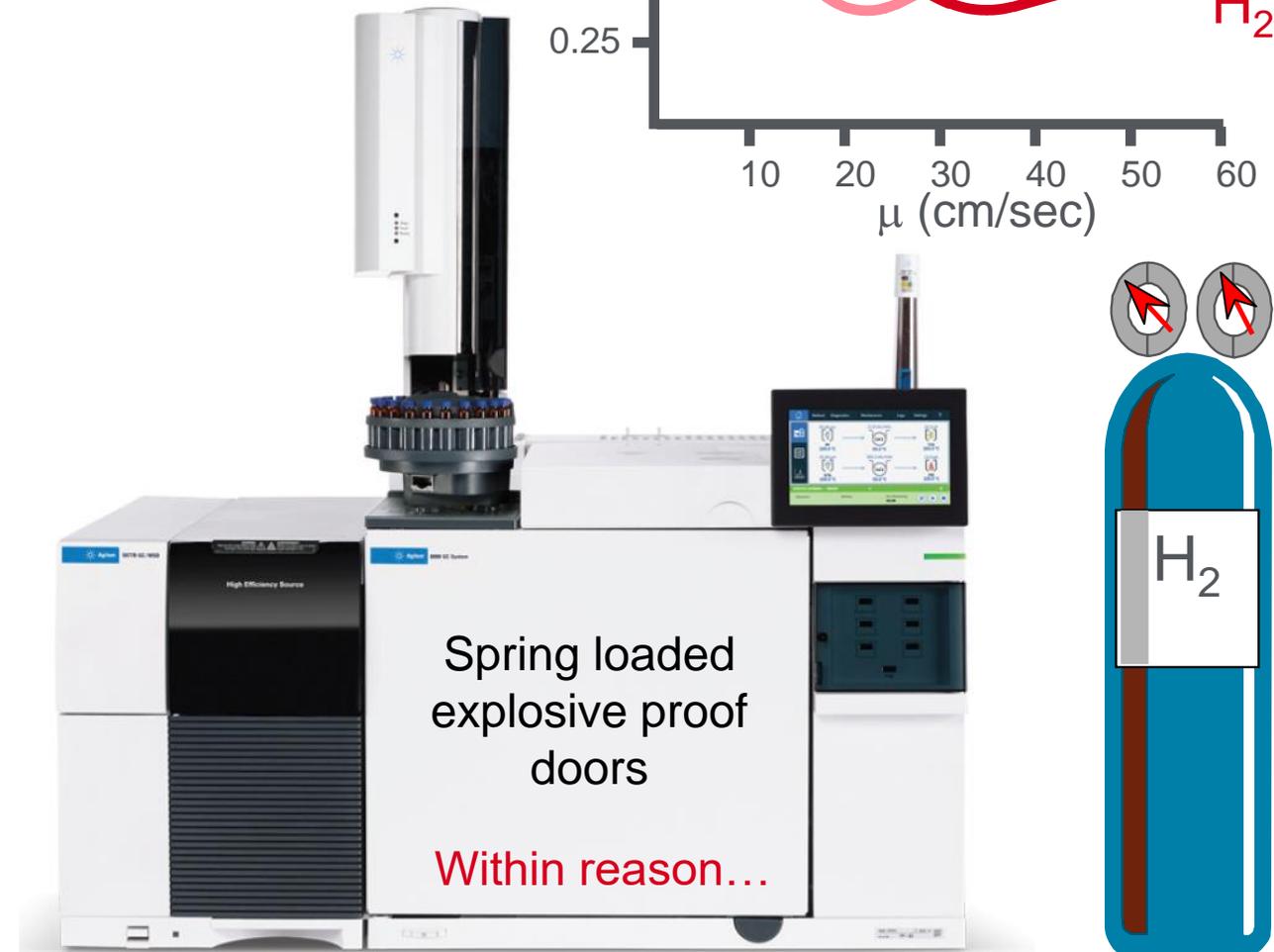
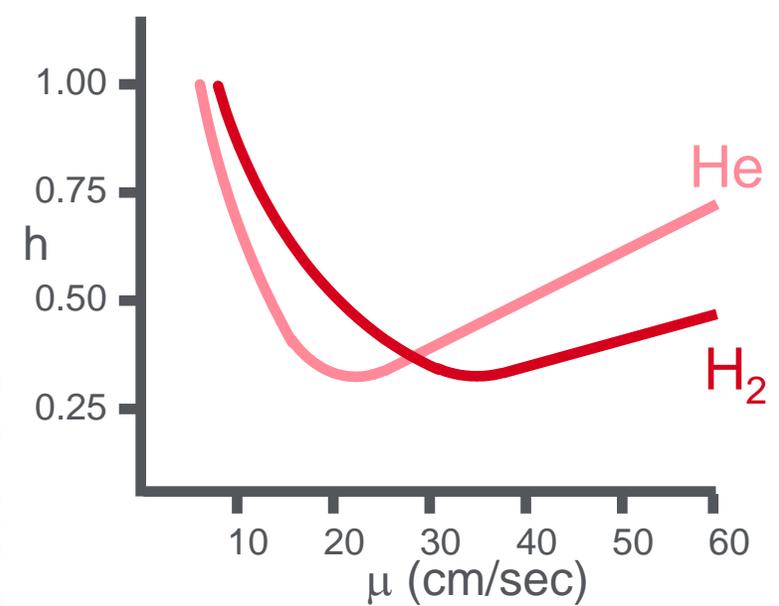
- Faster analysis
- Lower temperature separation possible
- Move to “more efficient” columns
  - 30 m x 0.25 mm x 0.25 μm → 20m x 0.18 mm x 0.18 μm
- H<sub>2</sub> available “on demand”
- Hydrogen generator
- H<sub>2</sub> already used for FID and other detectors



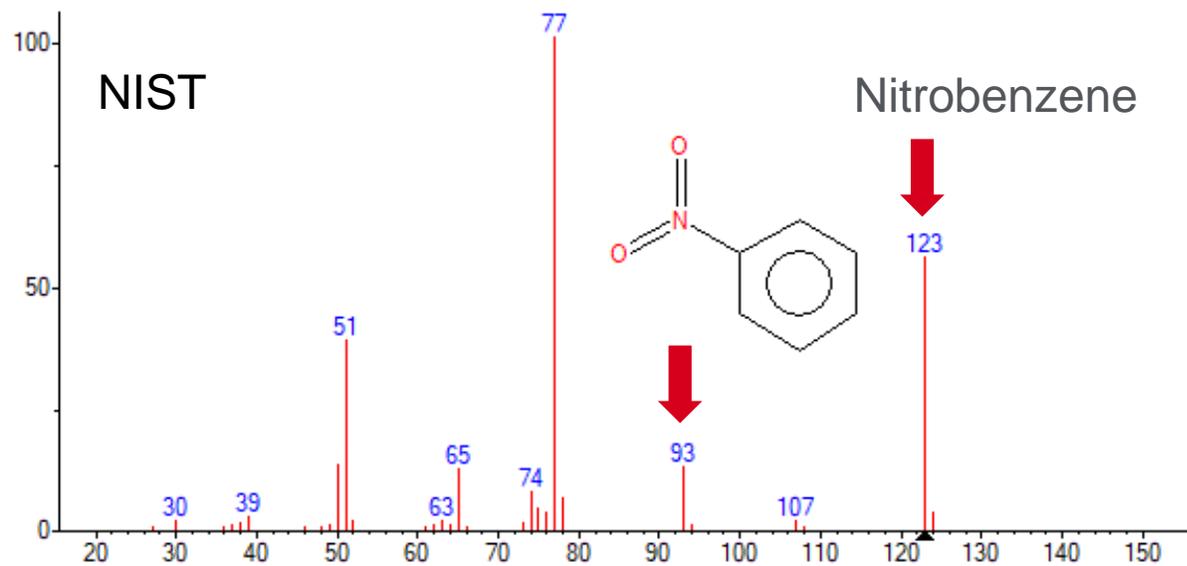
# Notes on Using Hydrogen with Your Mass Spectrometer

## Hydrogen is Safe

- ✓ Hydrogen is extremely diffusive
- ✓ Hard to reach explosive level 4%
- ✓ Flow regulator has a safety shut down
- Still needs Gas Clean filter
  - Add a large moisture filter
- Decrease flow for source
  - Inert 0.7 mL/min
  - HES 0.5 mL/min
- Mass ratios may be different
  - Tune will look different
- Avoid using DCM and Carbon Disulfide as a solvent
- Hydrogen is *not* inert
  - Start inlet cool to decrease interactions

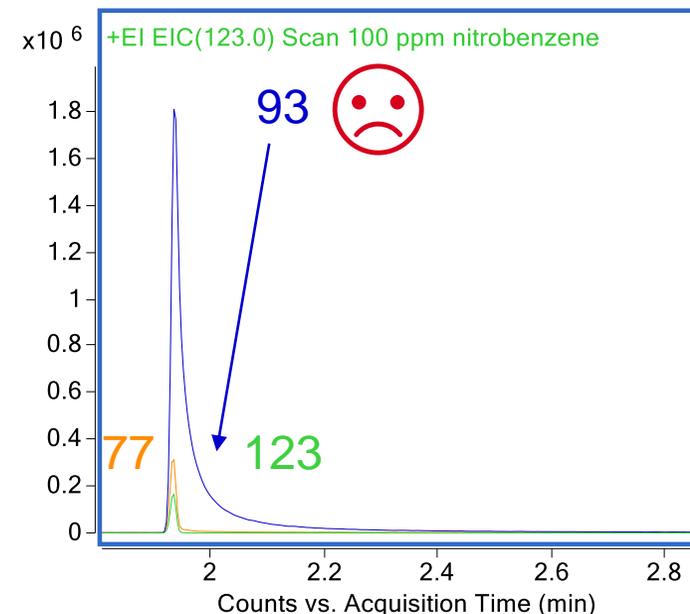
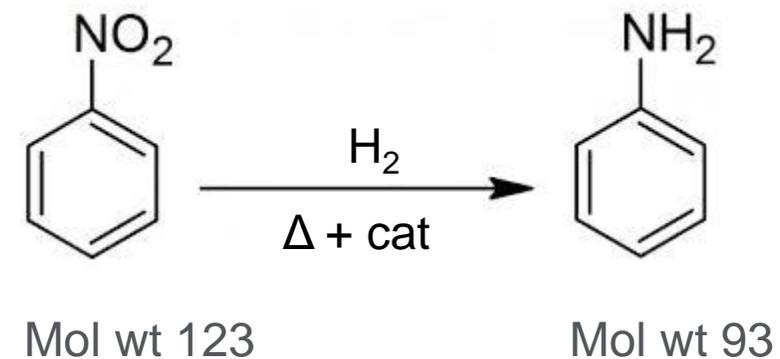
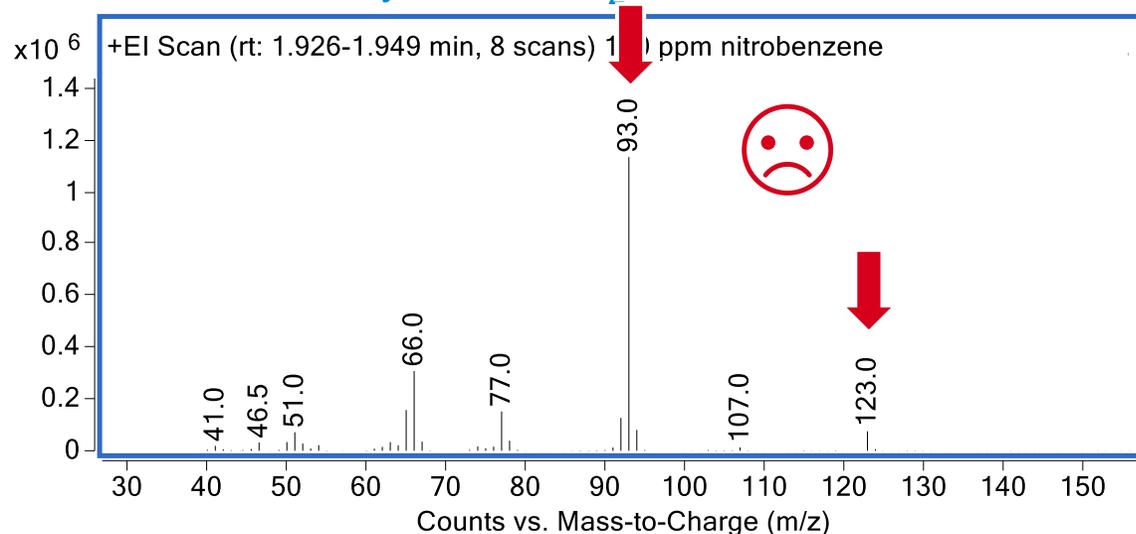


# H<sub>2</sub> + GC/MS is Not for Every Application



(replib) Benzene, nitro-

## GC/MS analysis with H<sub>2</sub> carrier



# Recommendations for H<sub>2</sub> GC/MS

## DO

- Use a 9 mm extraction lens
- Column flow rate: 0.5 – 1.2 mL/min
- Switch to a “more efficient” column
  - 30 m x 0.25 mm x 0.25 μm → 20 m x 0.18 mm x 0.18 μm
- Use gas filters, especially with H<sub>2</sub> generator!
- Allow the system to bake out longer
  - May require running system overnight with filaments on

## It's best to avoid

- Chlorinated solvents and hot inlets
  - DCM + hot inlet + H<sub>2</sub> + (tiny bit of H<sub>2</sub>O) = HCl
- Heavily chlorinated compounds
  - Dechlorination potential
- Nitrocompounds (such as nitrobenzene)
  - Hydrogenation

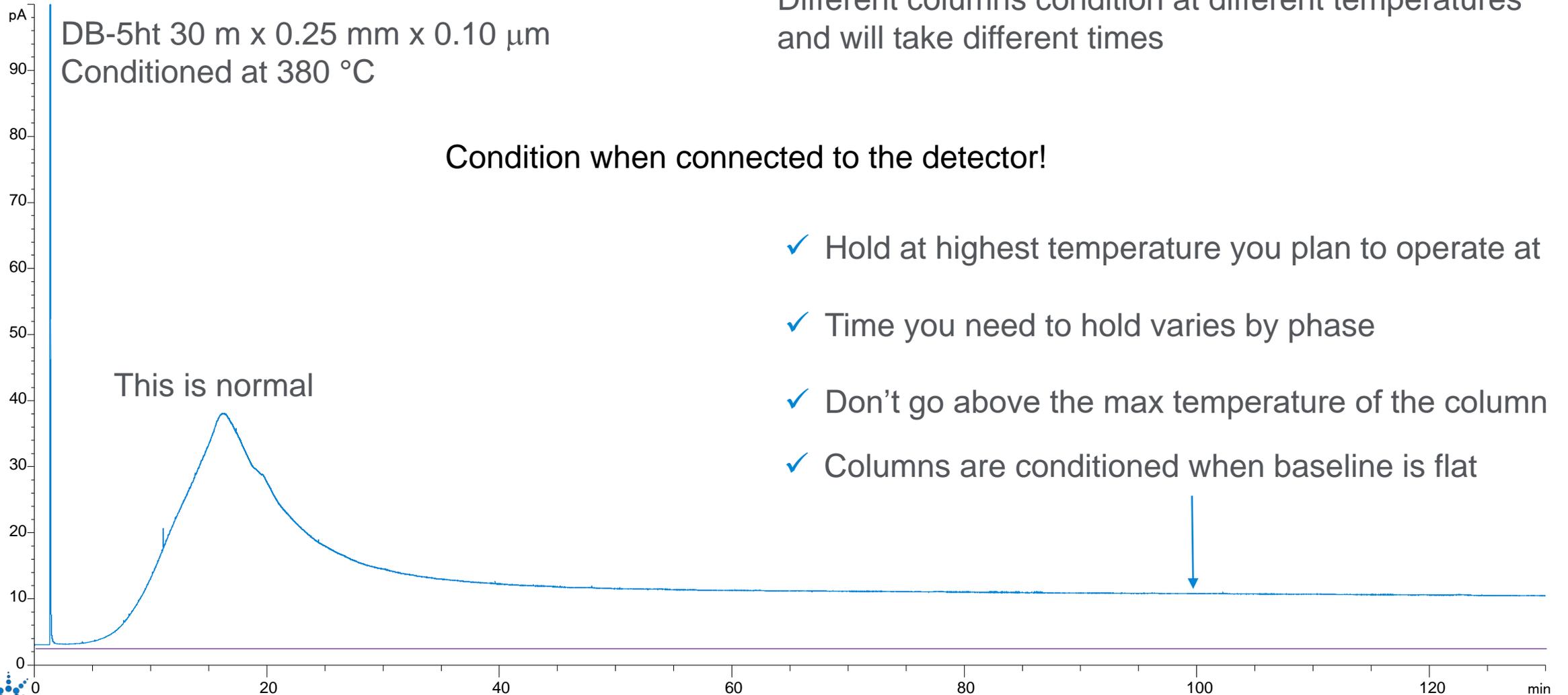
Where can I easily use H<sub>2</sub> GC/MS?

Hydrocarbon or PAH analyses



# How Long Should I Condition my Column For?

# How to Condition your Column





# Pulsed splitless optimization and why use it?

# Why would I use pulsed splitless injections?

1. Pressure pulse reduces sample expansion volume, and transfers analytes to the column faster

## When do I use it?

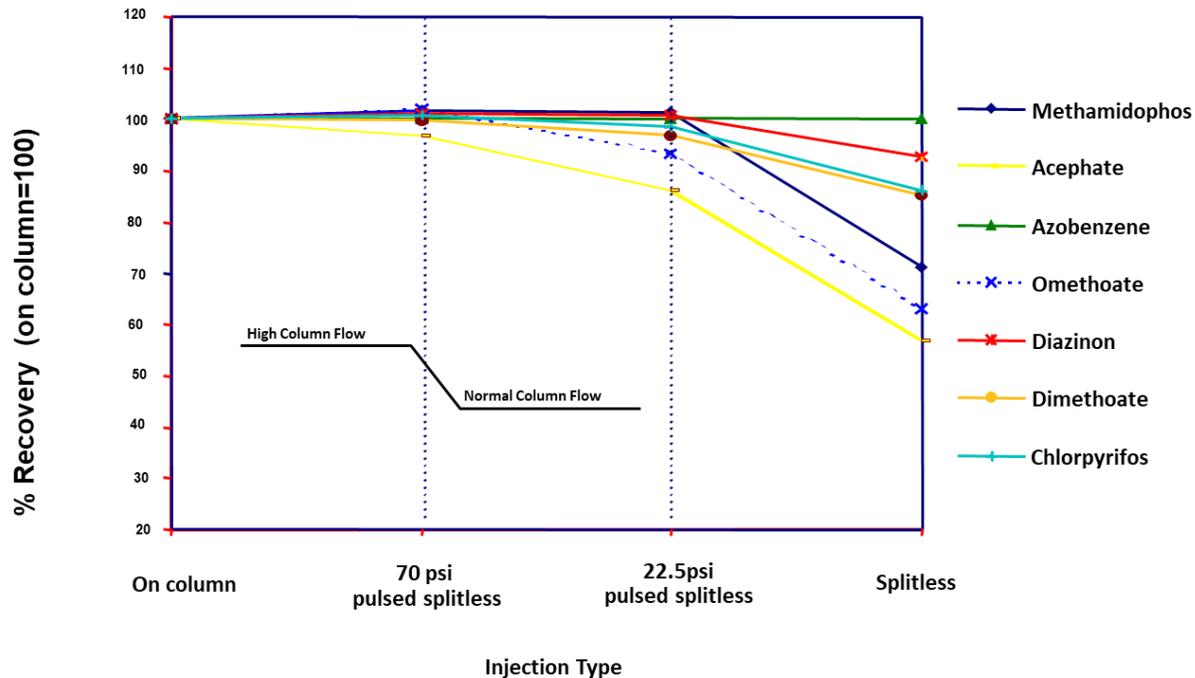
- Trace level analyses
  - When you want analytes out of liner fast
2. Sample containment more critical than in split injection
  3. Sharper peaks than in traditional splitless injection
  4. Two new parameters to set:
    - Pulse pressure
    - Pulse time

## Typical starting point:

- **Pulse pressure = double the normal inlet pressure**
- **Can set pulse time and purge time to same value (e.g. 0.75 min)**

# Benefits of Pulsed Splitless Injections

% Recovery of Each Labile Pesticide Relative to Cool On-Column Injection



What applications have you used pulsed splitless injections in?

- Environmental analyses, e.g. EPA 8270
- Pesticides analyses
- PAH analyses

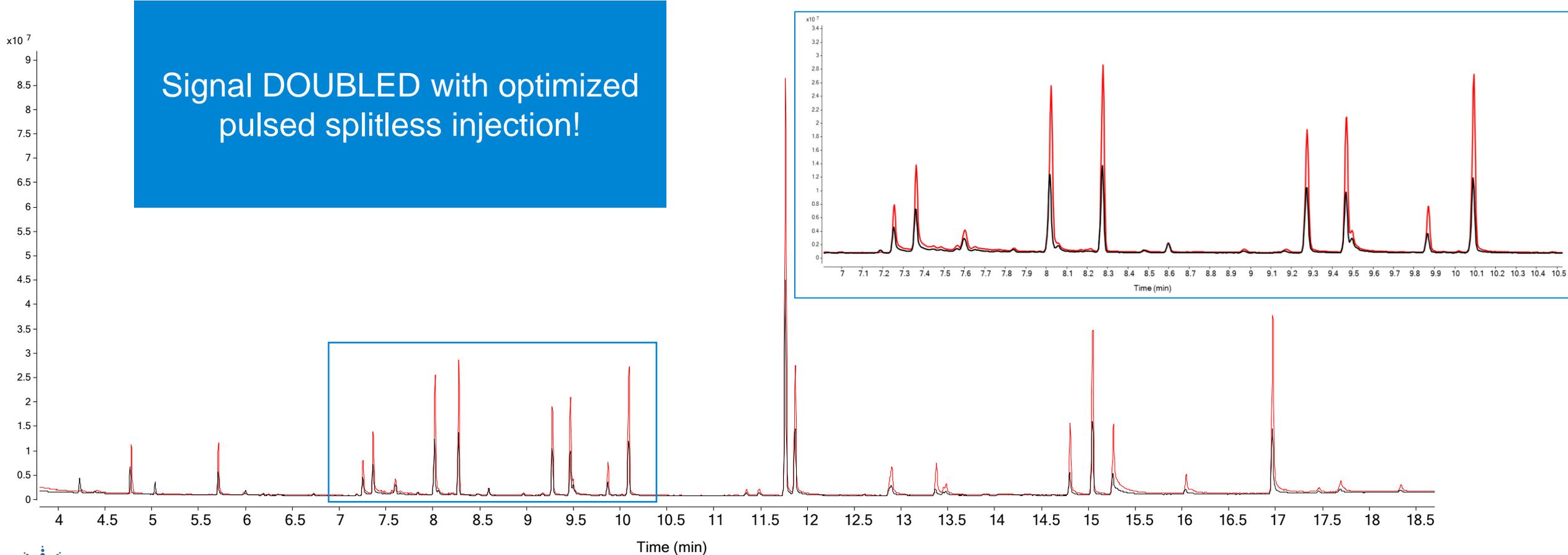
# Can a pulsed splitless injection really be that different?

## Pesticides analysis

Splitless

Pulsed splitless: Pulse 50 psi until 0.75 min; Purge Flow to Split Vent: 50 mL/min at 0.7 min

Signal DOUBLED with optimized pulsed splitless injection!



# For More Information

**Agilent CrossLab**  
From Insight to Outcome

## The Right Connections Make All the Difference

Agilent gas chromatography connection supplies



 **Agilent**

Publication Number 5991-4215EN

PRODUCTS APPLICATIONS & INDUSTRIES TRAINING & EVENTS SERVICES SUPPORT & RESOURCES ORDER CENTER

Home > Products > GC Columns > GC Column Selector Tool

## GC COLUMN SELECTOR TOOL

Welcome to Agilent  
GC Column Selector Tool

A series of tools to help you navigate to the best column choice for your application. Guidance can be by manufacturer's part number cross-reference, desired phase chemistry, USP designation, regulatory method, or by application.

Contact Us

Select an option to find your GC Column

 PART NUMBER SEARCH	 PHASE CHEMISTRY
 AGENCY METHODS	 APPLICATION
 USP DESIGNATION	

GC Column Selector | Agilent

# 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 USA and Canada 8–5, all time zones**



[gc-column-support@agilent.com](mailto:gc-column-support@agilent.com)

[lc-column-support@agilent.com](mailto:lc-column-support@agilent.com)

[spp-support@agilent.com](mailto:spp-support@agilent.com)

[spectro-supplies-support@agilent.com](mailto:spectro-supplies-support@agilent.com)

[chem-standards-support@agilent.com](mailto:chem-standards-support@agilent.com)