

Ask the Agilent Experts

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

Vanessa Abercrombie
Mark Sinnott

How Long Should My Liner Last?

That's a Tough Question...

What is the matrix (or matrices)?

Are QC samples being used?

What are the analytes?

What is the sample preparation?

What liner is being used?

Deactivated?

Barrier (frit or wool)?

What are the injection parameters?



What is a Barrier (Glass Wool or Frit) Used For?

Filtration

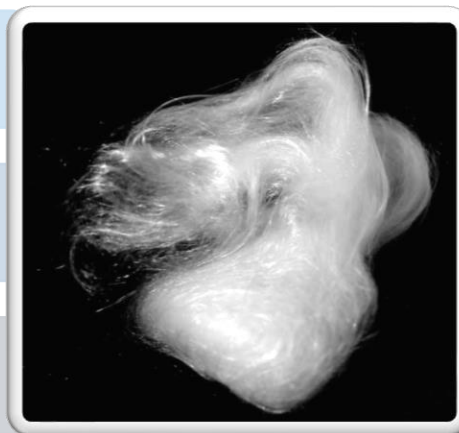
- Prevents nonvolatile matrix from entering column

Vaporization

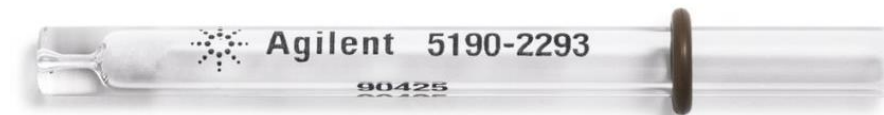
- Provides volatilization surface for liquid injections, promotes mixing with carrier gas

Needle wiping

- Increases reproducibility by wiping needle after injection



Frit



Glass wool

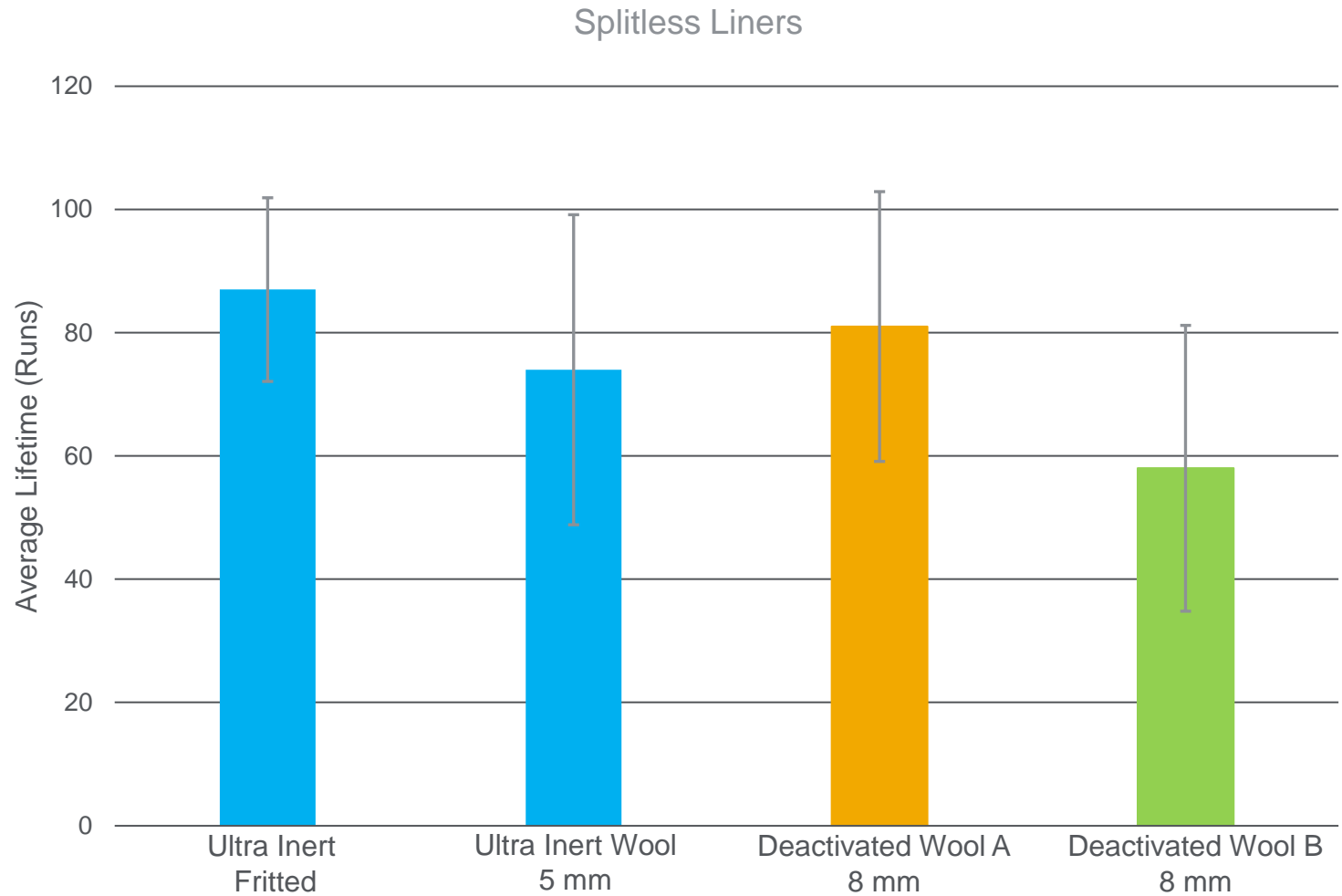


When should I use a liner with a frit or glass wool?

- Complex (dirty) matrices
- Viscous matrices



What Matters? ...Barrier? ...Size of Barrier? ...Deactivation?



Frit



Glass wool



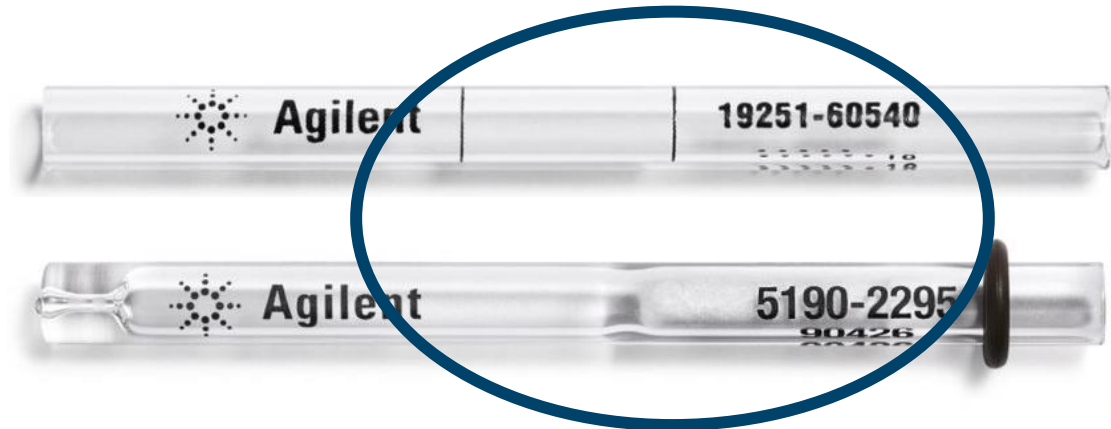
...But How Do I Pick the Right Liner?

Splitless injections

- Ultra Inert deactivation
- Bottom taper
- Barrier/wool not required
- Barrier (wool, dimpled, or frit)
 - Positioned at bottom

Split injections

- Ultra Inert deactivation
- Straight or tapered
- Barrier (wool or frit)
 - Positioned upper-middle



How Often Should You Tune Your Mass Spectrometer?

Tuning Frequency



Follow lab SOP



Follow (regulated) method criteria

A calendar grid showing a 5-day cycle. The days are labeled: Monday - 1, Tuesday - 2, Wednesday - 3, Thursday - 4, Friday - 5. The text "QuickTune or Tune Eval" is written in green on the Thursday and Wednesday cells of each cycle.

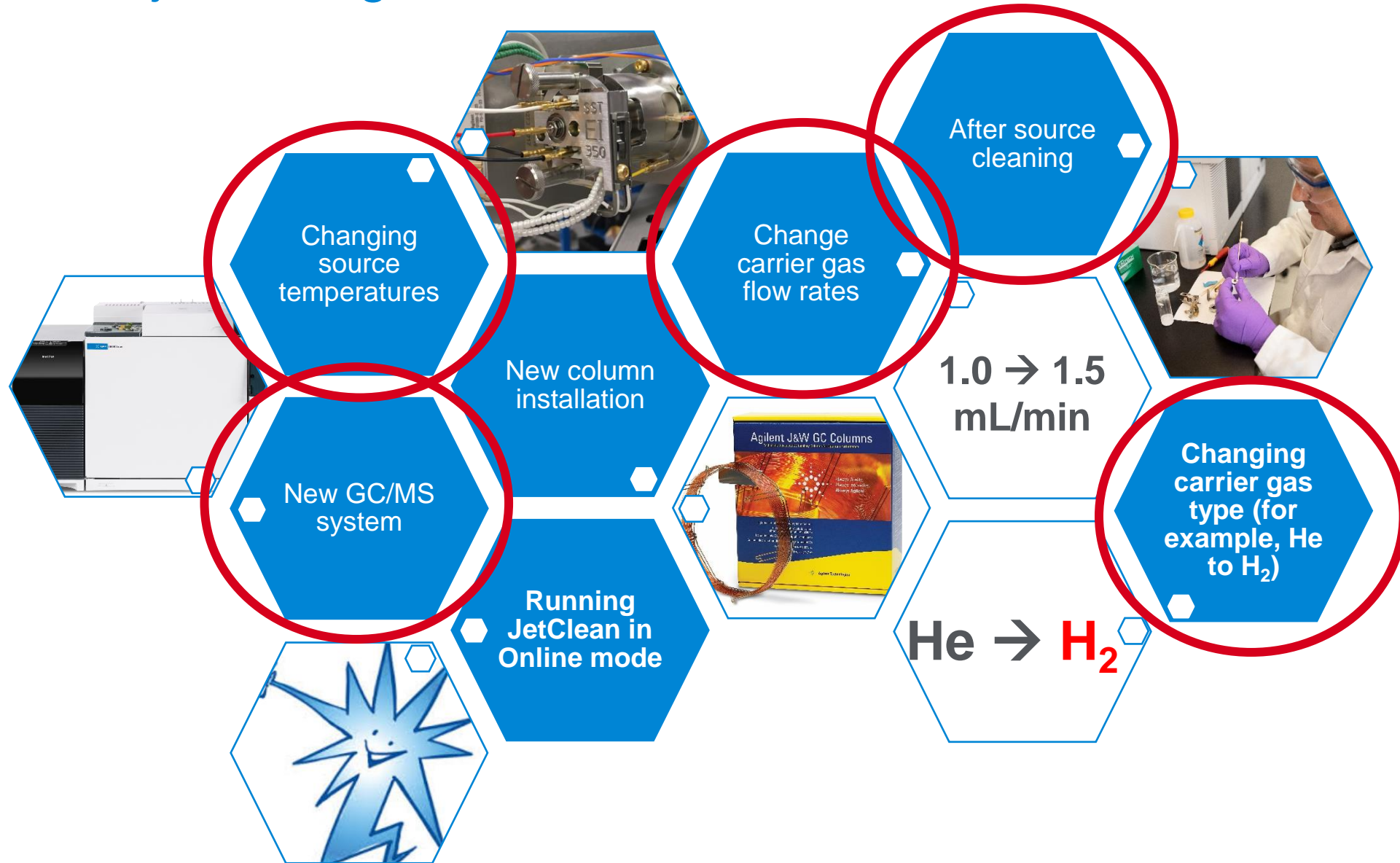
Monday - 1	Tuesday - 2	Wednesday - 3	Thursday - 4 QuickTune or Tune Eval	Friday - 5
Monday - 8 QuickTune or Tune Eval	Tuesday - 9	Wednesday - 10	Thursday - 11	Friday - 12
Monday - 15	Tuesday - 16	Wednesday - 17 QuickTune or Tune Eval	Thursday - 18	Friday - 19
Monday - 22	Tuesday - 23	Wednesday - 24	Thursday - 25 QuickTune or Tune Eval	Friday - 26
Monday - 29	Tuesday - 30	Wednesday - 1	Thursday - 2	Friday - 3

“It depends” (but not daily)



Major changes

What is a Major Change?



How Do I Determine That I Need to Tune?

1. Have a Baseline

System start-up




- Tune system
- Run Tune Evaluation
- Run QC and cal samples

2. Review QC and CCV

What are peak responses doing?

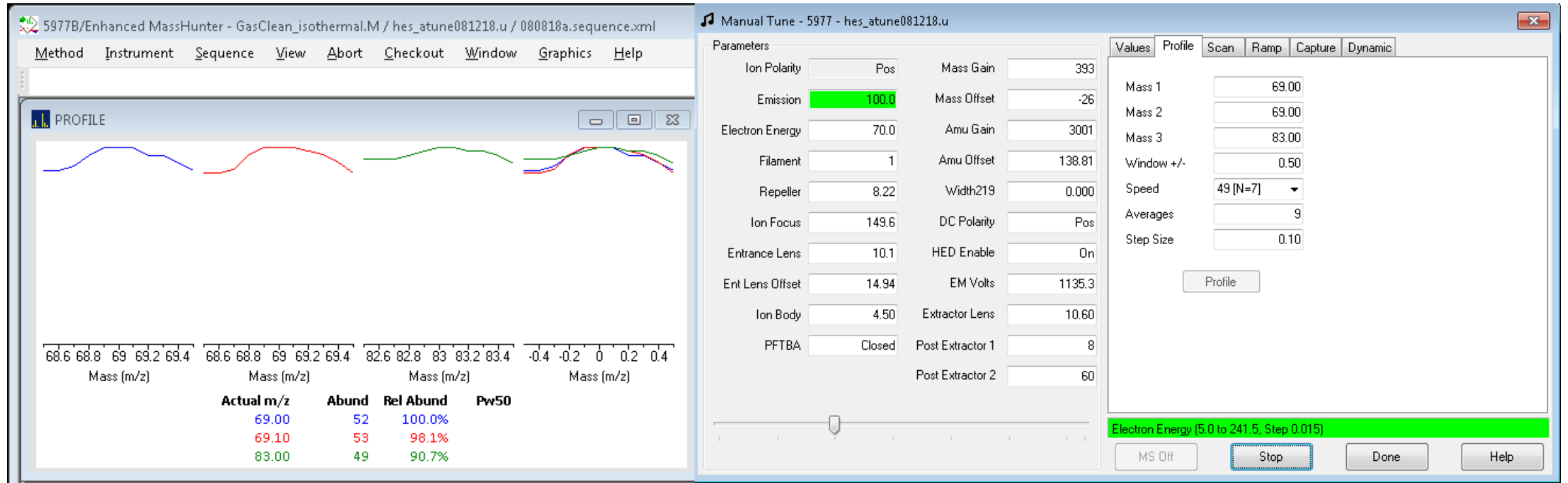
3. Keep records

Build a profile of tuning occurrence

Monday – 1	Tuesday – 2	Wednesday – 3	Thursday – 4	Friday – 5
 New system start-up			GC maintenance	
Tune Eval + update gain coeff.		GC maintenance		Tune Eval + QuickTune
	GC maintenance + Tune Eval + Quick Tune	 New tune		
				Tune Eval + update gain coeff.
GC maintenance				
		Failed ISTD areas = clean source	 Clean source = new tune	

I Must Tune to Check for Leaks, Right?

Using electronics duster to find system leaks with Manual Tune



Navigate to MSD Manual Tune in the Data Acquisition

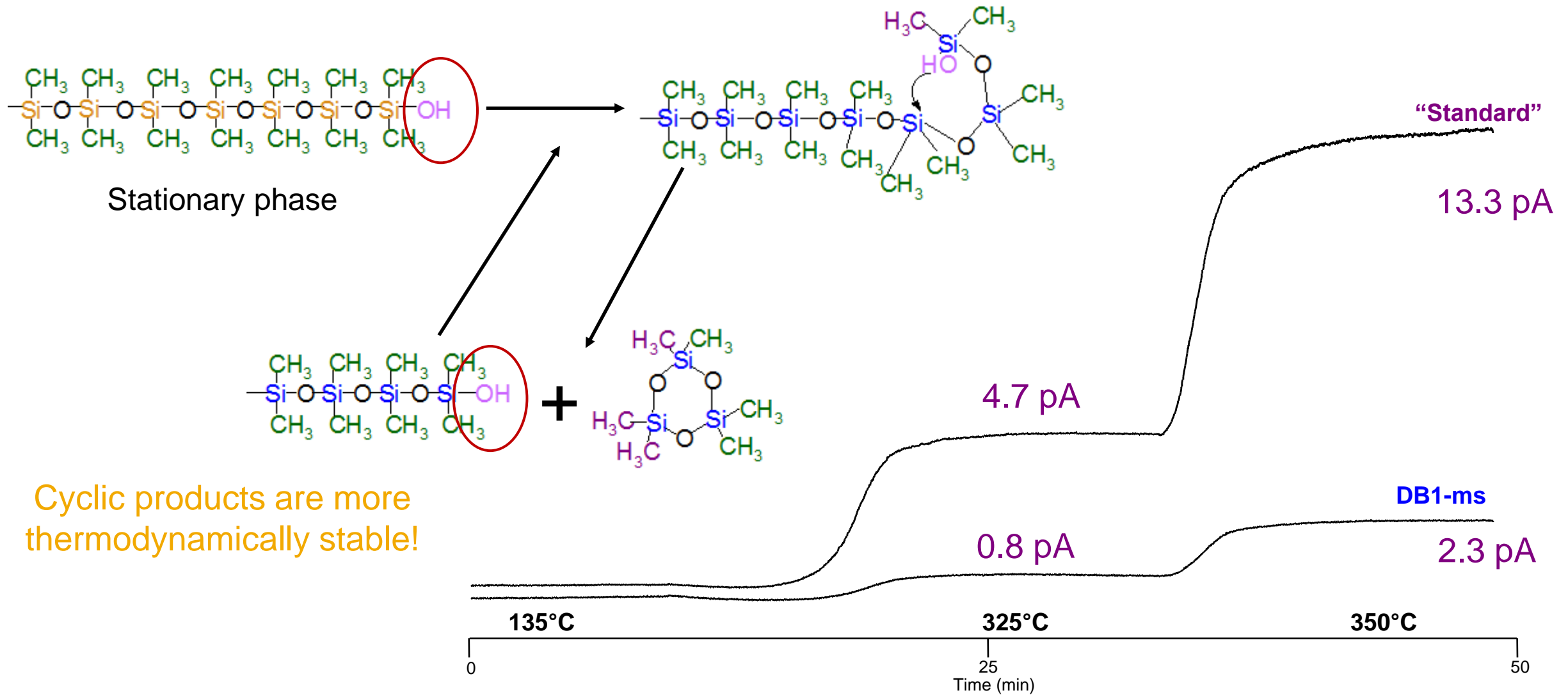
- Instrument > Edit Tune Parameters

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

Why Does the Baseline Increase at the End of the Run?

What Column Bleed Looks Like

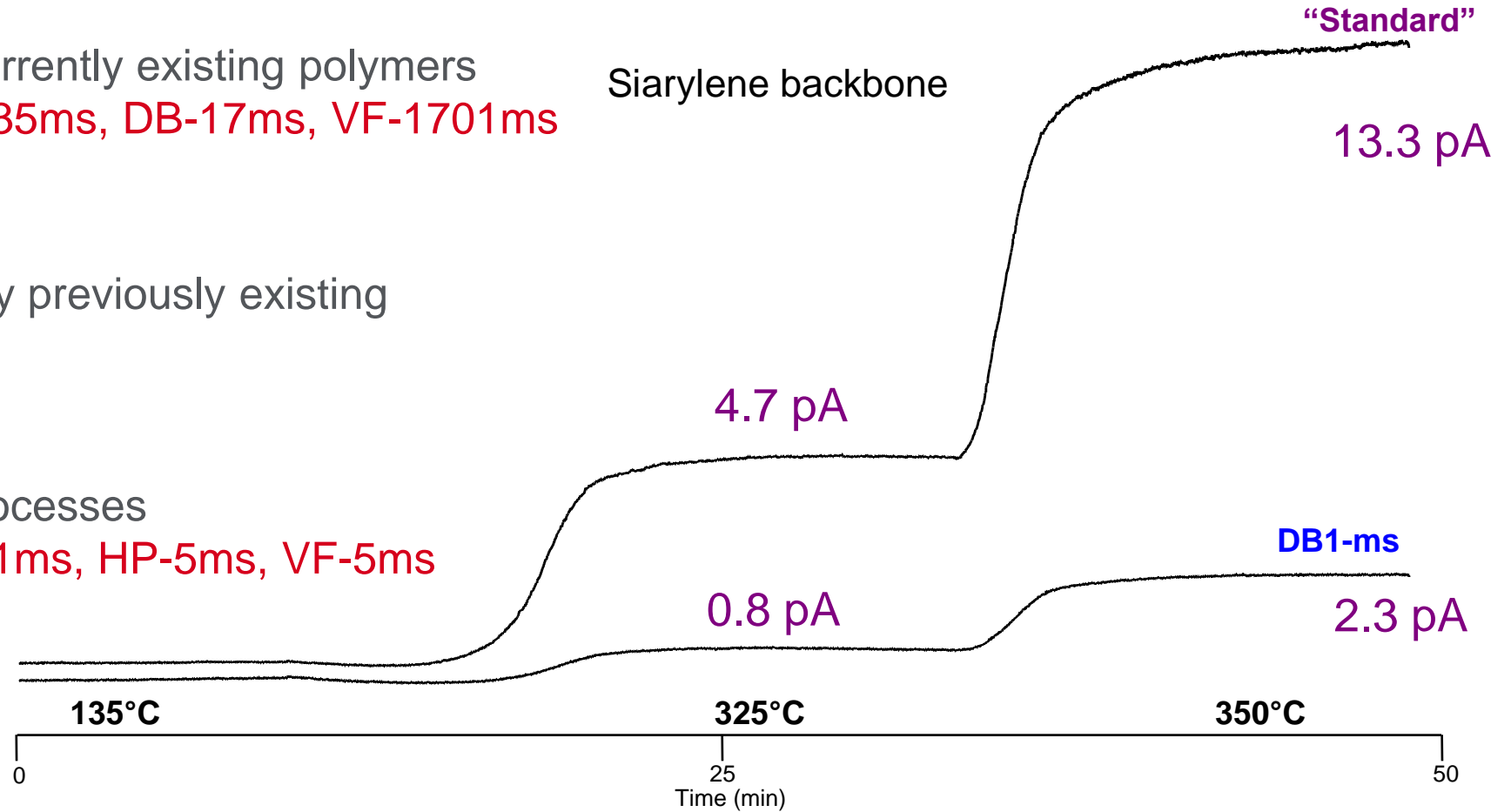
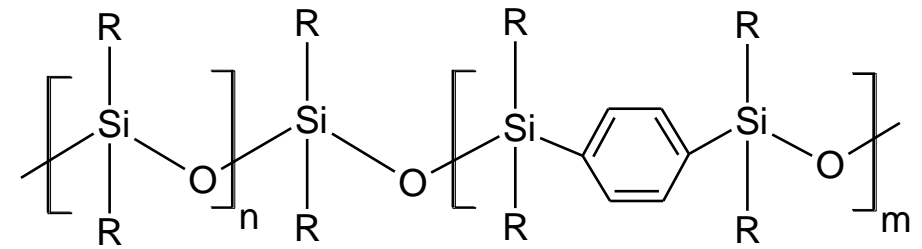


[How Does Bleed Impact GC/MS Data and How Can It Be Controlled? \(agilent.com\)](http://www.agilent.com)

Columns: 30 m x 0.25 mm I.D., 0.25 μm film

Low Bleed Phases

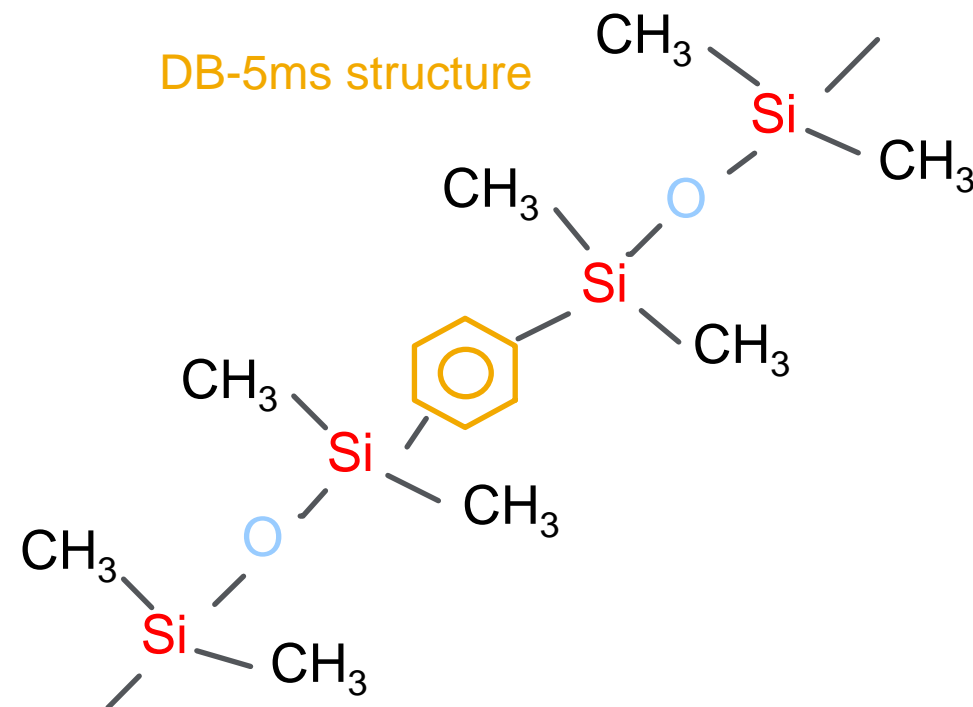
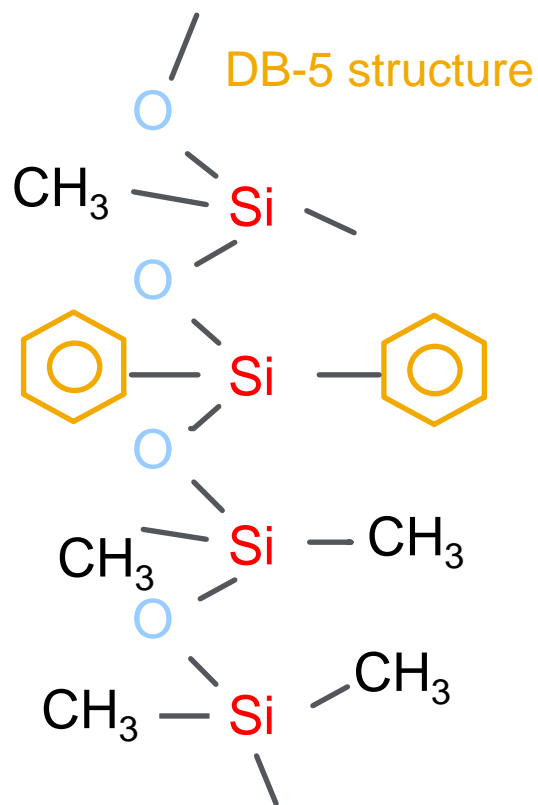
- Phases tailored to 'mimic' currently existing polymers
Examples: DB-5ms, DB-35ms, DB-17ms, VF-1701ms
- New phases unrelated to any previously existing polymers
Examples: DB-XLB
- Optimized manufacturing processes
Examples: DB-1ms, HP-1ms, HP-5ms, VF-5ms



Columns: 30 m x 0.25 mm I.D., 0.25 μ m film

[How Does Bleed Impact GC/MS Data and How Can It Be Controlled? \(agilent.com\)](http://agilent.com)

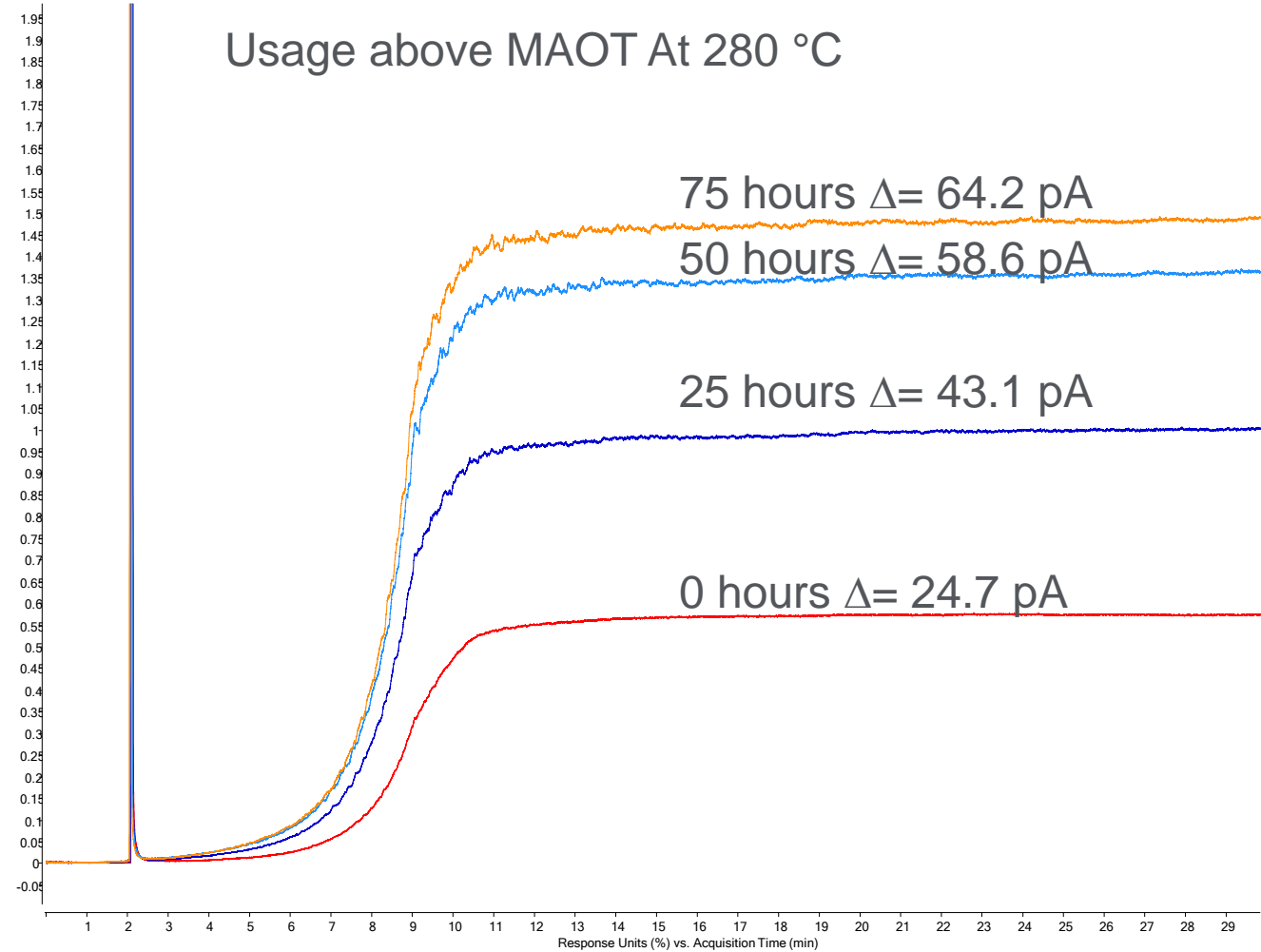
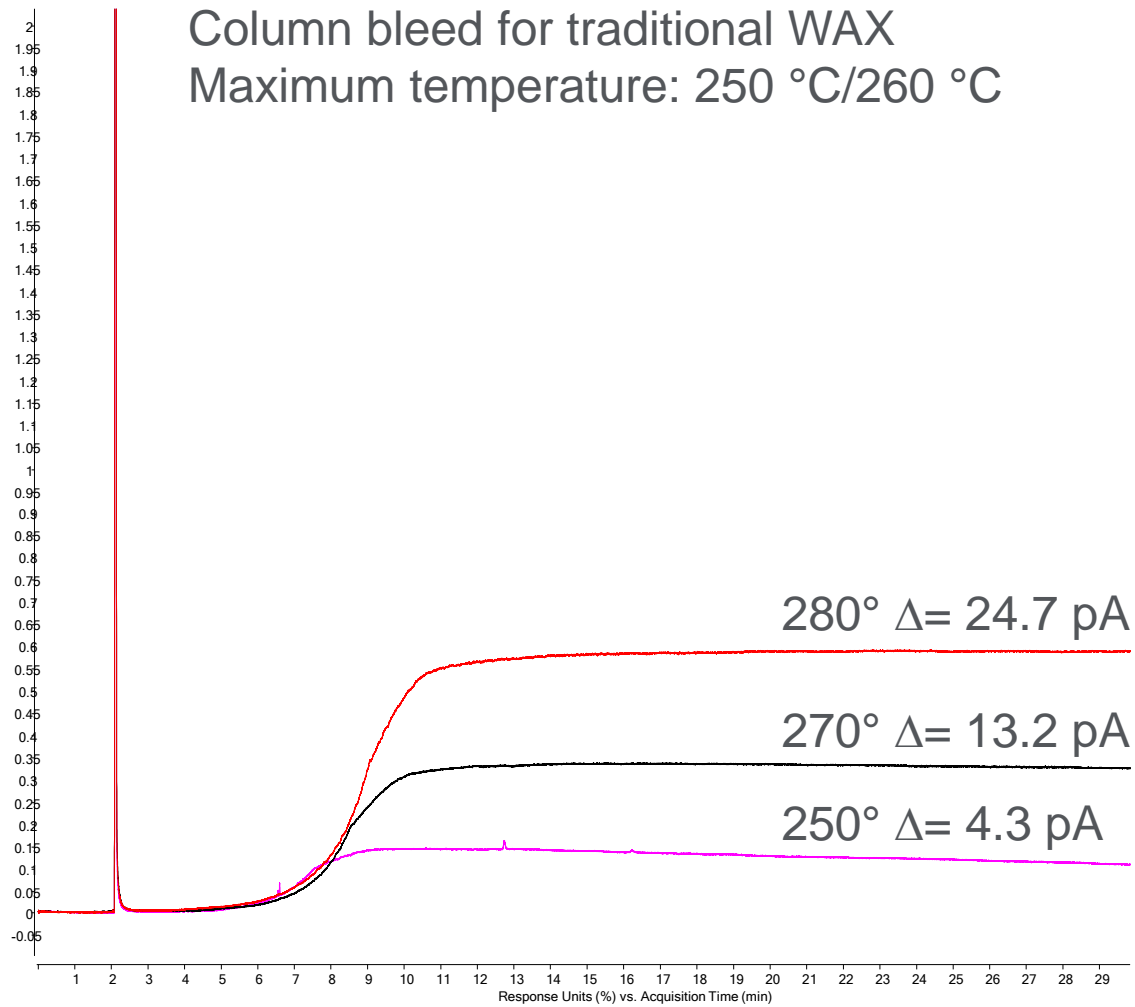
Agilent J&W DB-5ms Structure



DB-5ms

- Increased stability
- Different selectivity
- Optimized to match DB-5 as much as possible

Traditional WAX and Going Above the MAOT



What is a Bleed Problem?

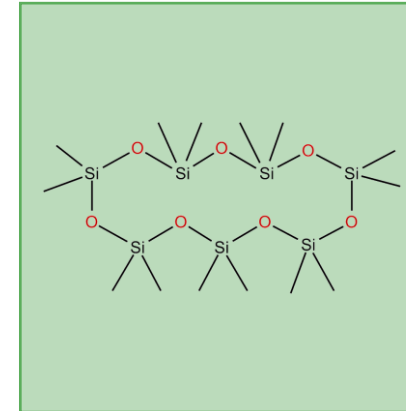
An abnormal elevated baseline at high temperature

It is not:

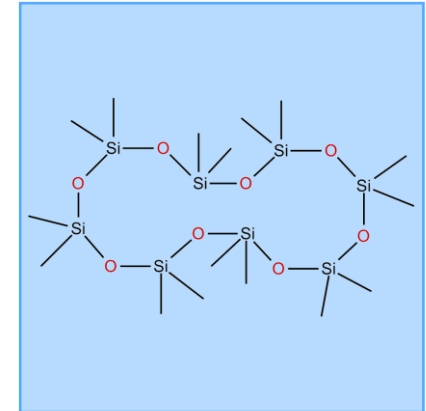
A high baseline at low temperature

Wandering or drifting baseline at any temperature

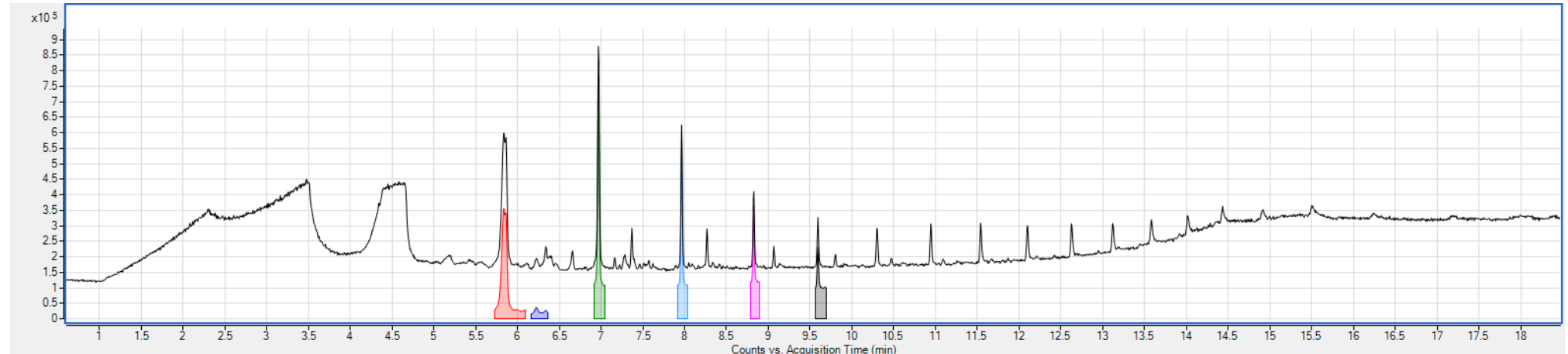
Discrete peaks



Tetradecamethyl
cycloheptasiloxane



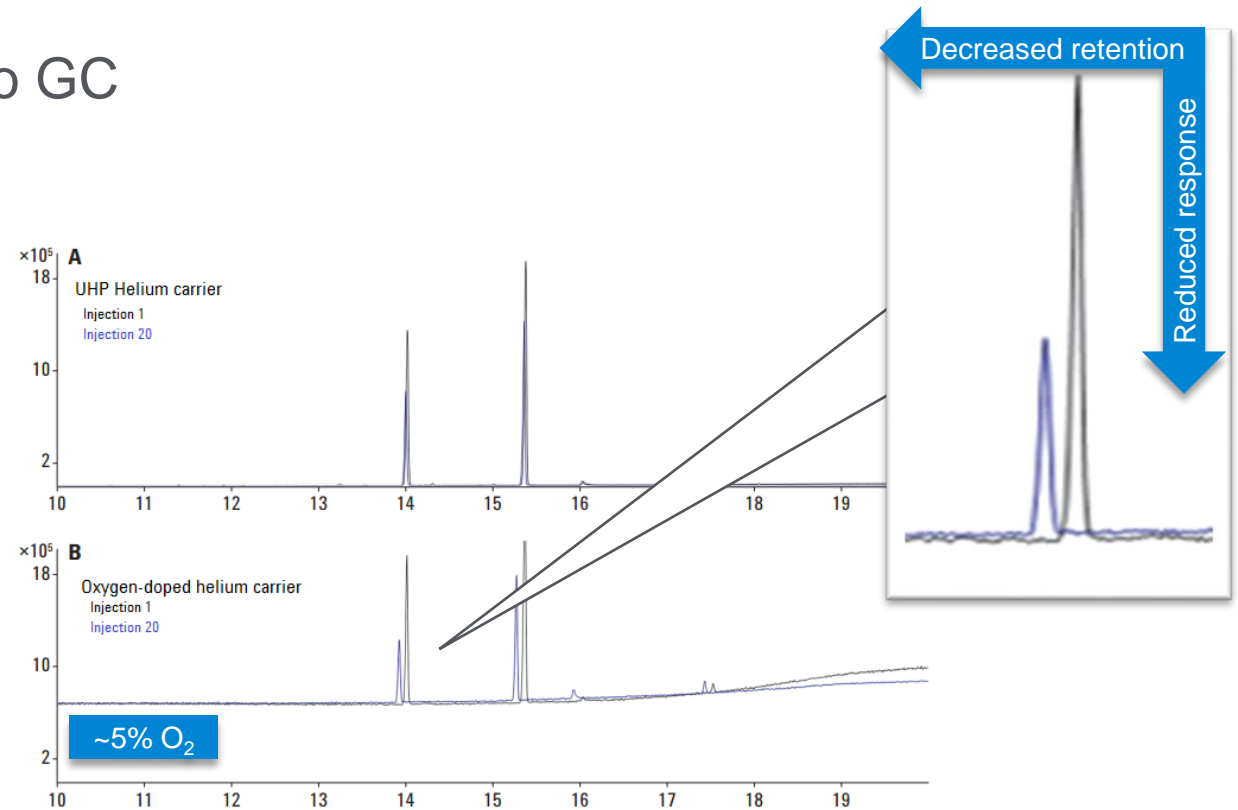
Hexadecamethyl
cyclooctasiloxane



What Impacts Do Leaks Have on My Mass Spectrometer?

Let's Talk About Gas Quality and Filters

- Oxygen in carrier gas is detrimental to GC
 - Reduced response
 - Elevated background
 - **Irreversible** column damage
 - Impaired electron multiplier function
 - Premature filament, liner lifetime
 - Excessive source maintenance
- Use UHP carrier gases
 - **99.999% or greater**
- Use Gas Clean carrier gas filters



GC/MS filter
Agilent p/n
CP17973

Gas Clean Filters:

The first line of defense against leaks

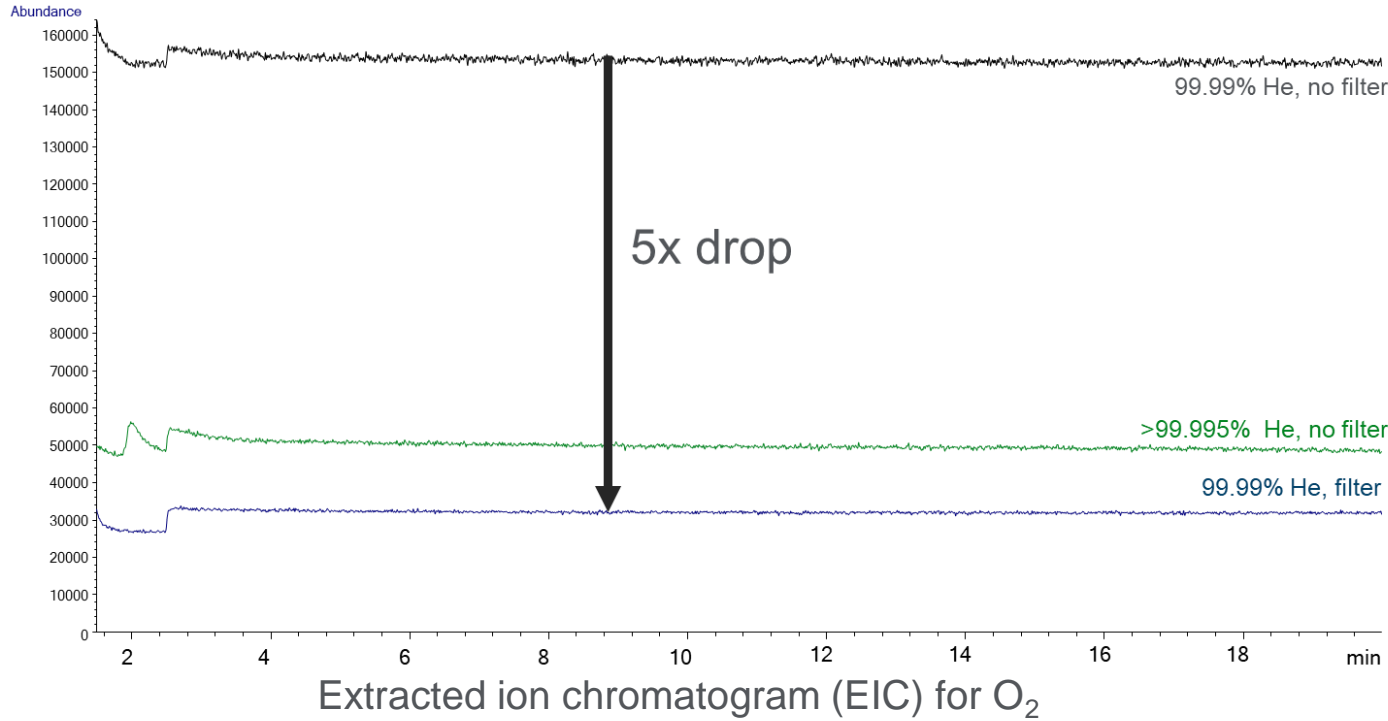


p/n CP17973



Let's Talk About Gas Quality and Filters

If you used lower quality gases, how much O₂ could the filter clean up?

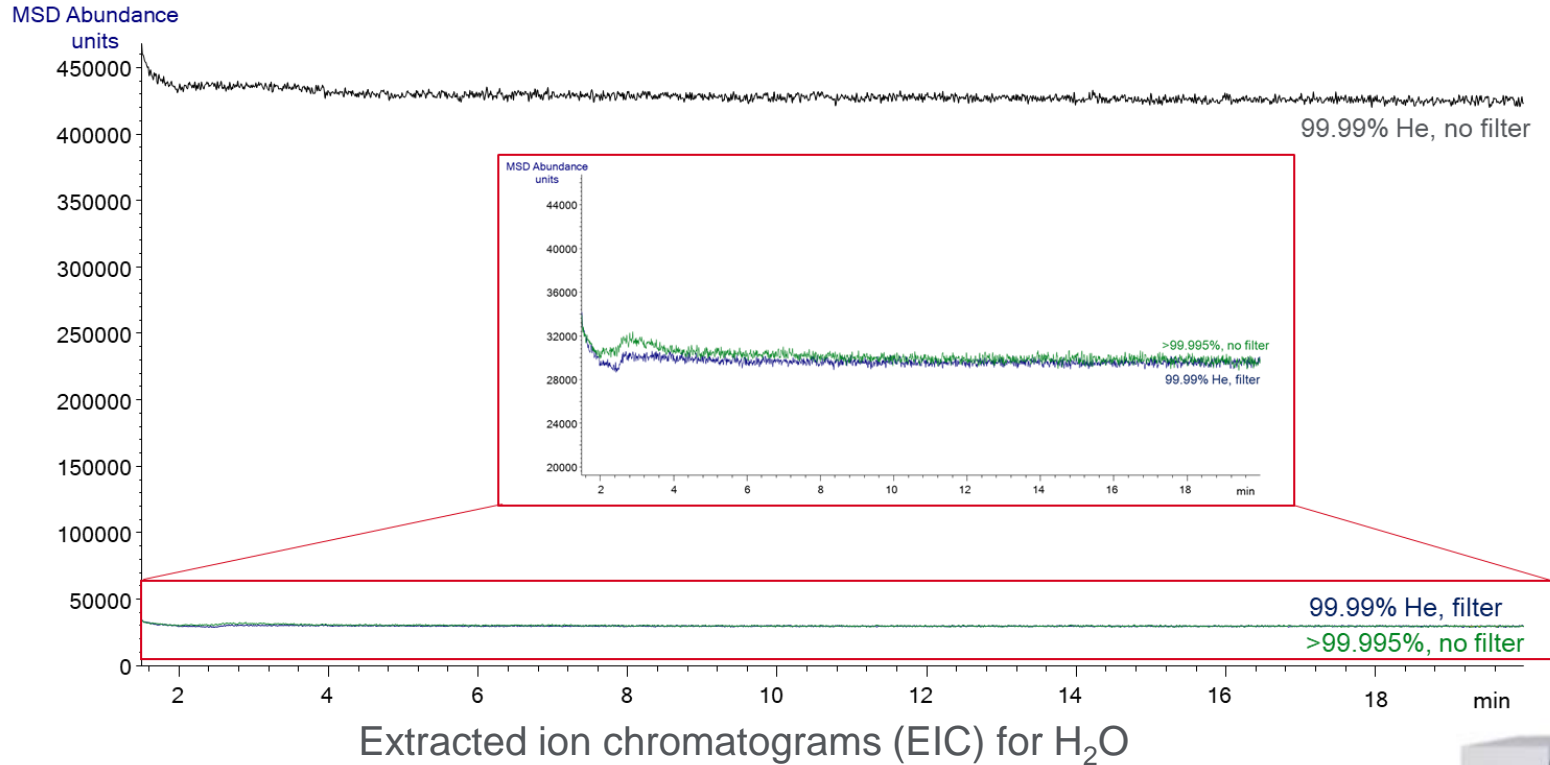


Less O₂ reaching column = longer column lifetime and less system maintenance (and lower costs)



Let's Talk About Gas Quality and Filters

If you used lower quality gases, how much H₂O could the filter clean up?



Extracted ion chromatograms (EIC) for H₂O

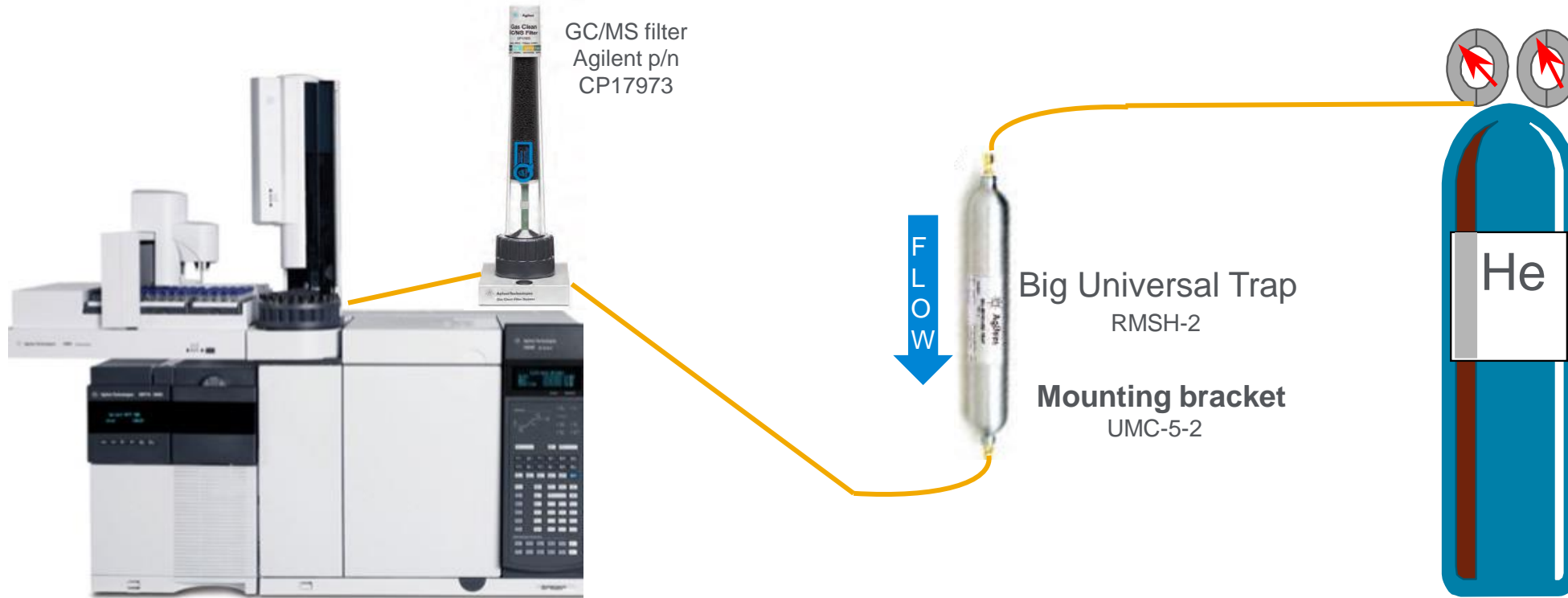
Gas Clean filter lowered the H₂O signal by a **factor >10**

Further increase Gas Clean and column lifetime with a Universal filter, installed before the Gas Clean filter



Let's Talk About Gas Quality and Filters

If lower quality gases were used, how much background could filters clean up?



- Install the Universal trap **vertically** – use the mounting bracket(s)
- Extend the lifetime of your Gas Clean (indicating) filter **and** (most importantly) your column

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 N₂ 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>



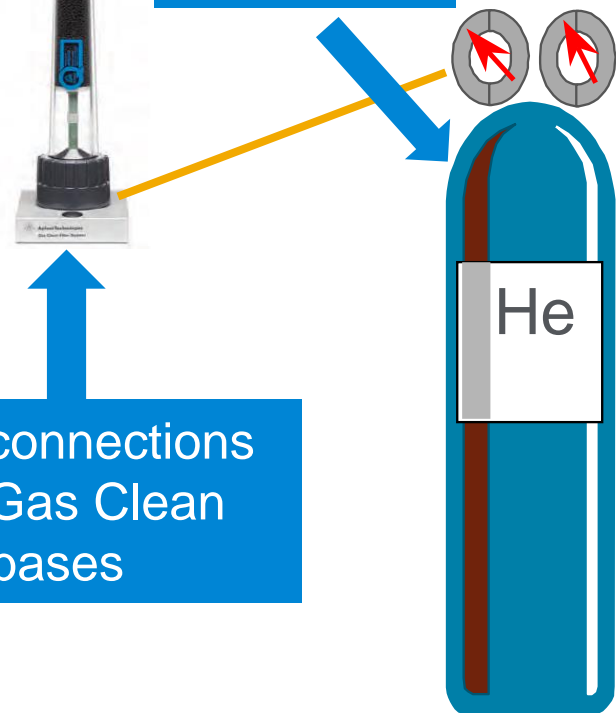
All fittings in the back of the GC



Gas source



Filter connections and Gas Clean bases



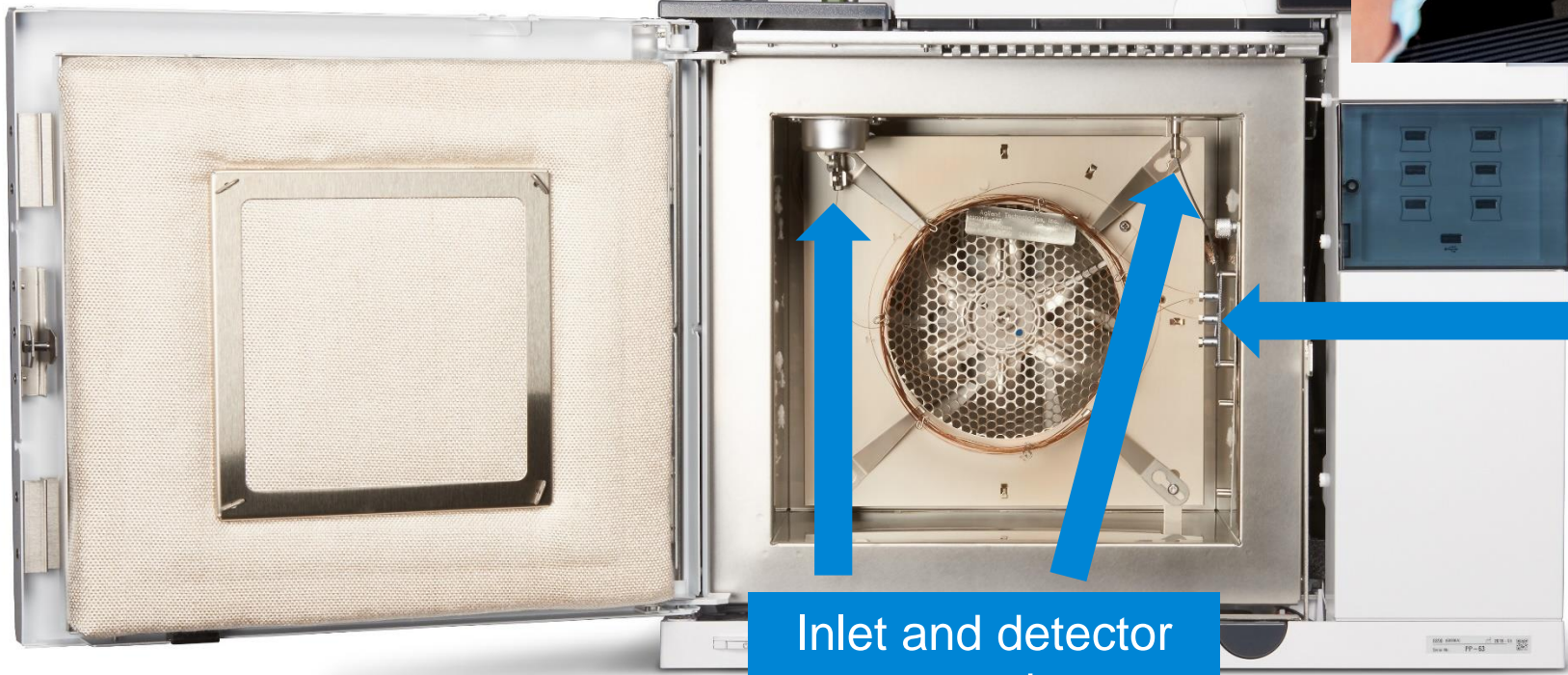


MS transfer line

Septum nut and turn-top



CFT connections



Inlet and detector connections

How Leaks Affect MS – Filaments

- Leaks will reduce filament life
- Cause more variation in the data
- Increase downtime



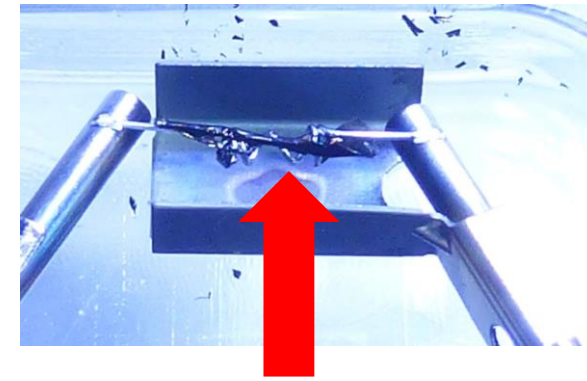
High temperature filament p/n: [G7005-60061](#)



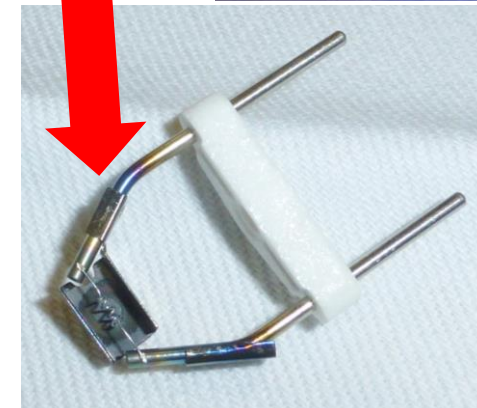
HES filament
p/n: [G7002-60001](#)



Oxidizing



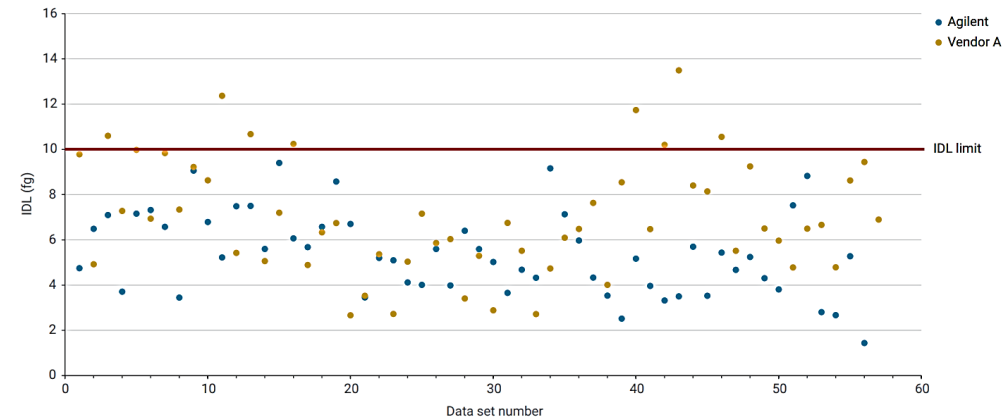
Coil destruction



Agilent MS Filaments Lifetime

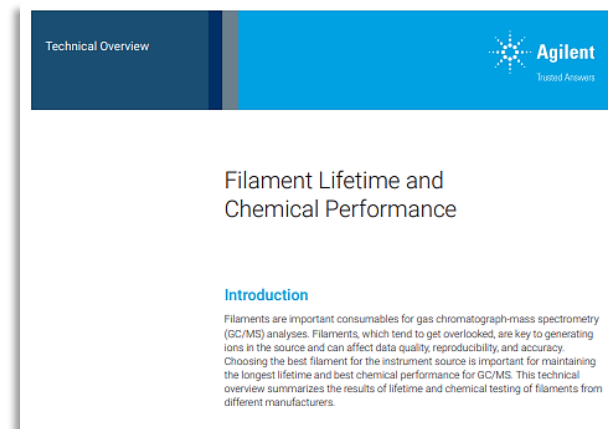
Agilent filaments last 6x longer

Filament Manufacturer	Average Lifetime (Hours)	%RSD
Agilent	94.2	9.0 %
Vendor A	16.0	35.0 %



14% of IDLs failed with non-Agilent filaments

Agilent filaments are more consistent



More info: [5994-4719EN](tel:5994-4719EN)

How Leaks Affect MS – Vacuum

- Leaks will reduce vacuum
 - Reduced sensitivity
 - Can cause damage to oil pump
 - Pump may smoke
- Alternative dry pumps may be preferable

Failed turbo pump – increased friction due to leak



Quiet Cover p/n:
[G6014B](#)



IDP-10 – TQ
p/n: [X3810-64000](#)



IDP-3 – SQ
p/n: [X3803-64301](#)

Common Issues with Traditional Pumps

- Disposing of oil
- Leakage
- Smell of oil when it gets old
- Heat from pumps
- Noise level in lab
- Maintenance costs




Inland 45
p/n: [6040-0834](#)

PM Parts	Approximate Price
Oil	\$25.00
Mist filter	\$348.00
Oil disposal	\$115.00
Total cost	\$488.00



[Tip Seal Replacement Kits for Agilent Dry Scroll Pumps | Agilent](#)

PM Parts	Approximate Price
Tip seal	\$407.00 



Mist filter
p/n: [G1099-80039](#)

PM twice a year = **\$976.00** a year on consumables alone

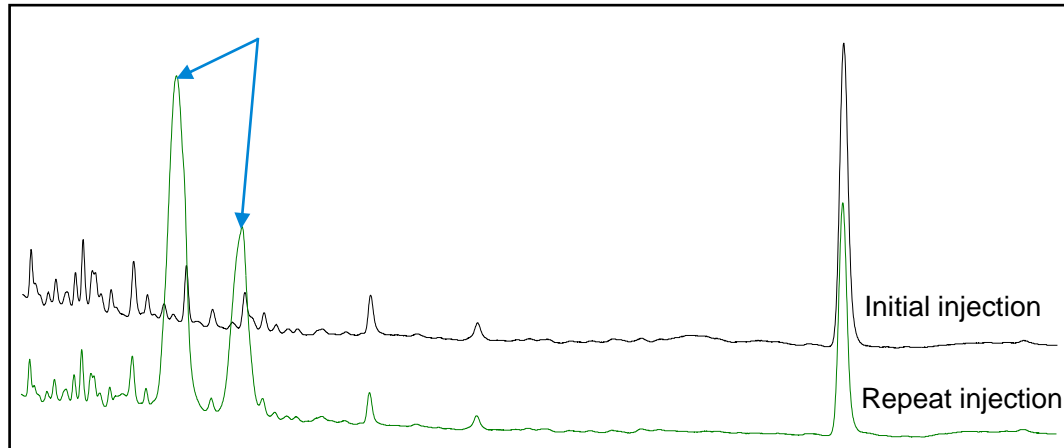


How Can I Tell If a Peak in a Blank Is Carryover or Contamination?

Carryover or Contamination?

Carryover

- After concentrated sample
- Analyte does not elute before end of run
 - Final oven temperature not high enough
 - Needs longer final hold time
 - Implement backflush

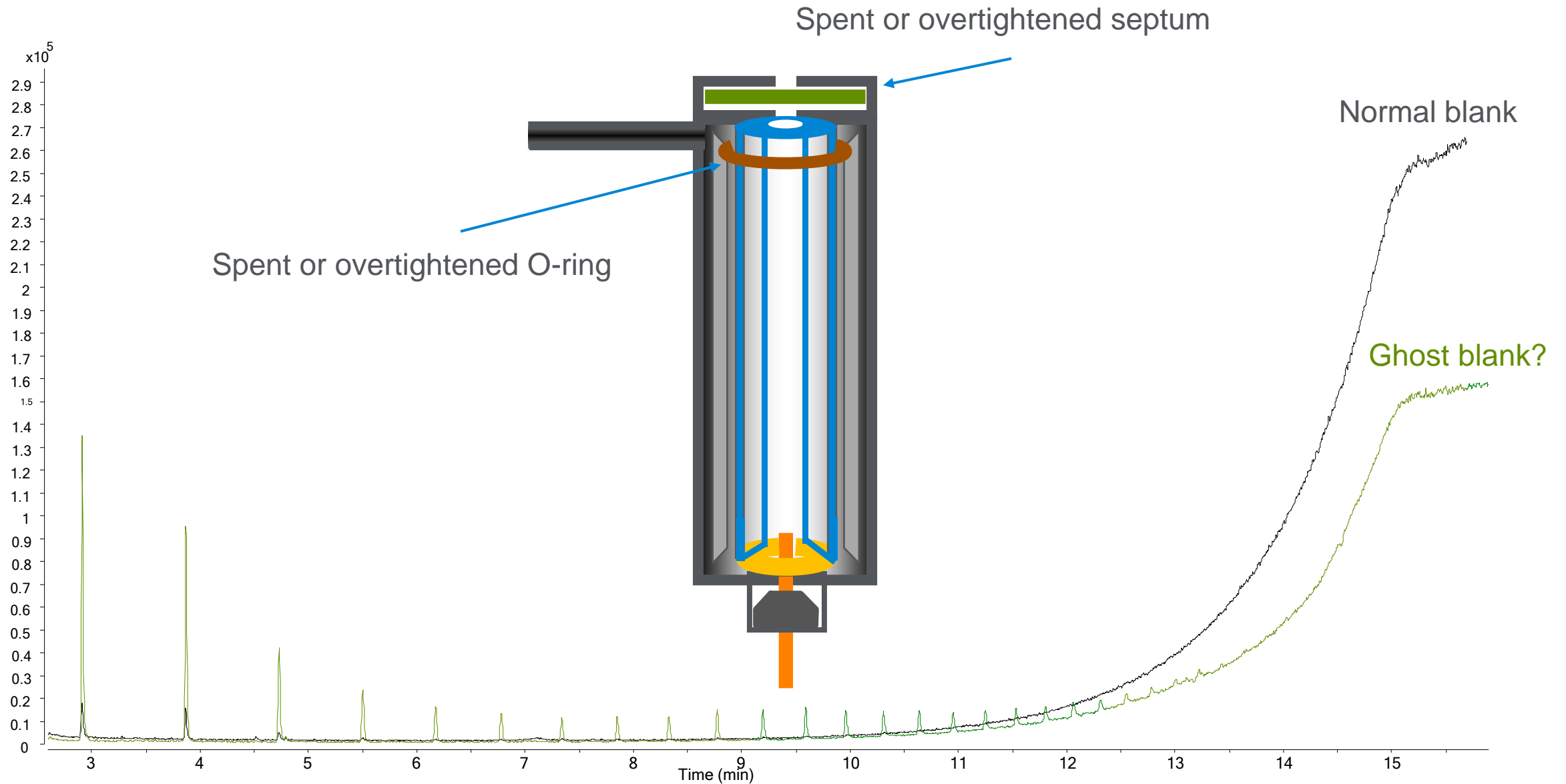


Contamination

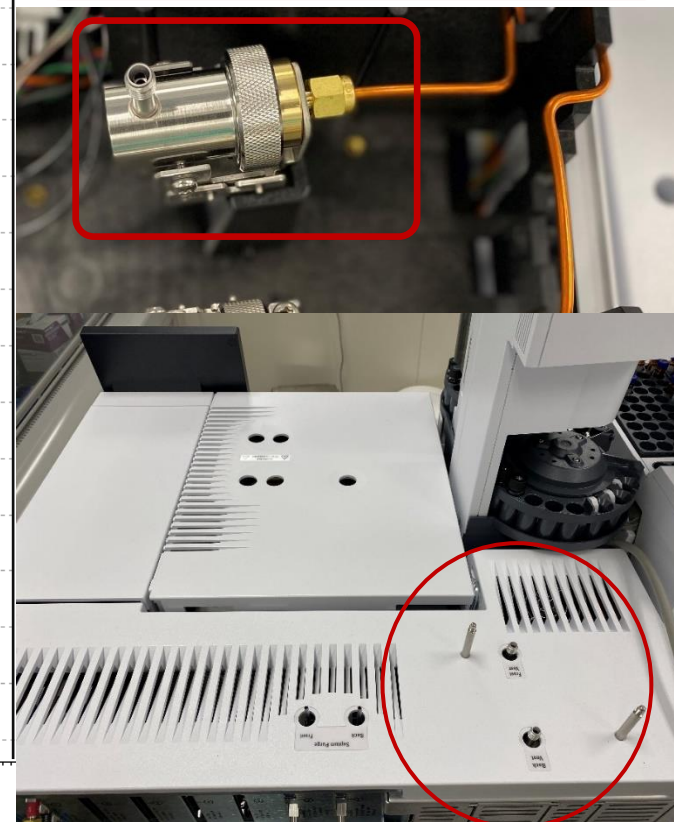
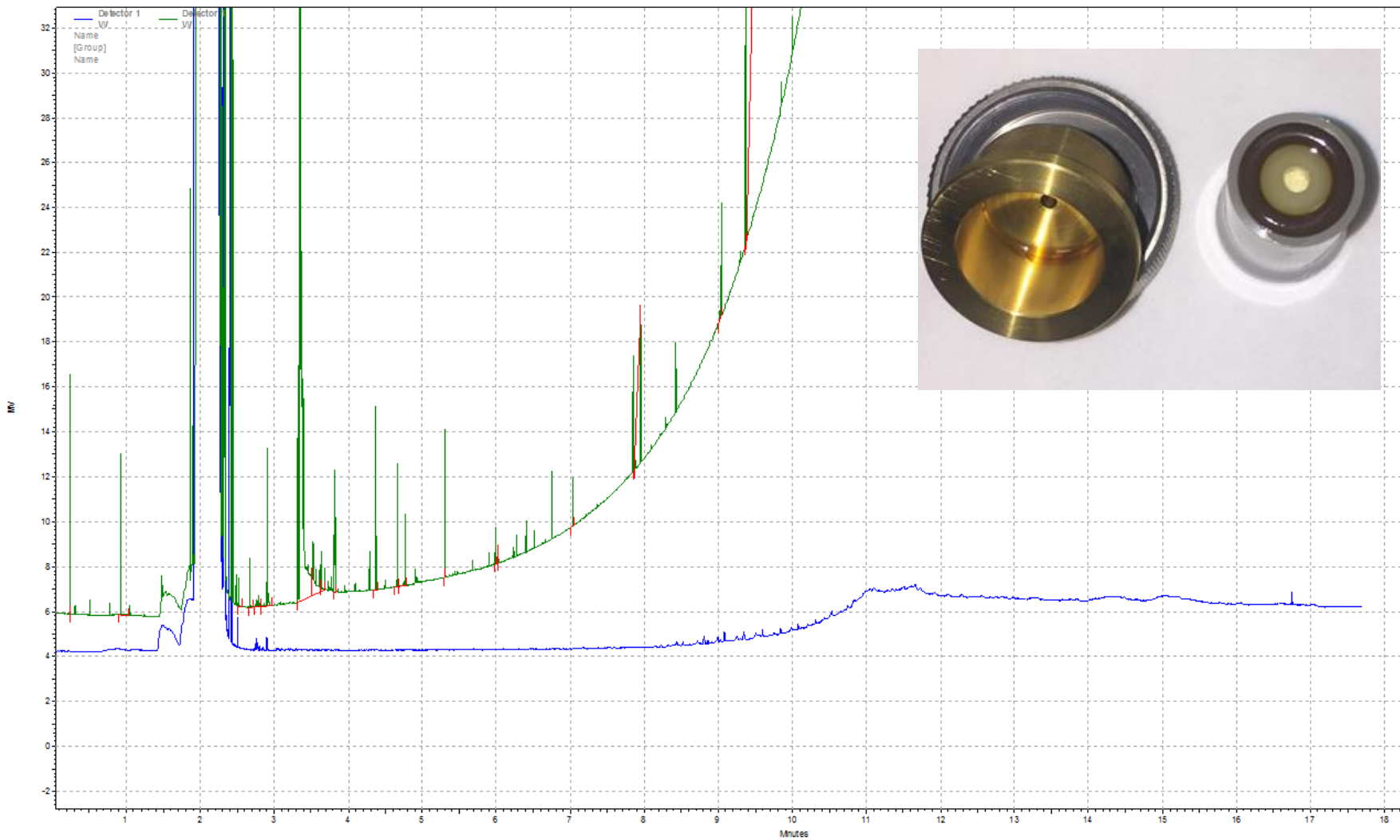
- Contamination somewhere in the flow path
- Run instrument blank to determine if contamination is from **inside** or **outside** the GC



Are There Apparitions in My GC?

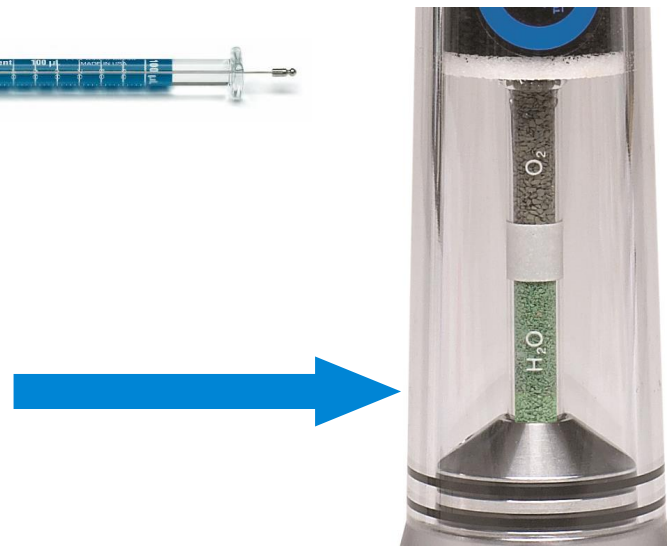
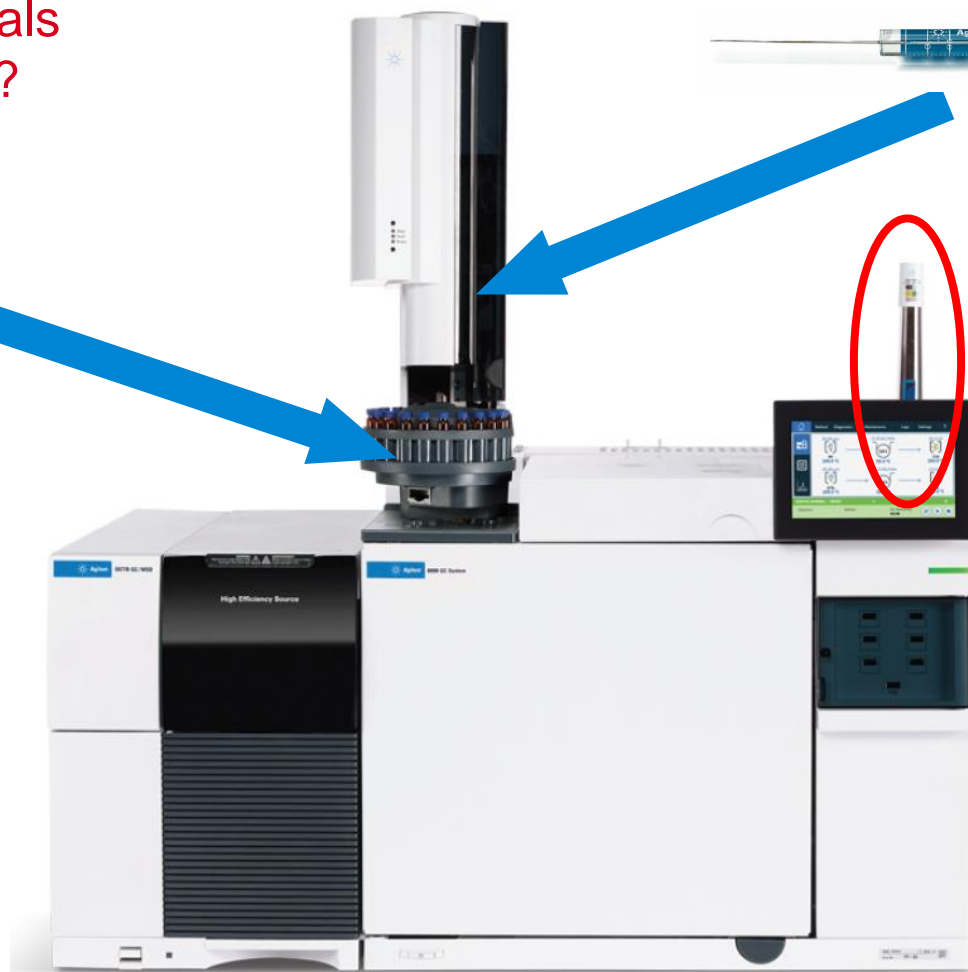
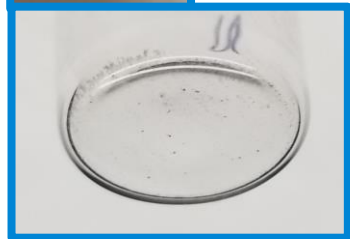


Split Vent Trap Changed (Column Bleed?)



What Parts Do I Need to Consider to Bring My GC System Back to 100%?

Are the wash vials contaminated?

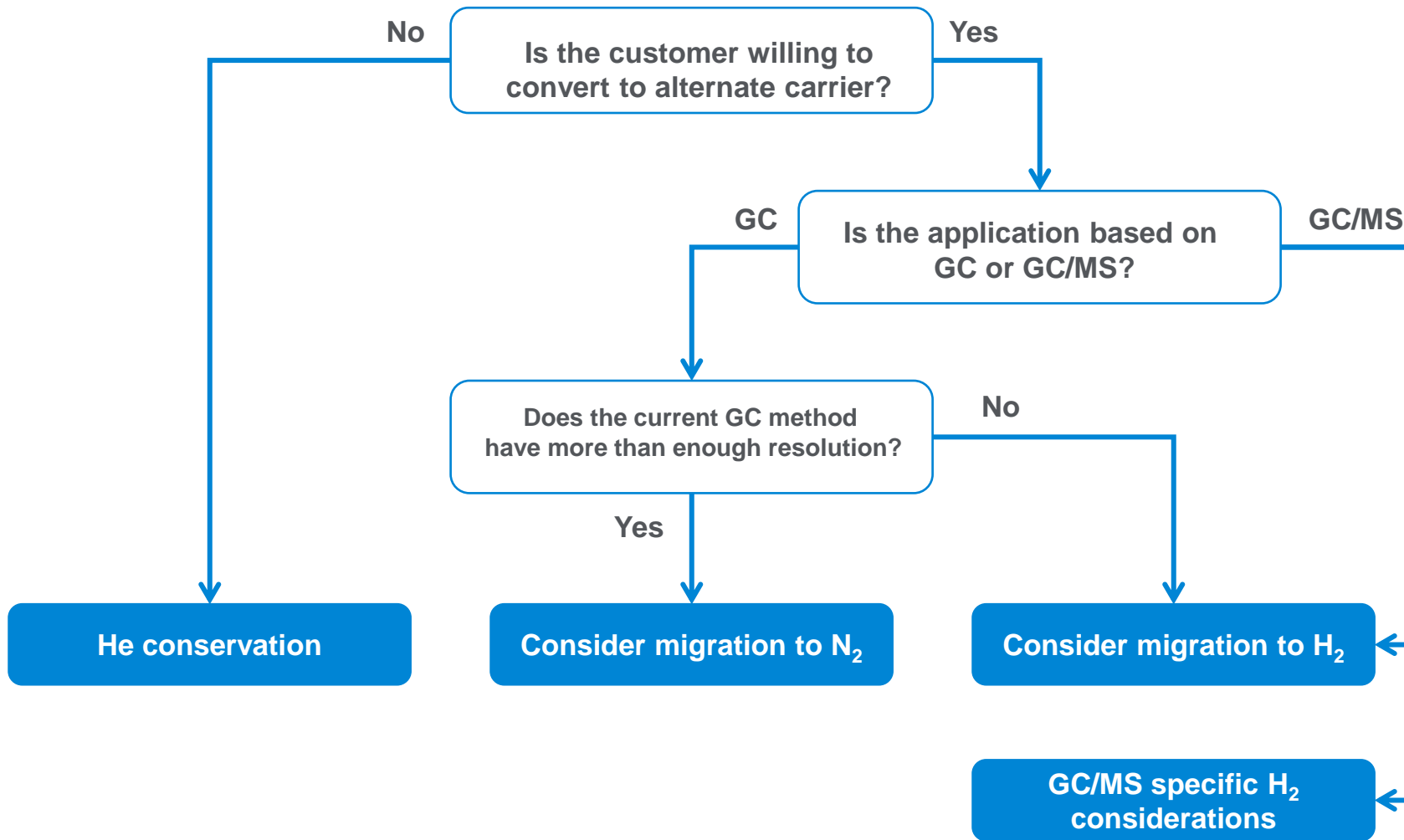


Are the indicators saturated?



If Can't Get Helium, Should I Use Nitrogen or Hydrogen Carrier Gas?

Carrier Gas Decision Tree



Reasons to Use Hydrogen Carrier Gas

- Readily available (H_2 already used for FIDs and other detectors)
- Sustainable
- Lower cost
- Cleans source during use
- Available on demand (H_2 generator) or by cylinder
- Faster analysis
- Lower temperature separation possible
- Move to “more efficient” columns
 - 30 m x 0.25 mm x 0.25 μm \rightarrow 20 m x 0.18 mm x 0.18 μm



Safety Considerations for Hydrogen Migration

Both GC and GC/MS offer H₂ enabled features:

Safety shutdown

- When gas pressure setpoints are not met, the valve and heater are shut off to prevent hydrogen leakage

Flow limiting frit

- If the valve fails in the open position, the inlet frit limits the flow

Oven ON/OFF sequence

- The fan purges the oven before turning on heater to remove any collected H₂

H₂ ignition “ready”

- GC and MS are designed to contain parts in case of hydrogen ignition (for example, spring in GC door)

H₂ oven sensor available

- Sensor passively samples the oven to check for hydrogen leaks

Considerations for Hydrogen Gas Sources

H₂ generator – preferred

- Very clean H₂, >99.9999% available
- Consistent purity
- Built-in safety features
- Make sure to buy a good generator which minimizes the production of water and oxygen
- Parker's H-MD are used at Agilent sites
- Use Gas Clean filter

H₂ cylinder

- Use Gas Clean filter
- Possible to add safety device



Considerations for Hydrogen Gas Plumbing

Tubing

- Use chromatographic quality stainless steel tubing (required)
- Do not use old tubing (H₂ is known as scrubbing agent)
- Especially don't use old copper tubing (brittleness is a safety concern)

Venting

- Connect split vent and septum purge vent to exhaust

Leak checking

- Recommended leak detector is G6699A/G6693A

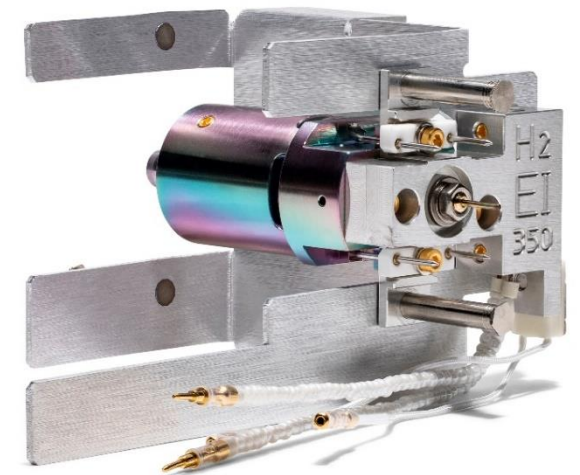
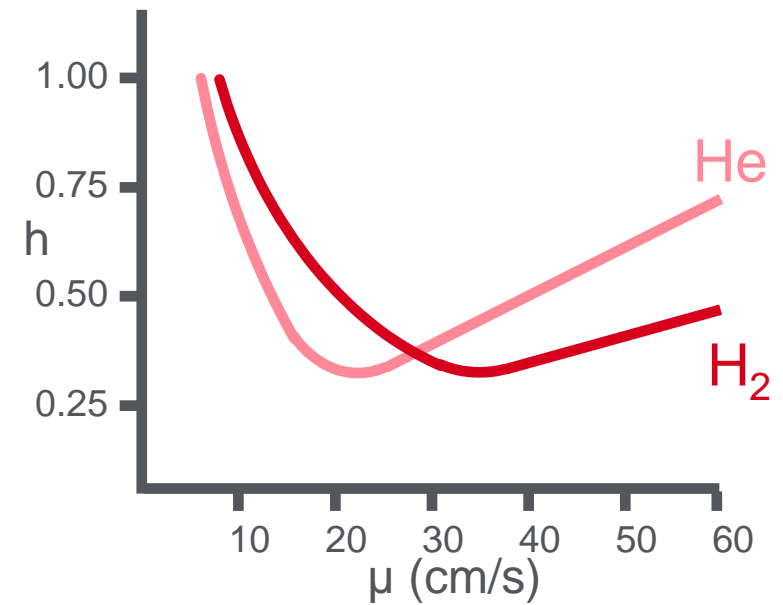


H₂: Method Considerations

Method development and optimization

- H₂ is a challenge, but not impossible
- Resolving potential problems will take longer than expected
- Have patience – it probably won't work perfectly the first time
- Think through plans and contingencies ahead of time: what are your must haves?
- Watch the inlet temperature and sample preparation
- Change to smaller diameter columns
- Adjust method flow to suit hydrogen
- Use the method conversion tool to easily predict flows

Use gas filters even on a H₂ generator



Use N₂ As Carrier Gas

Many methods are suited to nitrogen

- Readily available and less expensive gas
- No safety concerns
- Suitable for simple routine analysis (with sufficient resolution)
- More inert than H₂, especially with PLOT/micropacked columns
 - Some compounds are catalytically reduced in H₂
- 2-D GC is ideally suited to nitrogen
 - Resolution issues solved using two different columns

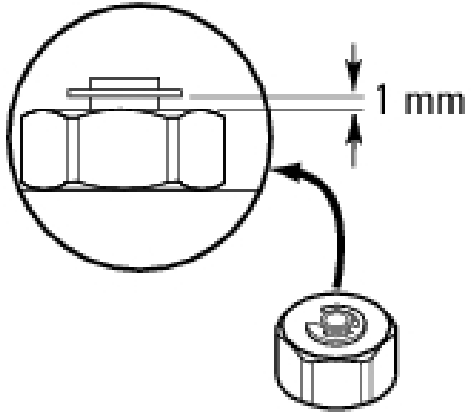


Potential issues

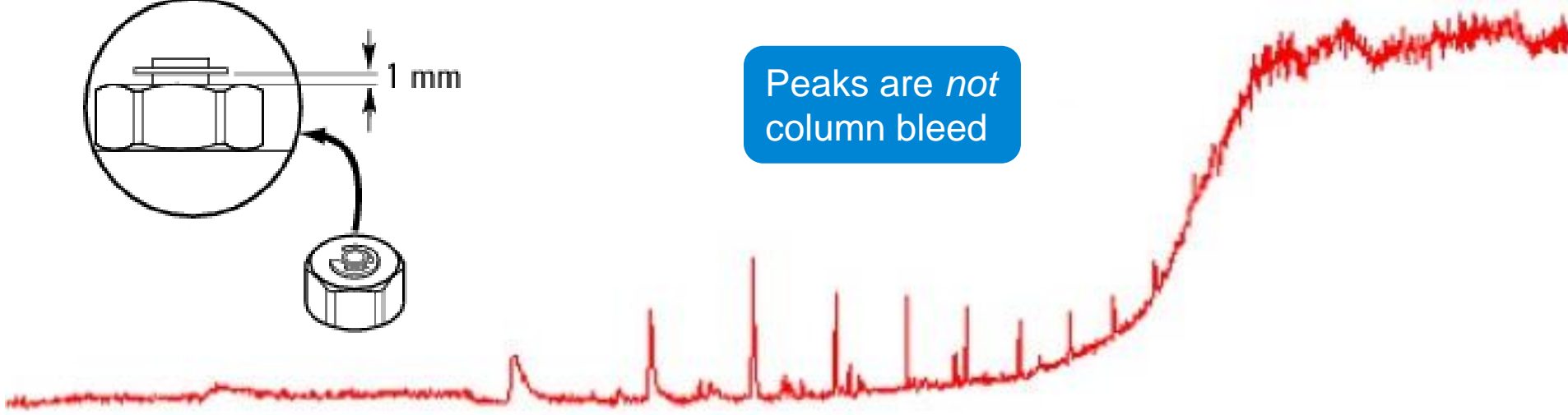
- Reduced chromatographic resolution at higher flows
- Not suitable for GC/MSD and certain GC detector applications

When Do I Need to Change My Septa?

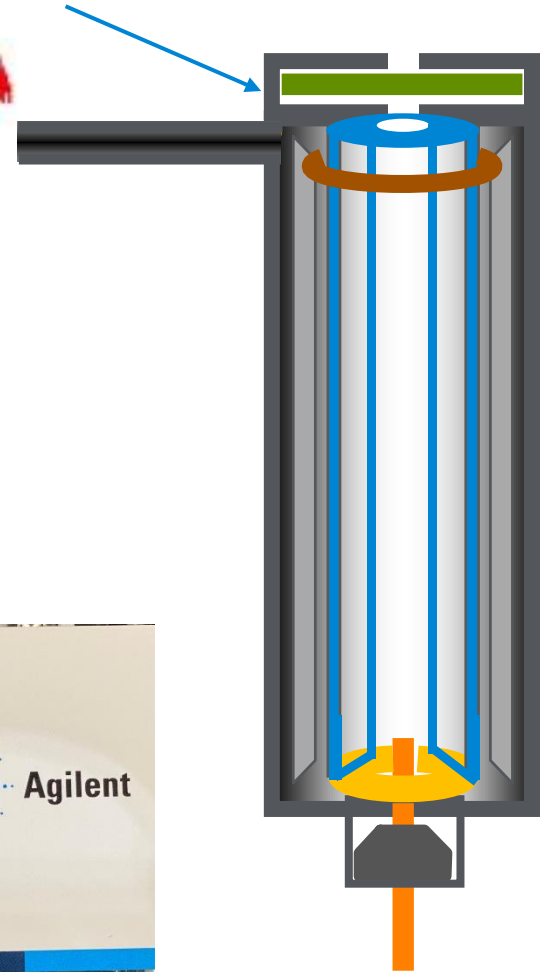
Siloxane Peaks → Time to Change Your Septum



Peaks are *not* column bleed

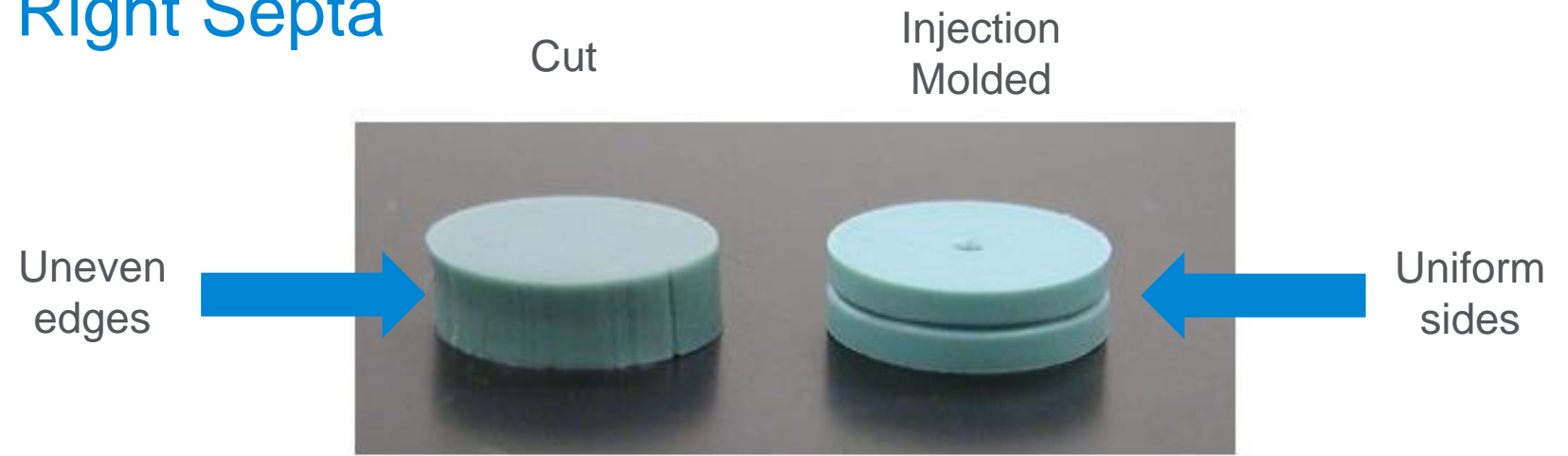


Spent or overtightened septum

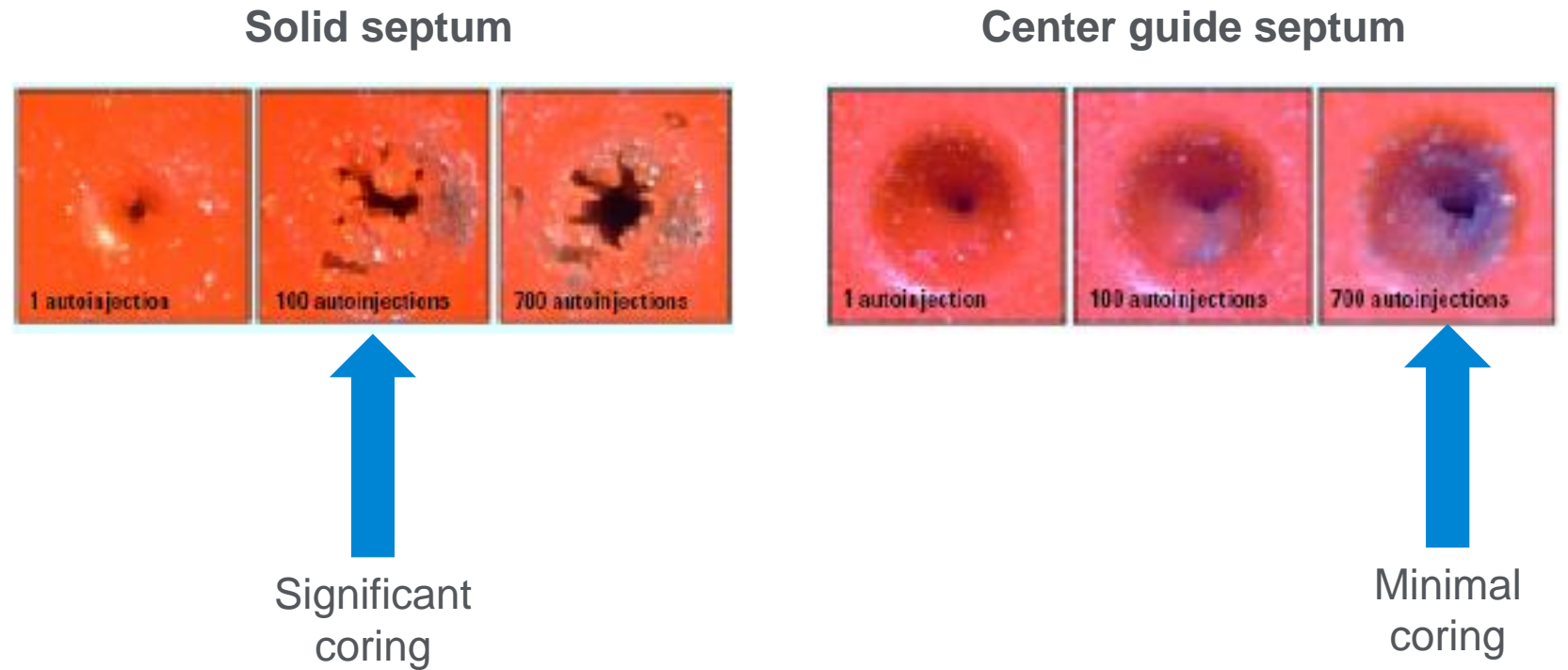


Step One: Choose the Right Septa

Injection molded
versus
cut



Solid septum
versus
Center guide septum

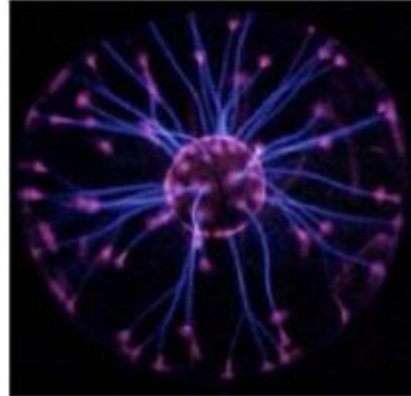


Don't Get Stuck With Nonplasma-Treated Septa

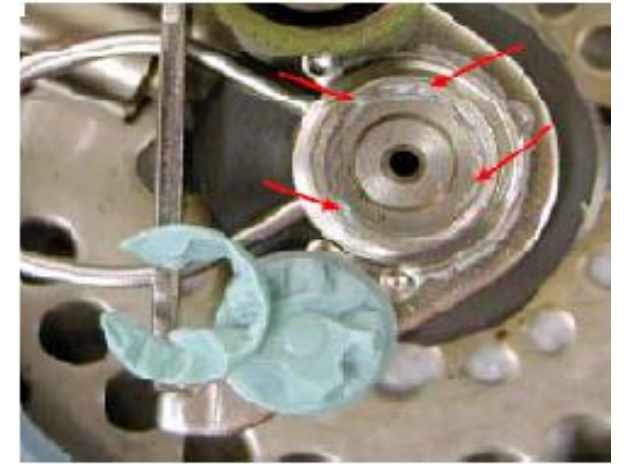
Traditional septa get stuck in your inlet



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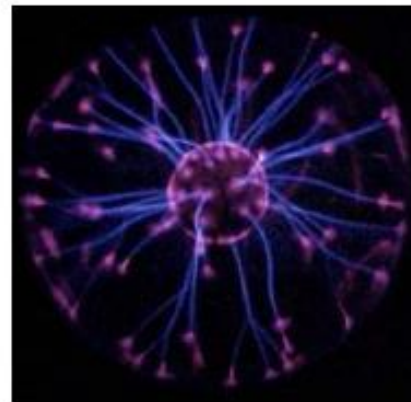
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Plasma treated septa are nonstick



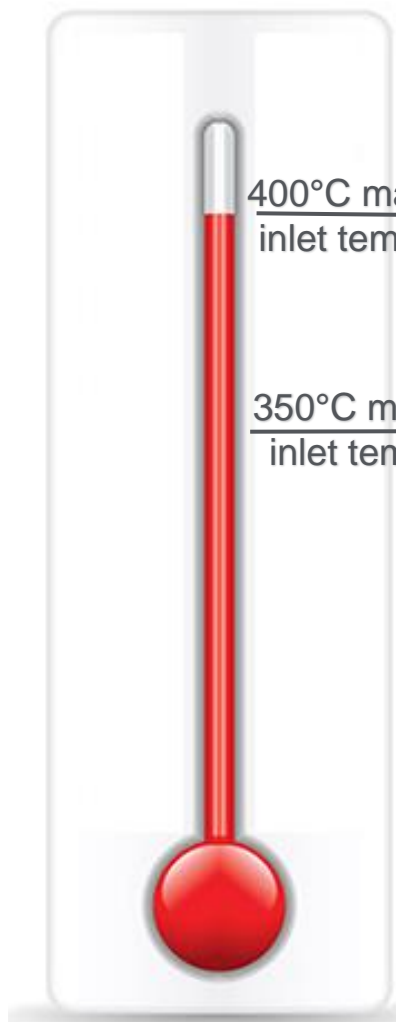
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Which Is the Right Septa for Your Analysis?



400°C max
inlet temp



Bleed and Temperature Optimized (BTO) septa

- Optimized for trace analysis
- Extended temperature range
- Low bleed

350°C max
inlet temp



Long-life septa

- Optimized for longest lifetime
- Extended puncture lifetime
- Excellent for autosamplers

Advanced green septa

- General purpose septa
- Optimized temperature range/puncture lifetime



Septum Type	Bleed	Lifetime	Temp. Limits
BTO (Bleed and Temp. Optimized)	✓✓✓	✓	to 400° C
Advanced Green	✓✓	✓✓	to 350° C
Long-Life	✓	✓✓✓	to 350° C

✓✓✓ = Best ✓✓ = Very Good ✓ = Good

How Often Should You Clean the HydroInert Source?

How Often to Clean a Hydrolnert Source

Short answer...don't

- Cleaning experiment
 - Standard extractor source with 9 mm lens vs a Hydrolnert source with 9 mm lens
 - Extracted soil matrix and running EPA 8270
 - Cleaning was required on the standard extractor after 365 matrix injections
 - 4000 injections were completed on the Hydrolnert before the source failed acceptance criteria
- Cleaning the Hydrolnert is not recommended
 - Replacing the 9 mm extraction lens (G7078-20909) and repeller (G7078-20902) is recommended when GC maintenance alone will not restore conditions.
 - Labs can replace the extraction lens and repeller yearly at the PM to truly prevent MS downtime.



Repeller



G7078-20902

Extractor lens



G7078-20909

Technical Overview

Agilent
Trusted Answers

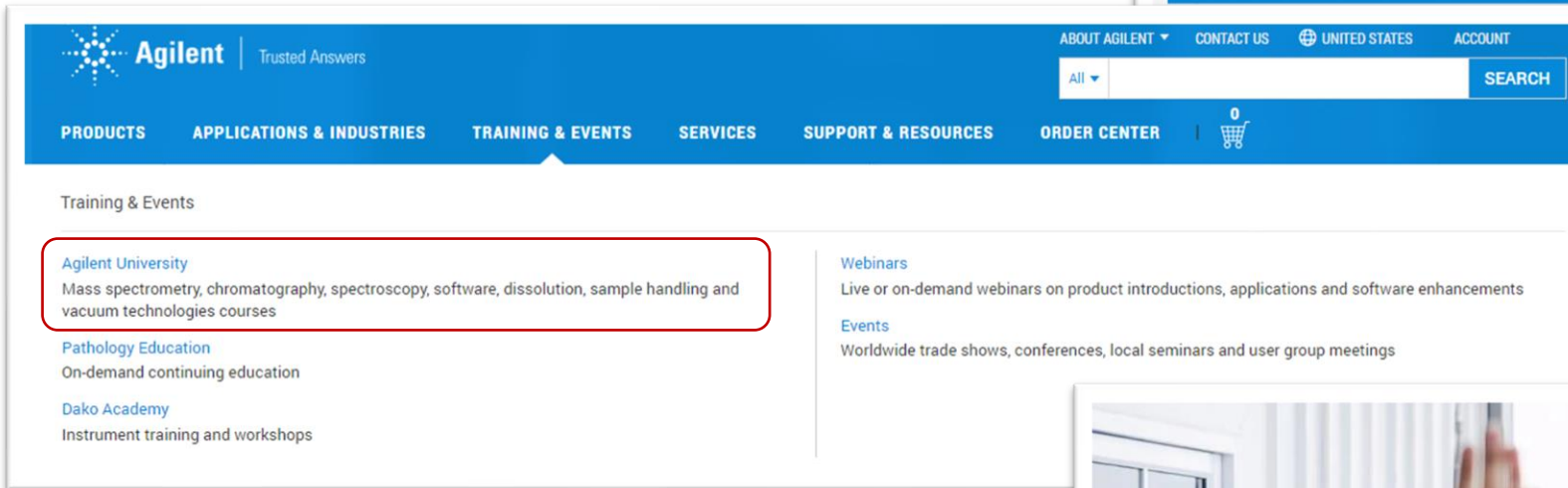
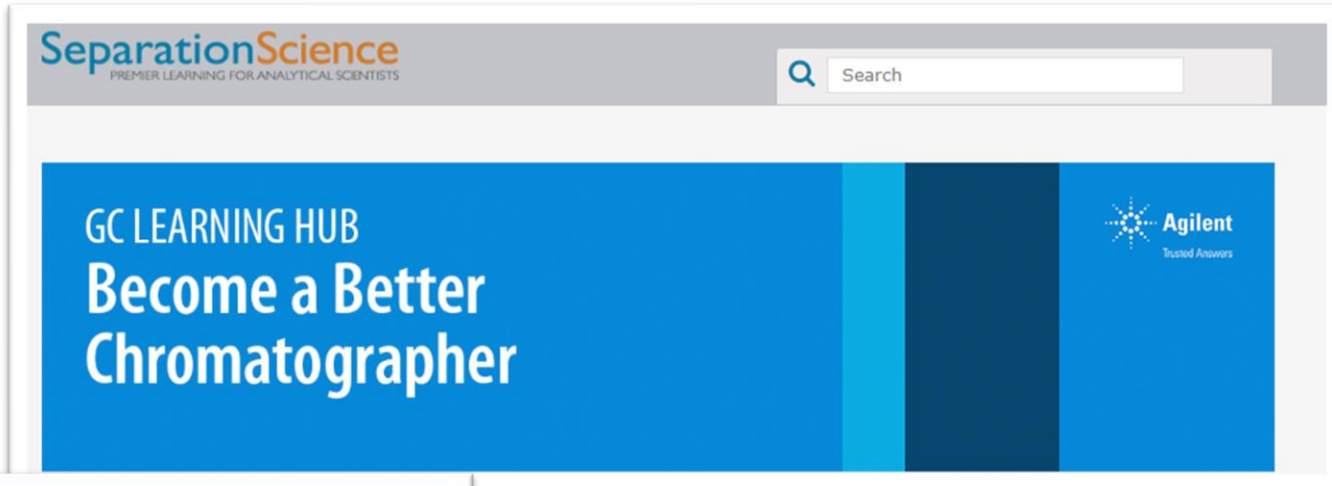
Agilent Inert Plus GC/MS System with
Hydrolnert Source

Applying H₂ carrier gas to real-world GC/MS analyses

Agilent publication [5994-4889EN](#)

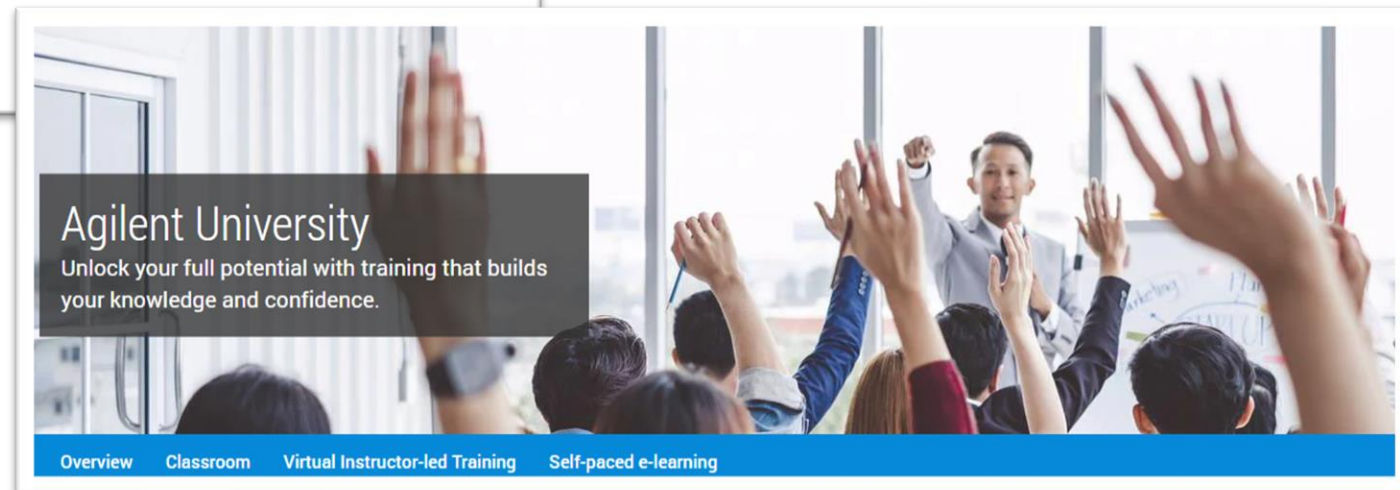
Where to Find More Information

For More Training Information



[GC Learning Hub - Become a Better Chromatographer \(sepscience.com\)](https://sepscience.com)

[Agilent Education & Training | Agilent](#)
[GC Column Installation Guide | 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



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