

Sealing the Deal: Strategies for Leak-Free GC

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What Will We Cover Today?

- Signs and symptoms of a leak
- Instrument diagnostics
- Finding the leak
- Ensuring no leaks in the future

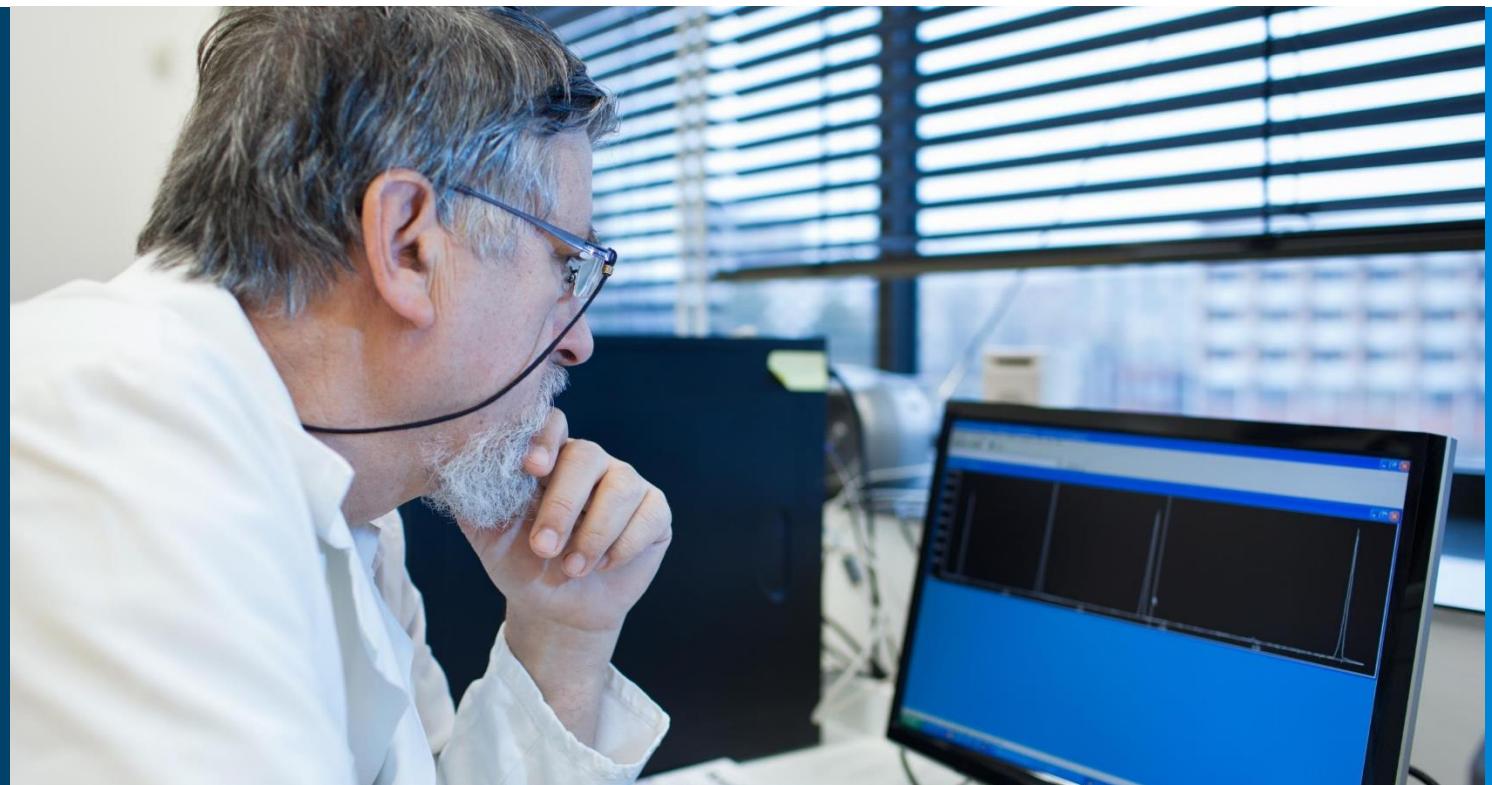


The Cost of Leaks

- Cost of gases
- Contamination from exposure
- Reduced consumable lifetime
- Reduced productivity from downtime
- Detector noise and elevated baselines
- Time in troubleshooting

It is critical that every customer checks for leaks and they should have the best tool for the job. Check valves, fittings, and traps for leaks after every maintenance, and after thermal cycling, as these can loosen some types of fittings.

Signs and Symptoms of a Leak

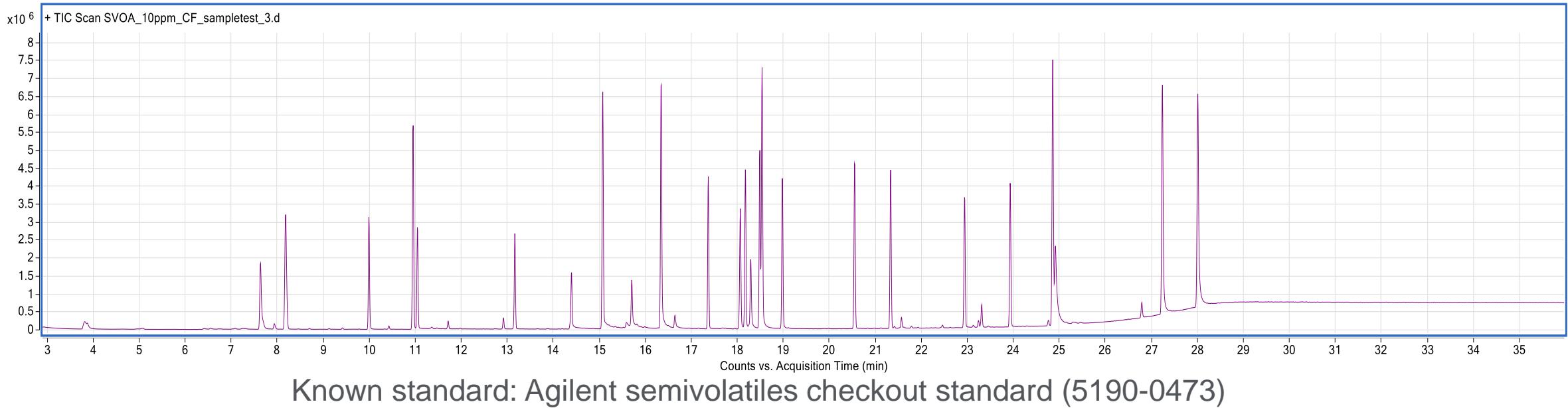


What Are Some Signs or Symptoms of a Leak?

- Elevated bleed
- Poor peak shape
- Spent gas traps
- Instrument diagnostics



Good Habit: Have System “Baselines”



What kind of system baselines?

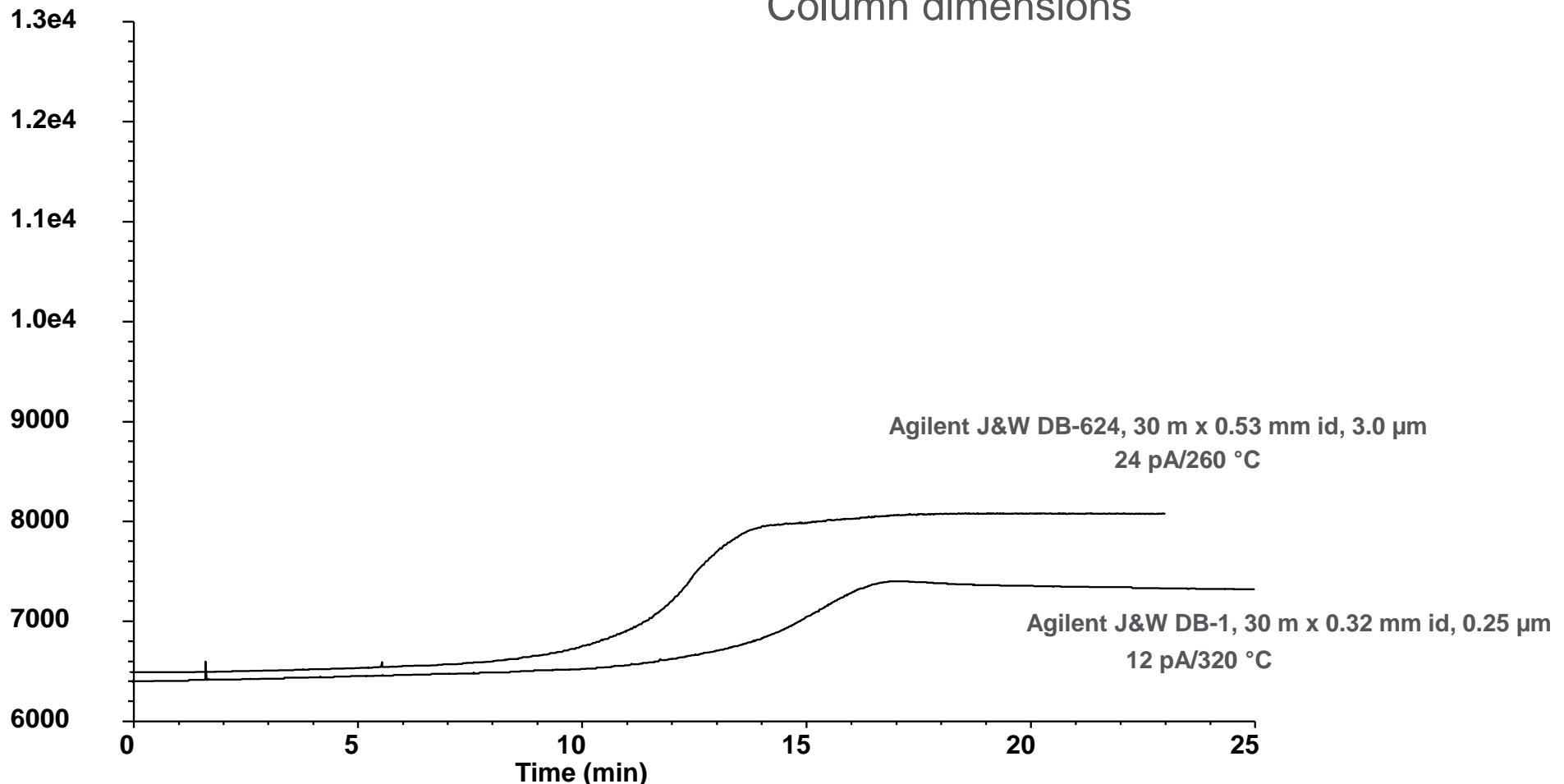
- System blank – Should only see a rise in the baseline with temperature (column bleed)
 - Should not see any peaks
- Solvent blank – May contain contaminant peaks (for example, phthalates, siloxanes)
 - Best practice: Use the same bottle of solvent that was used for any dilutions/extractions
- Known standard – GC/MS checkout standard, DFTPP tuning mix, or known calibration standard at easily detectable levels for your system (for example, 1 to 10 ppm)

What Is Normal Column Bleed?

Normal background signal is generated by the elution of normal degradation products from the column stationary phase. Column bleed is influenced by: Phase type

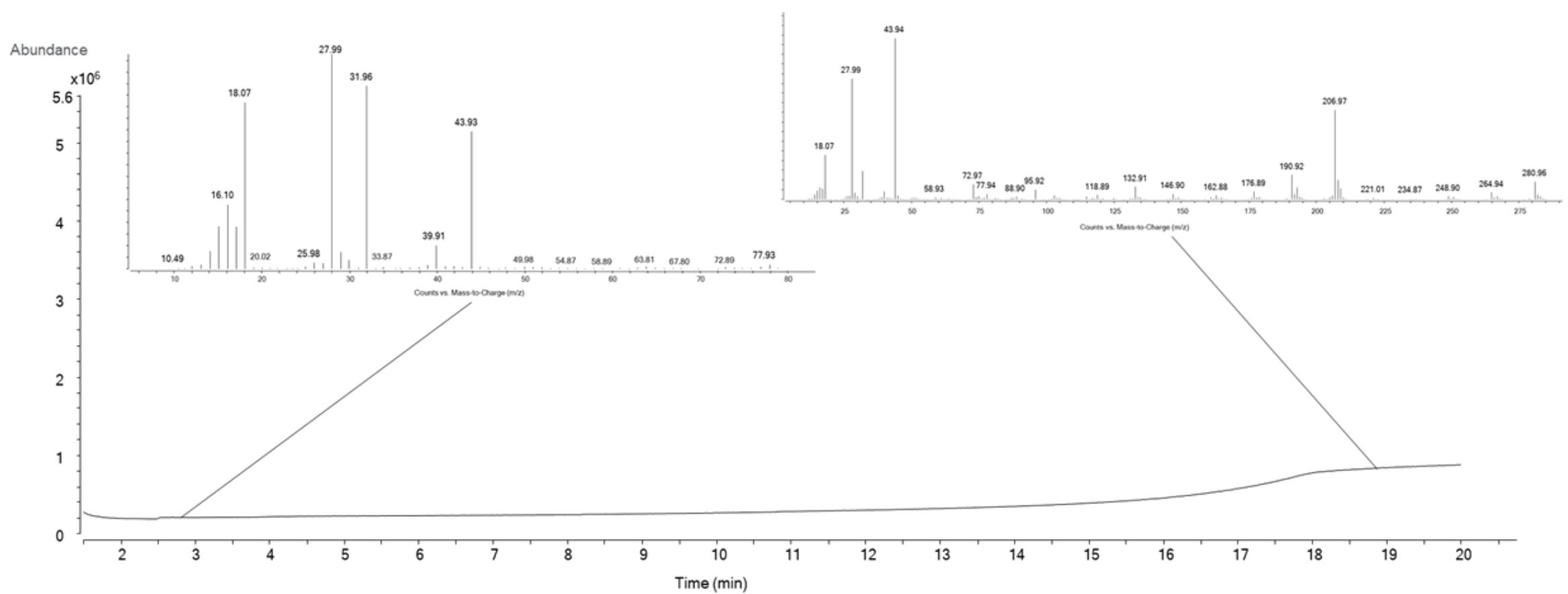
Temperature

Column dimensions

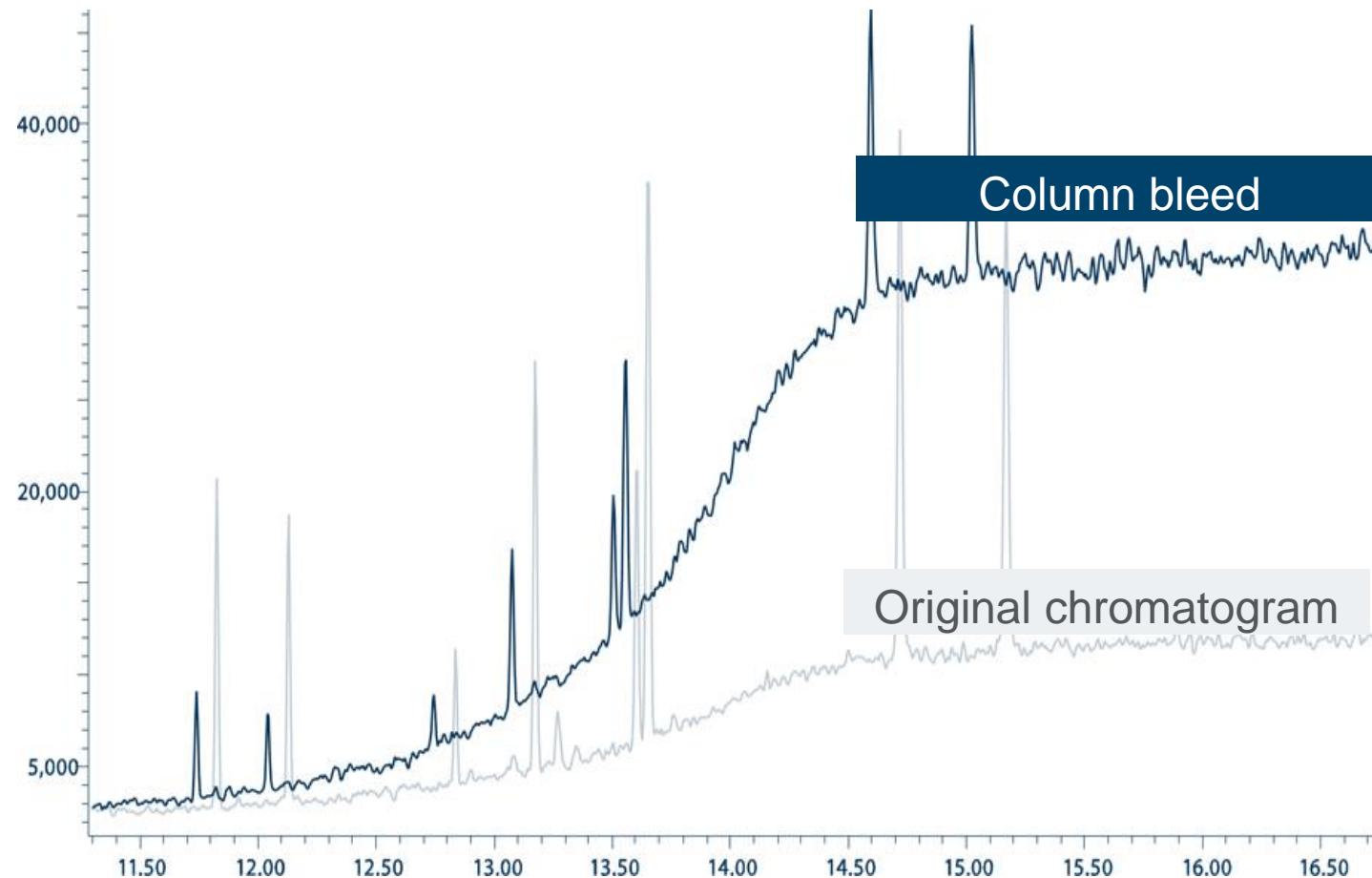


Mass Spectrum of Phenylmethylpolysiloxane Column Bleed

Normal background (HP-5ms UI)



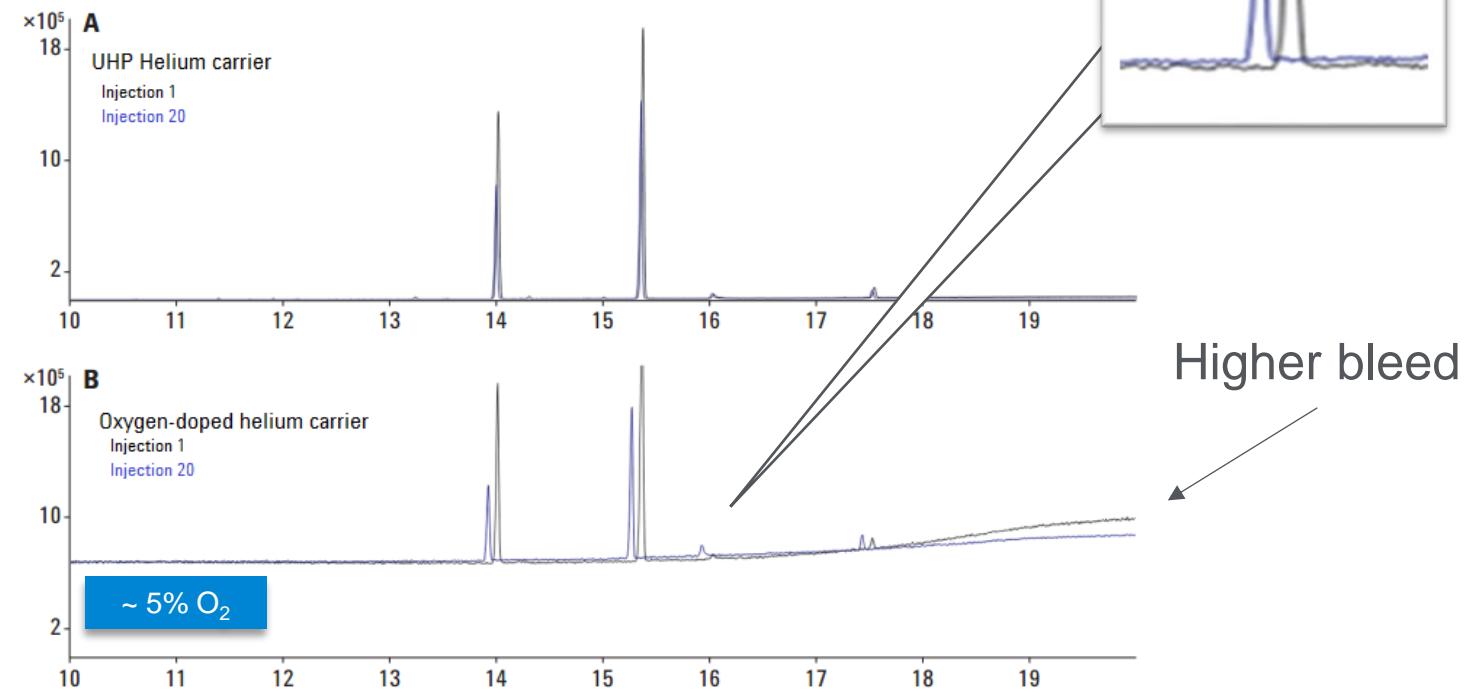
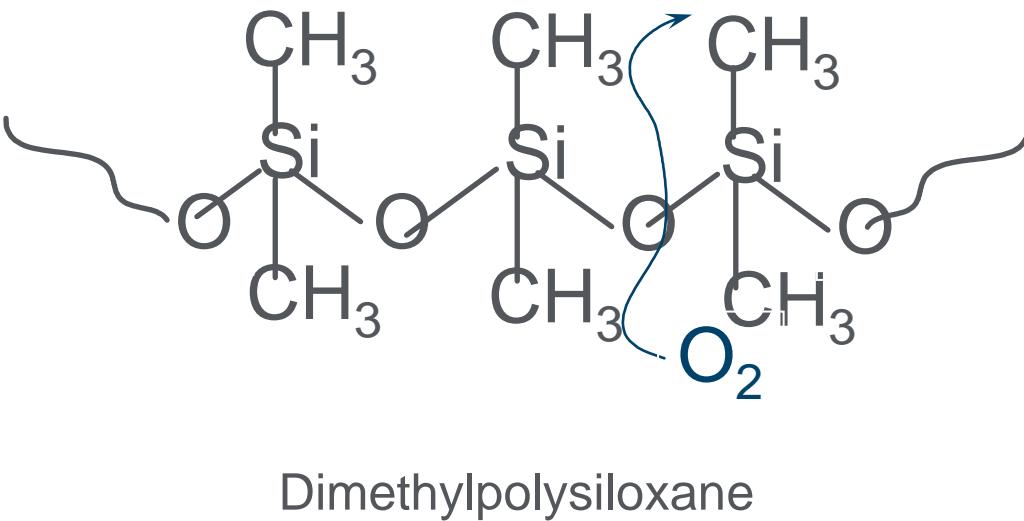
Troubleshooting Column Bleed



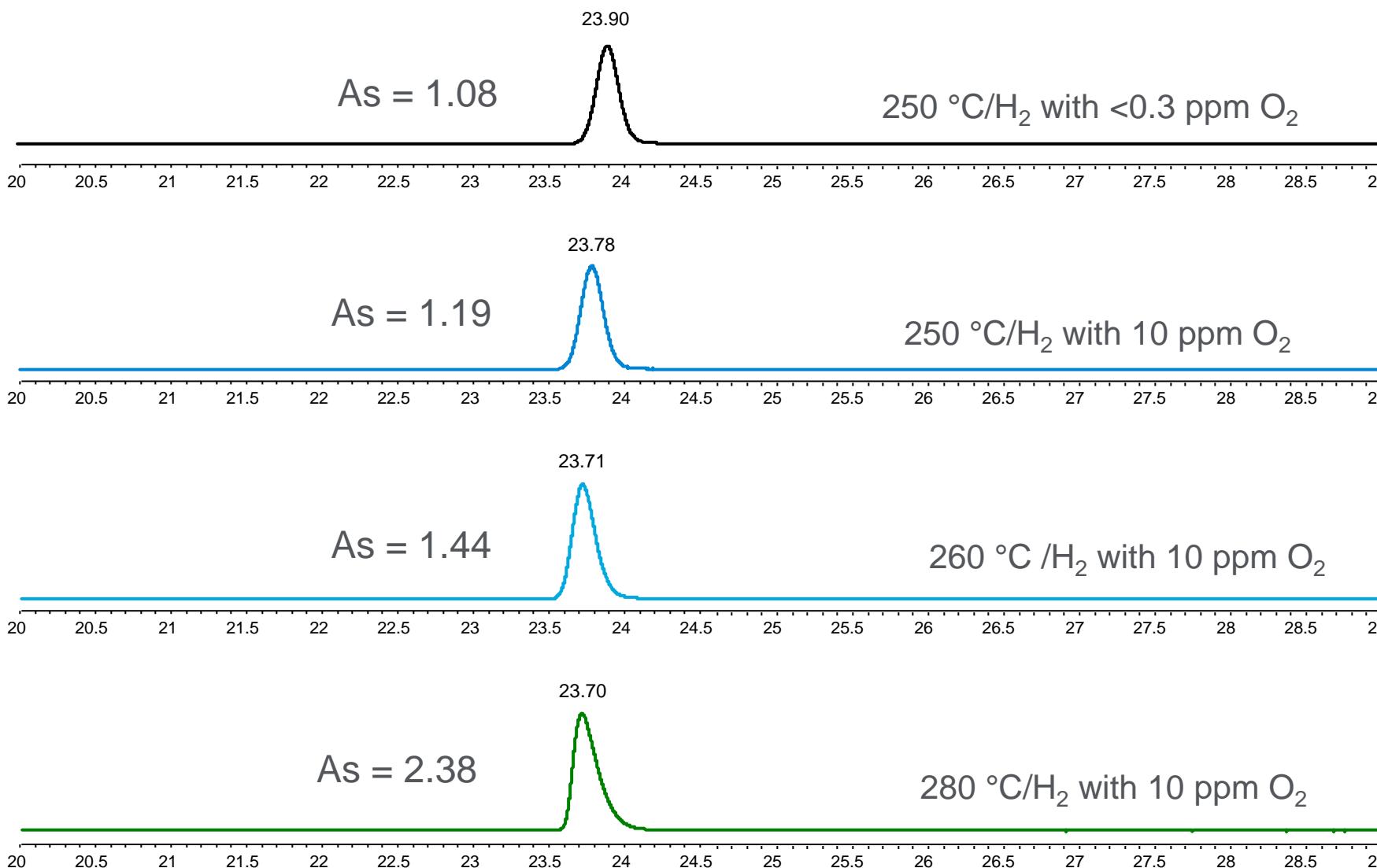
- Have you installed or conditioned the column?
- Are you exceeding the column's upper temperature limit?
- Is your column's film size too thick?
- Could leaks be present in your flow path, or are your carrier gases contaminated with air?
- Do you need to change your split vent trap?

Oxidation (O_2 Damage)

Oxygen in the carrier gas rapidly degrades the stationary phase. The damage is accelerated at higher temperatures. Damage along the polymer backbone is irreversible (premature filament failure/excessive source maintenance).



Effect of Oxygen on Peak Shape of 2-Ethylhexanoic Acid



Knowing If You Have a Leak Before Using Your GC



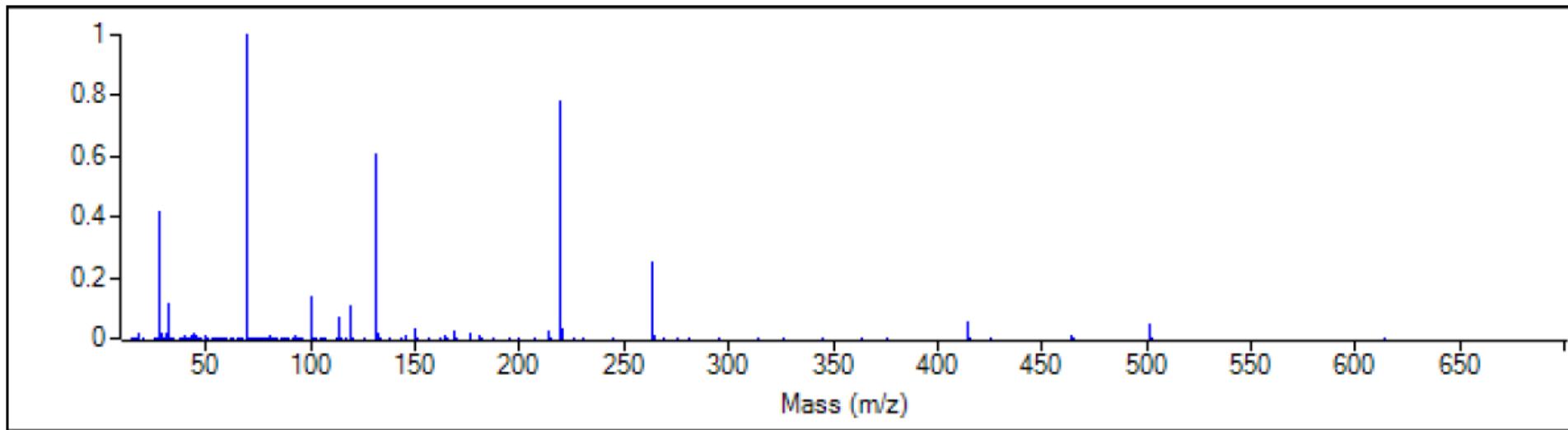
p/n CP17973

www.agilent.com/chem/gasclean



Remove plugs
before installation

Example Tune Report with Leak

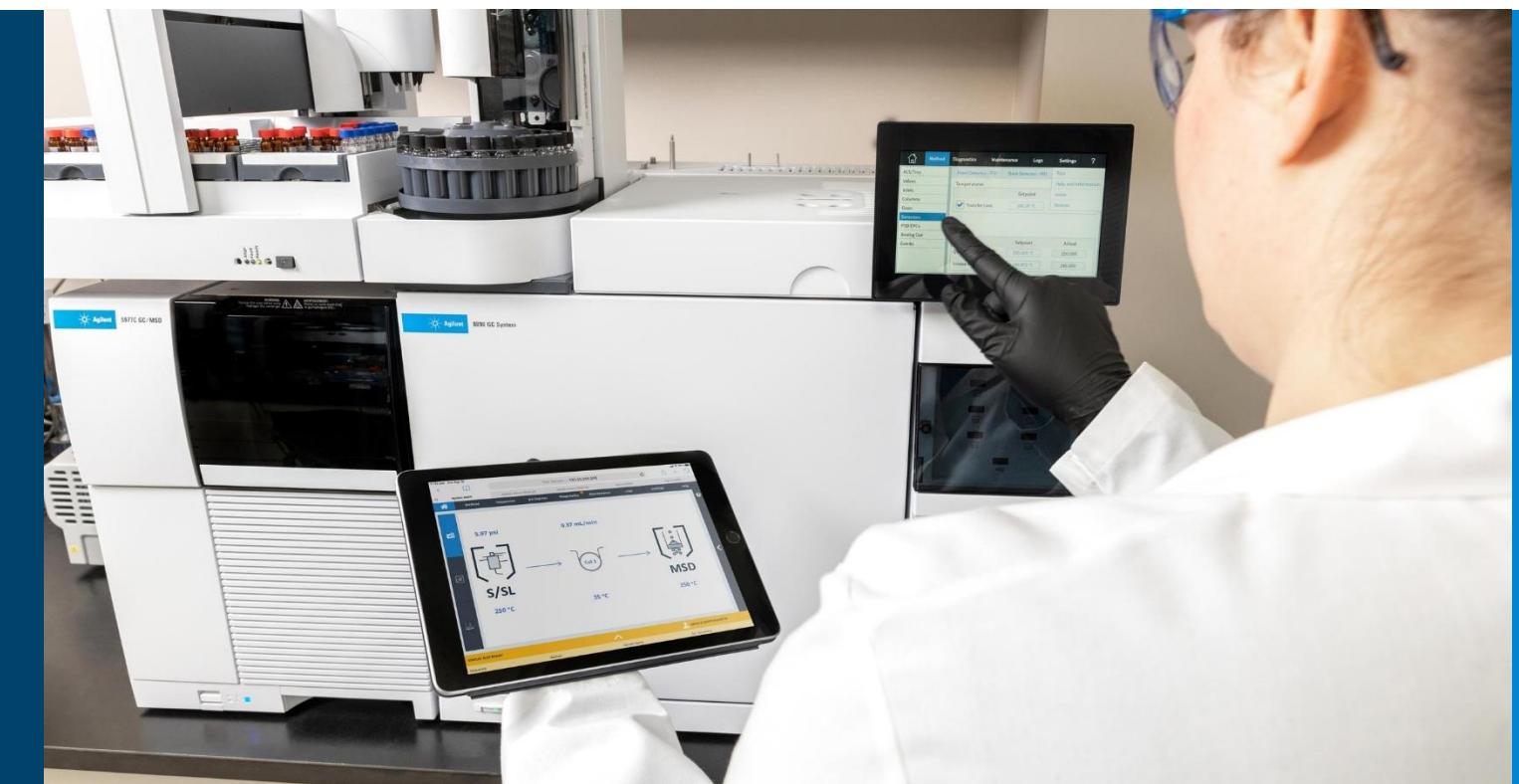


Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	498,432	100.0%	70.00	6,216	1.2%
219.00	219.00	391,232	78.5%	220.00	18,216	4.7%
502.00	502.00	23,680	4.8%	503.00	2,467	10.4%

Air/Water Check: H₂O ~1.8% N₂ ~42.1% O₂ ~11.4% CO₂ ~1.3% N₂/H₂O ~2325.0%

Column(1) Flow: 1.00 Column(2): 1.20 ml/min Interface Temp: 250

Instrument Diagnostics



Pressure-Decay Test

The pressure decay test checks for leaks from the inlet flow module up to the column fitting.

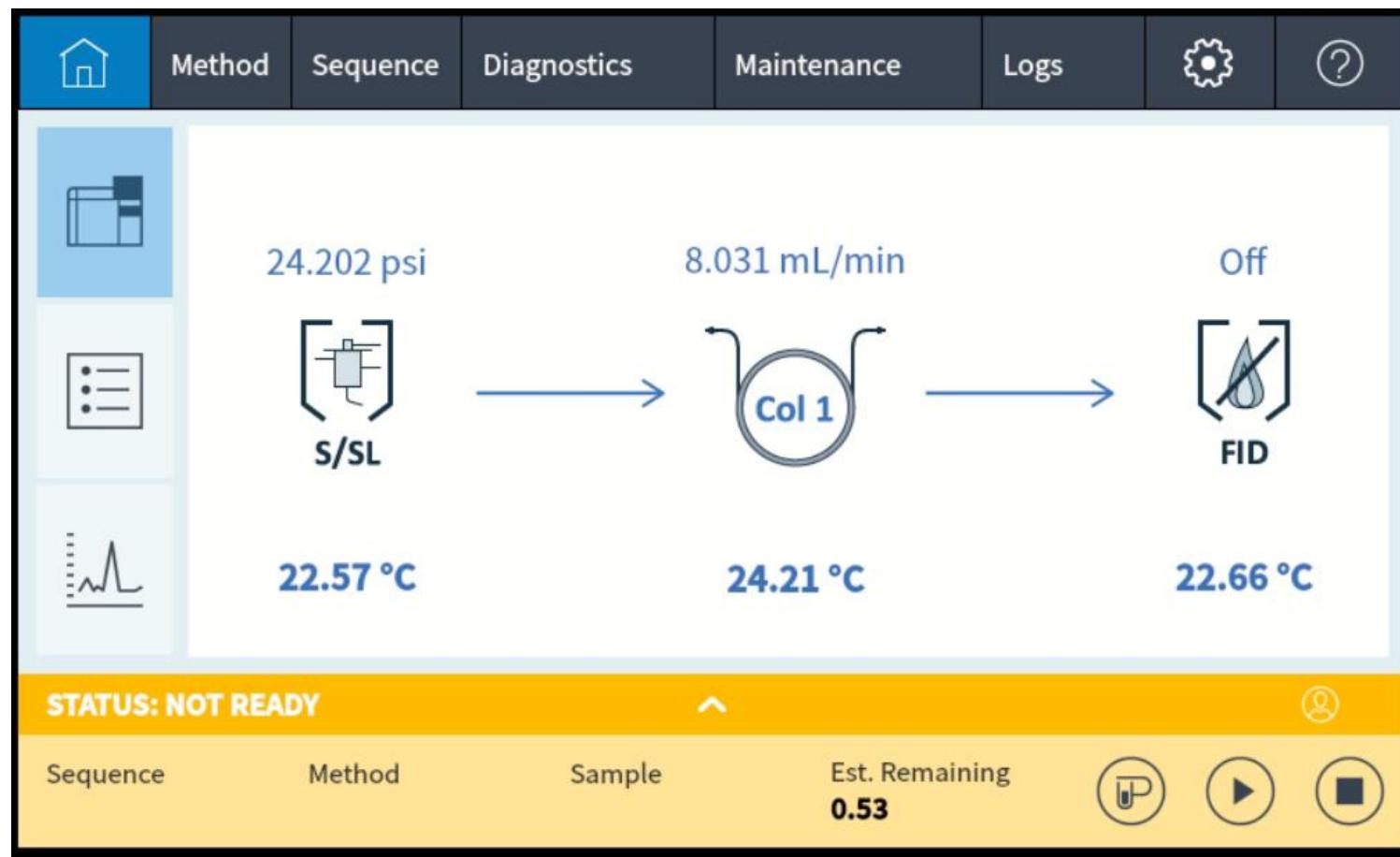
The test can find leaks at the:	The test cannot find leaks at the:
septum	column fitting
septum nut	gas supply bulkhead fittings to the flow module
liner O-ring seal	tubing and connections in a transfer line connected to the inlet
gold seal/washer and reducing nut	internal leaks in an EPC module (septum purge valve)
inlet body	
flow manifold split vent valve	
flow manifold septum purge valve	
split vent tubing and trap	
septum purge tubing	
seals within the tubing between the inlet flow module and the inlet body	

1. Gather the following:

- No-hole ferrule
- ¼ inch wrench
- Heat-resistant gloves (if the inlet is hot)
- Column nut
- New septum
- O-ring
- ECD/TCD detector plug (p/n: 5060-9055)

<https://www.agilent.com/cs/library/troubleshootingguide/public/7890B%20Troubleshooting%20Manual.pdf>

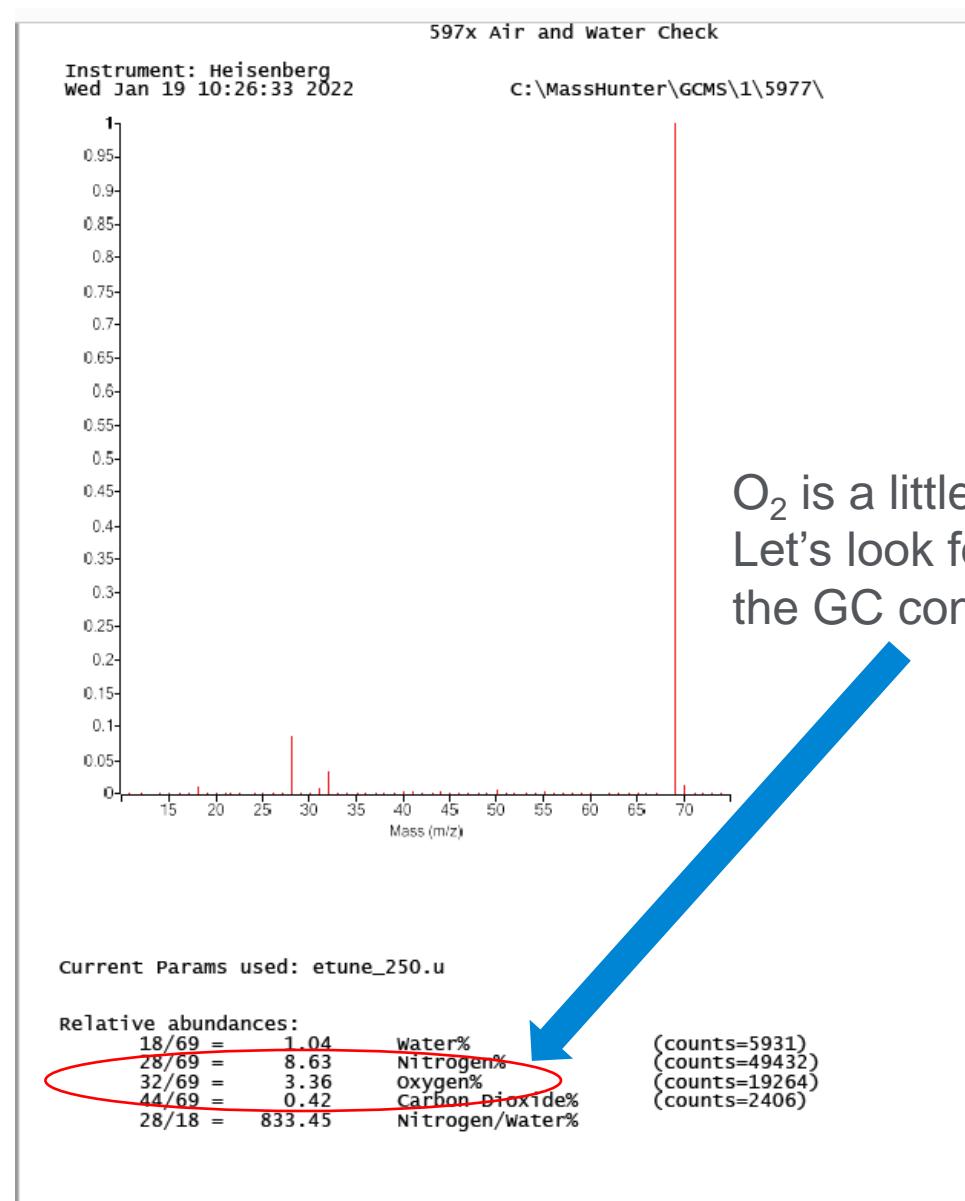
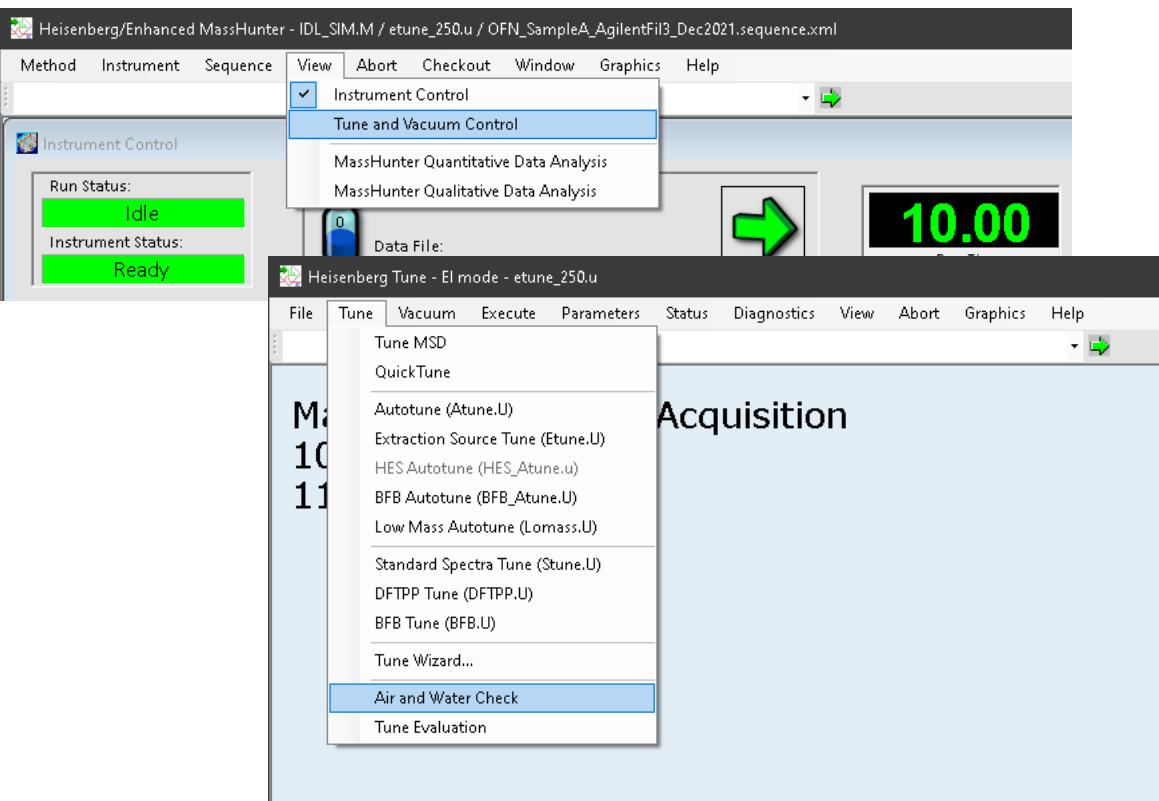
Pressure Decay Test on the 8890



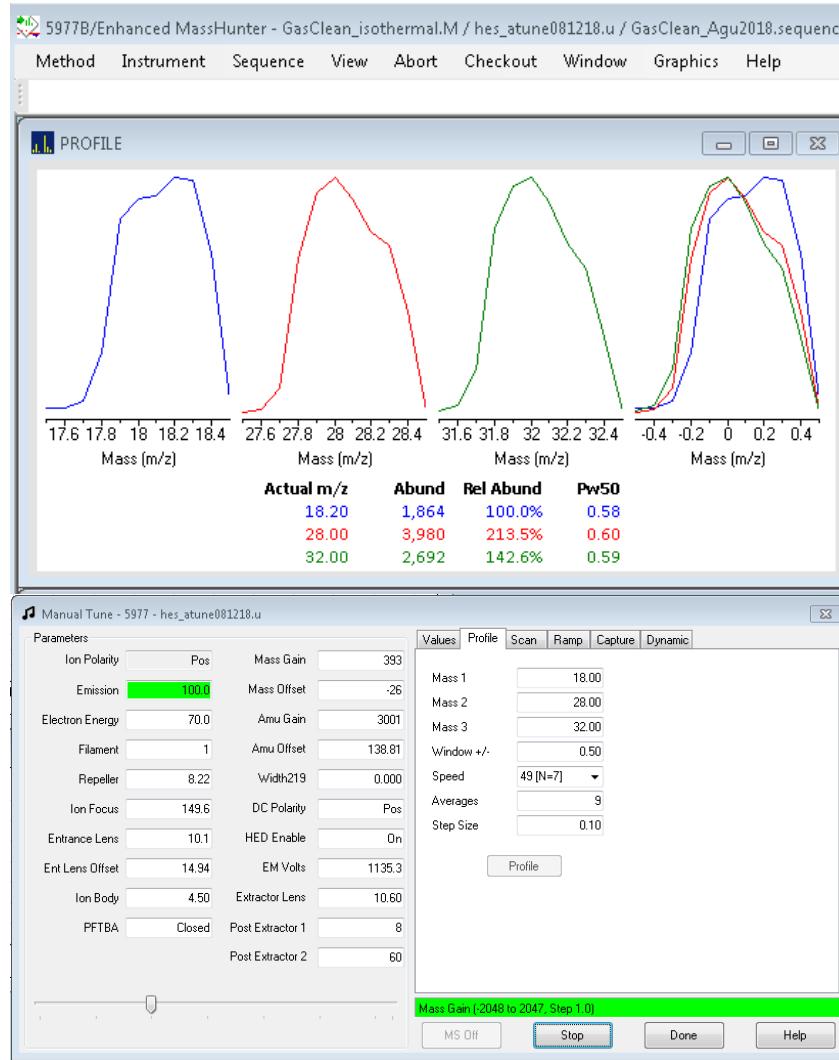
How to Run an Air/Water Check

Run an air water check

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



If My System Is Leak-free, What Should My Air Ion Abundances Be?

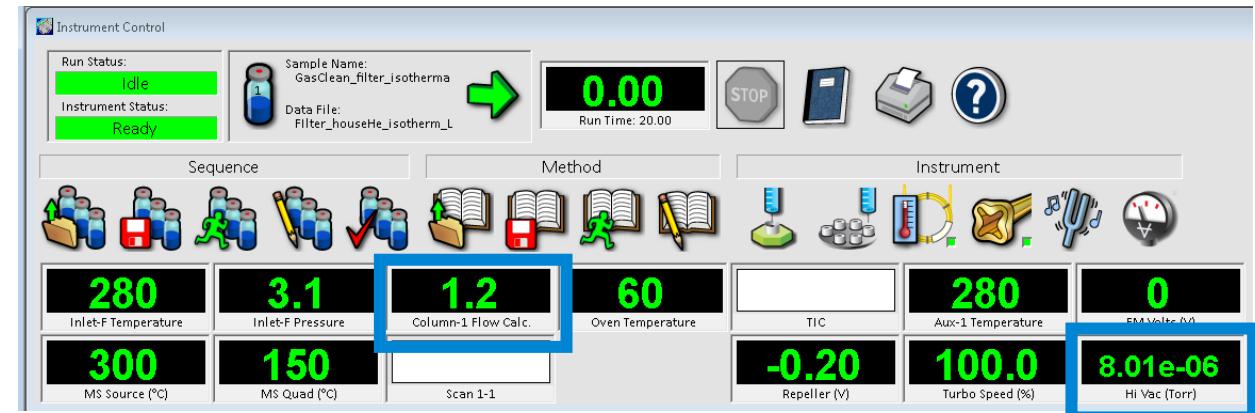


- These are just estimates
 - H₂O: ~2,000 counts (less is OK)
 - N₂: ~10,000 counts (less is OK)*
 - O₂: ~3,000 counts (less is OK)

*Make sure to purge your Gas Clean filter

High vacuum gauge pressure (for single quad):
 $\sim 1 \times 10^{-5}$ torr[†]

[†] dependent on flow rate



Atmospheric Gases and Instrument Performance Data

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

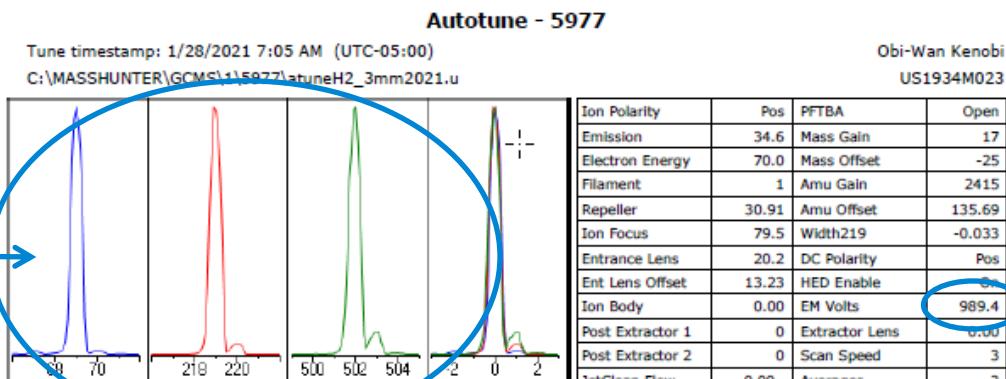
Mass Gain Values(Scan Speed): 23(3) 34(2) 41(1) 66(0) 118(FS1) 126(FS2)

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

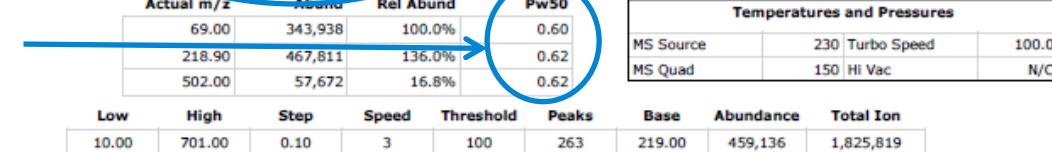
- Atmospheric gases (water, nitrogen, oxygen, and carbon dioxide) should be as low as possible.
 - The ratio of nitrogen to oxygen in atmospheric air is 3.7:1.
 - If the water is high, there will also be air present. Be patient and do not tune or acquire data until equilibrated.
- Why are two GC parameters included?
 - Tune parameters depend on flow rate and MS transfer line temperature.
- For single quadrupole instruments, the gain factor is the detector gain needed to achieve the target PFTBA abundance.
 - Gain will be discussed in detail in a future webinar.

What to Look For?

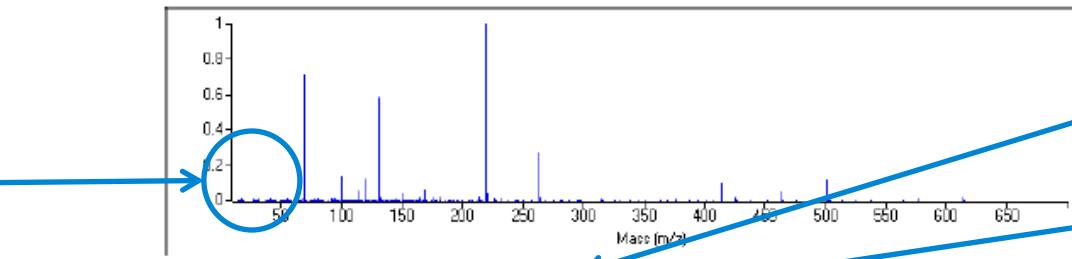
Symmetrical smooth peak shapes



Consistent mass peak widths



Low water and air



Correct mass assignments

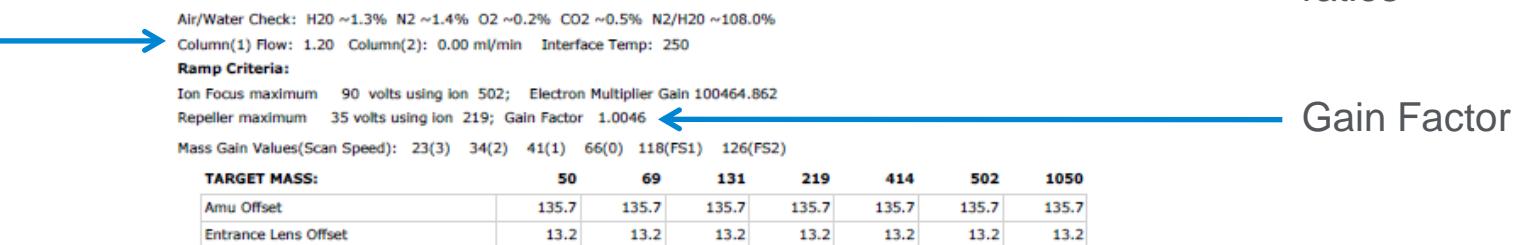


Proper absolute abundance

Typical relative abundance

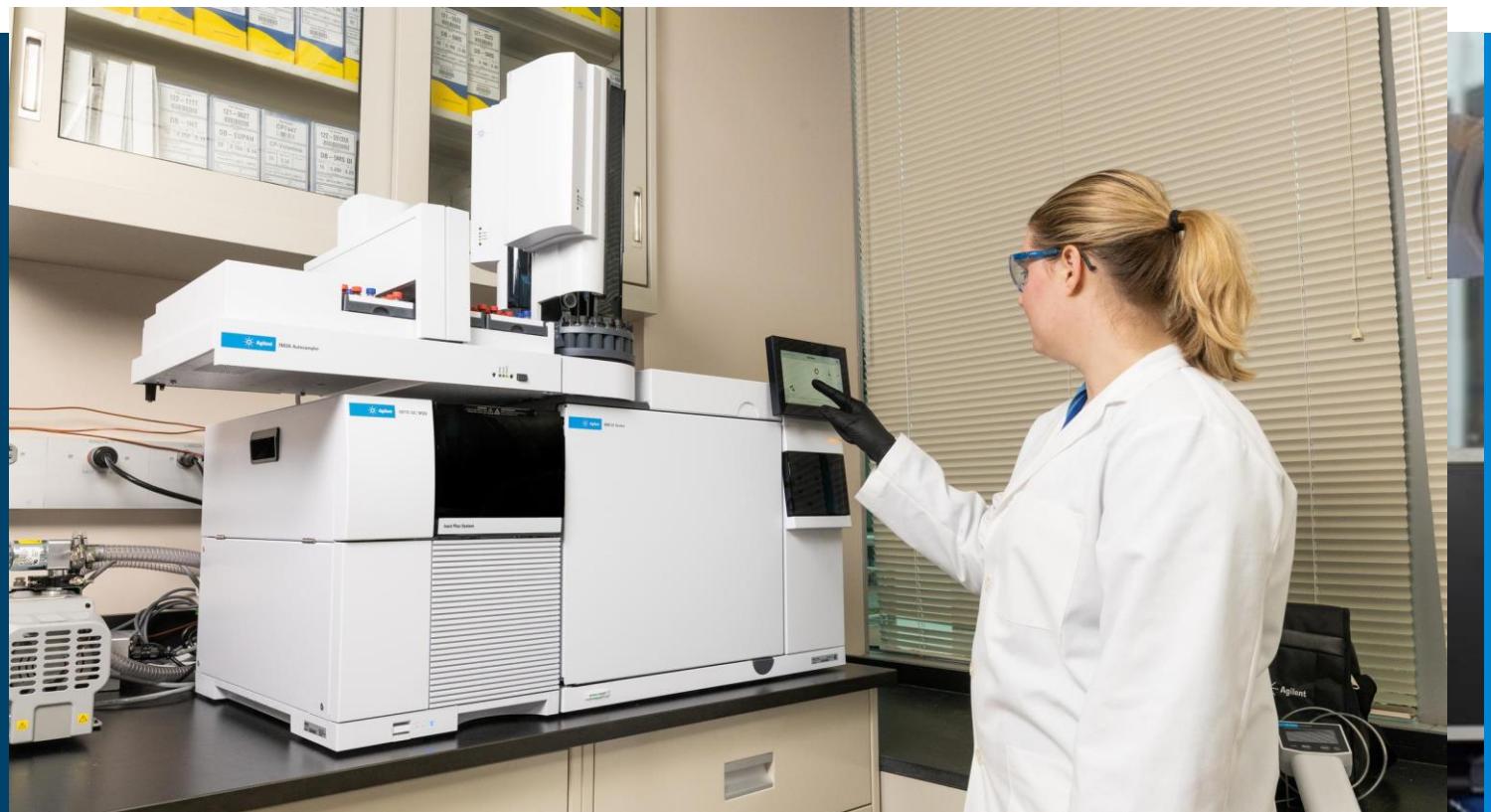
Proper isotope ratios

Acceptable column flow rate



Gain Factor

Finding the Leak



Agilent CrossLab CS (Cartridge System)

No peaks from leaks

Features:

- Exchangeable cartridge with ADM Flow Meter
- Automatic notification of probe filter replacement
- Ergonomic and robust design
- Universal 3AA or USB power
- USB connects to Web interface for added functionality and firmware updates
- Easy to view OLED screen
- Kickstand

Leak detector
cartridge



Handheld

ADM Flow Meter
cartridge

Information About the Leak Detector

- **Agilent.com CrossLab CS Leak Detector**

www.agilent.com/chem/gas-leak-detector

- **Agilent.com – ADM Flow Meter**

Agilent CrossLab CS Cartridge System | Agilent

- **Installation manual**

*Agilent CrossLab CS
Electronic Leak Detector manual*

Part number: G6693-90000

The installation manual is available on Agilent.com.

- **Innovation minute video**

The video is available on Agilent.com.

- **Technical overview**

*Agilent CrossLab Cartridge System
(CS) Electronic Leak Detector*

Publication number: 5994-4262EN

The technical overview is available on Agilent.com

- **Brochure**

*GC Troubleshooting in
the Palm of Your Hand*

Publication number: 5994-3607EN

The brochure is available on Agilent.com

- **Flyer**

*Is a Leak Causing Your
Inaccurate Results?*

Publication number: 5994-4202EN

The flyer is available on Agilent.com

Ordering Guide

One-year warranty

- G6693A – CrossLab CS Electronic Leak Detector
- G6694A – Electronic Leak Detector Cartridge
- G6699A – CrossLab CS Bundle: ADM Flow Meter and Electronic Leak Detector
 - The bundle will include one handheld, two cartridges, and a **free** carrying case.
- G6694-60005 – Replacement probe filter
- G6691-40500 – Carrying case



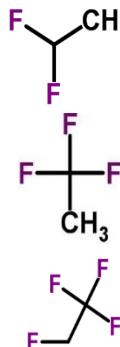
Existing products:

- G6691A – CrossLab CS ADM Flow Meter
- G6692A – ADM Flow Meter cartridge*
 - Note that the ADM Flow Meter cartridge is ordered annually for calibration. The Electronic Leak Detector does not need to be recalibrated.

Use Electronics Duster to Find Your Leaks with an MSD

- Hold can upright (don't spray liquid)
- Spray short bursts around likely leak suspects
- Spray just enough
- “Live” tune scanning for ions to pinpoint leak

Typical electronic duster components and ions



1,1-difluoroethane

m/z
51,65

1,1,1-trifluoroethane

m/z
83

1,1,1,2-tetrafluoroethane

m/z
101

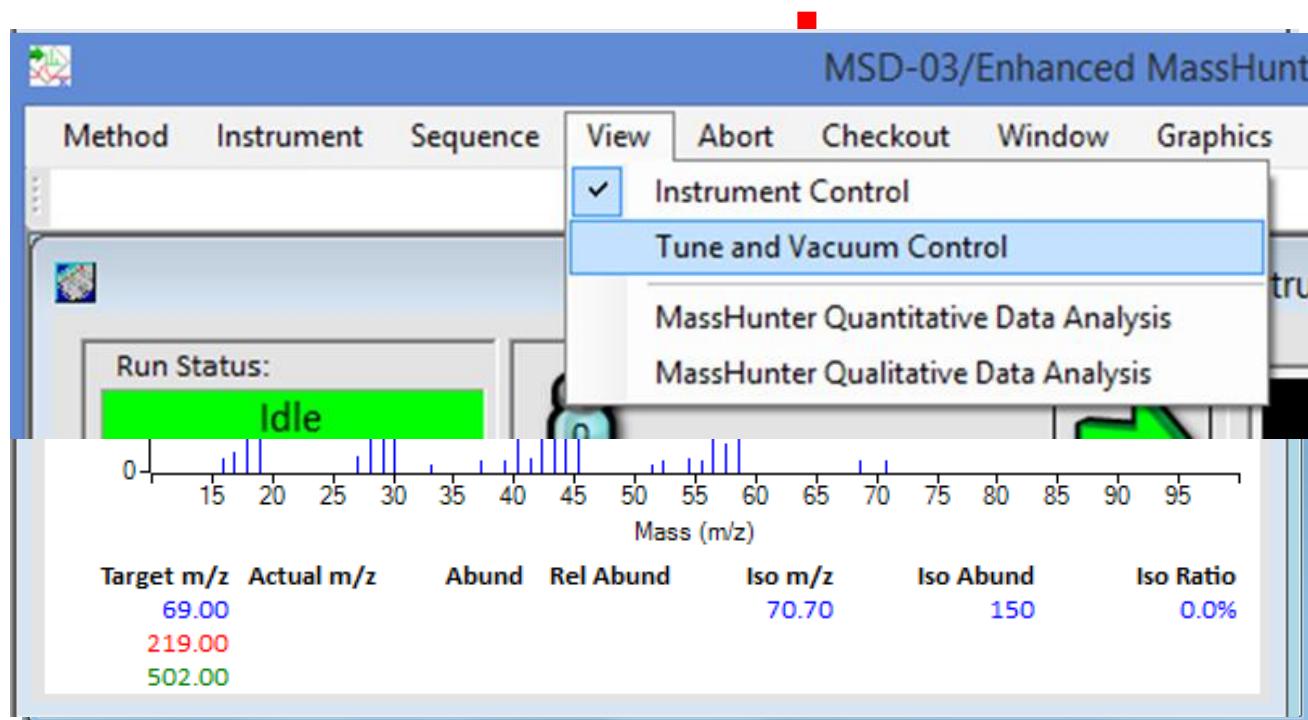


Hunt for Leaks in Manual Tune Mode

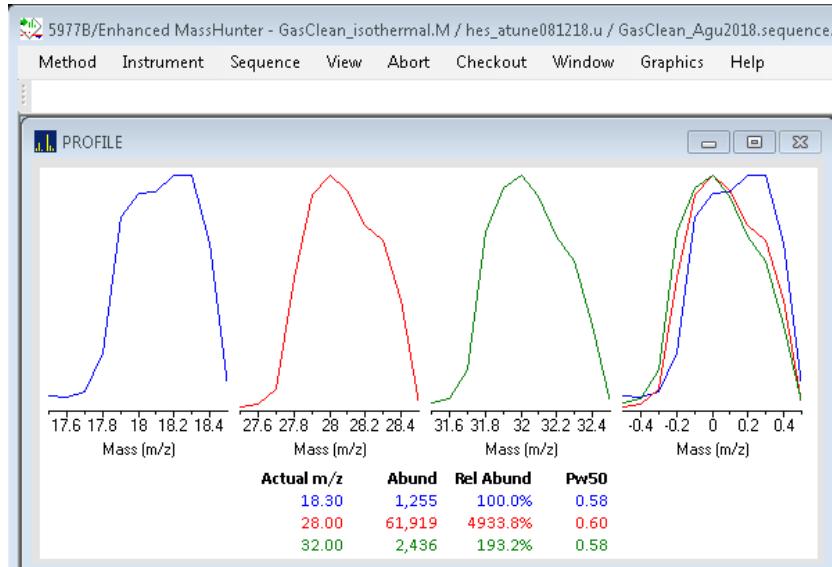
From MS tuning interface

In the Agilent MassHunter Acquisition,
Vacuum/Tune Control window:

1. Select Parameters > Manual Tune.
2. Click the Scan tab on the right side.
3. Set low m/z = 10, high m/z = 100 amu.
4. Ensure the PFTBA valve is closed.
5. Click Scan.
6. Spray common leak sites and watch the scan window for large duster ion spikes.
7. Exit the manual tune screen and do not save the parameters.
8. Return to acquisition.



I Have Gas Filters and High-quality Gas, But I Still Have a High Background



Use manual tune (before any experiments) to check for leaks/background.

- Ions m/z 18, 28, 32

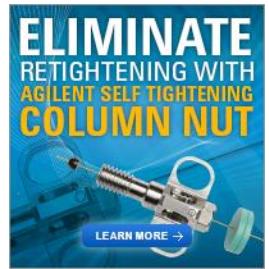
60,000 counts for N_2 is definitely high

What to check:

1. Gas line fittings (done).
2. Check the vent valve, MSD transfer line nut, and side door.



Check the Transfer Line Nut for Leaks



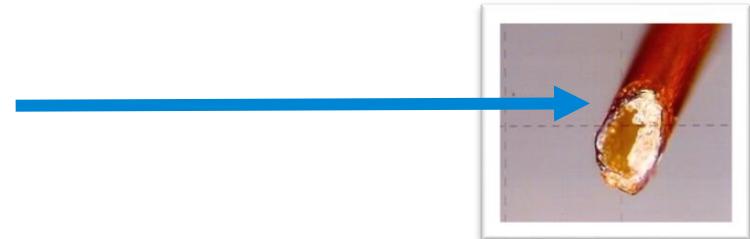
- Using a graphite/Vespel ferrule
 - Install with the flat end of the ferrule facing the MSD
- Nut “loosens” with heat cycles
- If you find a leak:
 - Tighten in small increments and then check again until there is no leak
 - Try to not overtighten the nut
 - If you have to apply a lot of pressure, vent and check the ferrule/threads



Be careful with the transfer line nut

- Over-tightening damages transfer line threads and column
- Audible squeaking → over-tightening

Crushed end of column



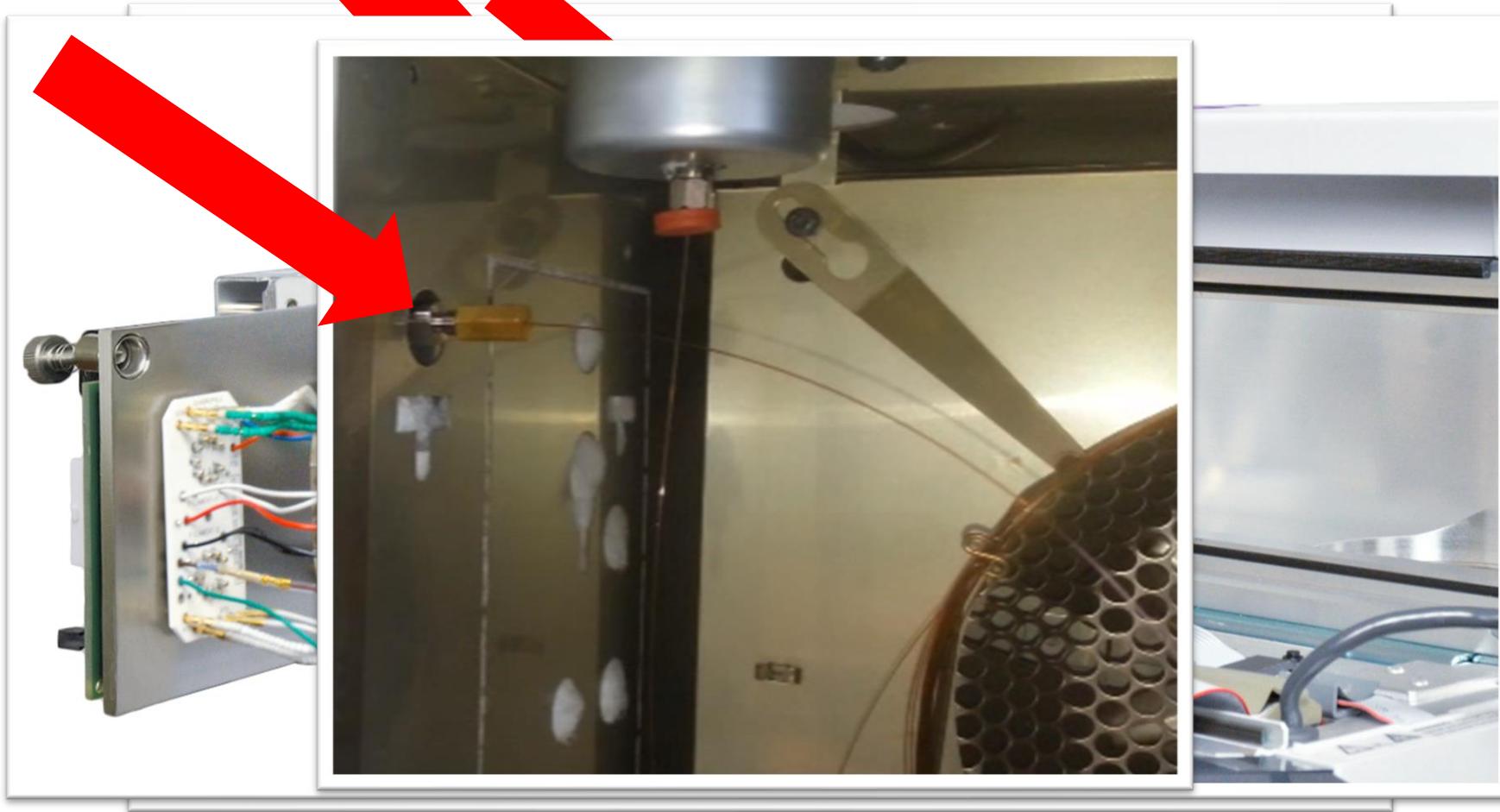
Good Habit: Check Other Connection Points in the GC



- **Check the inlet**
 - Installed with a quarter turn with a wrench
 - Inlet nut may need slight tightening after heating cycles
 - Or, preswage flexi-metal ferrules
 - Or, use Self Tightening column nut for inlet
 - Use graphite/Vespel ferrules with Self Tightening nuts
- **Check CFT connections**
 - Use CS Electronic Leak Detector or electronics duster
 - Short bursts of electronics duster at each connection point



Where to Look: Common Leak Sites



Water/Alcohol to Find Leaks

- You can spray a water/alcohol solution around fittings and look for bubbles
- Mainly for those that do not have an MSD or leak detector
- Can be difficult to see bubbles



How to Protect Your Instrument in the Future?



Gas Management



Inline gas purifiers

- Big traps, 750 mL
- Combination traps
- Universal traps

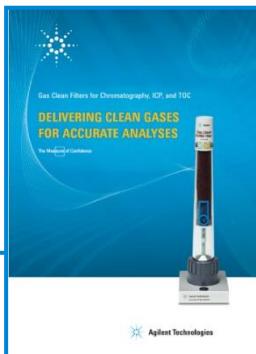


Gas Clean purification system

Carrier gas filter: O₂, H₂O, HC
Shipped with every GC/MS SQ

- O₂ specific
- H₂O specific
- Hydrocarbons

Ordering guide
5990-8243EN



ReNEWable filter

Single GC/MS cartridge for
O₂, H₂O, hydrocarbons

General Installation and Use of Filters

- Install all traps vertically – use the appropriate mounting bracket (or brackets) for your trap
 - Flow should go from the top down
 - Do not place the filter horizontally (mounting clips available)
- For disposal, always check the safety data sheet (SDS) and dispose of the contents and container in accordance with all local, regional, national, and international regulations

Filter Types

Inline gas purification

Connected directly to the gas lines

- To change or install a filter, you must stop the gas flow
- Typically, they have a large capacity compared to the other options
 - Example of our big universal traps (last ~13 “K” sized tanks):

Capacity Data

High Purity Helium-99.997%

	Removal Capacity	
O ₂	< 5 mg/L	1.07 L
THC*	< 1 mg/L	20 g
H ₂ O	< 5 mg/L	46 g

*Total Hydrocarbons, analysis limited to three contaminant groups

Effluent Concentration

Research Grade Helium-99.9999%

Impurities	< 1 mg/L	H ₂ O	< 0.2 mg/L
N ₂	< 0.5 mg/L	H ₂	< 0.2 mg/L
O ₂	< 0.5 mg/L	Ar	< 0.1 mg/L
THC*	< 0.1 mg/L	Ne	< 0.5 mg/L
CO+CO ₂	< 0.1 mg/L		

*Total Hydrocarbons, analysis limited to three contaminant groups



Big moisture trap

Hydrocarbon Moisture trap

Refillable moisture trap MT120

Filter Types

GasClean purification system

Connect to a baseplate, which is plumbed inline with the gas

- Does not require turning off the gas upstream
 - Internal valving system turns off the flow when the filter is not in place
 - Quick and easy to change
 - Less chance of leaks or atmosphere entering the line
 - Indicators for oxygen and moisture

Gas Clean Filters Technical Specifications

Description	Function	Indicator Color Change	Capacity	Outlet Concentration (at operating flow of 1-10 L/min)
Oxygen Filter	Removes oxygen as well as traces of sulfur and chlorine compounds from carrier gas	From green to gray	150 mL oxygen	< 50 µg/L
Moisture Filter/Process Moisture Filter	Removes water, oil, and other foreign material from the carrier gas	From green to pale brown	7.2 g water	< 0.1 mg/L
Charcoal Filter	Removes organic compounds from gas streams	No indicator	Approximately 7 g, depending on impurities	< 0.1 mg/L
GC/MS Filter	Single combination filter, removes water, oxygen, and organic compounds	Oxygen, from green to gray; Moisture, from green to pale brown	100 mL oxygen, 1 g water, organics depending on impurities	Oxygen < 50 µg/L Moisture < 0.1 mg/L Organics < 0.1 mg/L
CO ₂ Filter	Removes CO ₂ from the gas stream; use with moisture filter	From white to violet	9 g CO ₂	< 1 mg/L



GC/MS filter
Agilent p/n
CP17973



Filter Types

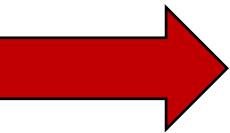
GasClean

GasClean uses a base plate and a filter, ordered them separately.

- Base plates come with 1/4 or 1/8 in fittings.
 - Options of one, two, or four positions for the filters.
- Filters come as:
 - Carbon dioxide
 - Oxygen
 - Moisture
 - Charcoal
 - Triple filter of oxygen, moisture, charcoal
- P/ns found in our brochure -
<https://www.agilent.com/cs/library/brochures/5990-8243ENGasCleanFilters.pdf>



Gas Trap Indicators Help Tell You When It's Time to Change Them



If there are no indicators, then change them periodically or according to your SOP.

Agilent Gas Clean sensor
(Agilent 8890 and 8860 GC systems)

Filter Types

ReNEWable gas purification system

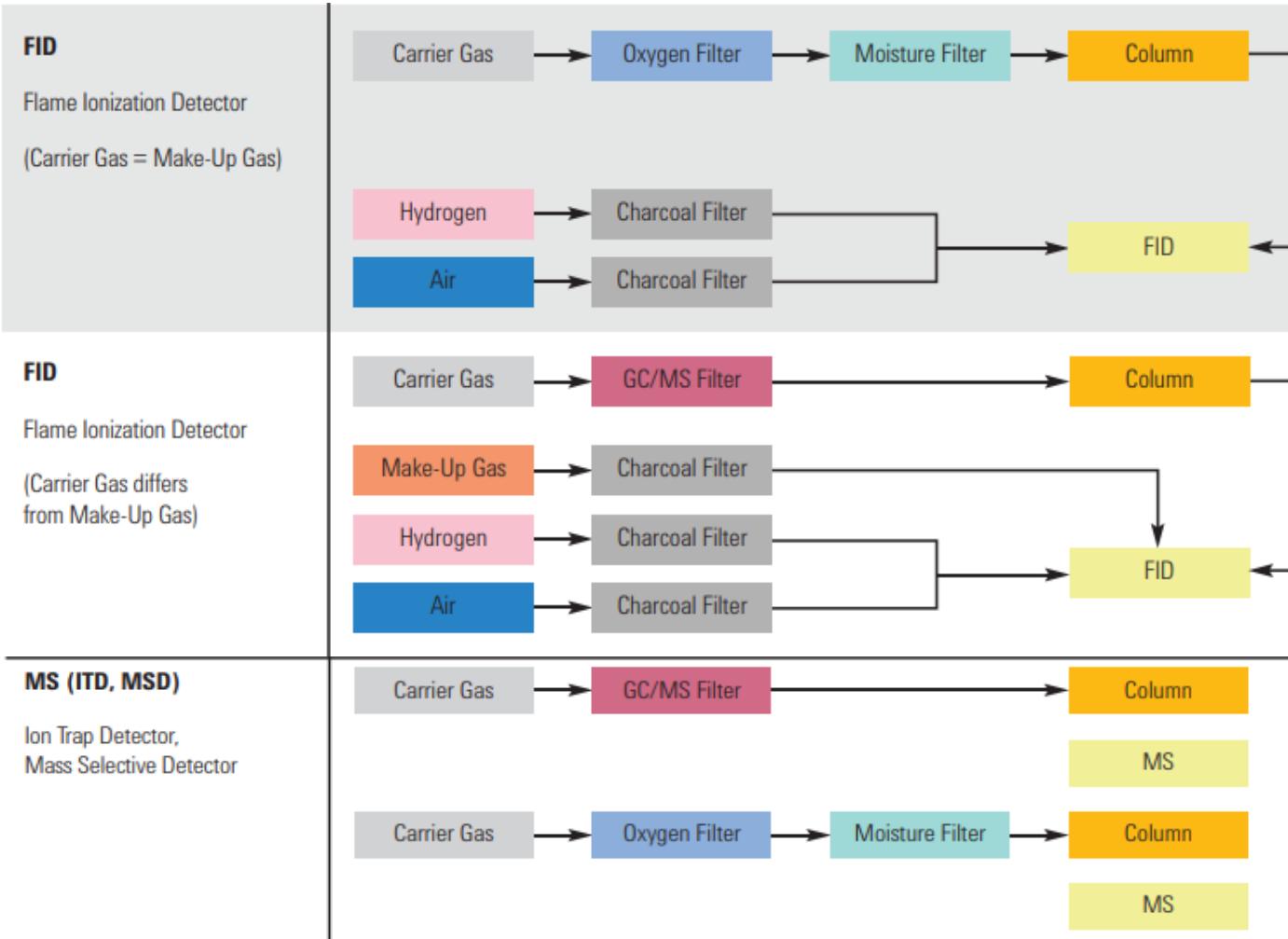
Connects to a special mounting bracket

- Combination trap for oxygen, moisture, and hydrocarbons
 - Can be returned to Agilent and recycled
 - This process is supported by our ordering team, please contact them for returns and reordering
 - Large capacity
 - 850 cm³ or more oxygen filtration, 12 g H₂O, and approximately 8 g hydrocarbon filtration per cartridge



Examples of Different Setups for Systems

Different filters are required, based on the gasses used and how they are being used:



- Moisture traps are typically last in line
- Always mount traps vertically, plumbed top down

https://www.agilent.com/cs/library/usermanuals/public/GasCleanFilter_5973-1528.pdf

Find Recommended Filters

We have a few resources to find filters for your specific system:

- Agilent Gas Clean Filter System User Manual –
https://www.agilent.com/cs/library/usermanuals/public/GasCleanFilter_5973-1528.pdf
- GasClean Selection Tool – <http://gascleanfiltertool.chem.agilent.com/>
- Agilent General Chromatography catalog – <https://www.agilent.com/cs/library/catalogs/public/5991-1056EN%20General%20Catalog.pdf>



Gas Setup

Connections

- **Do not** use pipe dope, liquid sealant, PTFE tape, or chlorinated/halogenated solvents on tubing or fittings
 - An exception is PTFE tape — this is needed to seal the NPT connection at the regulator (0460-1266)
- Agilent uses Swagelok fittings for connections to the instrument
 - Our regulators ship with a 1/4 in NPT to 1/8 in Swagelok adapter
- We often recommend ordering an install kit that comes with the connections and parts used in the plumbing setup, such as:
 - 1/8 in brass nut and ferrule set, 20/pk, 5080-8750
 - 1/8 in tee, brass, 2/pk, 5180-4160
 - 1/8 in cap, brass, 6/pk, 5180-4121

Description	Part number
Swagelok 1/8-inch to female 1/4-inch NPT, brass	0100-0118
Swagelok 1/4-inch to female 1/4-inch NPT, brass	0100-0119
Reducing union, 1/4-inch to 1/8-inch, brass, 2/pk	5180-4131

Making Better GC Connections

- Select supplies appropriate to your instrument and application
- Follow the recommended assembly process
- Maximize productivity by using innovative tools and supplies
- Column installation preswaging tools
- Self Tightening column nuts
- Ultra Inert press fit connectors
- UltiMetal Plus Flexible Metal ferrules/Gold ferrules
- UltiMetal Plus tubing and fittings



How Do You Make Better GC Column Connections?

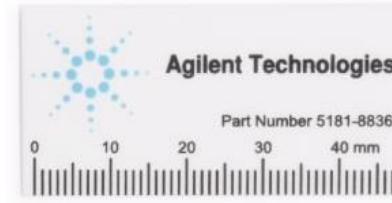
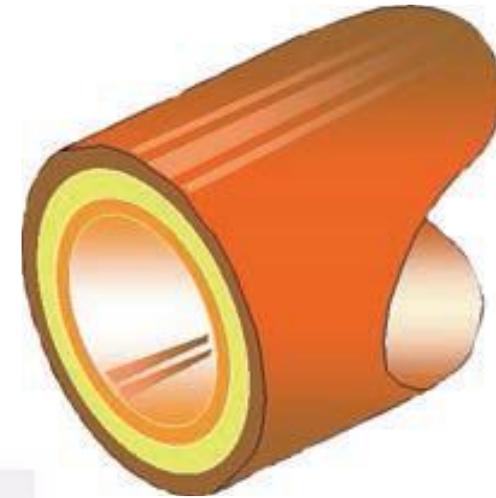
1. Start with the right tools and supplies

- Column nuts
- Material: stainless, but need brass for MS interface
- Choice of right ferrules

2. Proper assembly process

- Make a good clean column cut, every time
- After ferrule is installed
- Fused silica tubing cutters: ceramic, diamond-tipped
- Magnifier to inspect the cut – cracked fused silica and flaps of polyimide are active sites that ruin chromatography

3. Ensure the proper and consistent length of column into the fitting



5181-8836

Supply Selection: Which Capillary Ferrules to Use?



Composition	Re-use	Max T	Use	Limitation
Graphite	Yes	450	FID, NPD, inlets	Contamination, permeable to air – not for oxygen-sensitive detectors
Polyimide/graphite (85%/15%)	Limited	350	MS, ECD, FID, inlets	Still shrinks after thermal cycles, creating leaks; need to retighten regularly
Flexible metal	No	450	Capillary Flow Technology - CFT (backflush, splitters)	May not seal well with damaged fittings or rough surfaces
Gold flexible metal	No	450	Capillary Flow Technology - CFT (backflush, splitters)	Seals better, especially for scratched or damaged fittings (gold plating tends to fill microscopic scratches)
Polyimide (Vespel)	Yes	280	Easy seal	Shrinks after heating, causing leaks after thermal cycle; isothermal only



Dial packaging

Capillary Column Nuts

Column nuts are determined by the instrument fitting

Basic mechanical fittings with little enhancements

Brass for the MS (soft to reduce transfer line damage)

Some finger-tight designs for ease-of-use

New design addresses ease-of-use and productivity issues of leaking and over-tightening



Graphite/Polyimide Blend Capillary Ferrules

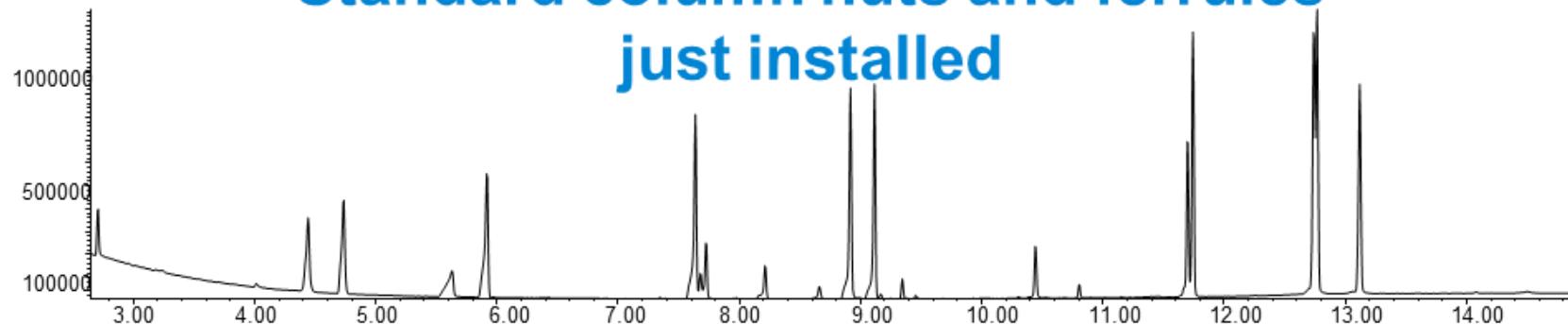
Unfortunately, there was a leak following normal temperature program runs.

Studies show that the leaking continues with use of the ferrules.

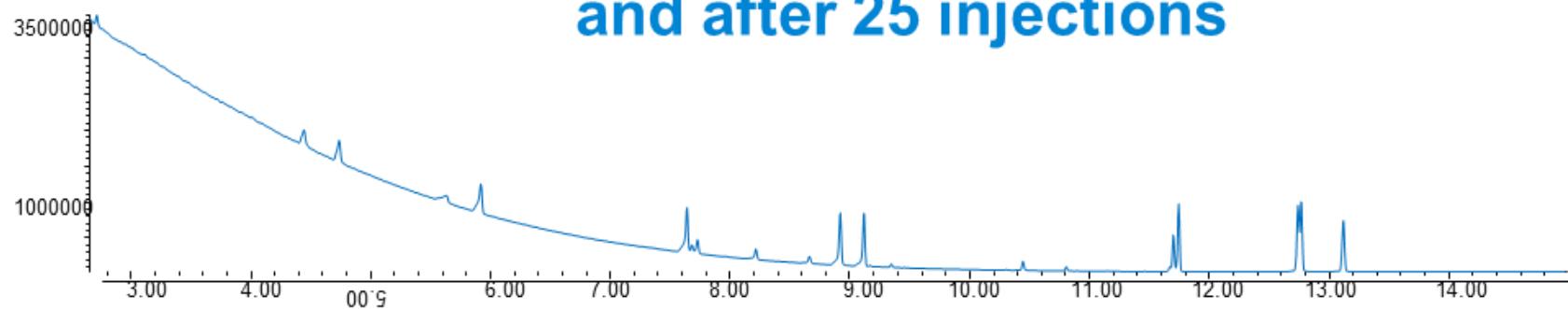
Not just after the first one or two runs.

Frequent retightening of the fitting is needed to maintain a leak-free seal, and system performance and productivity.

**Standard column nuts and ferrules
just installed**



and after 25 injections



Better Connections: Collared Self Tightening Column Nuts

Designed for use with short graphite/polyimide blend ferrules—both at the inlet and the MS interface—so only one type of ferrule is needed for both ends of the column.

Replacement collar

G3440-81012



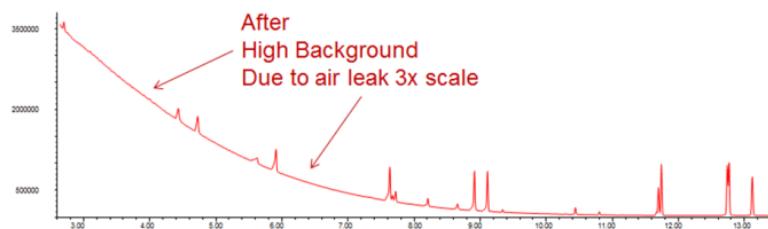
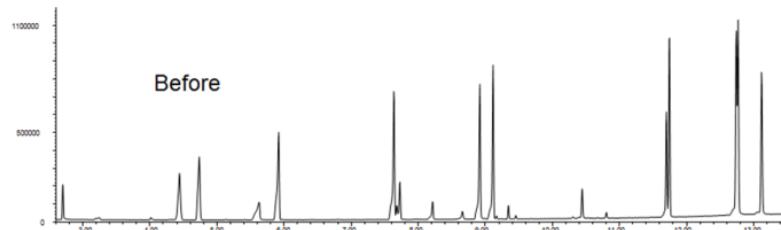
For inlet or detector
p/n G3440-81011



For mass spectrometry transfer line
p/n G3440-81013



The Benefit of Self Tightening Column Nuts

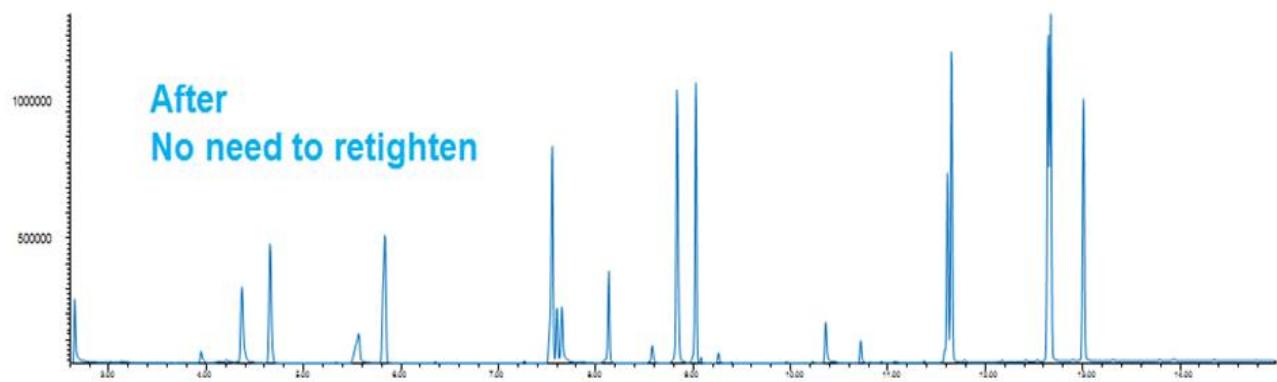
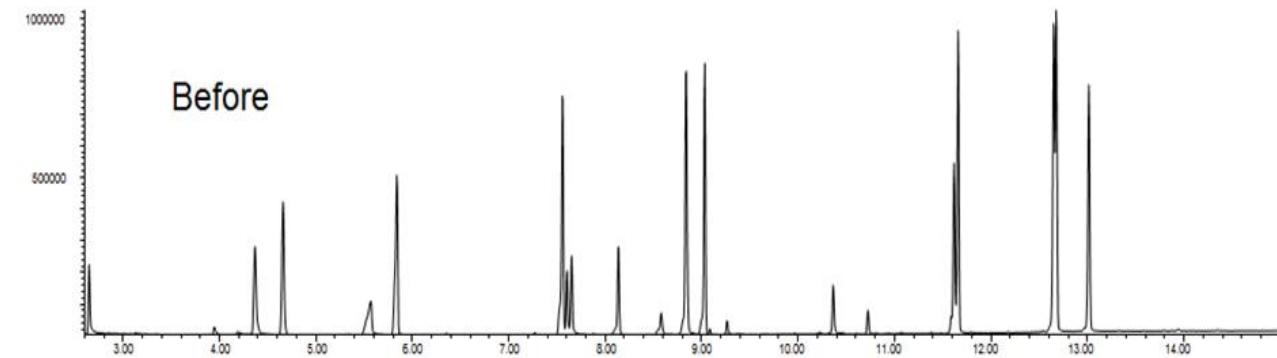


Without retightening, the baseline remains flat after 400 runs with no indication of leaks when using the Self Tightening column nut.

Technical note: 5991-3612EN

They can take you from this...

...to this

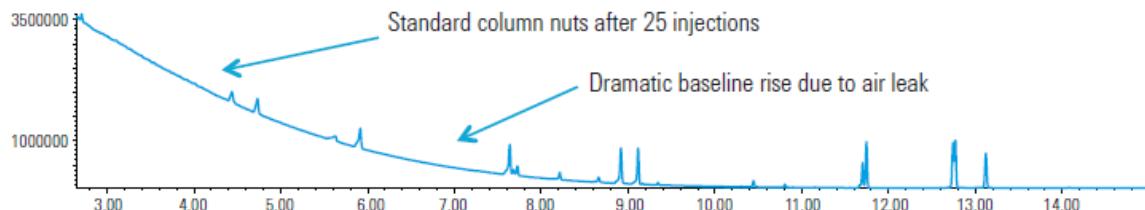
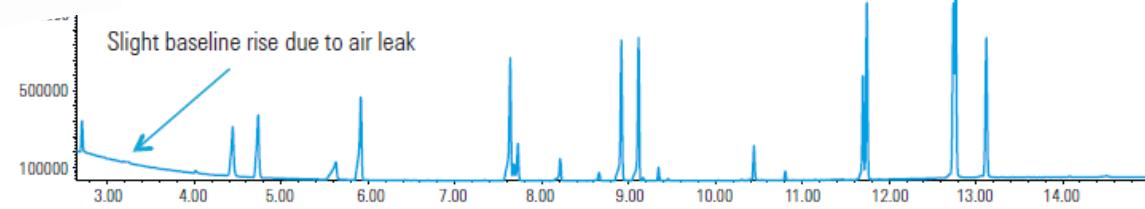


Self Tightening Nuts: No Leaks, No Downtime, No Frustration

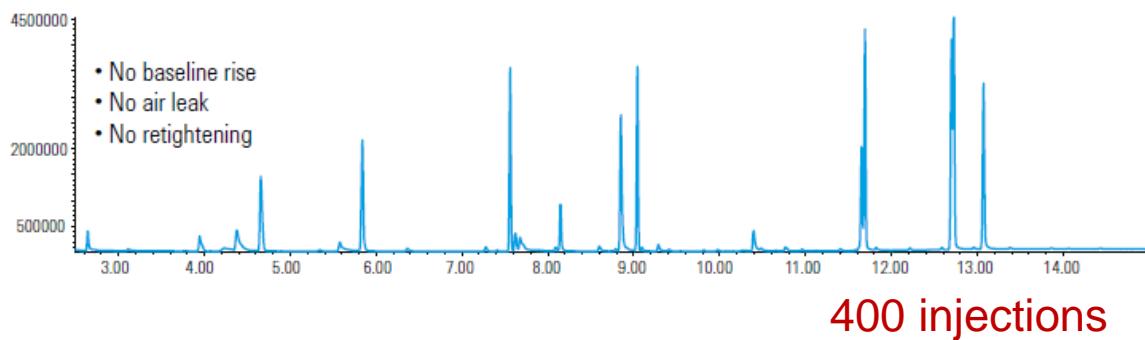
- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- No tools needed
- **Works only with short graphite/Vespel ferrules**



column nuts new fitting



Agilent Self Tightening Column Nuts after 400 injections



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>

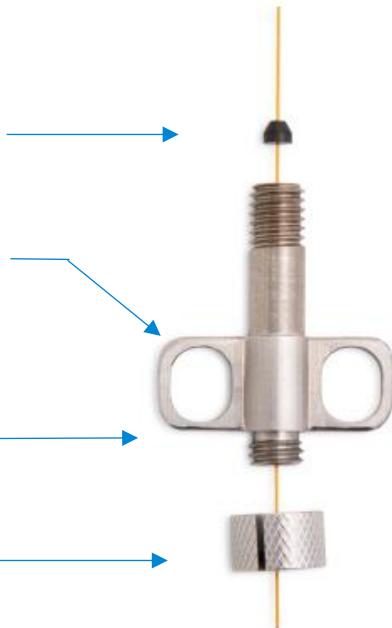
<https://www.agilent.com/en/video/stcn-mass-spec>

New Agilent Standard Winged Nut and Depth Guide

Agilent

EI MS

- Compatible with Agilent/HP style compact ferrules, including graphite ferrules
- Winged fastener design for easy engagement and tool-free installation
- Hollow-body design with low thermal mass mitigates thermal lag during temperature cycling within the GC oven
- Removable locking-collar with soft-PTFE insert to secure column placement during installation without damaging the analytical column



Don't confuse them with the Self Tightening nuts



Inlet / Detector
G3440-81018



MSD
G3440-81019



- Easy-to-use template provides critical capillary column installation for the most popular Agilent GC configurations
 - SSL, MMI, purge-packed inlets
 - FID, TCD, NPD detectors
 - EI MSD source
- Compatible with the Agilent Self Tightening and winged column nuts

G3440-88000

FID

MMI

SSL

PP

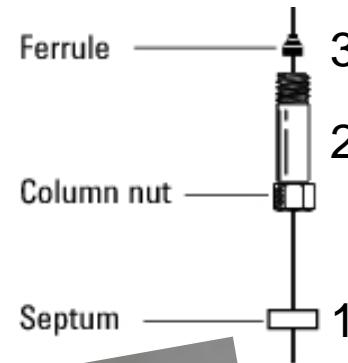


Matching the Correct Nut With the Correct Ferrule

<u>Nuts/Ferrules for Inlets and Non-MS Detectors (Male)</u>			
Photo	Nut	Photo	Ferrule
	Traditional/legacy nut		Short
	Winged nut		Short
	Self Tightening column nut		Short – G/V only

<u>Nuts/Ferrules for MSD (Female – G/V Only)</u>			
Photo	Nut	Photo	Ferrule (G/V Only)
	Traditional/legacy nut		Long
	Winged nut		Long
	Self Tightening column nut		Short – G/V only

Column Installation Assembly Process



Thread through an inlet septum.

Pass the column through the column nut.

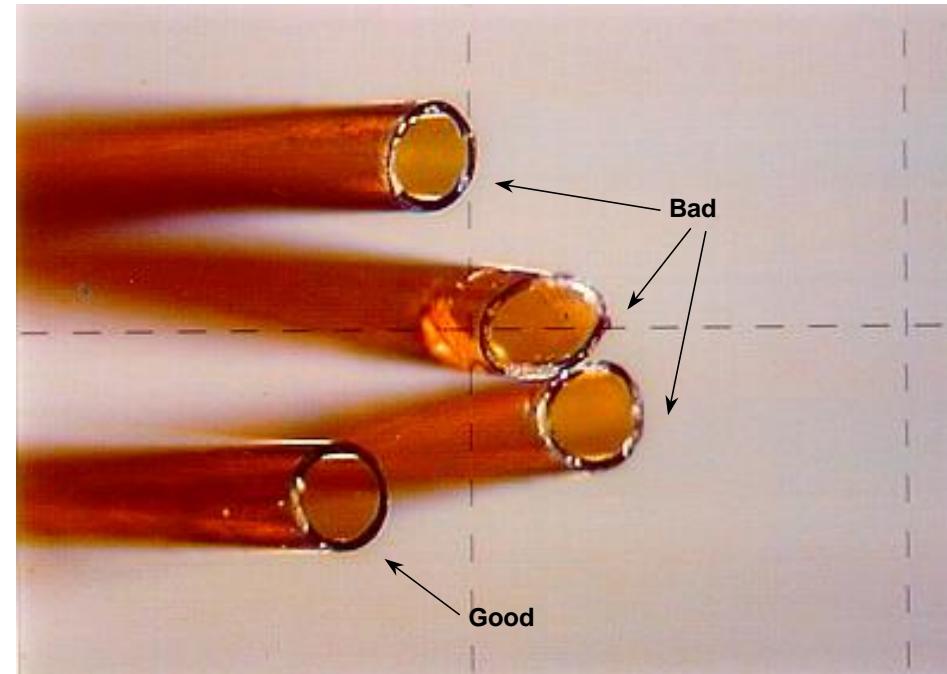
Install the ferrule onto the column tubing.

Then make a fresh cut.

Inspect the cut; repeat if there are any jagged or rough edges.



430-1020



Column Conditioning

The system must be leak-free before conditioning the column.

Heat the column to the lower of the isothermal maximum temperature, **or** 20 to 30 °C above the highest operation temperature.

- Temperature programming is not necessary.

Stop conditioning when a stable baseline is obtained:

After 1 to 2 hours, usually

Column Installation Videos

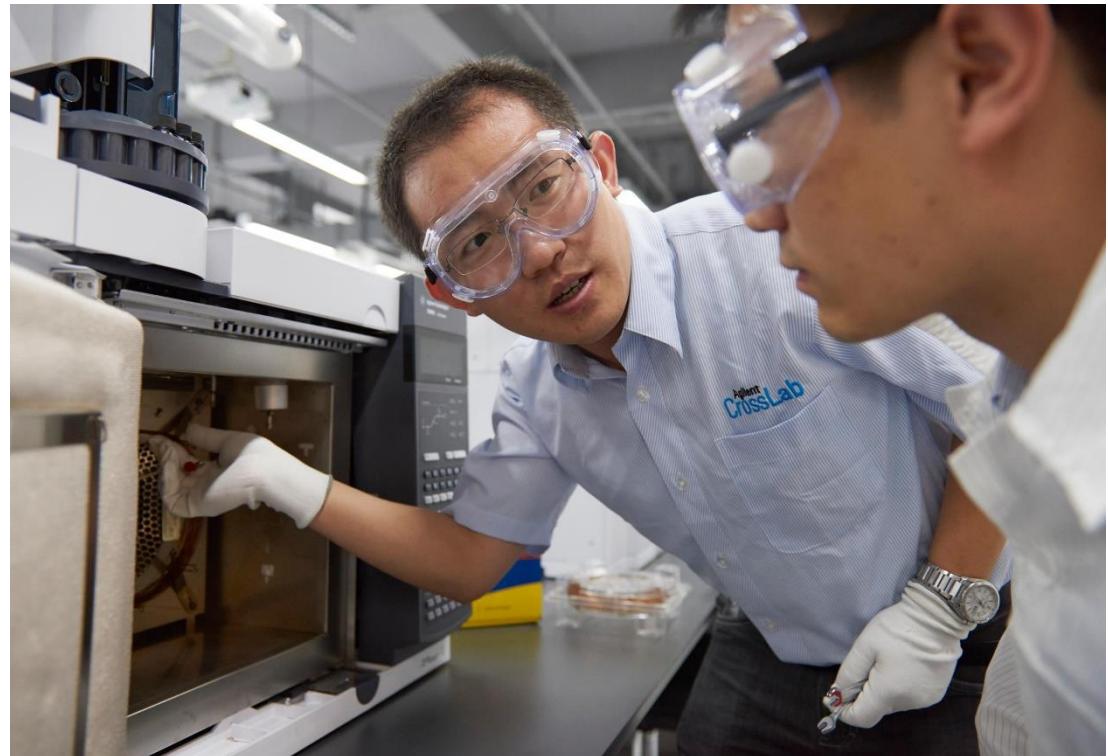
[Conditioning Your GC Column | Agilent](#)

[Front-End Maintenance for Your GC Column | Agilent](#)

[Installing a New GC Column into Your Inlet | Agilent](#)

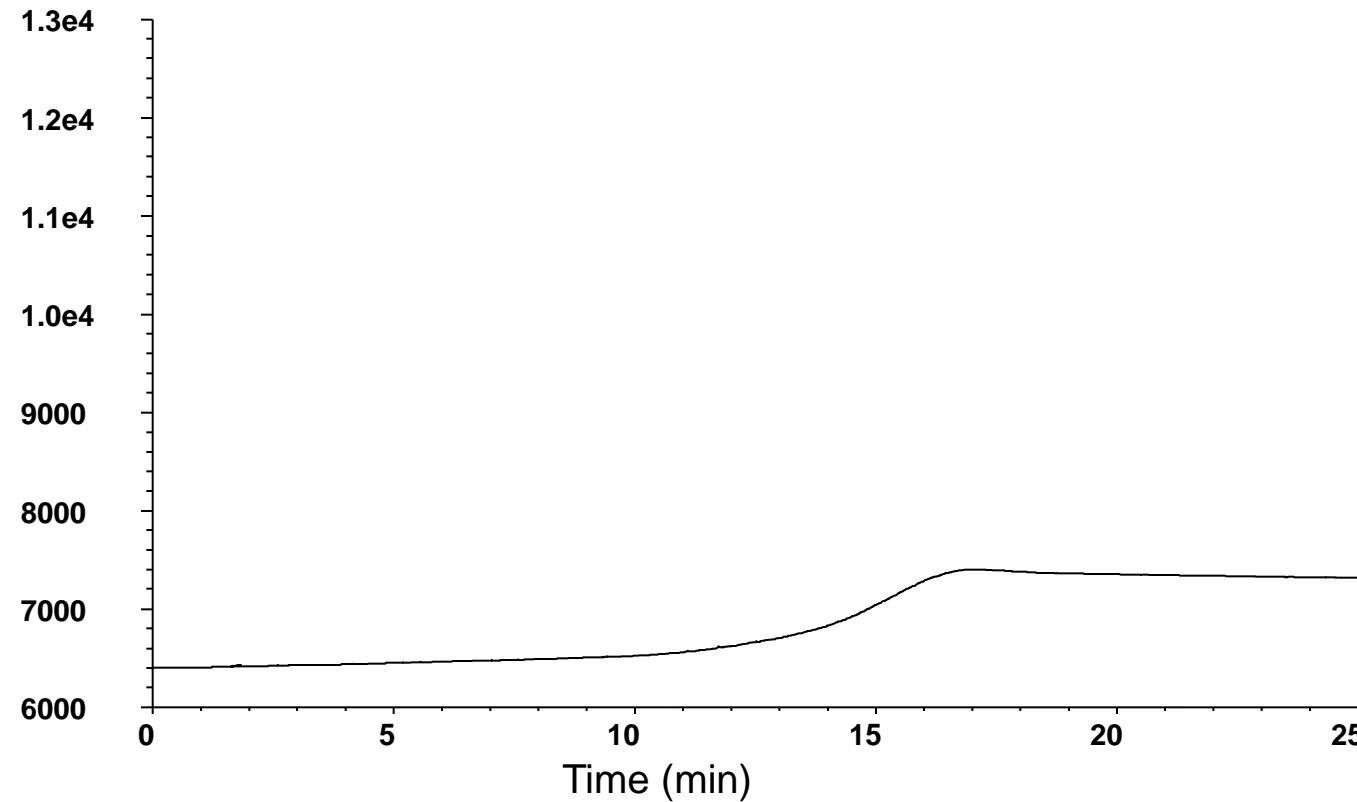
[Installing New GC Column into Your Atmospheric Detector | Agilent](#)

[Installing a New GC Column into Your Mass Spec | Agilent](#)



Generating a Bleed Profile

Temperature program the column without an injection*



*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

Always Remember

- Start with a good installation
- Maintain an oxygen-free system
- Avoid physical, thermal, and chemical damage
- Take steps to prevent contamination



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Overtasked staff

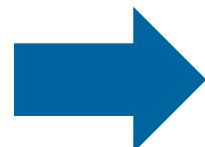
Staff turnover

Pressure to improve quality and productivity

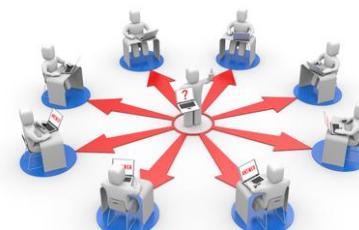
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In-person training



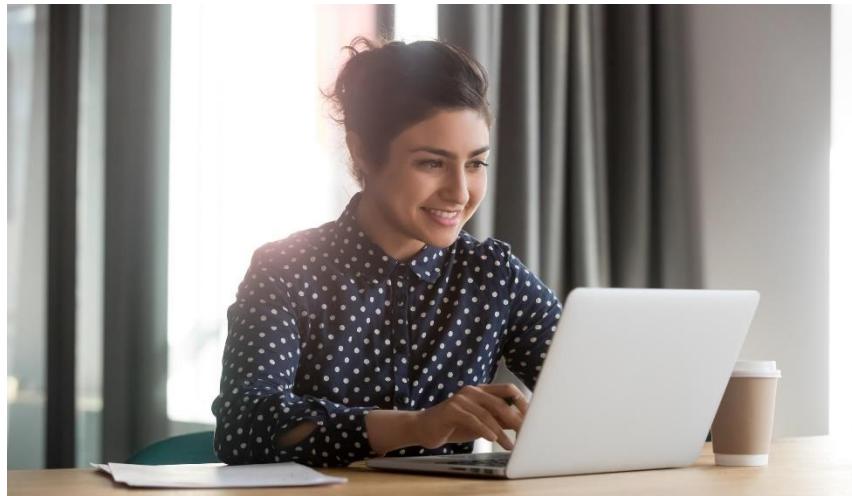
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