Returning your GC & GCMS to Peak Performance

Agilent CrossLab
From Insight to Outcome

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DE.4951967593

What parts do I need to consider to bring my GC system back to 100%?

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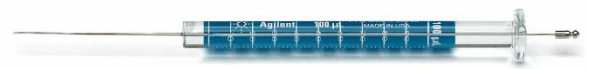
Are the wash vials contaminated?



Are the indicators saturated?

What is the age of the pump oil or tip seals?

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What happens to your chromatograms if you run with a spent filter?

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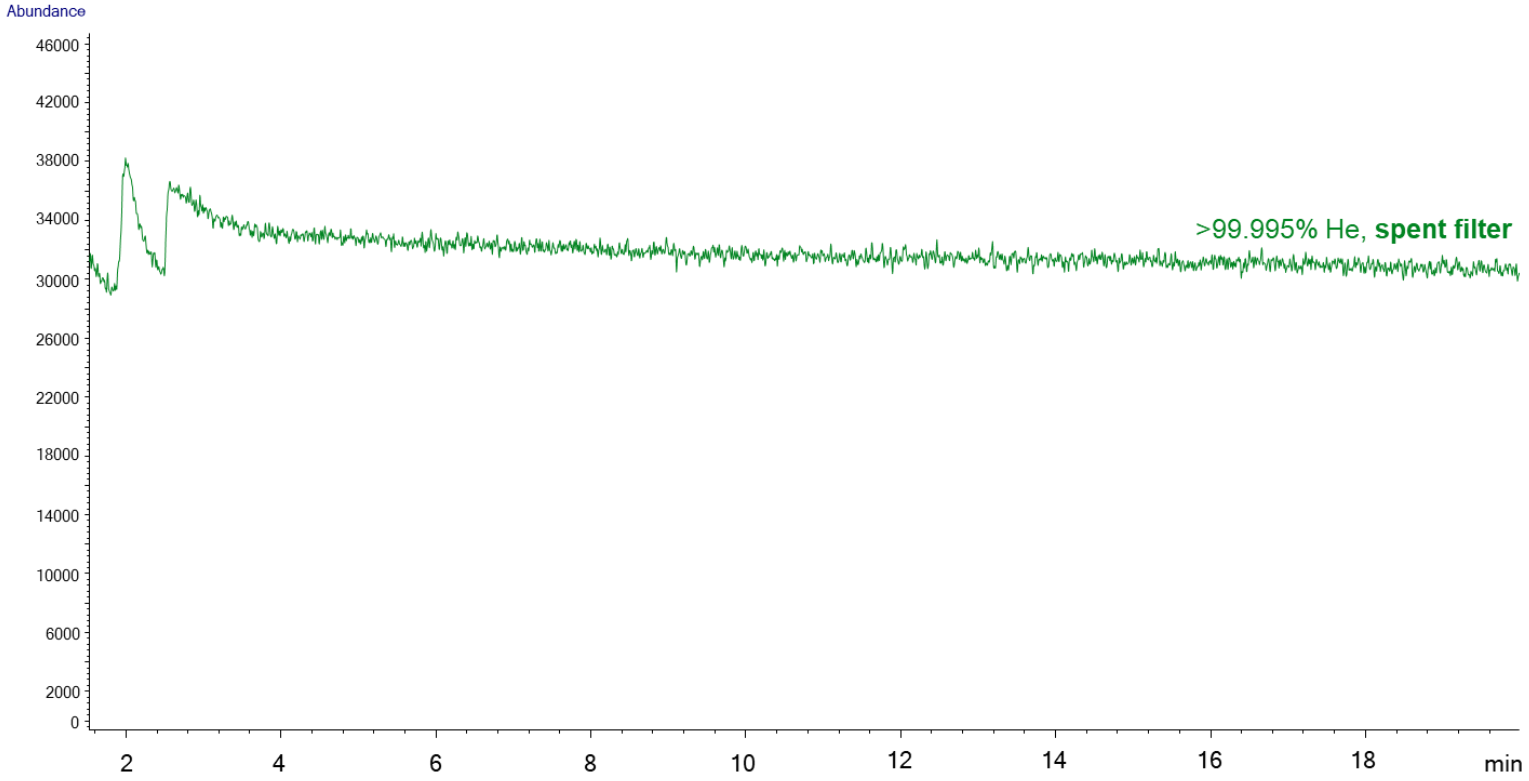

Extracted Ion Chromatogram (EIC) for O_2

Oxygen filter saturates at 0.1 ppm O_2
Moisture filter saturates at 0.2 ppm moisture

Remember to regularly look at the Gas Clean filter or use Gas Clean sensor on 8890 or Intuvo GC systems

Take the Guess Work out of Maintenance: Gas Clean Sensor on Intelligent GCs

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P/N CP17973

https://www.agilent.com/en/video/easy-clean-filter-replacement

www.agilent.com/chem/gasclean

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What happens to your chromatograms when you replace the filter?

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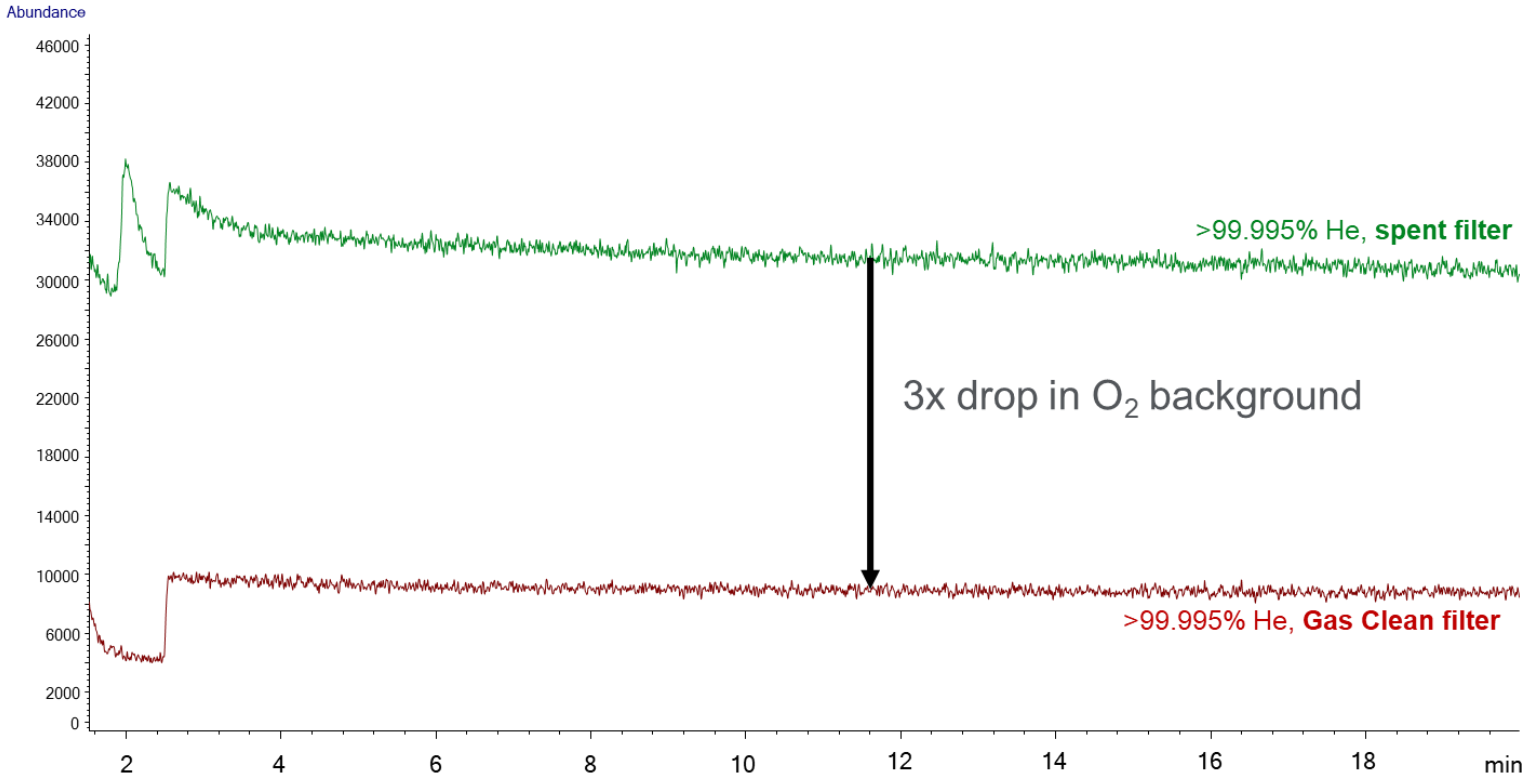
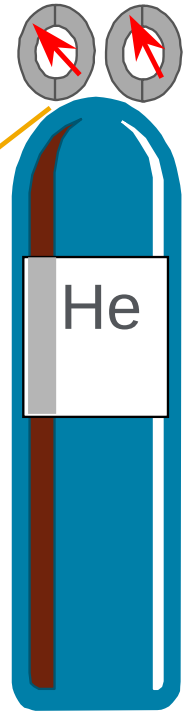


Extracted Ion Chromatogram (EIC) for O_2

Less O_2 reaching column = Longer column lifetime
and less system maintenance (and cost!)



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Traditional Rough Pumps vs. Oil Free Scroll Pumps

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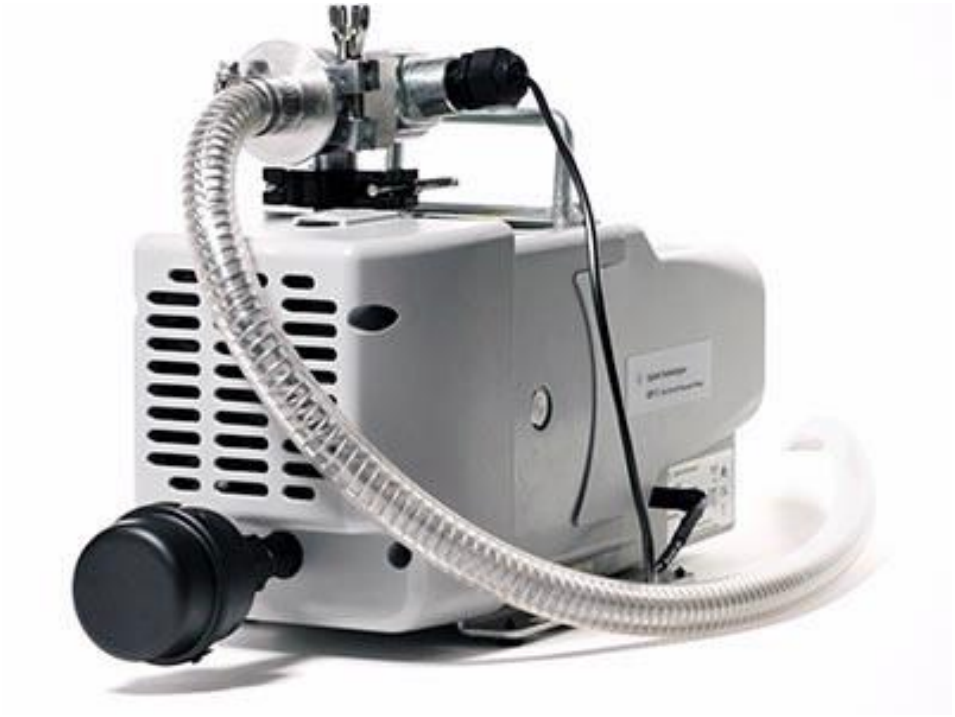

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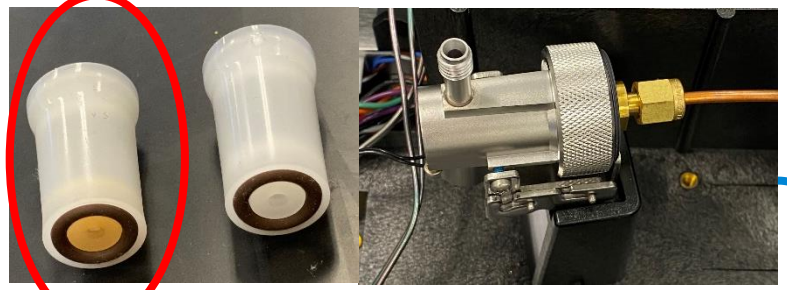
Remember to look under the covers at these parts, too!

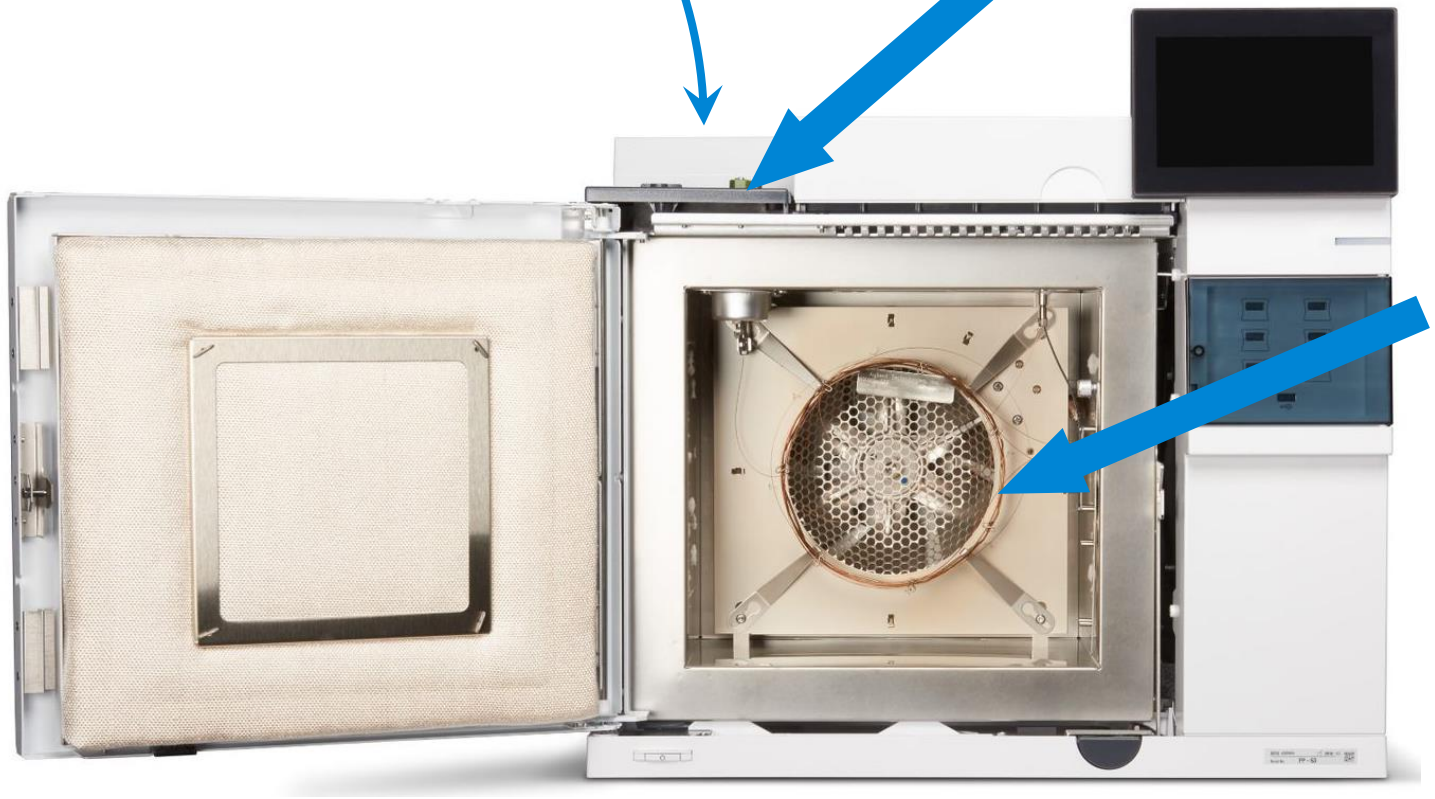
When was the split vent trap replaced last?

Dirty trap



Was the inlet exposed to atmosphere for an extended time?

Remember to look under the MS covers and clean around the fan!



Was the column exposed to atmosphere for an extended time? Are you failing QC and normal inlet/column maintenance isn't recovering responses?

If GC/MS was off for 1+ week (no carrier gas flow)…

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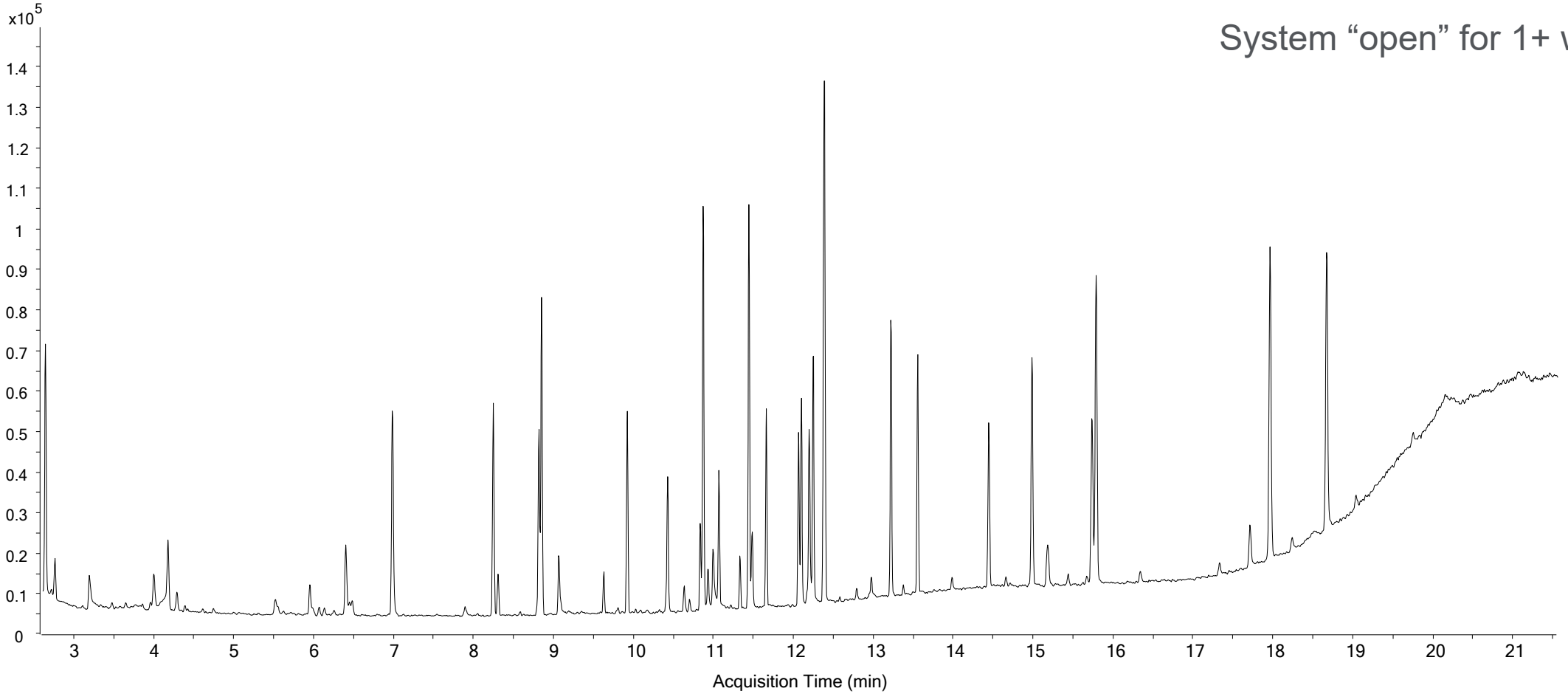

System “open” for 1+ week

TIC looks okay (I think). How does it compare to a previous run of the same sample?

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If GC/MS was off for 1+ week (no carrier gas flow)…

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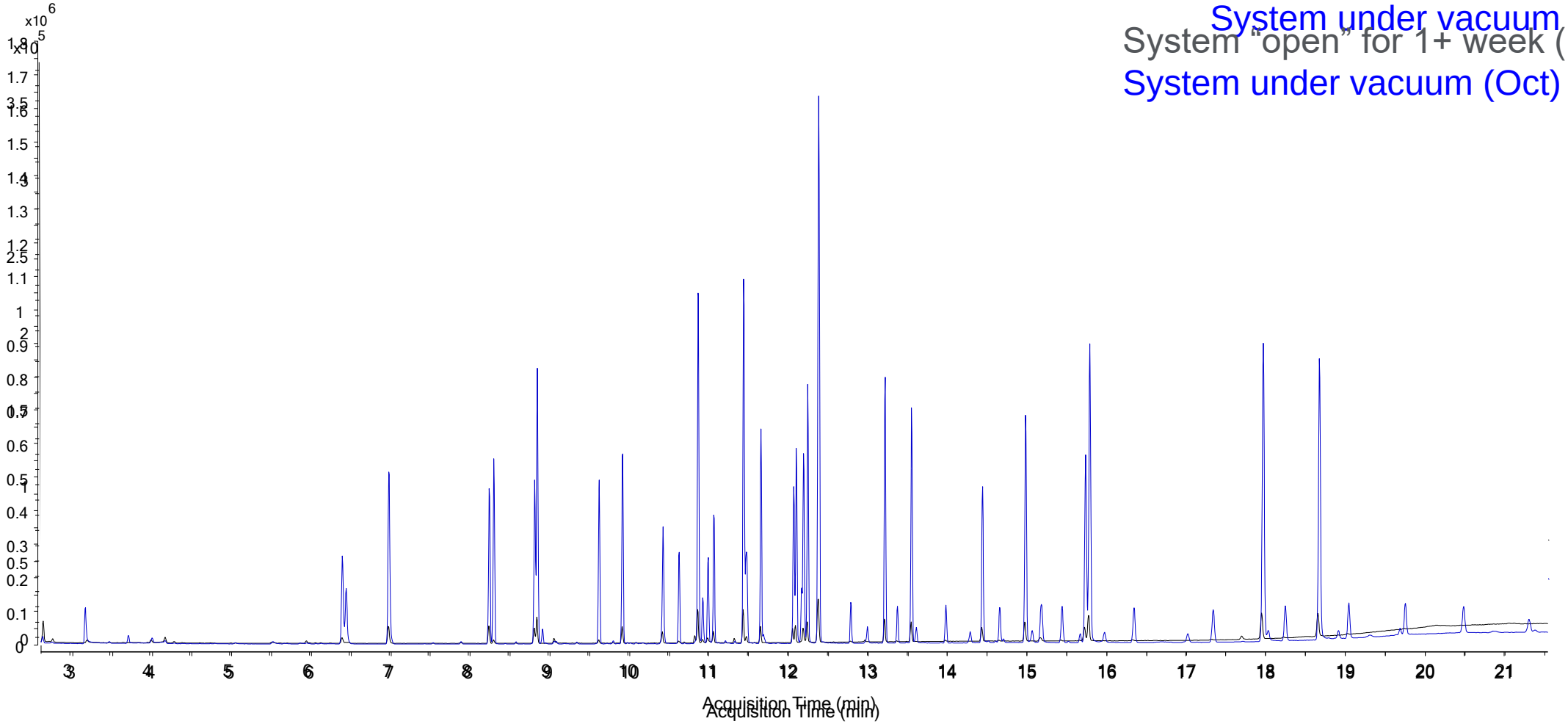
System “open” for 1+ week (Dec)
System under vacuum (Oct)

System “open” for 1+ week (Dec)
System under vacuum (Oct)



“Open system” TIC is ~10x lower than good run in the previous. What happens if we replace the column and liner?

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Recover peak response with new column and liner

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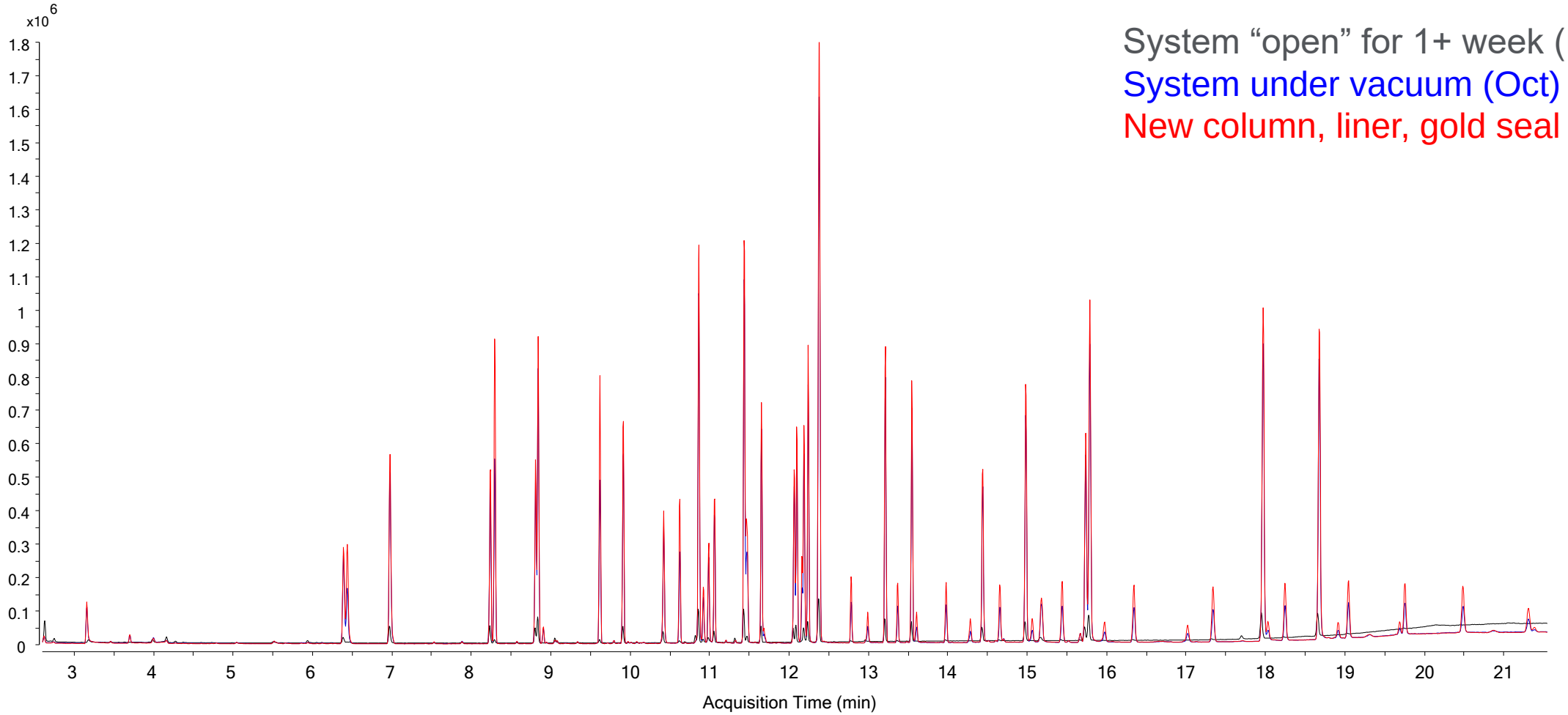

System “open” for 1+ week (Dec)
System under vacuum (Oct)
New column, liner, gold seal (Dec)

Try a new liner and re-conditioning column first.
If response doesn’t recover, a new column may be required.

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How to Change a Column

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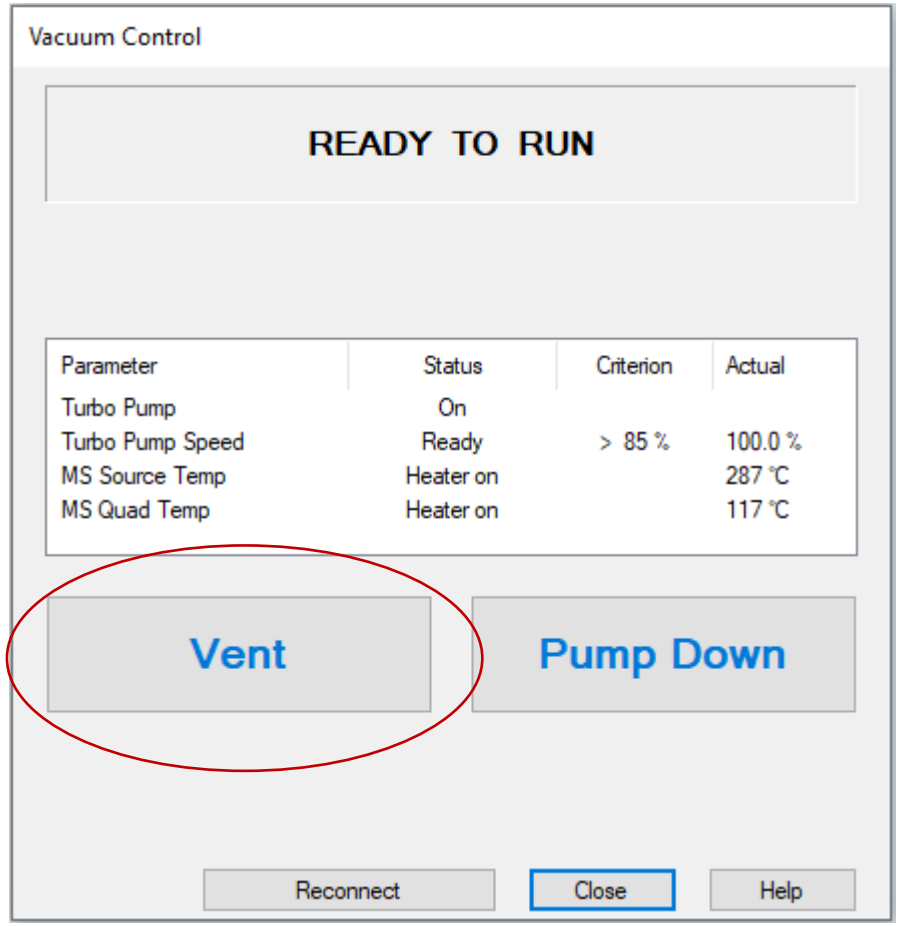
- ✓ Cool Inlet
- ✓ Turn off Mass Spec
- ✓ Turn off GC
- ✓ Verify Rough Pump is Off
- ✓ Open Vent Valve
- ✓ Remove Column



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Why does the length above the ferrule matter?

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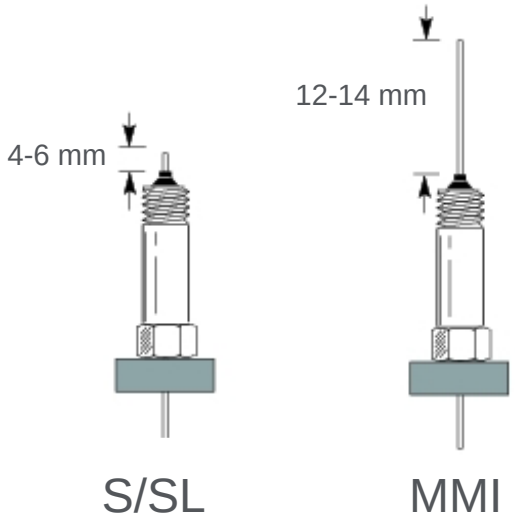
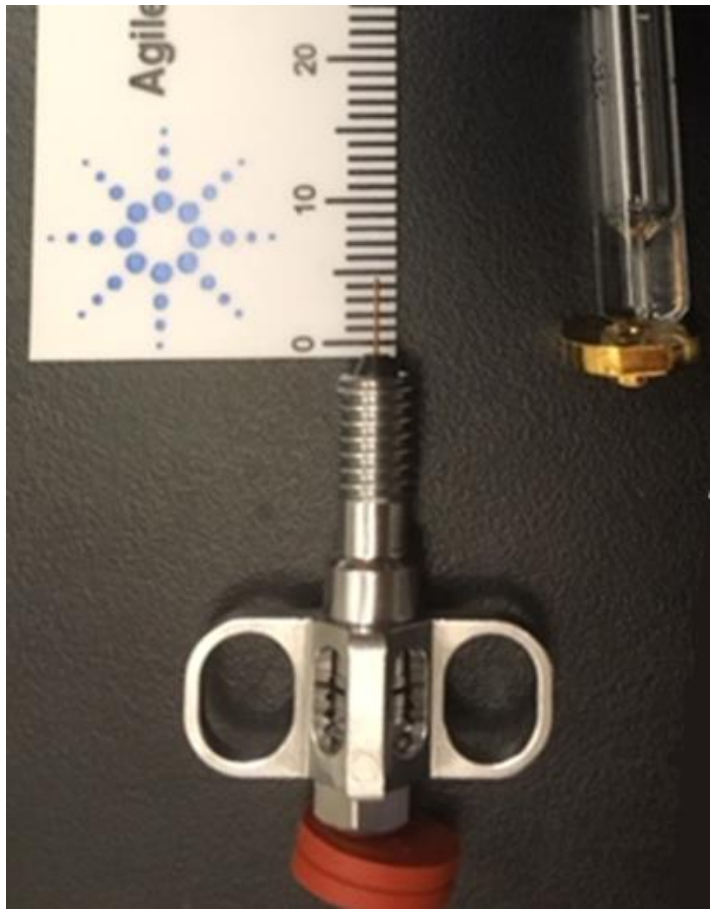
Tip of column enter the bottom of the liner but not past the taper





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What happens if the column sits too low?

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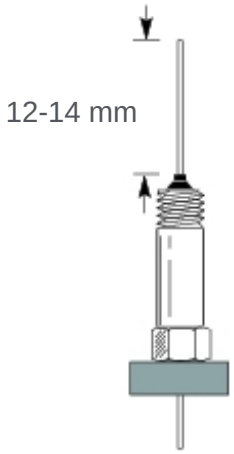

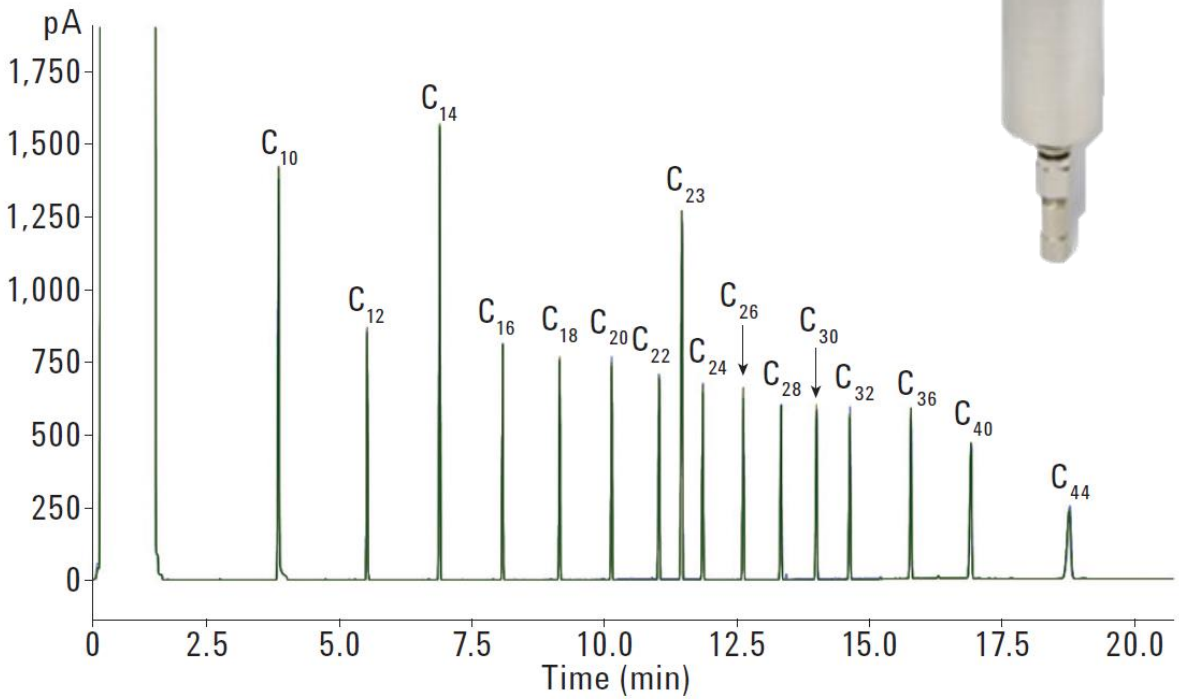

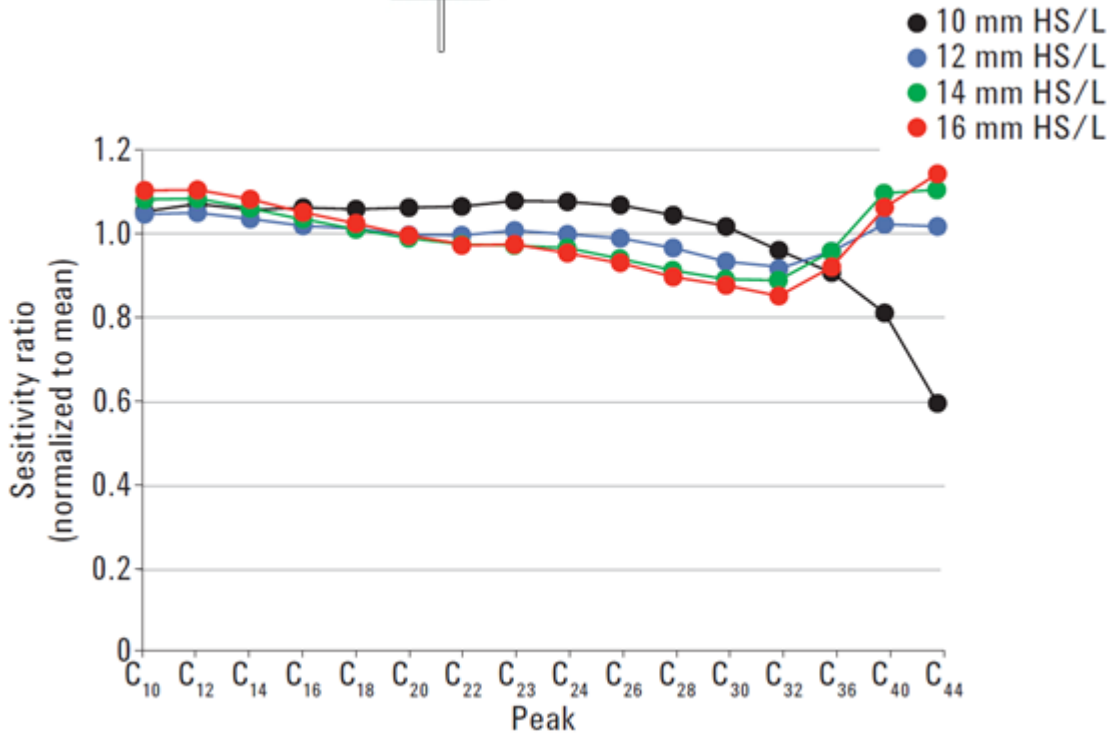

Figure 4. Overlay of four replicate chromatograms of the $C_{10–44}$ mixture in hot splitless mode at 14 mm install length.

Agilent Publication 5991-7619EN

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Use Self Tightening Column Nuts: No Leaks, No Frustration Holds installation depth

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- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- Collar holds column in place for easy and fast installation
  - Set the depth for inlet or detector, install, remove collar and it's ready to run
- No tools needed!

For mass spectrometry transfer line

For GC inlet or detector

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Detector depth for MSD, other detectors

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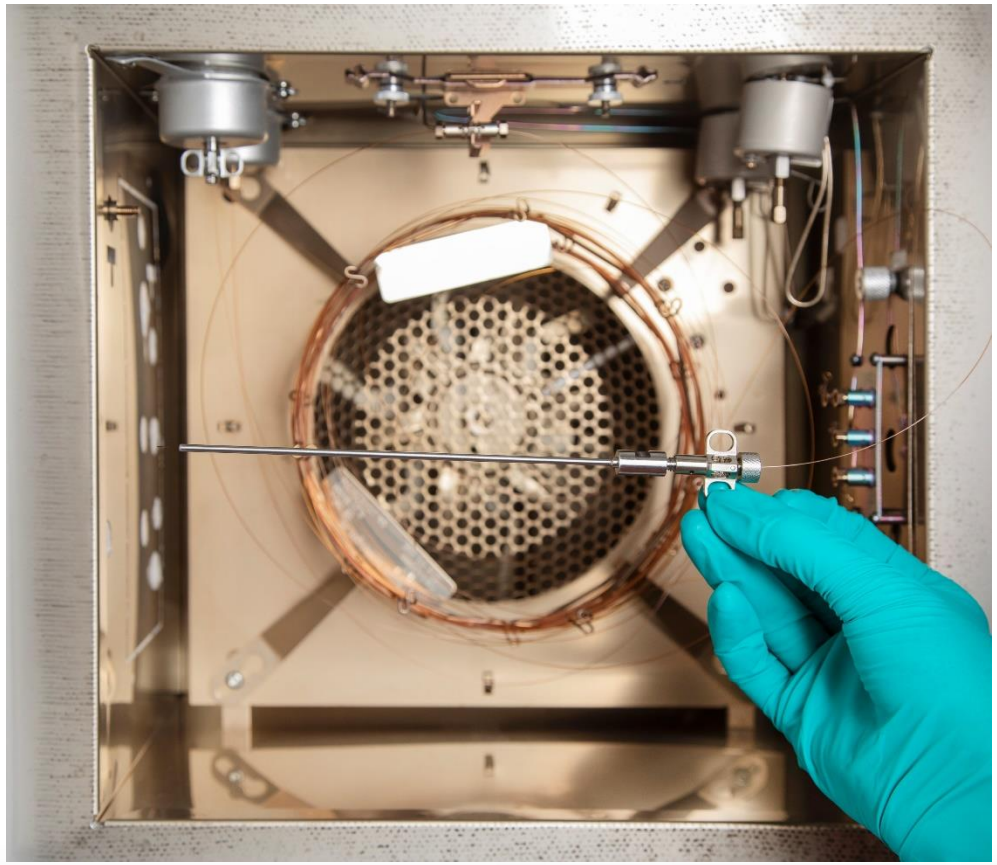
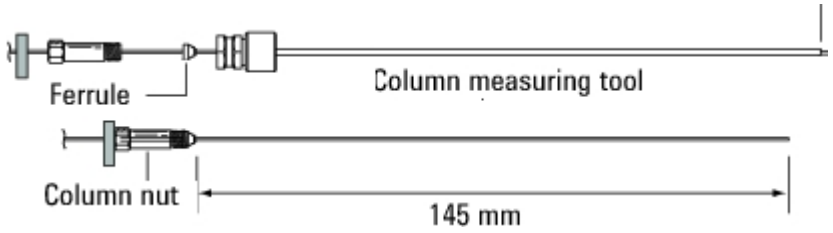
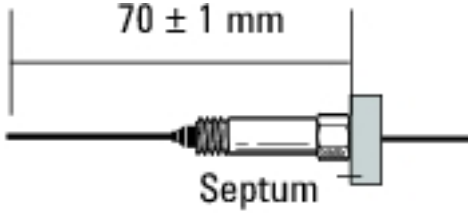
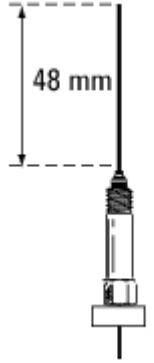




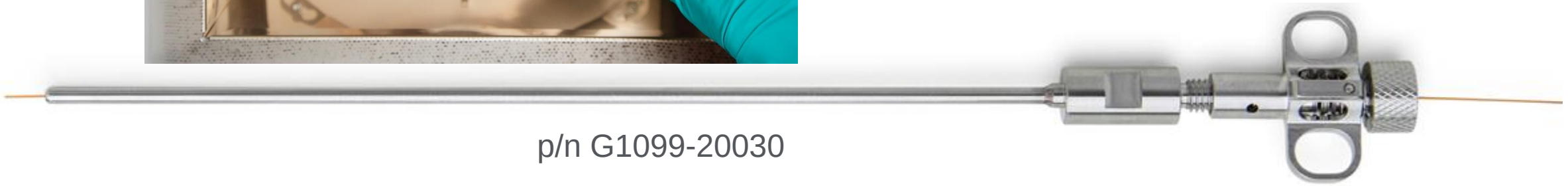
FPD



µECD



FID



p/n G1099-20030

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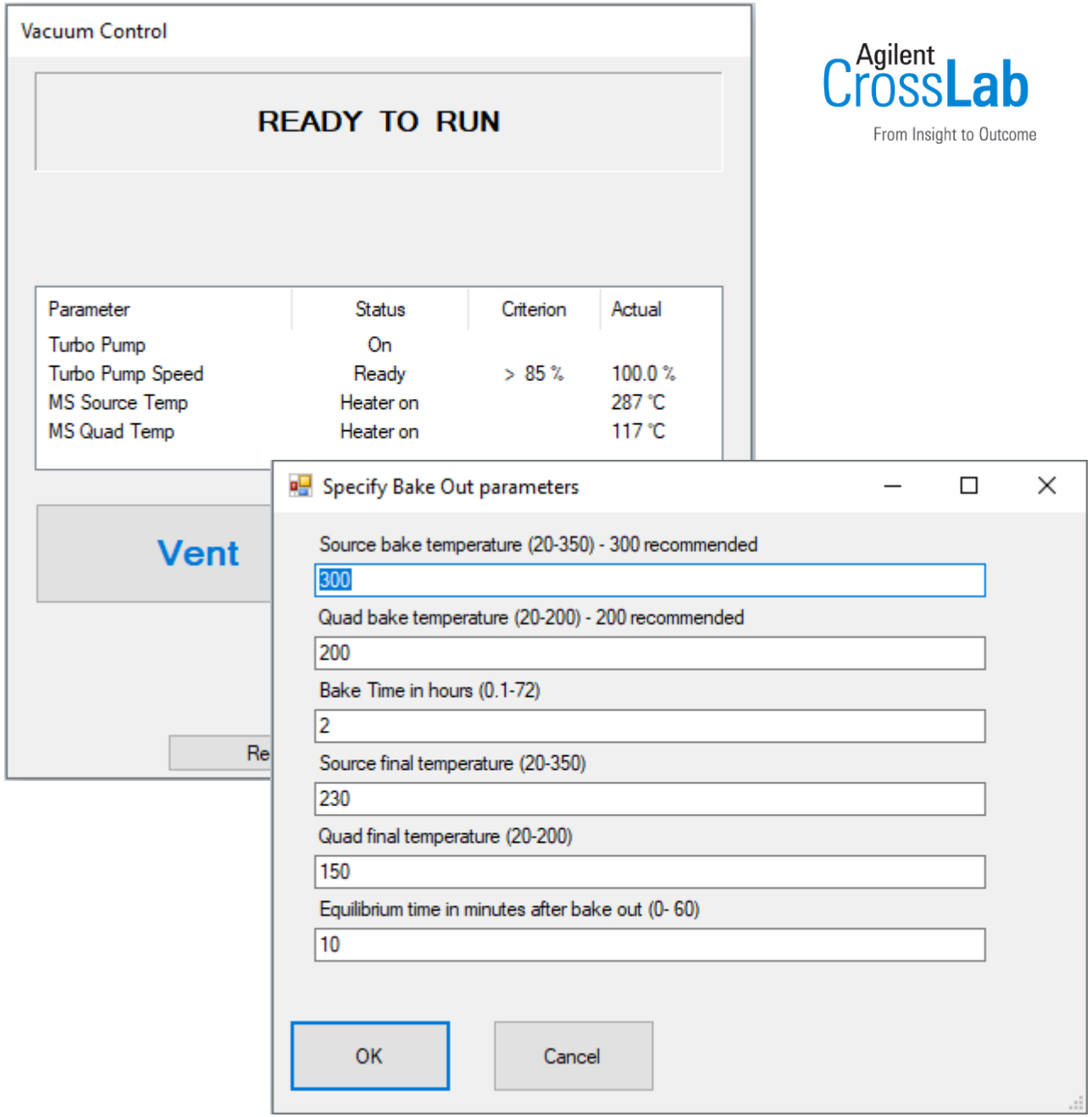
Pumping Down Your Mass Spec

- ✓ Apply set temperatures
- ✓ Initiate “pump down”
- ✓ Bake Out Mass Spec
- ✓ Check Vacuum gauge
- ✓ Air and water check
  - ✓ If any leak, may need to tighten STCN a bit more
- ✓ Condition Column



How to Condition your Column

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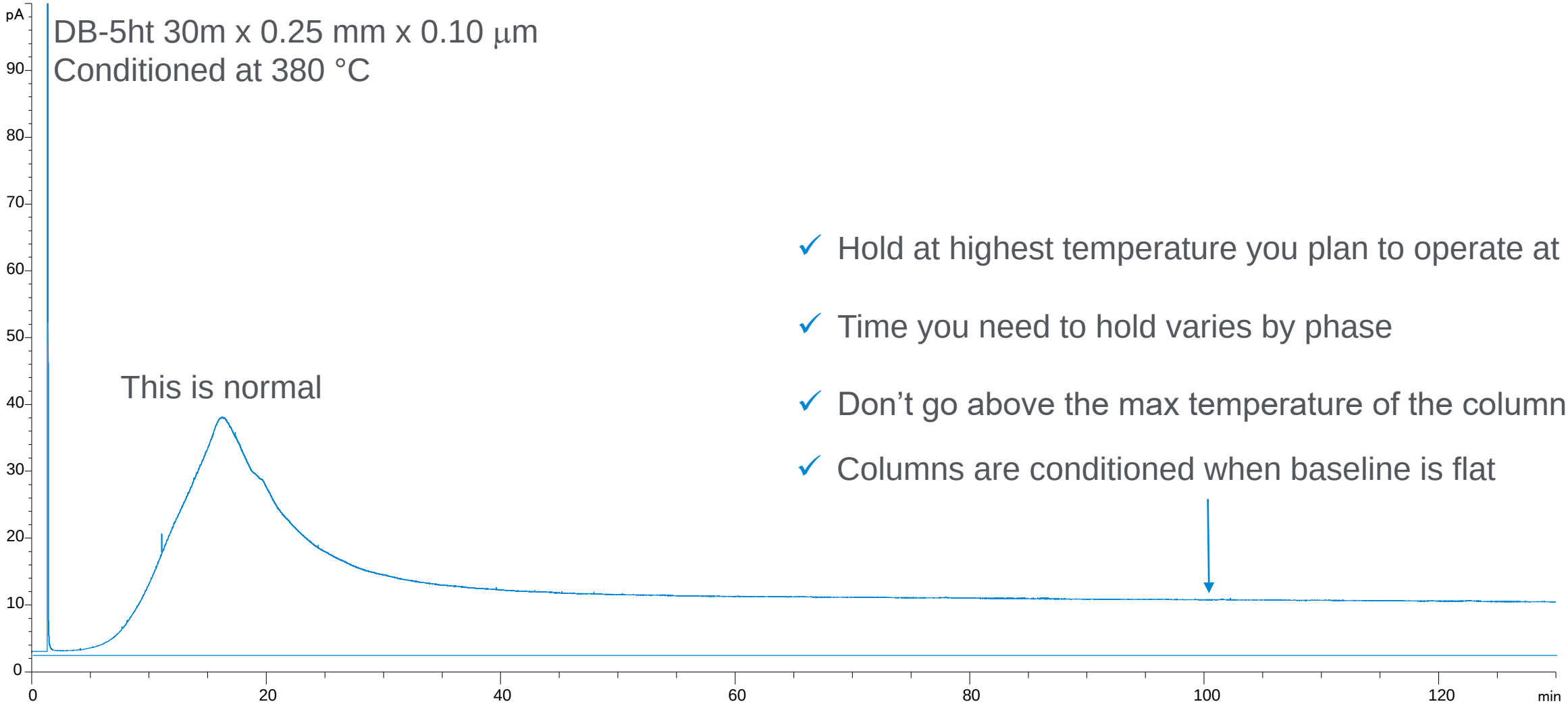

- ✓ Hold at highest temperature you plan to operate at
- ✓ Time you need to hold varies by phase
- ✓ Don’t go above the max temperature of the column
- ✓ Columns are conditioned when baseline is flat

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While we’re bringing the system back online, is it optimized for your analysis?

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MS Columns, and why you should use them

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DB-5

DB-5 high quality, non-polar, general-purpose columns are low bleed with a high temperature limit.

Quick View | BUY PRODUCTS

DB-5ms

DB-5ms non-polar, low-bleed columns feature an improved signal-to-noise ratio for excellent sensitivity and mass spectral integrity of aromatic compounds.

Quick View | BUY PRODUCTS

DB-5ms Ultra Inert Columns

Deliver consistent inertness, exceptionally low column bleed, great peak shapes, and effective performance for challenging active analytes.

Quick View | BUY PRODUCTS

Multi-Purpose → Decreased Bleed → Robust for Active Compounds

What Column Bleed Looks Like

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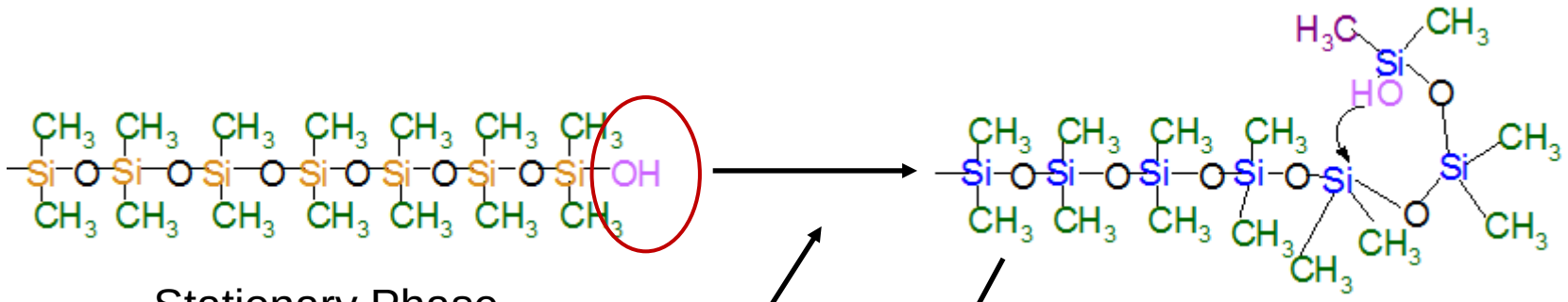
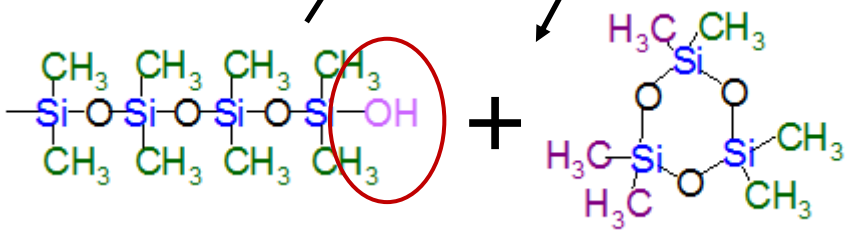


Stationary Phase



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

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Columns don’t make peaks….

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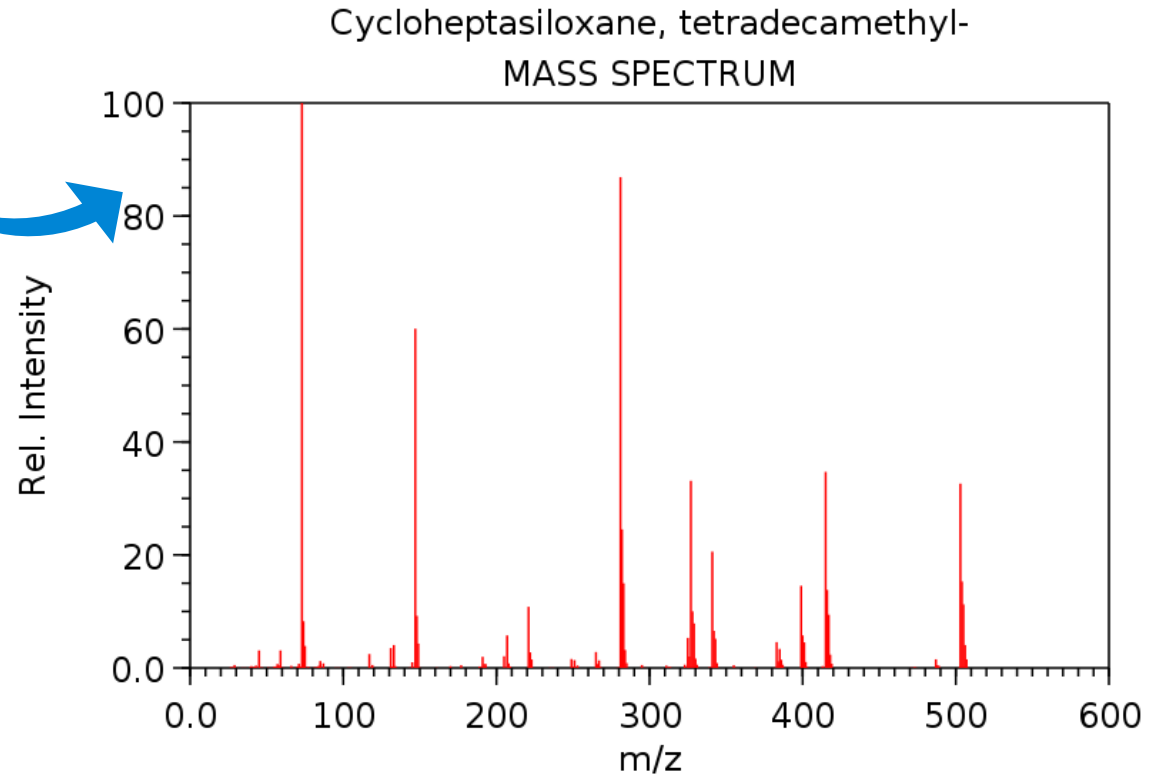
Peaks ARE NOT COLUMN BLEED!

New

Over Compressed and Spent



NIST Chemistry WebBook (http://webbook.nist.gov/chemistry)

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Improved peak shape for difficult compounds

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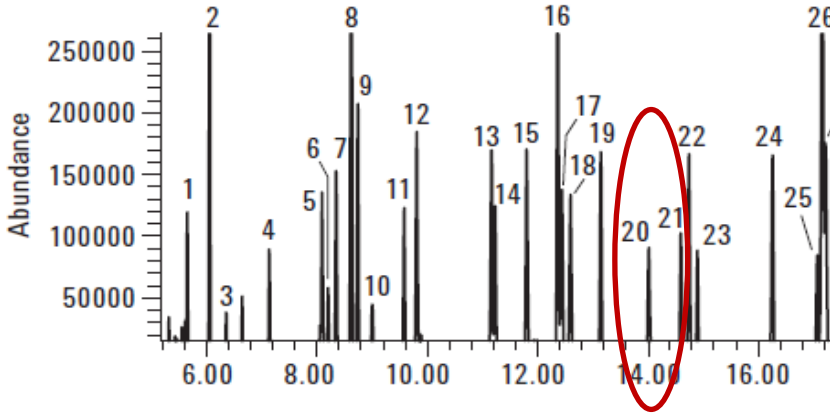
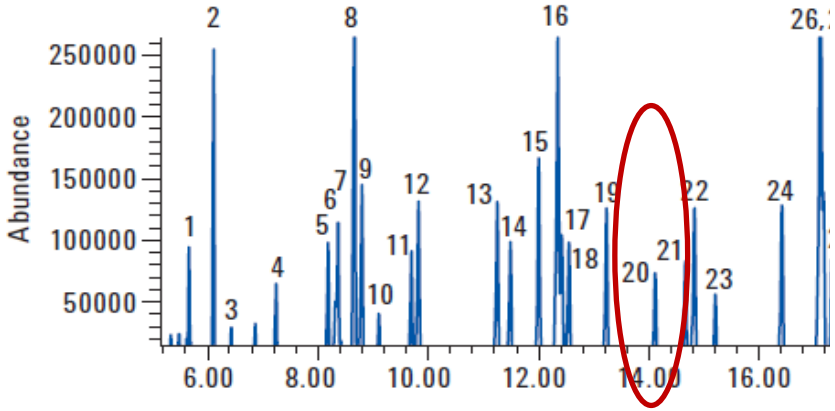
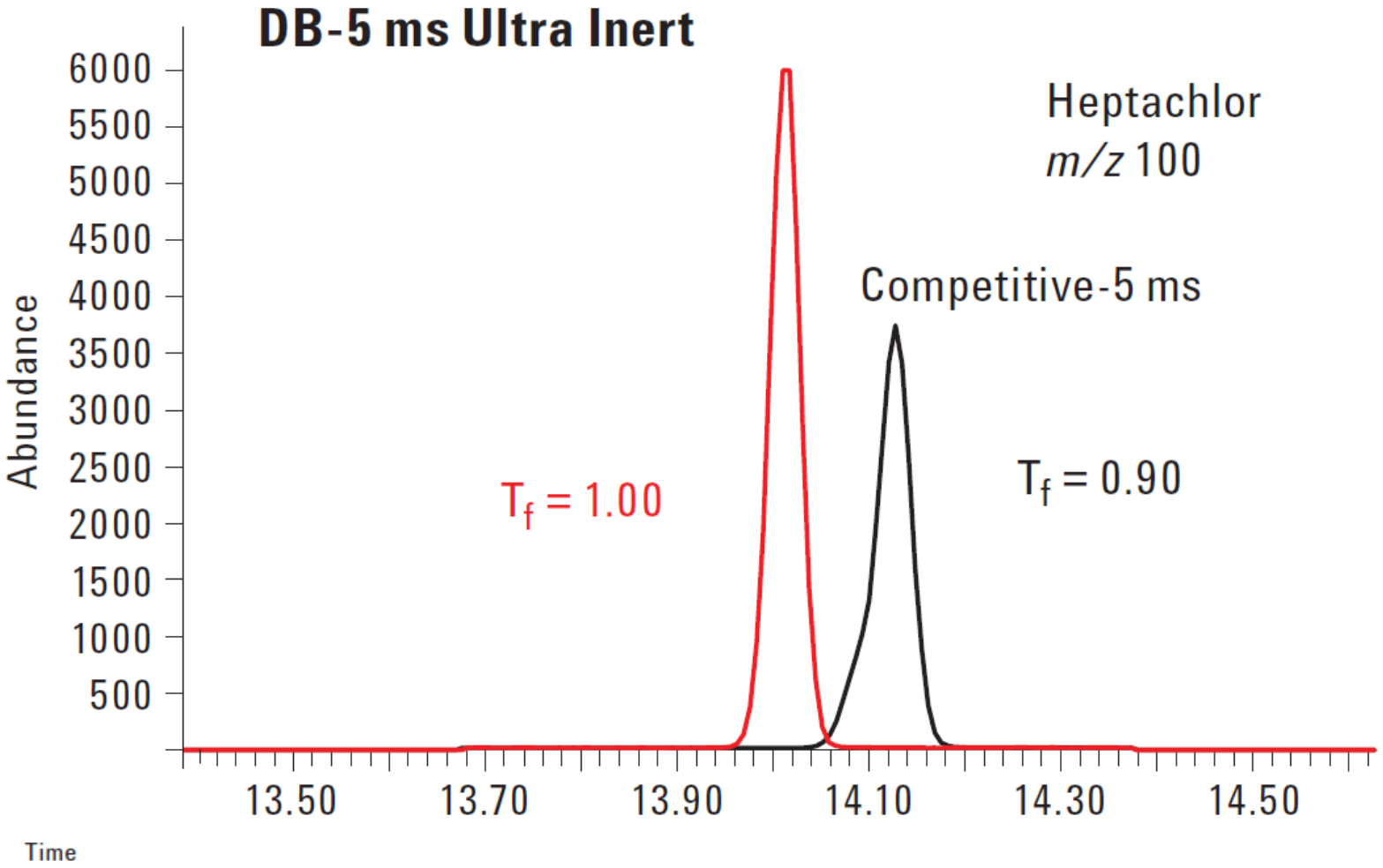
DB-5 ms Ultra Inert column



Other Mar





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Temperature Range, Applications, & **Detectors**

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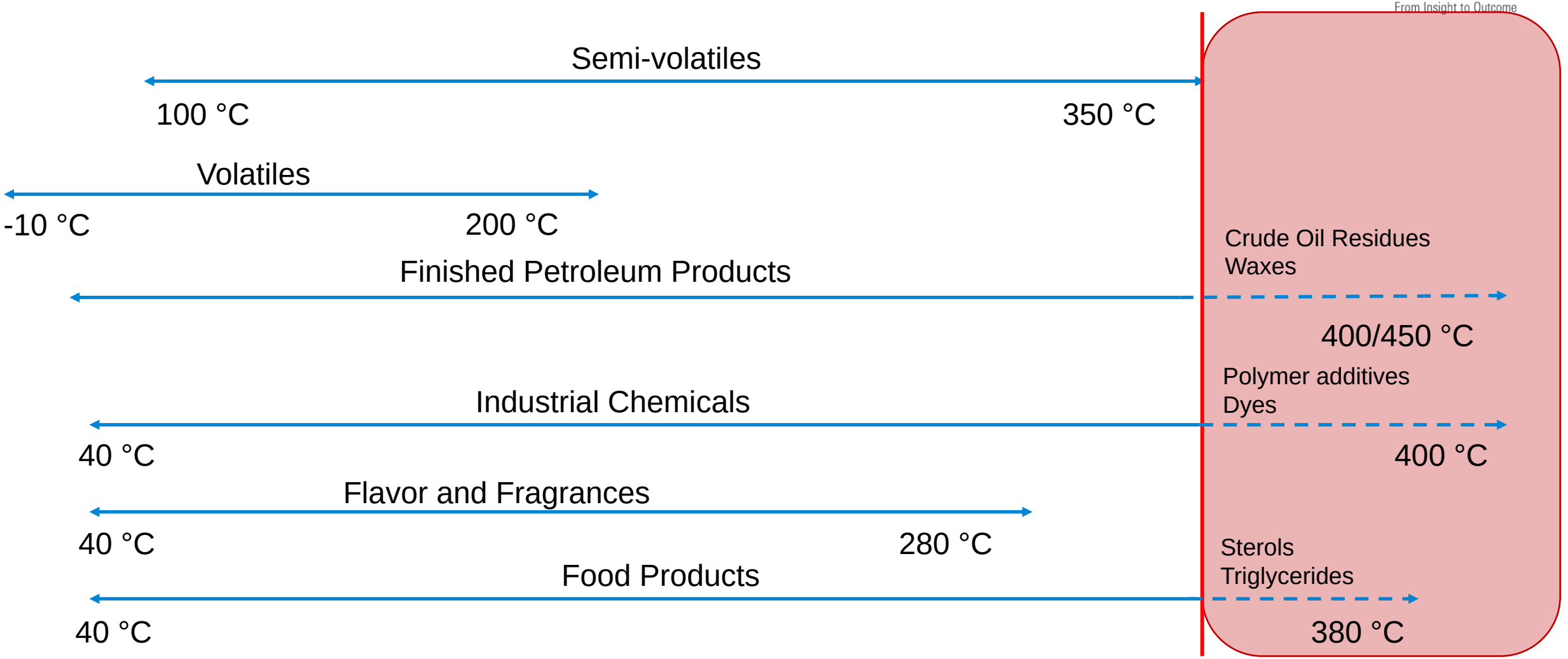

MSD/ FID/ ECD/ TCD/ FPD/ SCD

FID/SCD

Just because you can do High Temp GCMS…. Should you?

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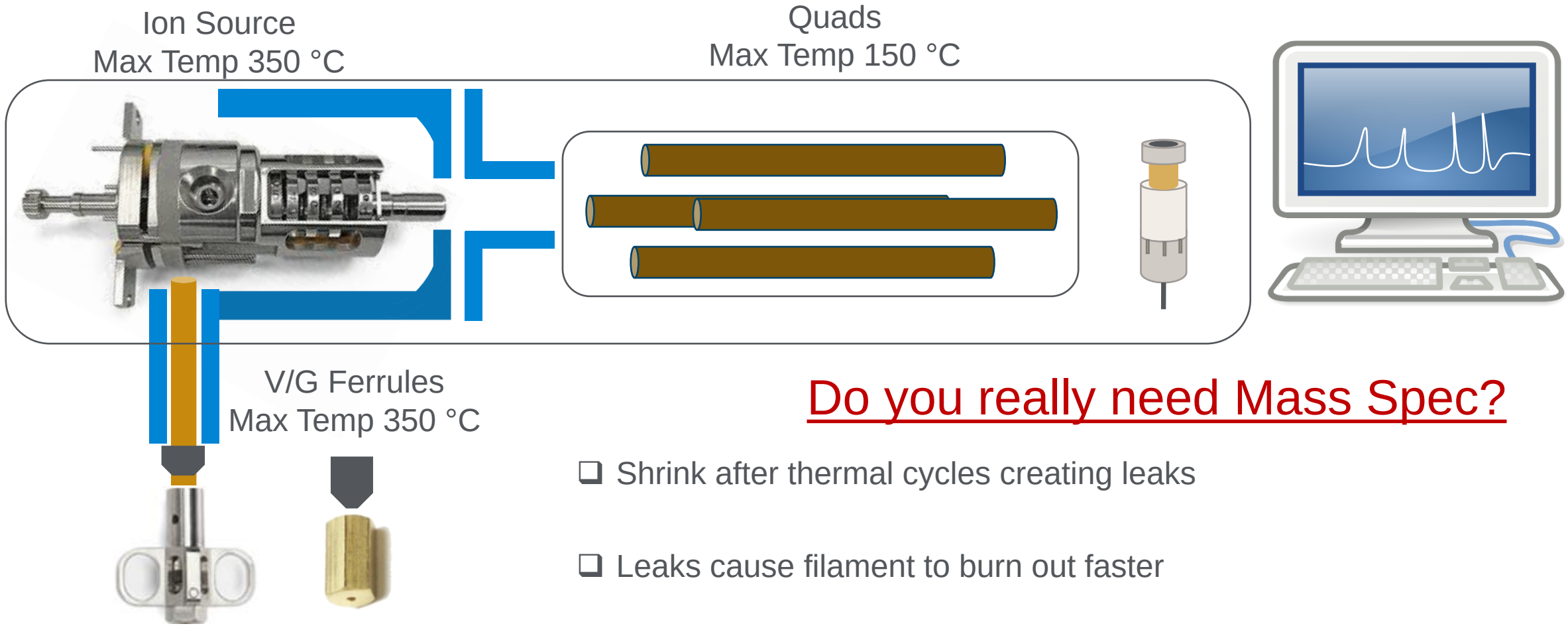


Do you really need Mass Spec?

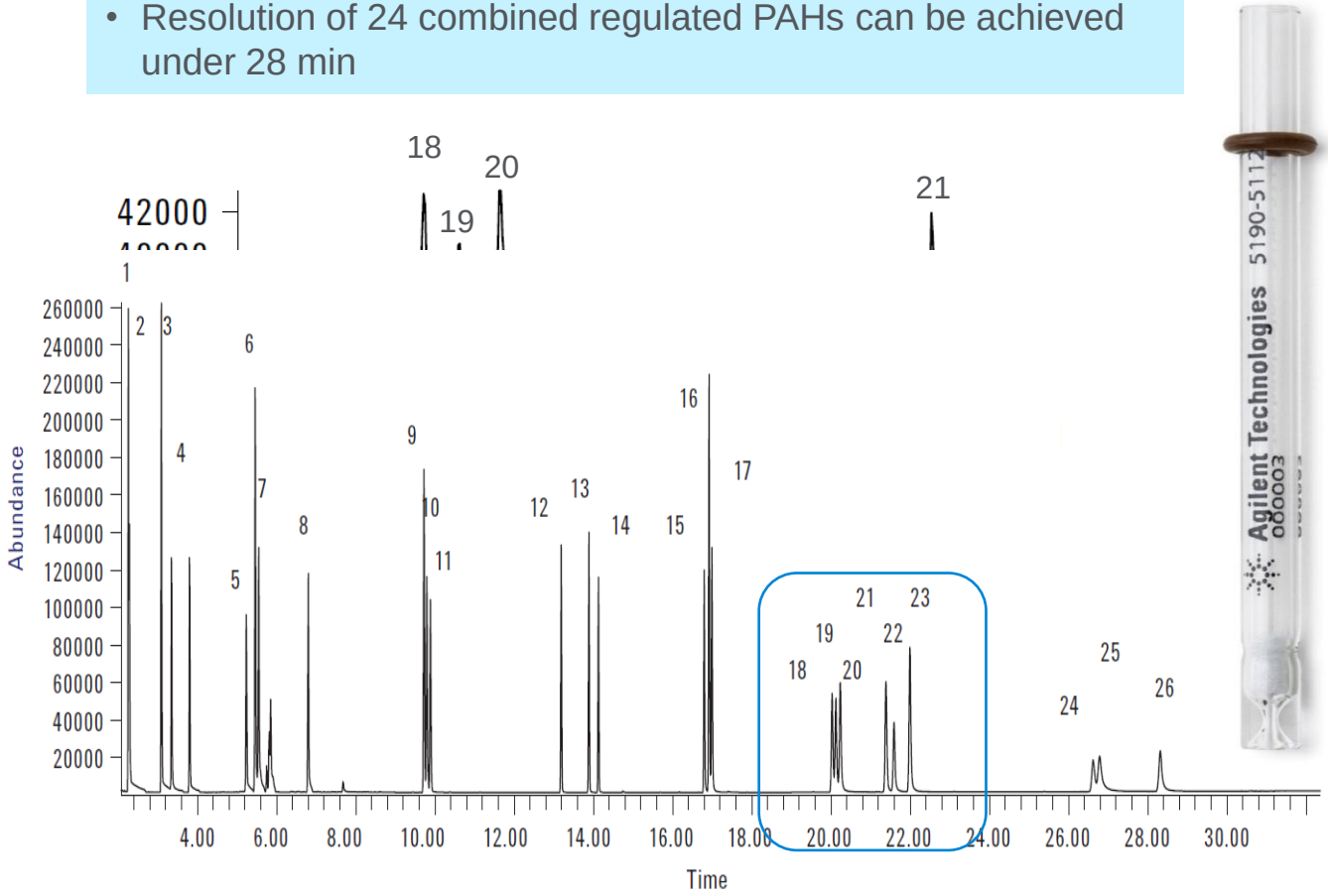
- ❑ Shrink after thermal cycles creating leaks
- ❑ Leaks cause filament to burn out faster
- ❑ Condensation in Quads…..

PAH analysis: Environmental or Food

Two columns for what you need

DB-EUPAH

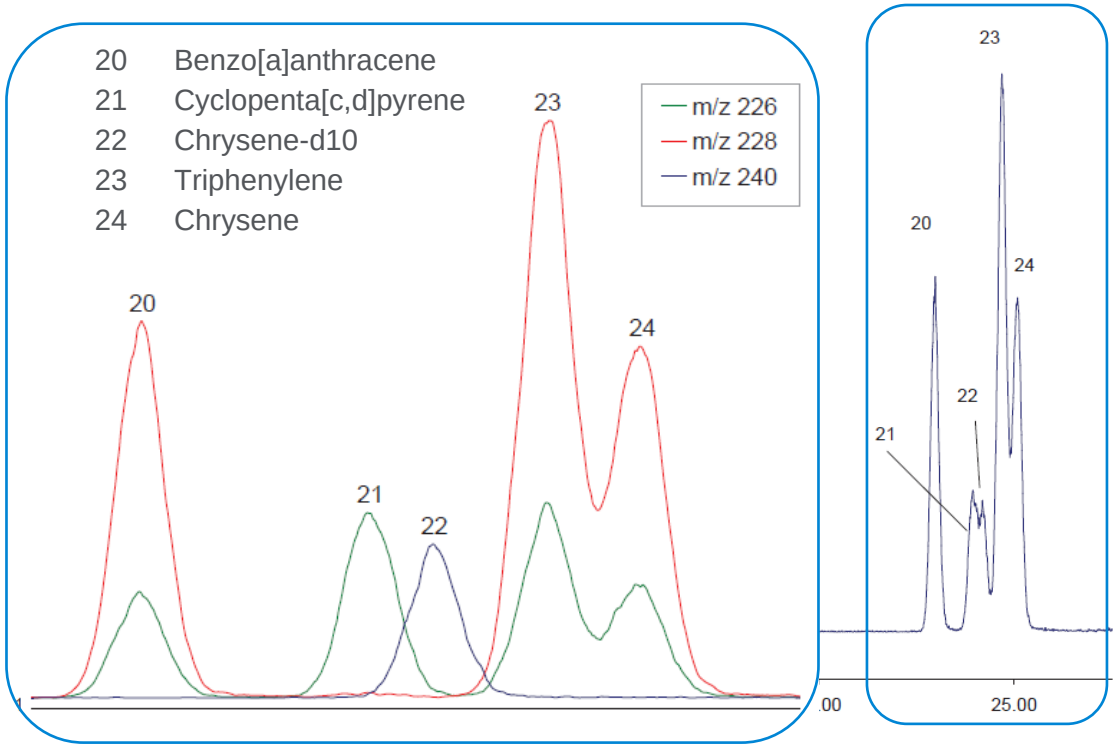
- Mid-polar column that resolves benzo (b,j,k) fluroanthenes
- Resolution of 24 combined regulated PAHs can be achieved under 28 min



Application Note: 5990-6155EN

Select PAH

- Mid-polar column that optimize chrysene/triphenylene resolution
- Resolution of 54 PAHs under 40 min



Application Note: SI-02232

Environmental: Drinking Water, Wastewater or soils

Semi-Volatiles

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Drinking Water

Splitless Injection + Clean matrix = Splitless Liner → Splitless Single Taper liner

Column: DB-5ms UI or DB-8270 UI

Extraction Lens: 9 mm

Soils, Wastewater, etc.

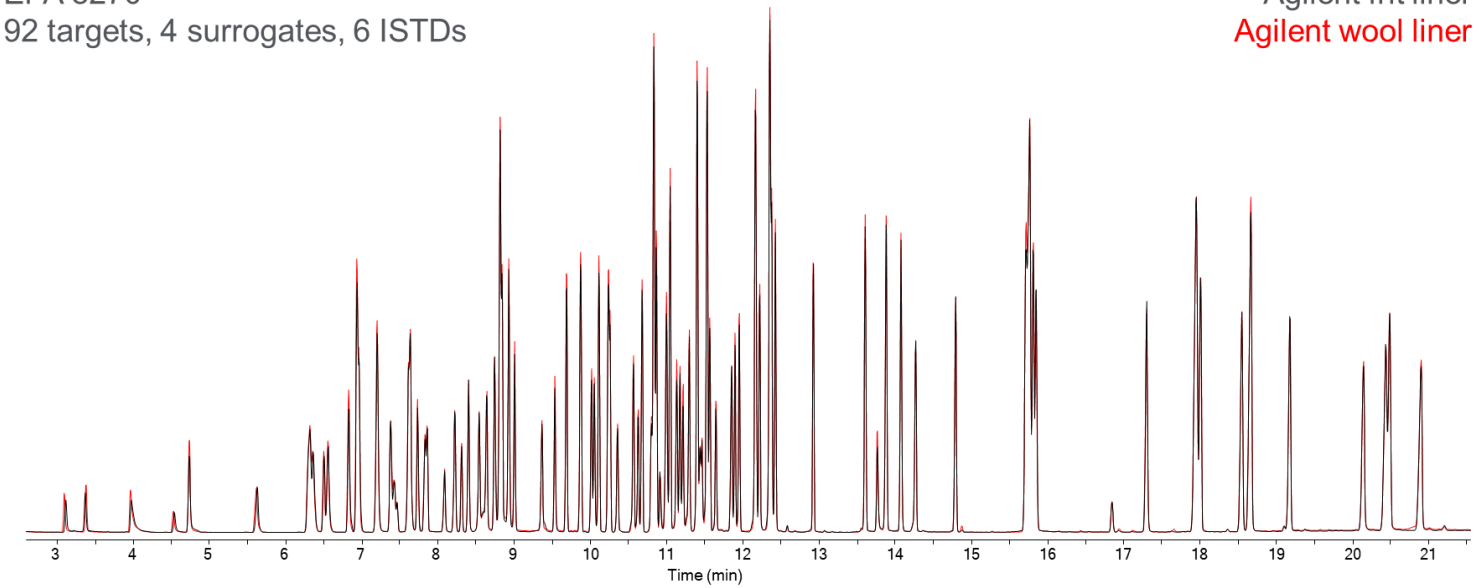
Splitless Injection + Dirty matrices = Splitless Liner → Splitless Fritted or Wool Liner

Column: DB-5ms UI or DB-8270 UI

Extraction Lens: 9 mm

EPA 8270
92 targets, 4 surrogates, 6 ISTDs

Agilent frit liner
Agilent wool liner



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Smart key performance tracking the column

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- Tells your GC what is installed
  - Individual to each columns
- Walks you through configuration
- Keeps track of:
  - # Injection
  - Max temperature taken to
  - Time at max temperature

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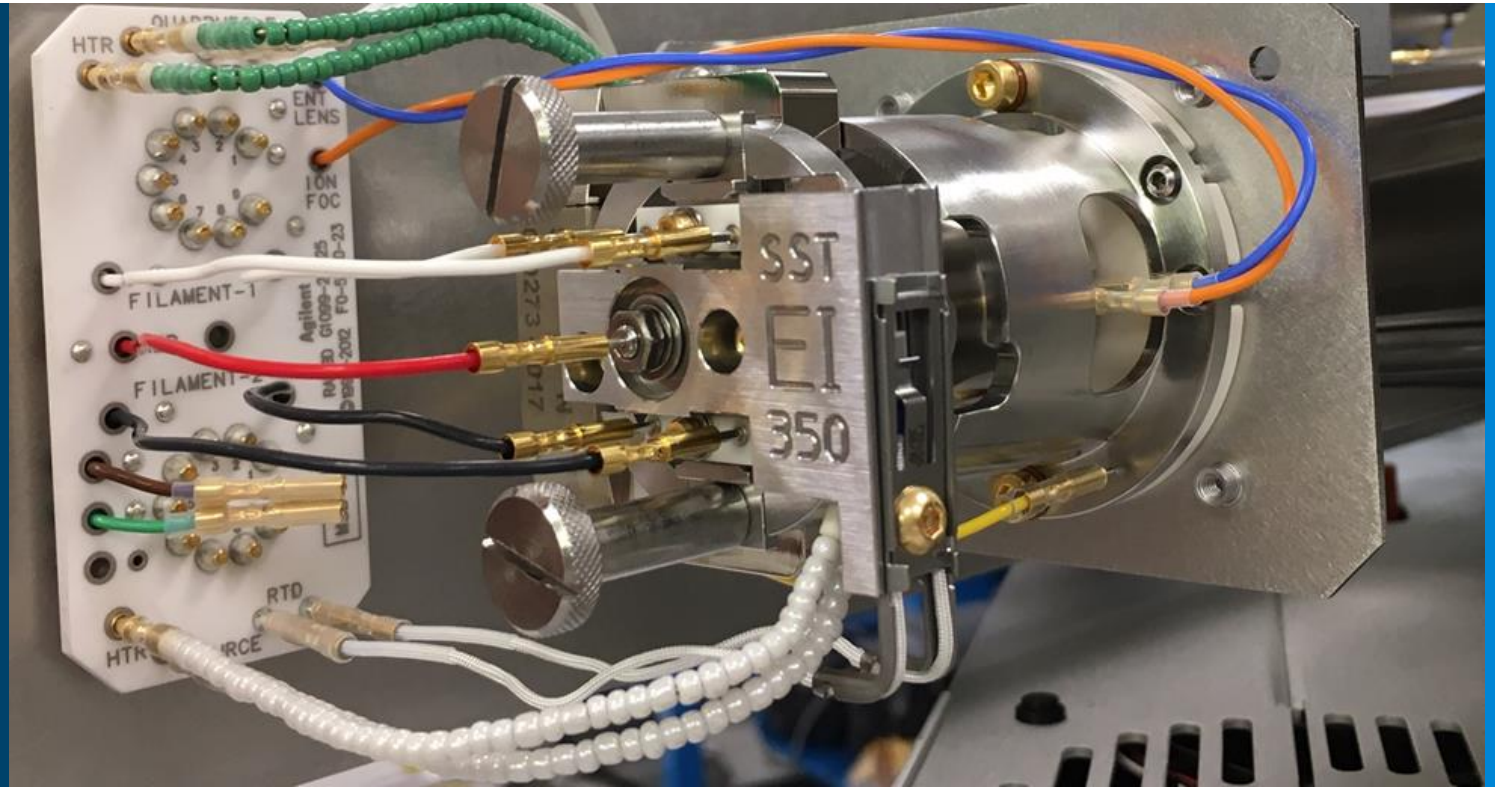
Match the MS source parameters to your analysis



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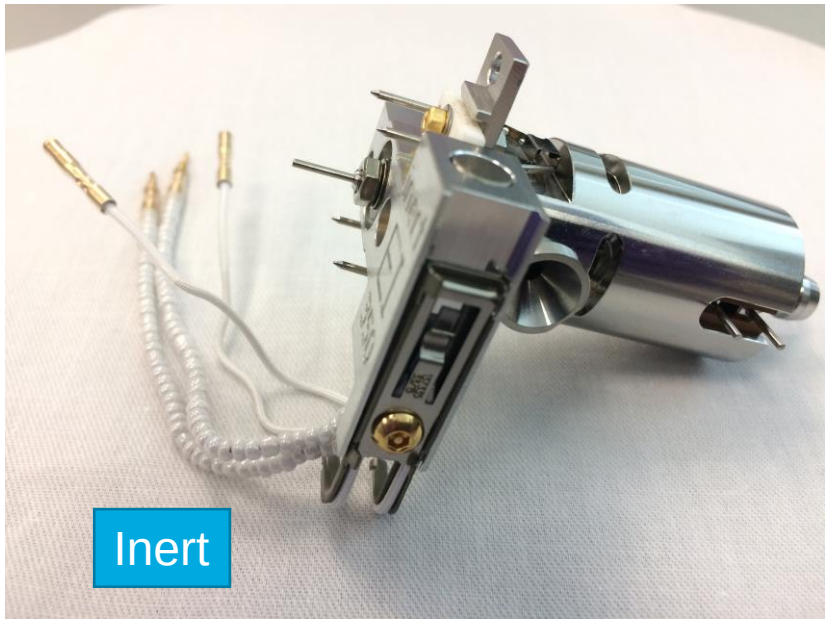
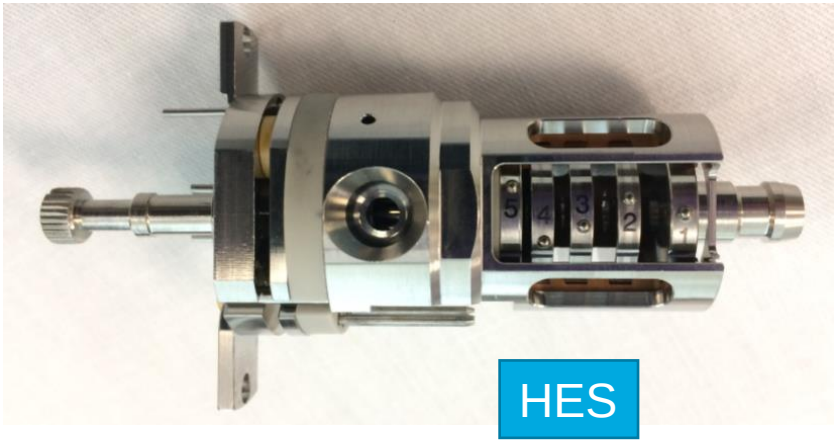
EI Sources



The same geometry



Draw out Lenses in Stainless Steel and Inert Sources and Extraction Lenses for Extractor (Inert Plus) Source

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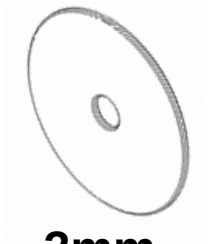
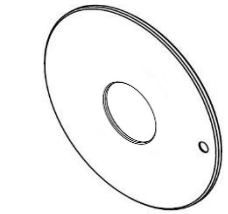
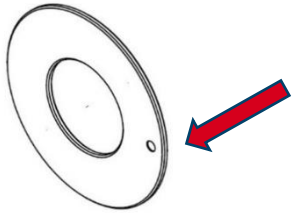
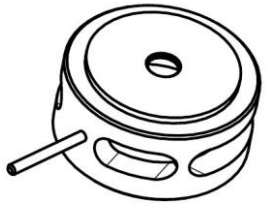
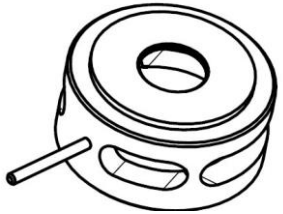
Draw Out Lens for SS	3mm 05971-20134	6mm G3163-20530	
Draw Out Lens for Inert	3mm G2589-20100	6mm G2589-20045	9mm G3440-20022
	 **3mm**	 **6mm**	 **9mm**
			
Extraction Lens	3mm G3870-20444	6mm G3870-20448	9mm G3870-20449
	Instrument checkout Pesticides	Volatiles (P&T and HS)	ASTM Aromatics in Gasoline PAHs Phthalates Semivolatiles Volatiles H_2 carrier

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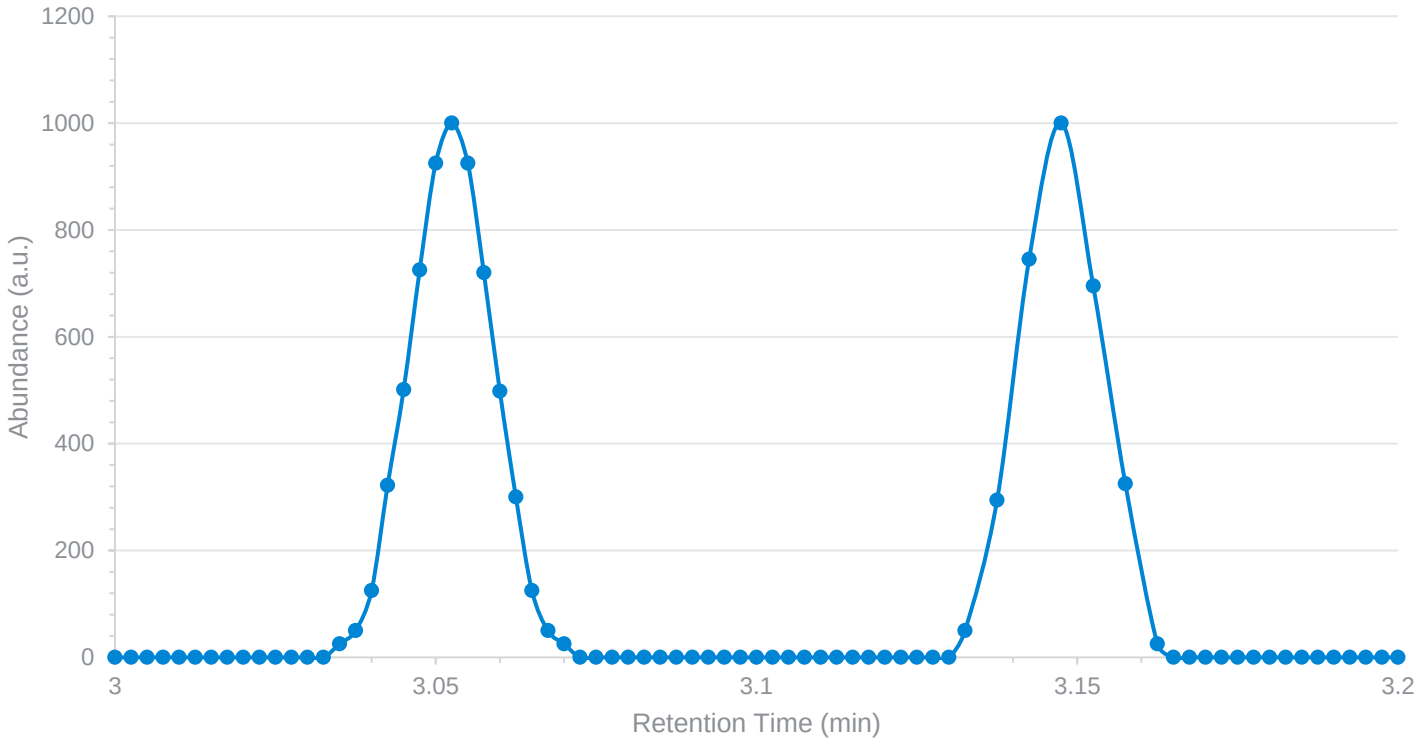
We've optimized the hardware components. Is there anything else to optimize?

Scan speed (data points across a peak)

Gain factor



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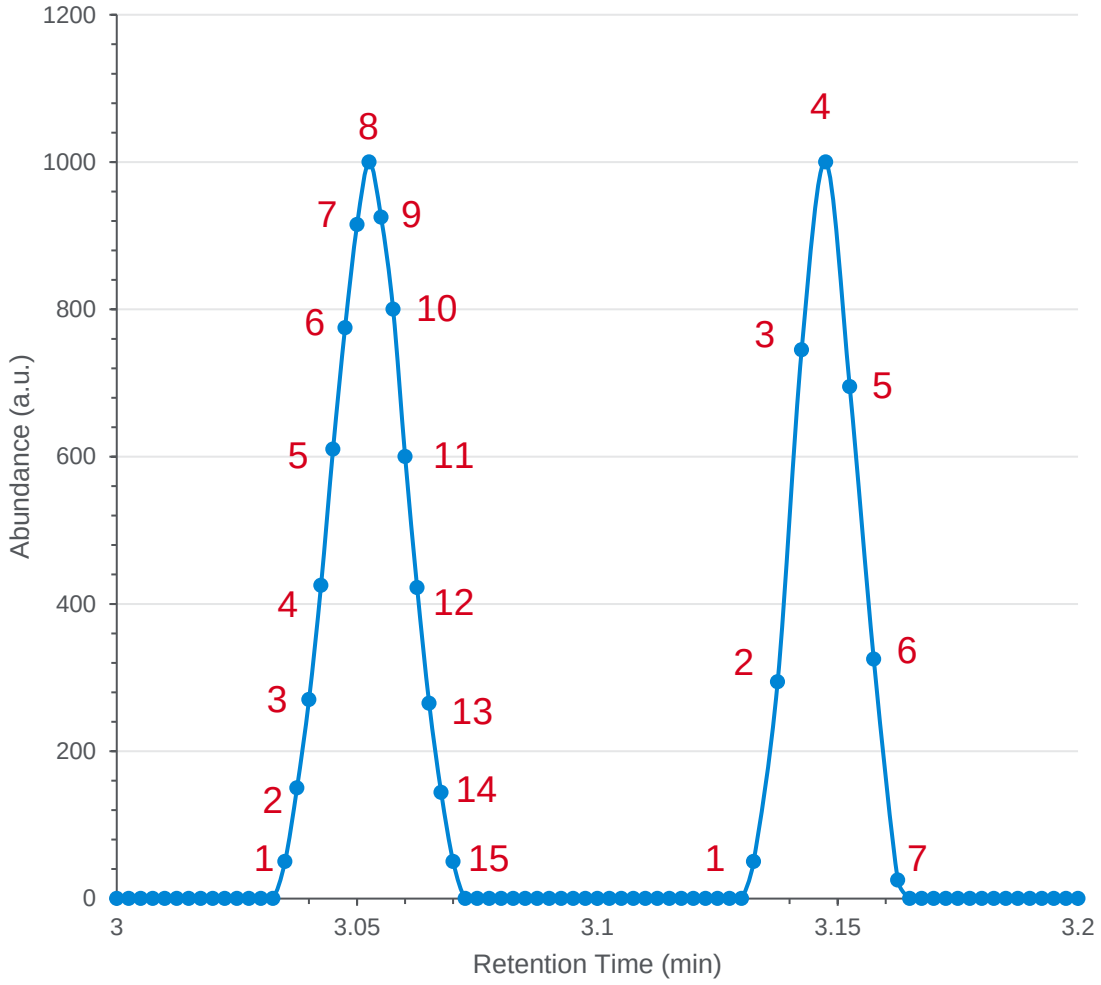
How many data points do I need across a peak?



Application requirement: **Quantification**

- 10-20 data points across peak
- ~2 to 5 data points per second (Hz) (depending on peak width).

Application requirement: **Identification**

- 5-10 data points across peak
- ~1 to 3 data points per second (Hz) (depending on peak width).

Decreasing scan speed
(increasing “N”)

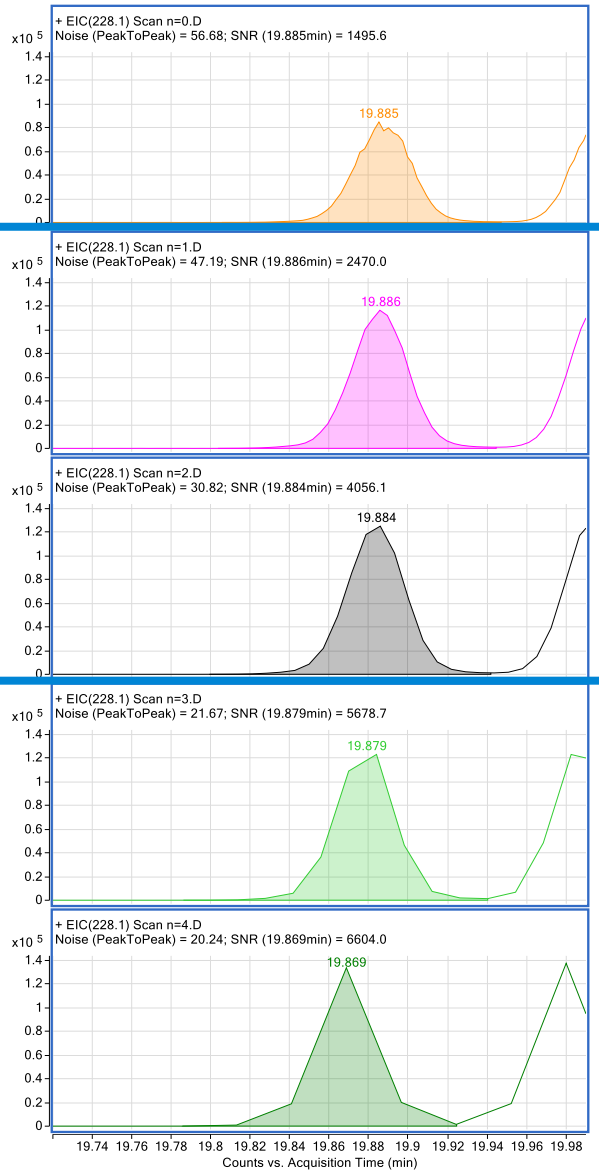
N=0
DP ~ 48

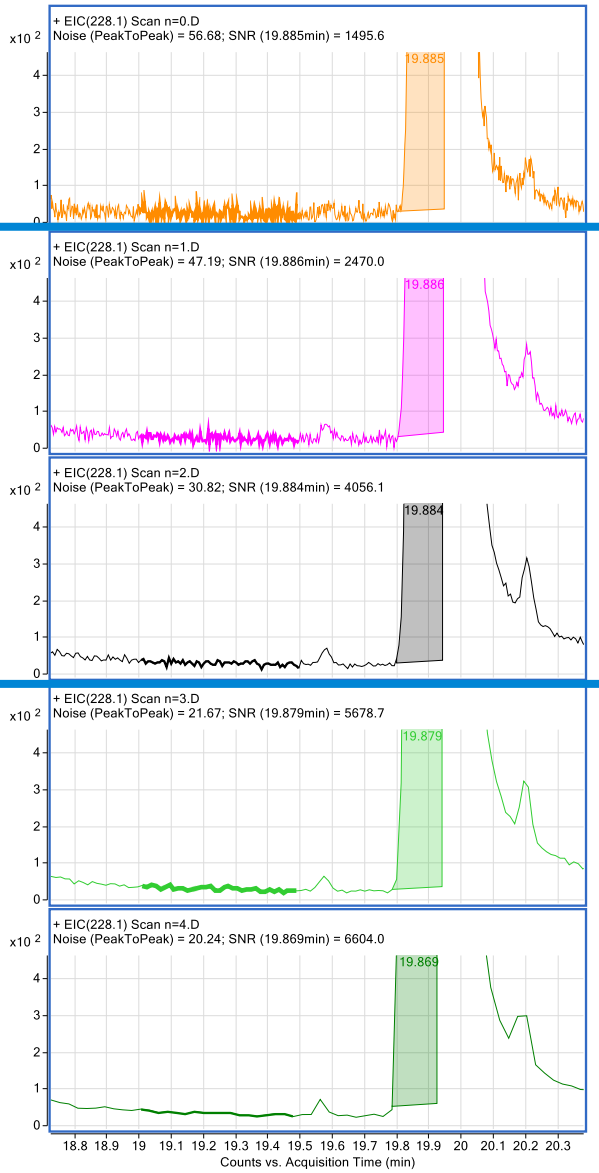
N=1
DP ~ 24

N=2
DP ~ 12

N=3
DP ~ 6

N=4
DP ~ 3





N=0
S/N ~ 1500
~8.1 Hz

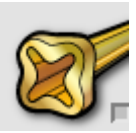
N=1
S/N ~ 2500
~4.4 Hz

N=2
S/N ~ 4000
~2.3 Hz

N=3
S/N ~ 5500
~1.2 Hz

N=4
S/N ~ 6500
~0.6 Hz

Agilent CrossLab From Insight to Outcome



Decreasing data points (DP) per peak

Increasing signal to noise ratio

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System optimization- calculating optimum gain factor

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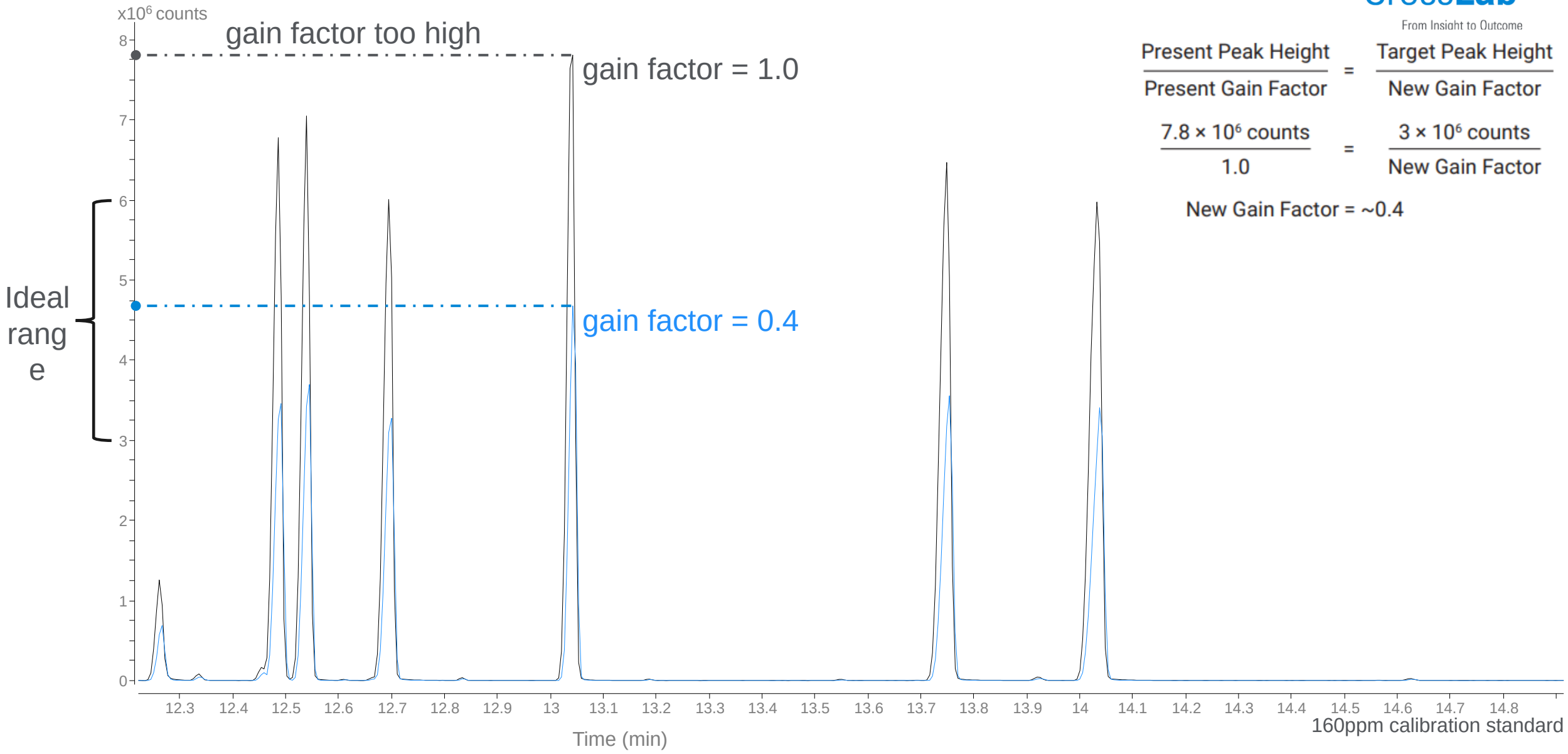

$$\frac{\text{Present Peak Height}}{\text{Present Gain Factor}} = \frac{\text{Target Peak Height}}{\text{New Gain Factor}}$$

$$\frac{7.8 \times 10^6 \text{ counts}}{1.0} = \frac{3 \times 10^6 \text{ counts}}{\text{New Gain Factor}}$$

New Gain Factor = ~0.4

160ppm calibration standard

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Gain factor

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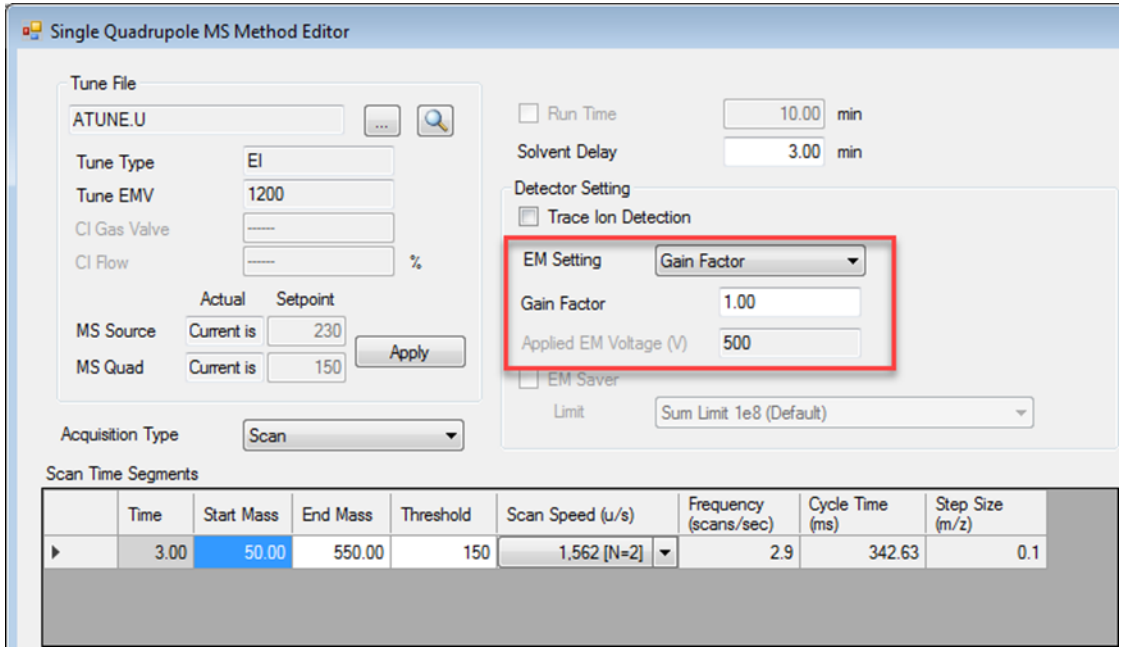
Instead of using very large “gain” values, Agilent converts them into smaller numbers that are easier to visualize.

$$GainFactor = \frac{Gain}{100{,}000}$$

Gain Factor is the number we type into a method.

So a gain factor of 1 = a gain of 100,000 electrons



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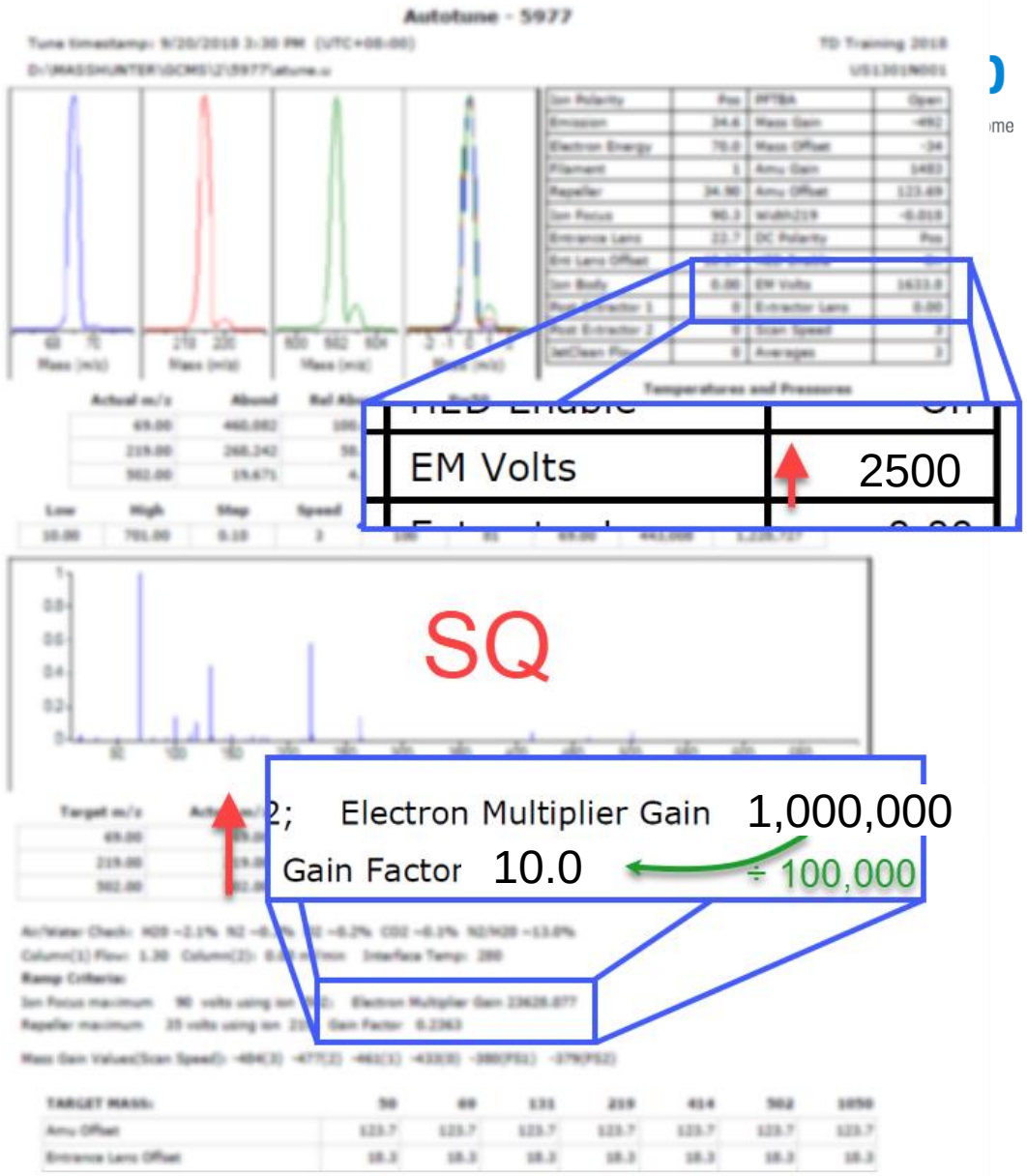
What can we learn from Autotune report (SQ)

Increasing Gain Factor… and EMV creeping up…

Generally indicates the **source is getting dirty**…

Generally happens over a shorter period of time… (depends on the application).



How to clean the source

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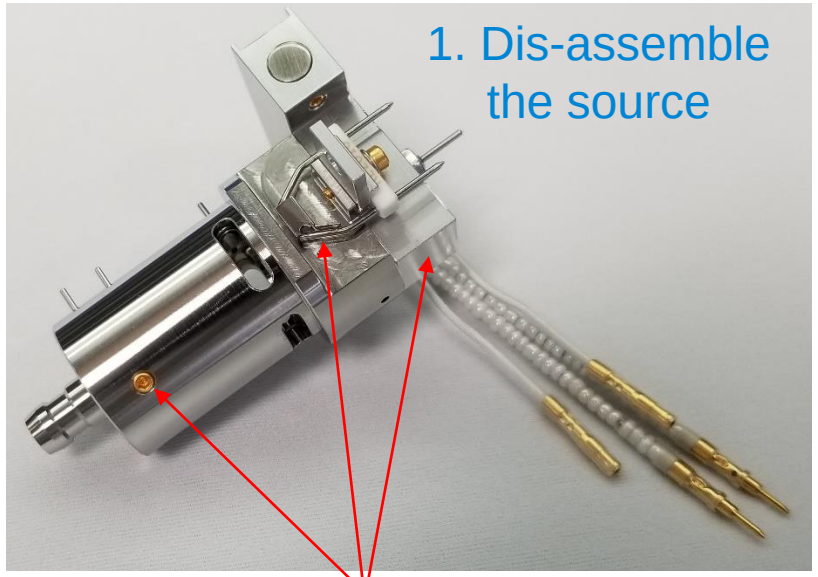

1. Dis-assemble the source

Do not clean the heater block, gold screws, nuts, ceramics, filaments or polyimide parts in this process

2. Clean metallic parts in the path of the ion beam and the source body

Use cotton swabs and slurry of alumina powder and DI H_2O

3. Rinse with lots of H_2O

You can't use too much water to rinse the parts.

Remove as much grit from parts as possible.

EI and CI source cleaning guide: 5989-5974EN

How to clean the source

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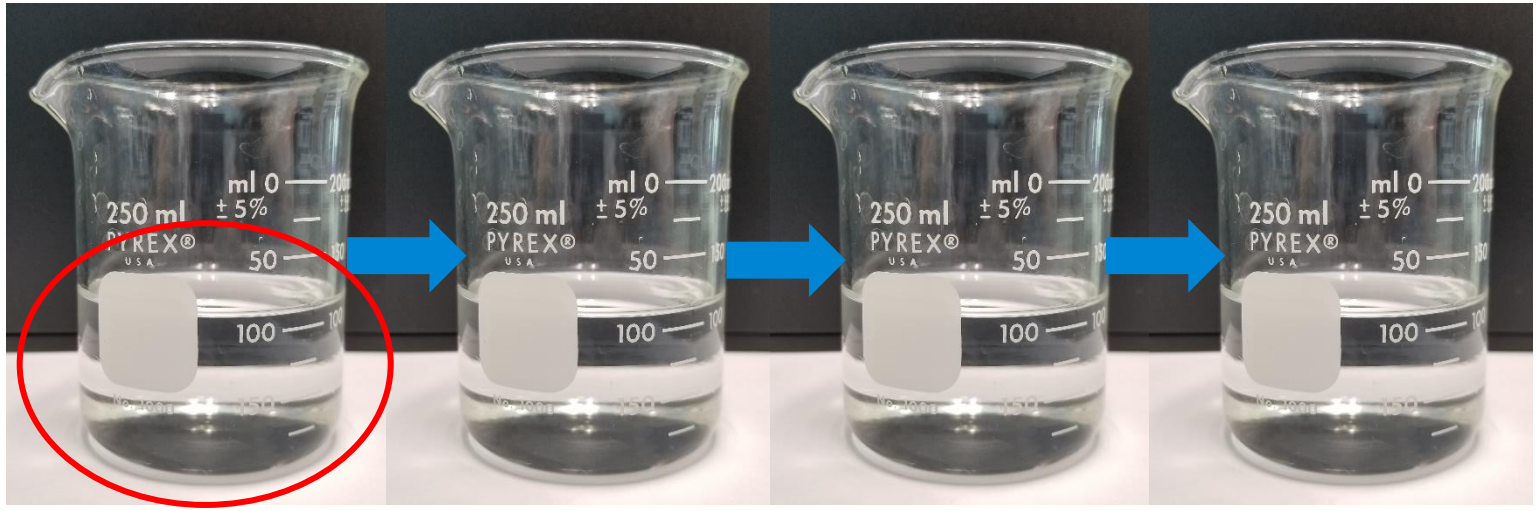
4. Sonication

- Submerge parts in beaker of DI H_2O
- Sonicate for 5 min

5, 6, 7. Sonication in other solvents

- Repeat step 4 with methanol, acetone and finally hexane
- Use different beakers between solvents

DI H_2O | Methanol | Acetone | Hexane

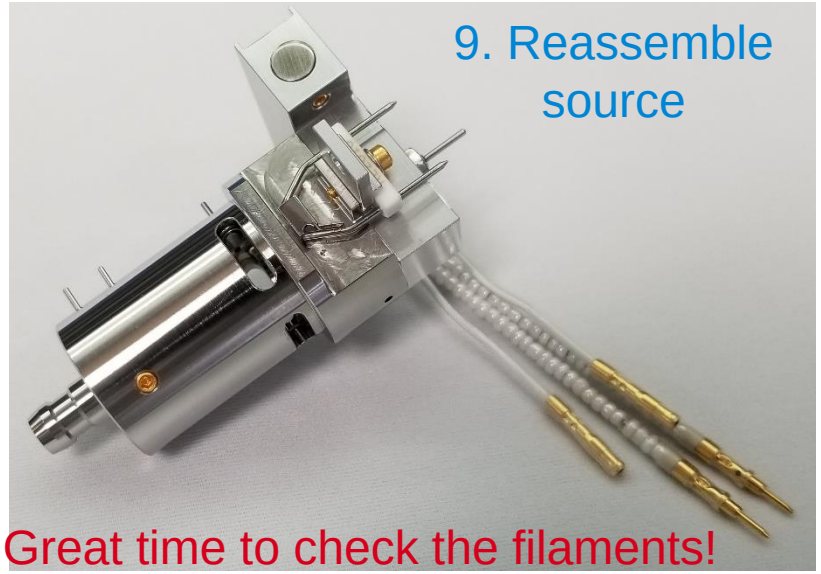


Enough solvent to cover all parts?

8. Dry source parts

- Remove parts from hexane beaker
- Place on clean foil or lint-free tissue/cloth
- Allow hexane to evaporate from parts

9. Reassemble source



Great time to check the filaments!

[EI and CI source cleaning guide](): 5989-5974EN

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Remember to check your filaments

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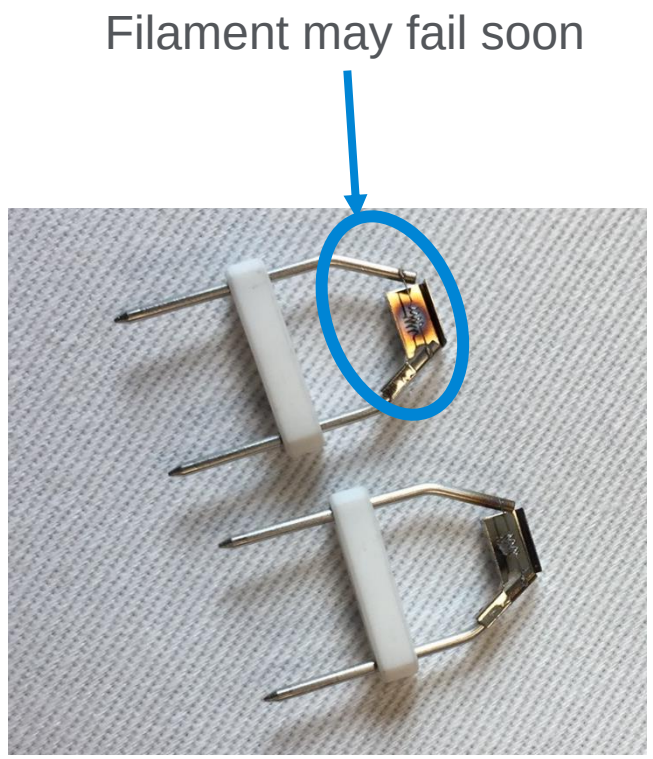
- Check filaments when you clean the source
  - Look for discoloration behind the filament and unraveling of the coil
  - Replace them as a pair
- End-of-life filaments may cause diminished response or odd artifacts in TIC
  - Keep them, just in case the problem is not the filaments
- Have (at least) 2 extra filaments on hand
  - More than 1 GC/MS system? Keep >2 on hand, depending on the number of systems.

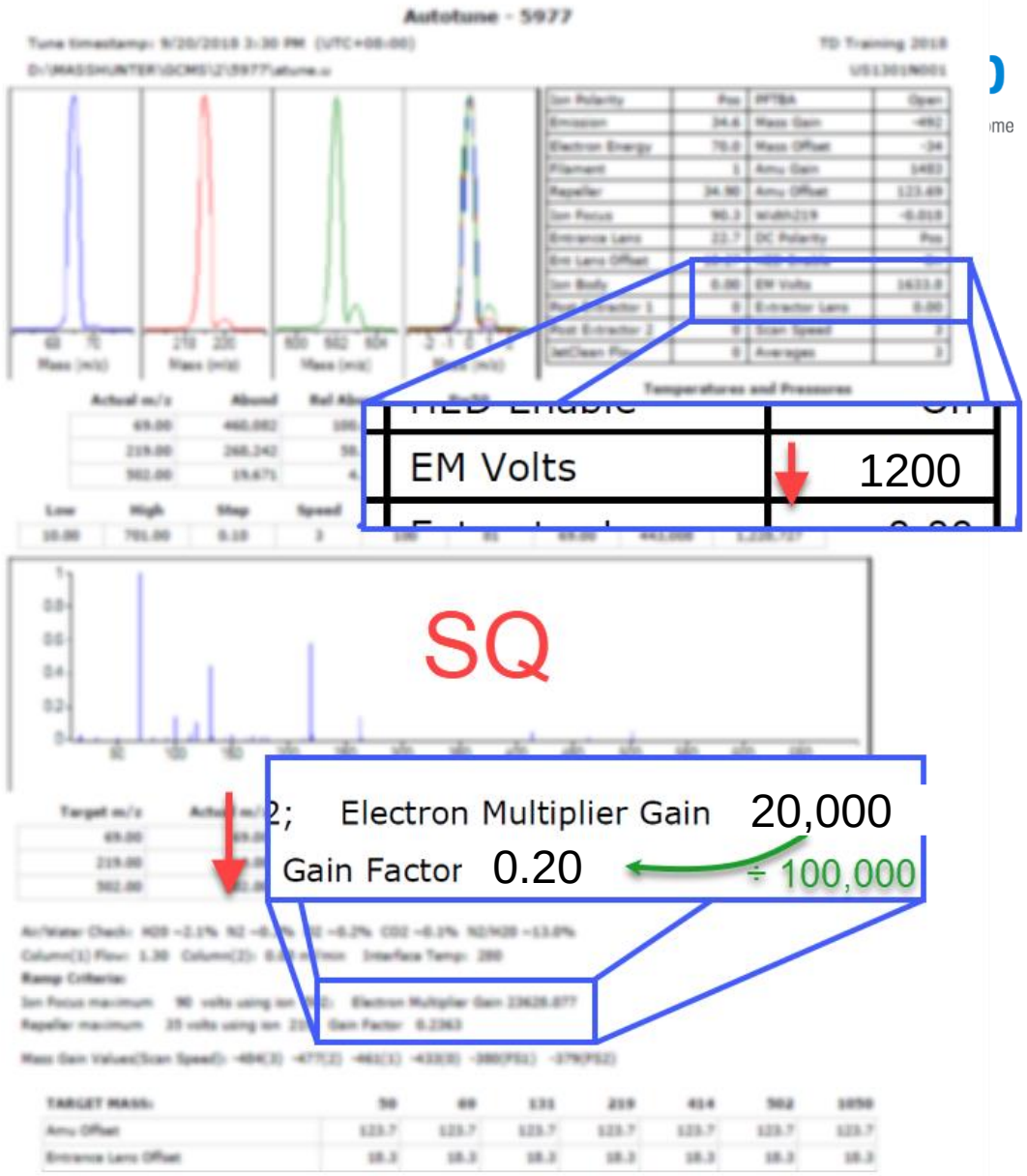
Careful! High Efficiency source (5977B HES single quad MS and 7010B HES tandem quad MS/MS) have different filament designs from 5977B InertPlus, extractor source and older MSD designs!



Agilent 5977 InertPlus, Extractor, & 5975 Filament Assemblies: G7005-6001

What can we learn from Autotune report (SQ)

Cleaning the source resets the values…

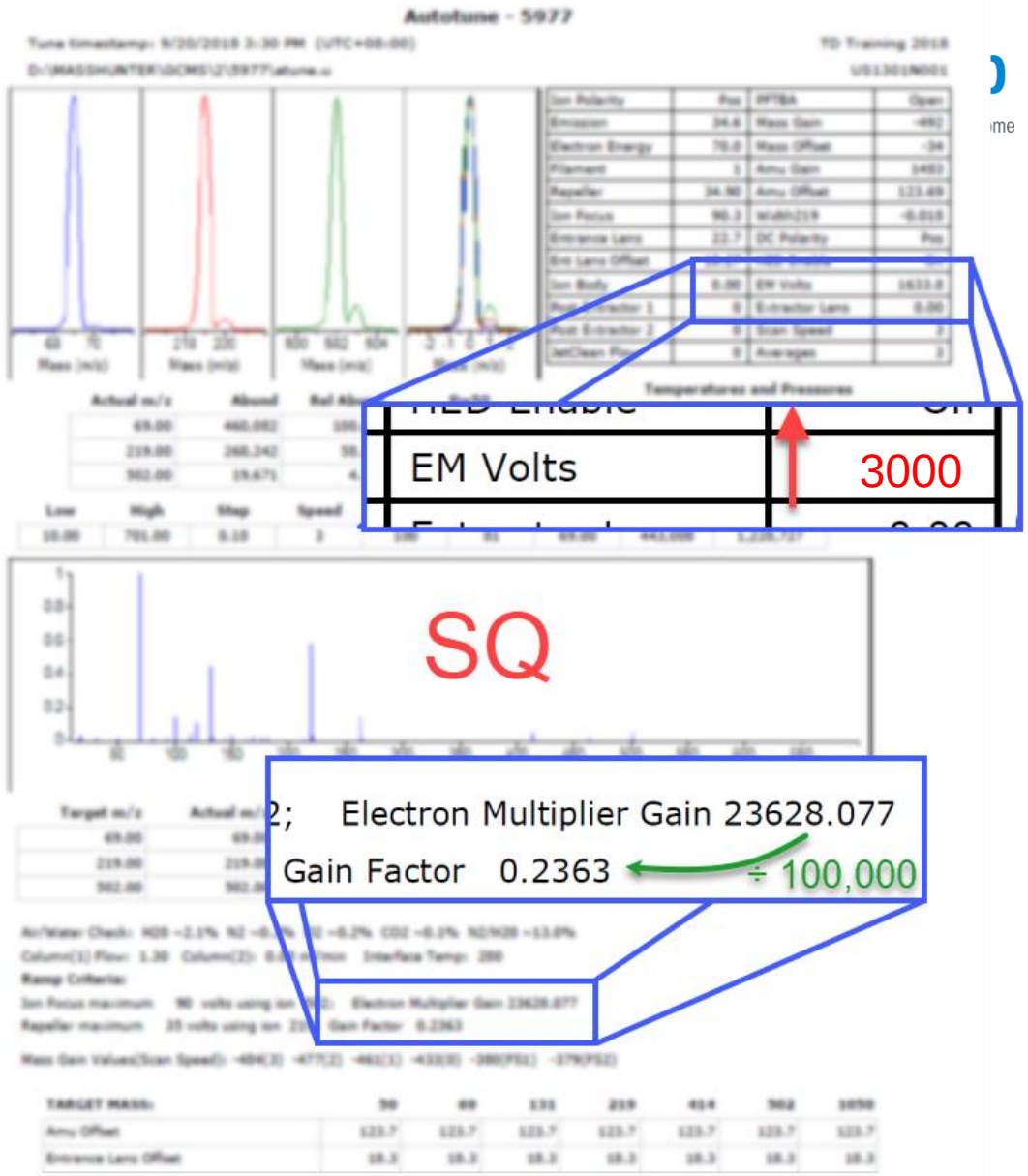


What can we learn from Autotune report (SQ)

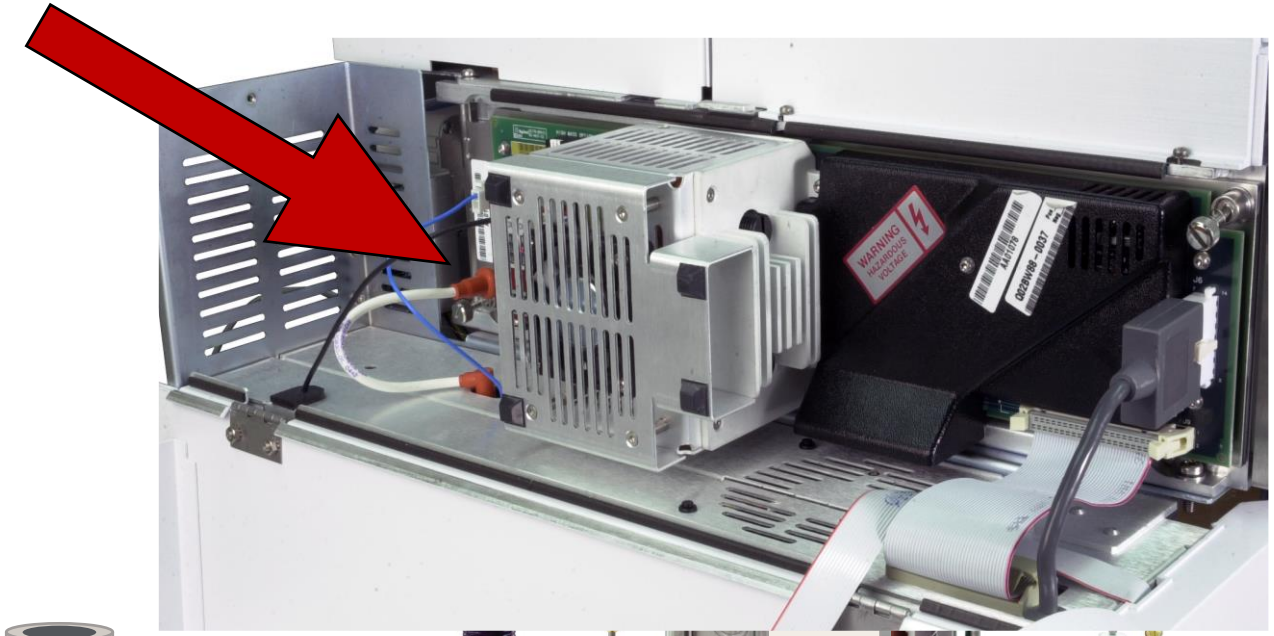
EMV increasing but Gain Factor remaining relatively unchanged…

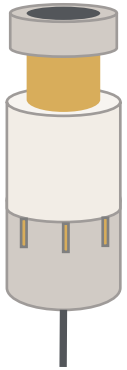
Indicates our EM is aging… happens over months…

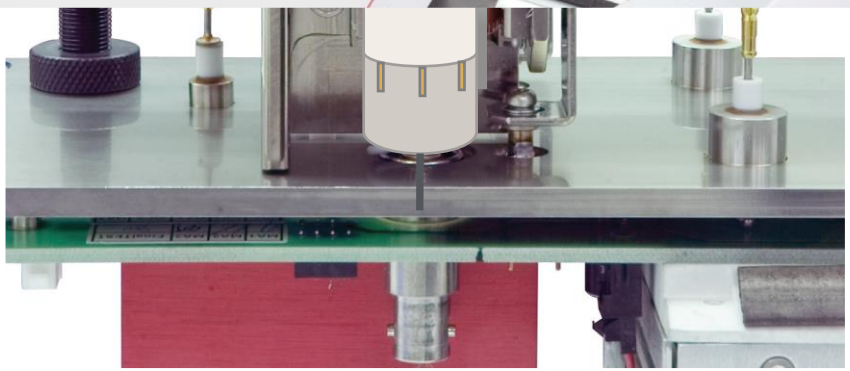
It is able to achieve the ~500K without needing to increase the gain, however an increase of EMV is required to maintain gain factor.



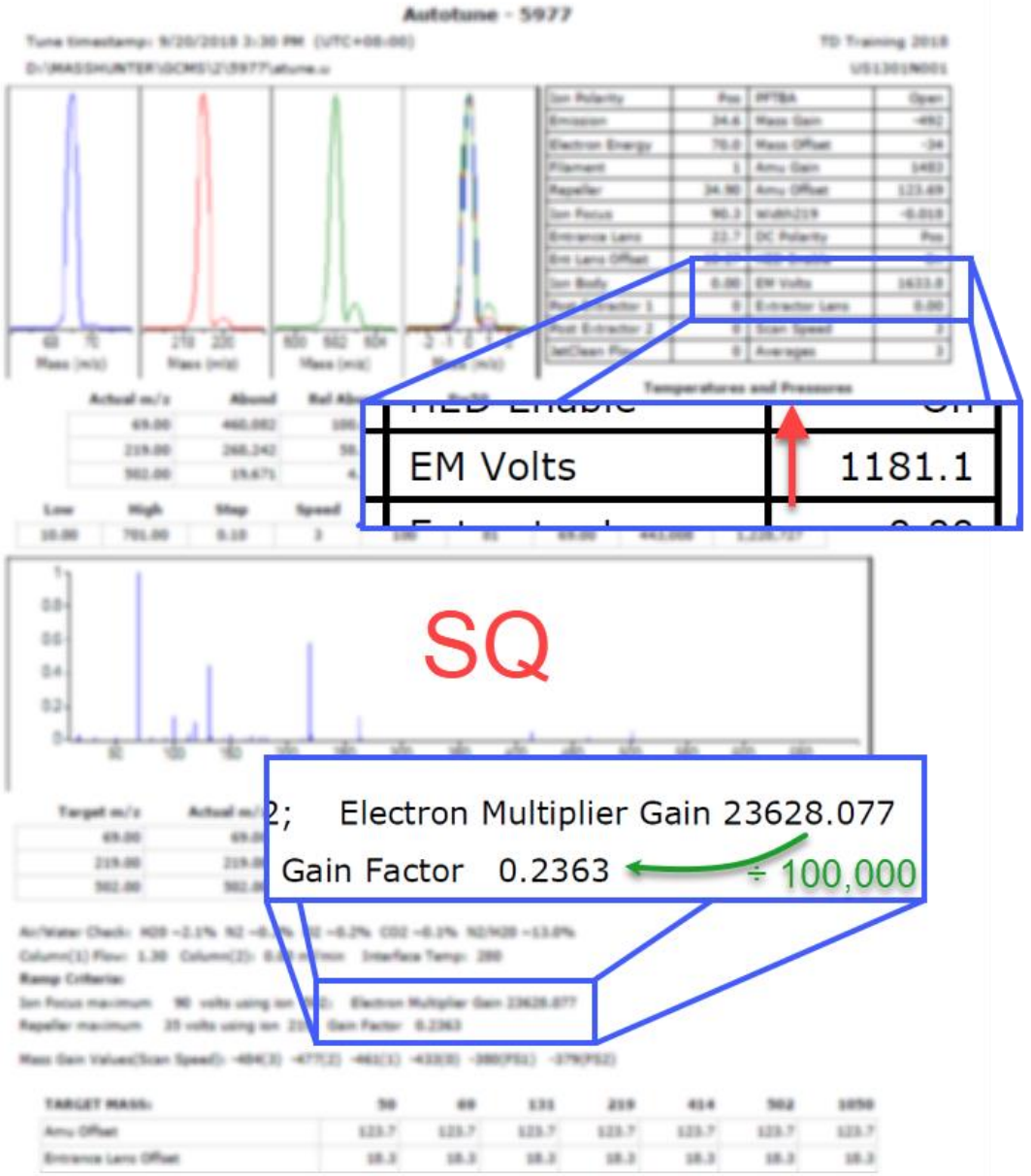
Replacing EM horn







p/n 05971-80103



Back to 100% with consumables and hardware to match your analysis

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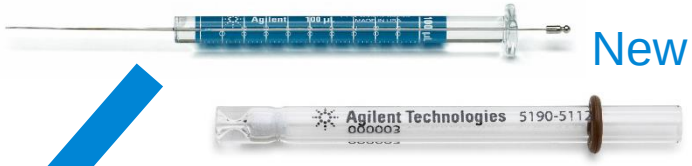
New inlet consumables

Wash vials filled with new solvent



New Gas Clean filter

New tips seals or fresh oil

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Contact Agilent Chemistries and Supplies Technical Support

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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

lc-column-support@agilent.com

spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com