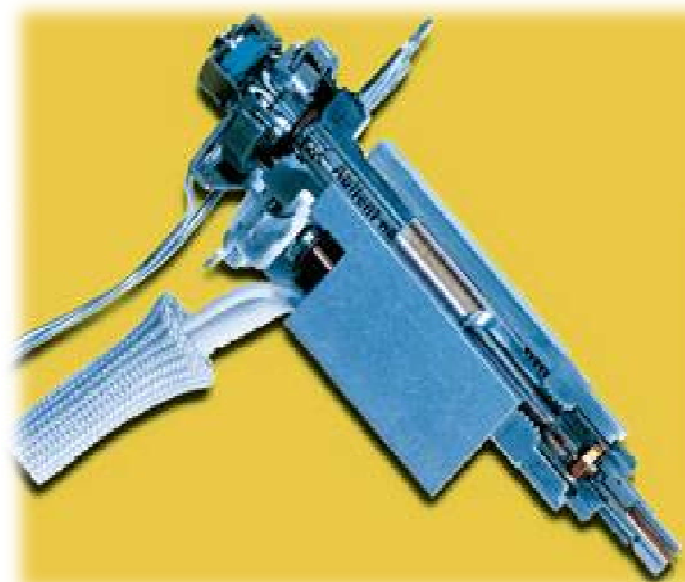


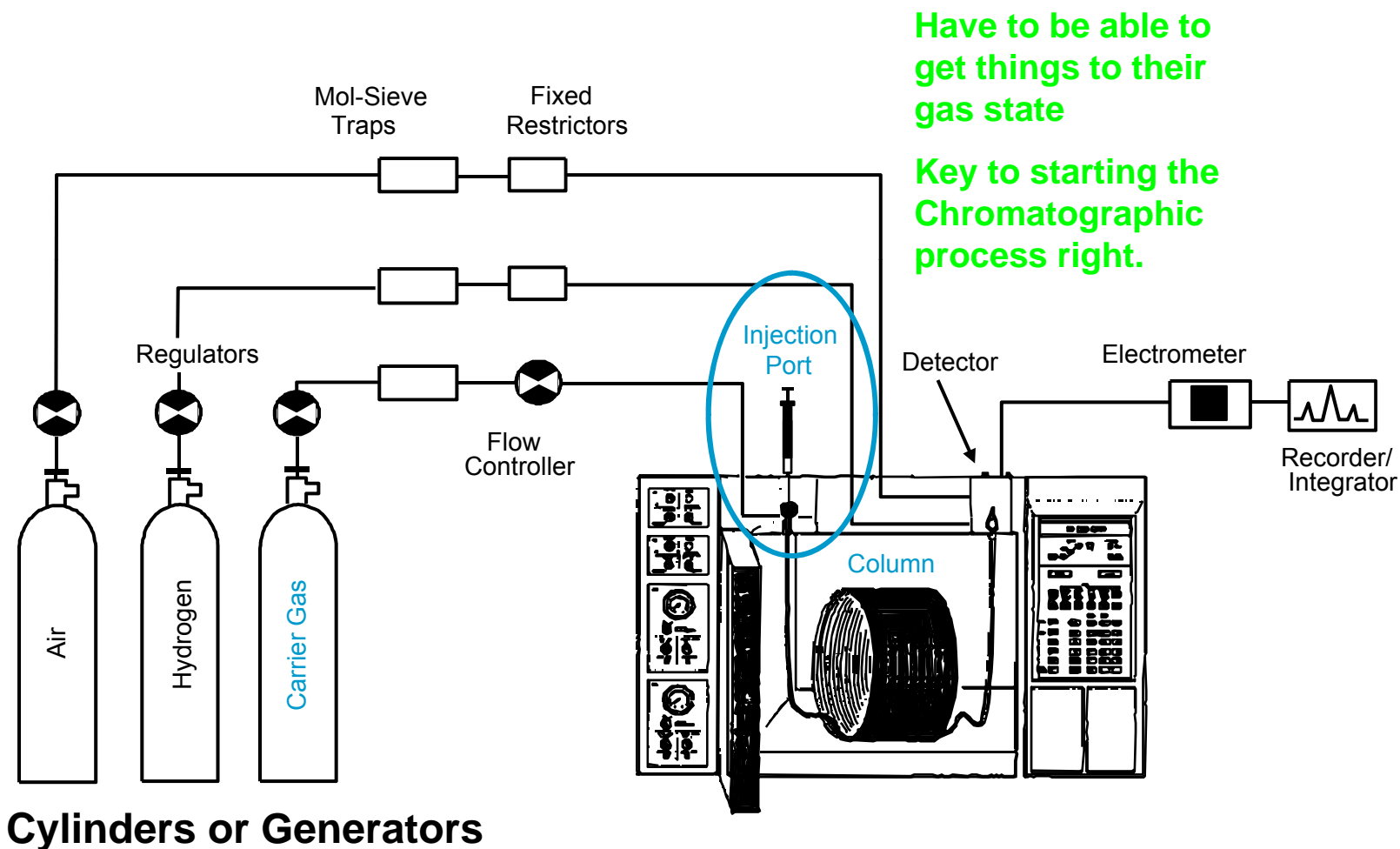
Tips and Tricks of Injector Maintenance

Abby Folk
Agilent GC Technical Support
January 2010



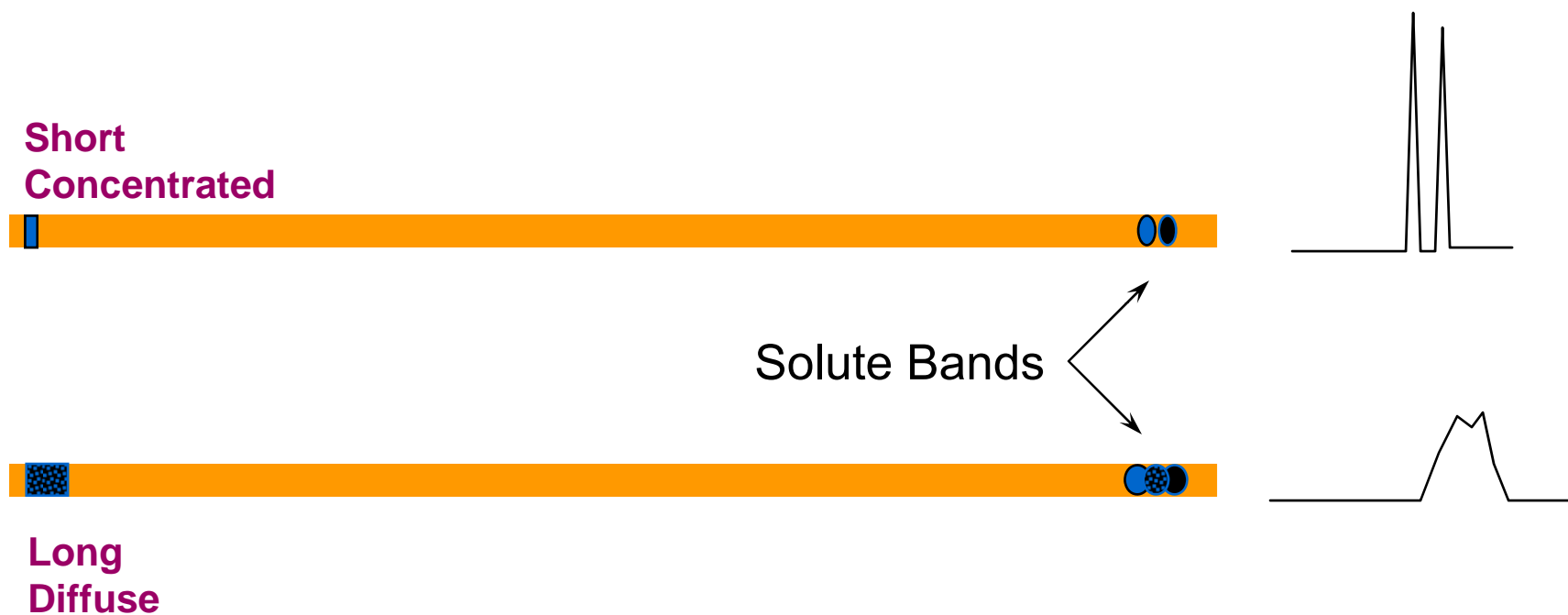
Agilent Technologies

Typical Gas Chromatographic System



Agilent Technologies

Influence of Injection Efficiency



Same column, same chromatographic conditions



Agilent Technologies

Injectors

Split

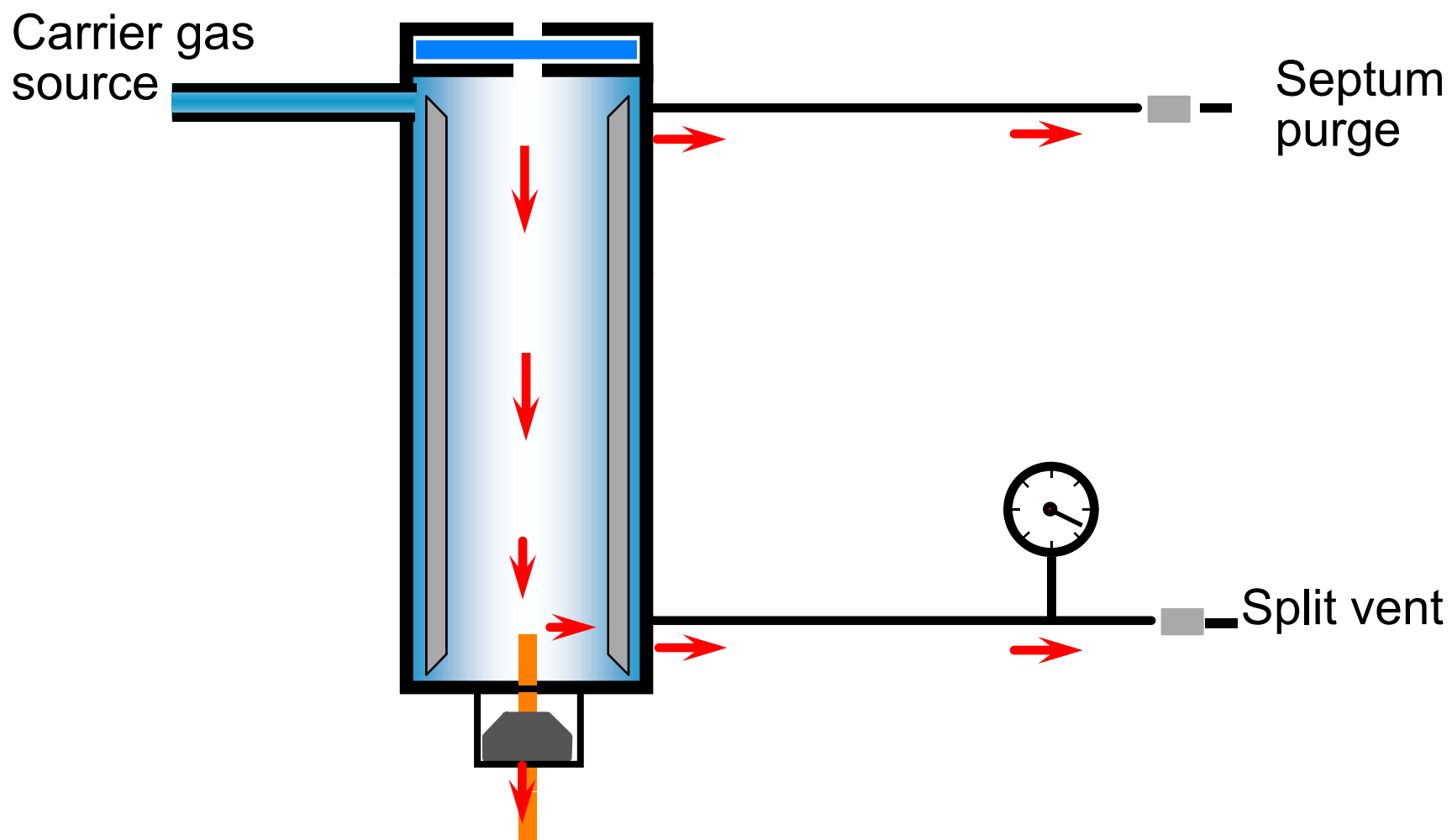
Splitless



Agilent Technologies

Split Injector

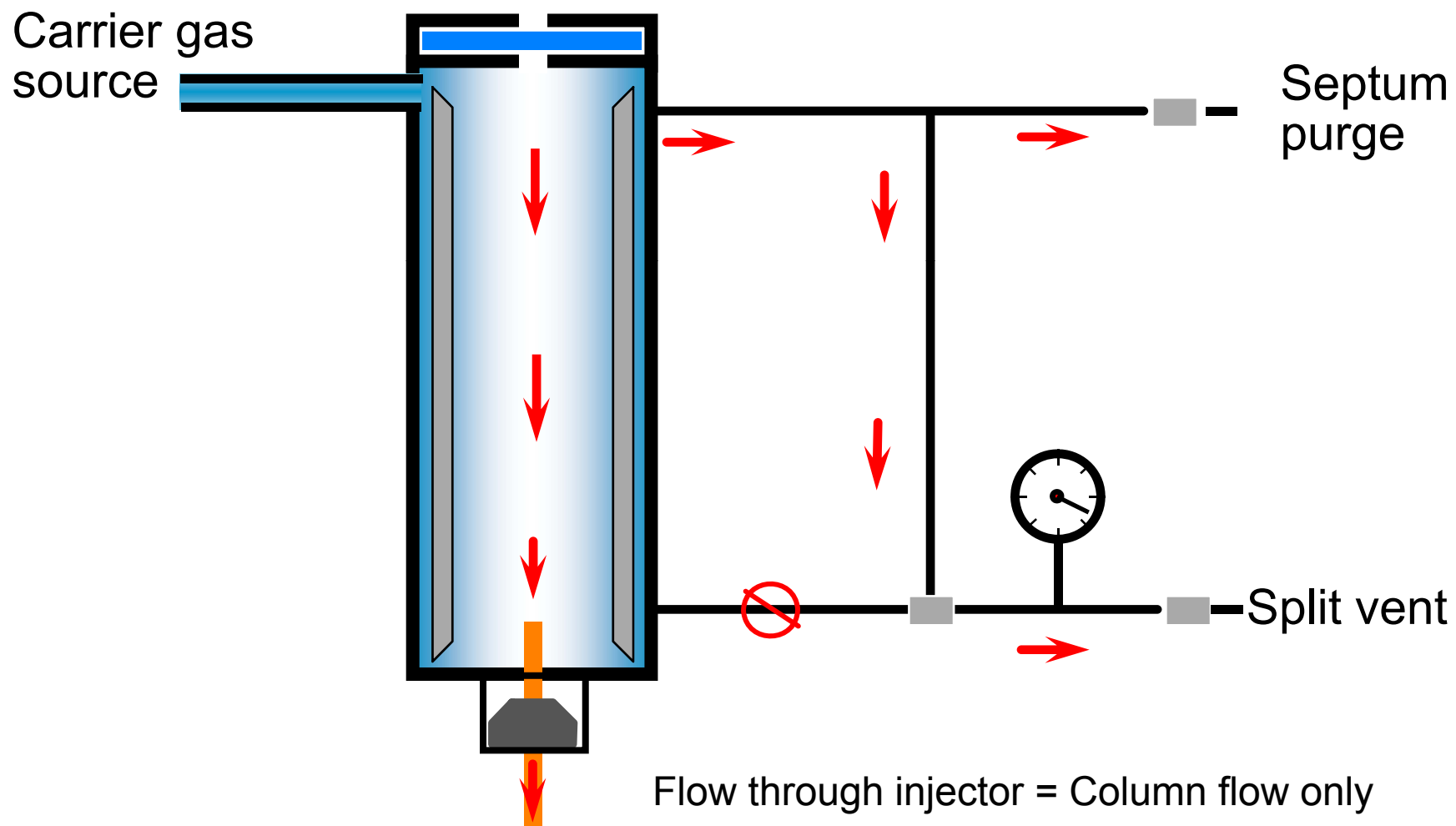
Flow Path



Agilent Technologies

Splitless Injector

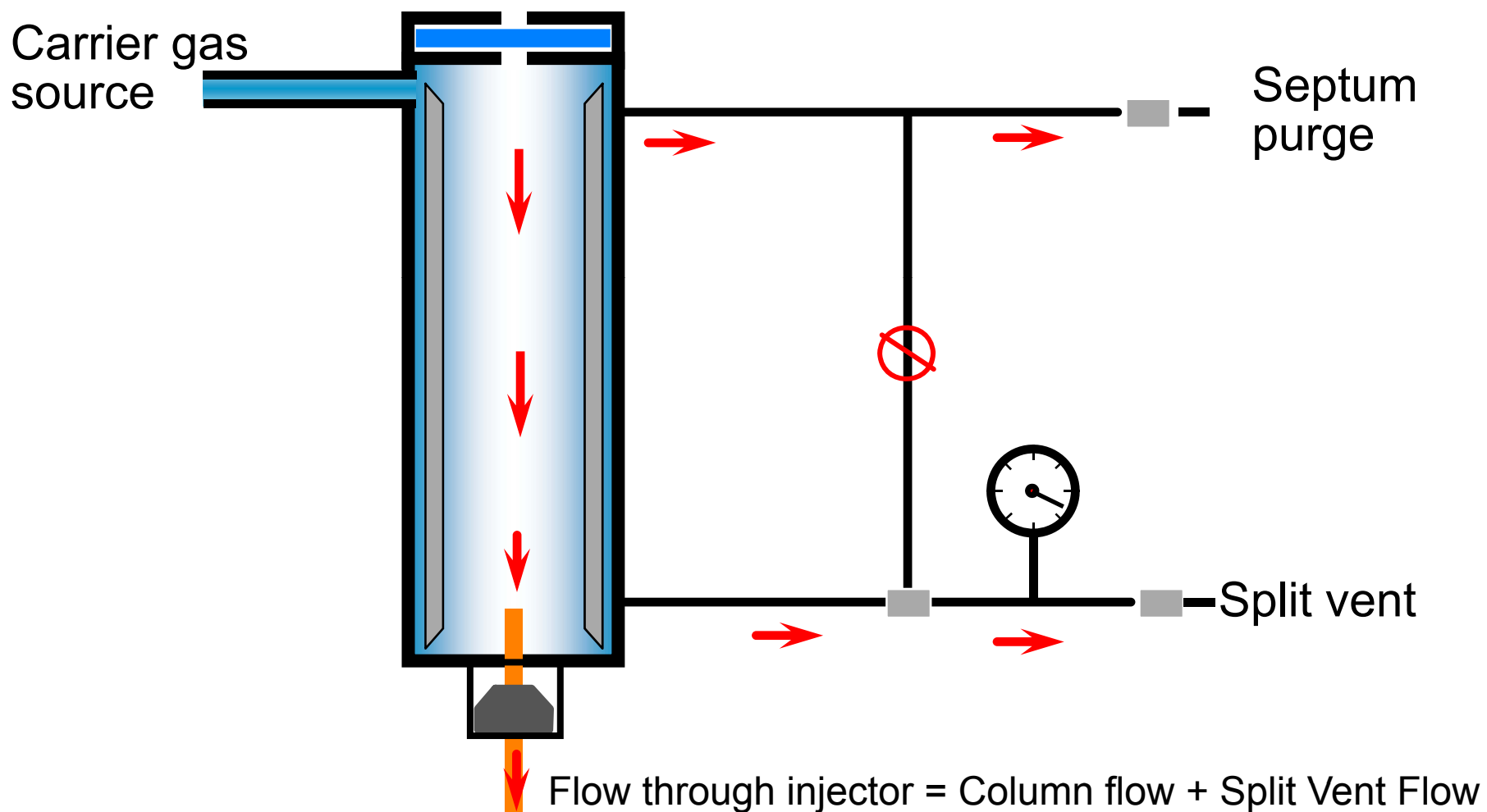
Purge Off At Injection



Agilent Technologies

Splitless Injector

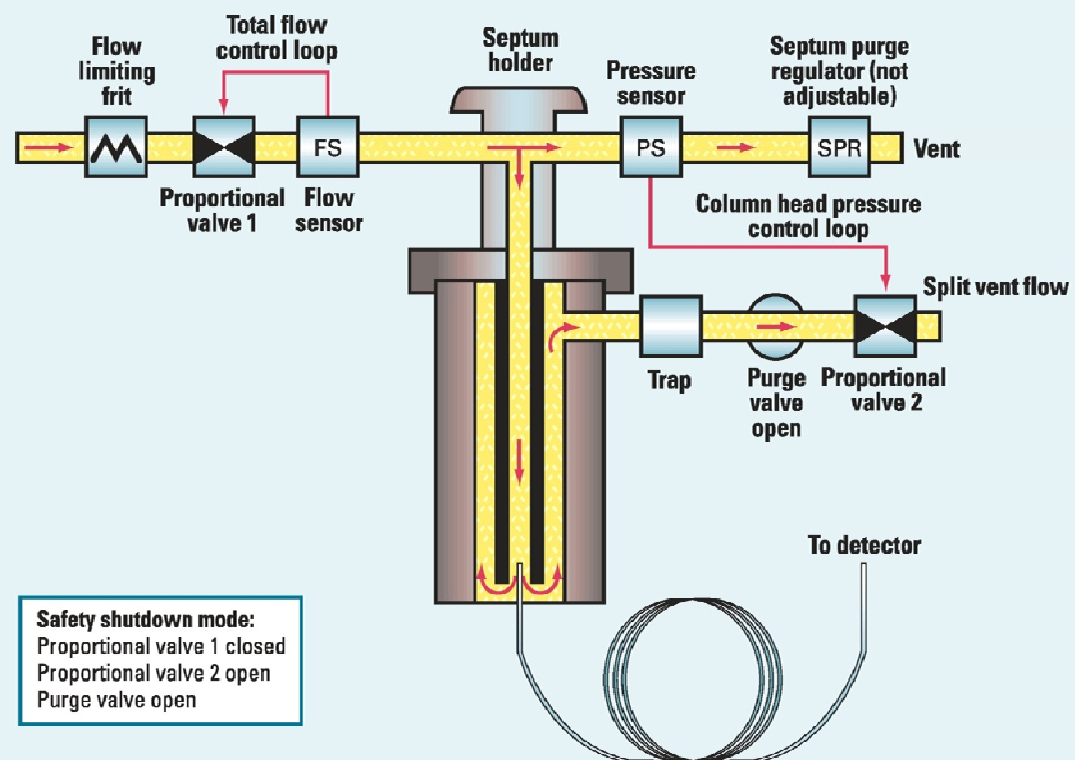
Purge On After Injection



Agilent Technologies

Split/Splitless Injector

Split/Splitless Inlet (Split Mode)



So, Why Do I Have To Do Maintenance?

1. Things wear out – septa, syringes, nuts, ferrules, o-rings, etc.
2. Things get dirty – liners, column, gas lines, traps, etc.



Agilent's Blue Line GC Autosampler Syringes

Supporting higher productivity features of 7693 ALS

- Improved plunger lifetime especially with PTFE tipped (aka Gas Tight) syringes
- Less septum coring due to finish of needle tip
- Better performance
 - Less carry over
 - More accurate sample volume delivered due to stroke alignment
 - Hamilton PTFE tipped syringe scale is different than Agilent
 - Setting 1 μ L on Hamilton ALS does not result in 1 μ L of sample injected

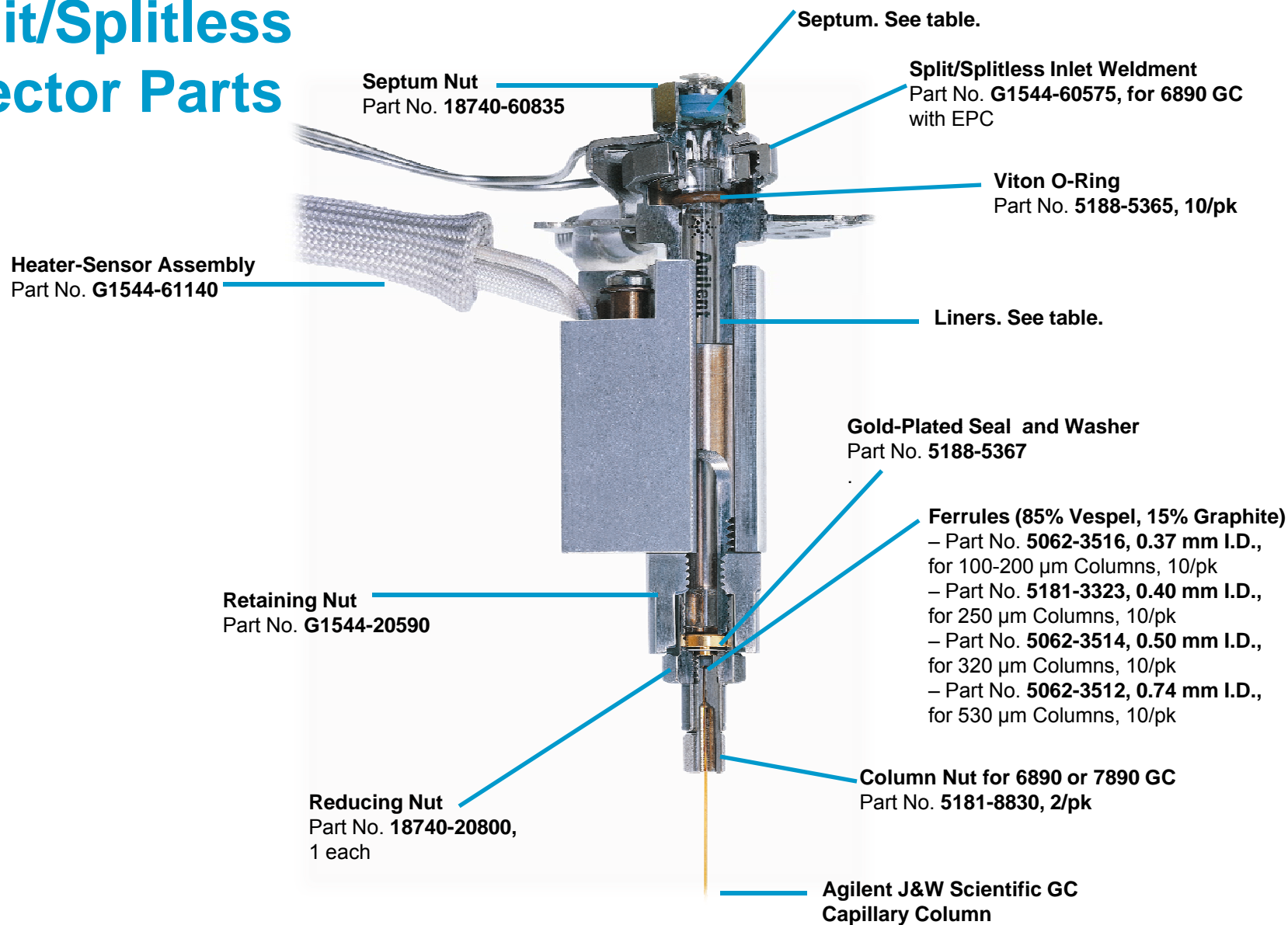


BUT...They Don't Last FOREVER!!



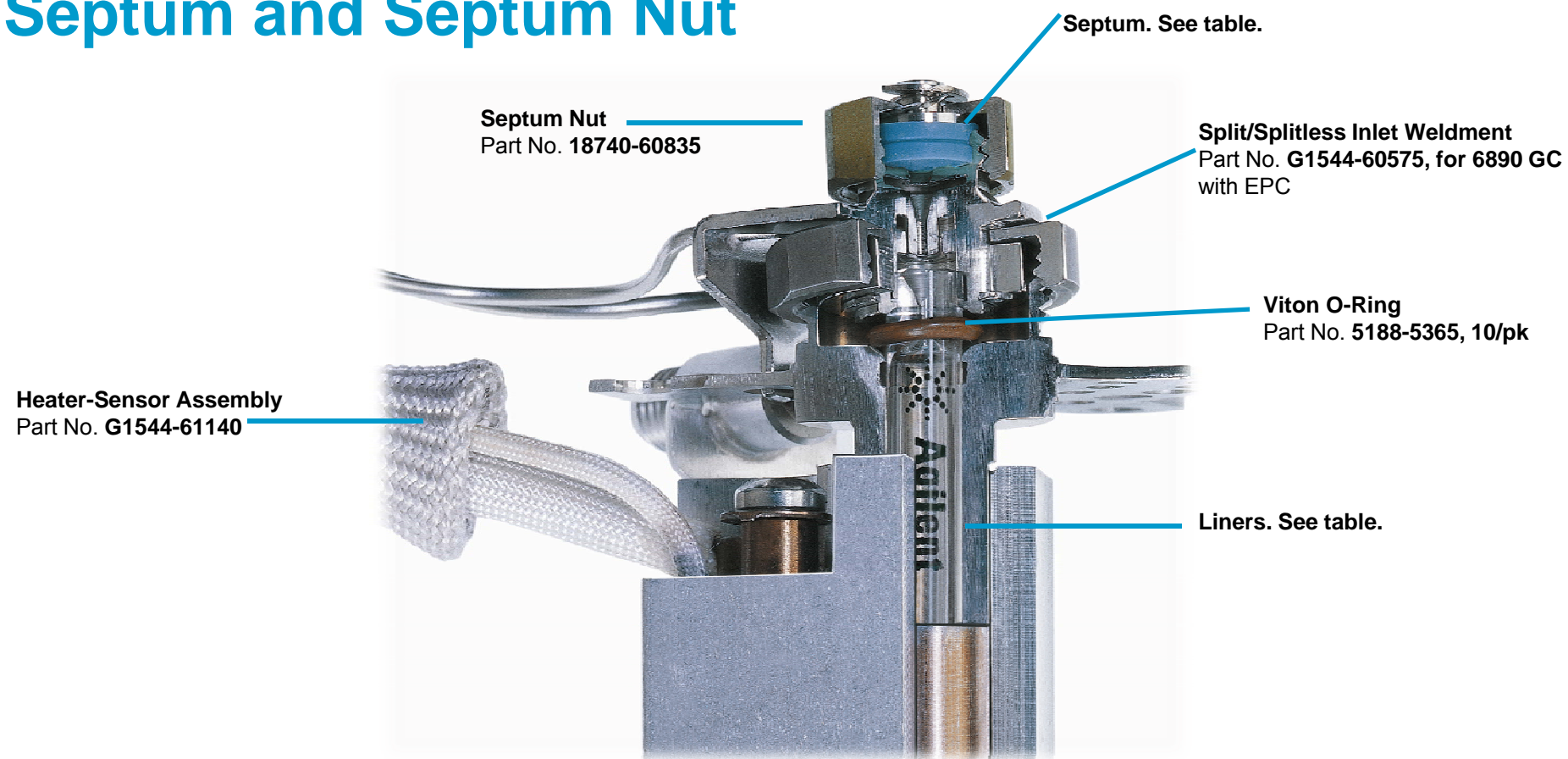
Agilent Technologies

Split/Splitless Injector Parts



Agilent Technologies

Septum and Septum Nut



Plasma treated Non-Stick fluorocarbon O-Ring
P/N 5188-5365



Agilent Technologies

Gold Seal & Washer

Retaining Nut
Part No. **G1544-20590**

Reducing Nut
Part No. **18740-20800**,
1 each

Gold-Plated Seal and Washer
Part No. **5188-5367**

Ferrules (85% Vespel, 15% Graphite)

- Part No. **5062-3516**, **0.37 mm I.D.**,
for 100-200 μm Columns, 10/pk
- Part No. **5181-3323**, **0.40 mm I.D.**,
for 250 μm Columns, 10/pk
- Part No. **5062-3514**, **0.50 mm I.D.**,
for 320 μm Columns, 10/pk
- Part No. **5062-3512**, **0.74 mm I.D.**,
for 530 μm Columns, 10/pk

Column Nut for 6890 or 7890 GC
Part No. **5181-8830**, 2/pk

Agilent J&W Scientific GC
Capillary Column

Column Ferrule & Nut



Agilent Technologies

Redesigned packaging: Agilent Convenience Dial packs

Individual packaging

- Maintains cleanliness
- Easy access to one at a time
- Doesn't go flying all over the floor
- Stores nicely in drawer

Inlet O-rings (Standard) 5188-5365

Inlet O-ring (Flip Top) 5188-5366



Agilent Technologies

Leak Checking

G3388A leak detector

Avoids loss of gases, damage to column and detector

- Portable, handheld unit shown is lightweight – only 310g/11oz
- Simple and easy to use
- Fast detection – 1 second
- Audible and visual alerts for 12 gases
- Minimum detection limit of 0.01 mL/minute for hydrogen and helium
- One year warranty
- RoHS compliant
- Rechargeable NiMH battery with over 5 hours of life



Agilent Technologies

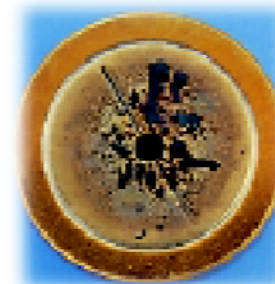
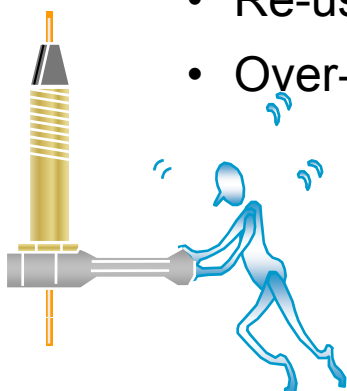
Replace Because...Most Common Causes of Leaks

Gold Seal P/N 5188-5367

Re-use and mis-installation.



- Leak from O-ring, Gold Seal, ferrules, column nuts
- O-rings are elastomer compression fittings designed for one use, not perfectly elastic.
- Gold seals are designed for one use, knife edge cuts into gold layer giving leak tight seal w/o shrinkage or potential organic contaminants from polyimide out-gassing/degradation.
- Manufacturing procedure of Agilent gold seals results in best sealing surface for leak free inlet
- Re-using could result in overlap in seal rings, resulting in a leak.
- Over-tightening of fittings

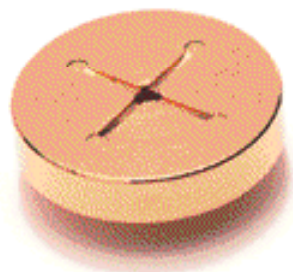


Agilent Technologies

Why Are There Different Gold Seals?

High Split Flows

- Limited by flow controller
- Range usually 1:200 to 1:1000



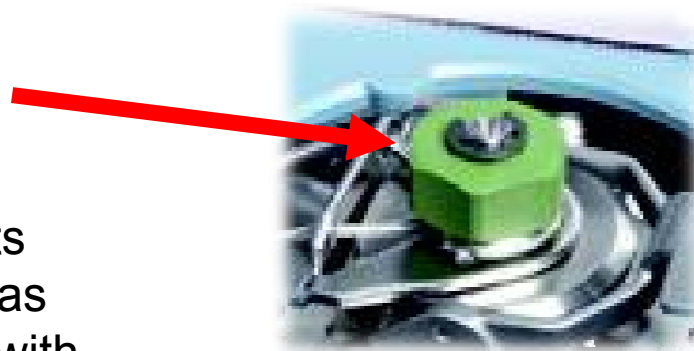
**Gold plated inlet seal with
cross, p/n 5182-9652**



Agilent Technologies

Leaks Due to Septum Nut

- With repeated use, conical needle guide gets worn, out of round, and needs replacement as septum can begin to “bulge” out, especially with excessive tightening,
- Septa fail faster because needle is not guided with as much precision.
- Under or Over tightening—tighten nut until c-clamp on top stops turning, then $\frac{1}{2}$ to $\frac{3}{4}$ turn more.
- Non-Agilent septa may be too thin, too thick, or out of round like die-cut septa and may not seal as well.
- “Use Environments” that decrease lifetime, like using non-Agilent Autosamplers (ours are precisely aligned), manual injection, larger gauge syringes
- Replace septum nut annually for peace of mind.



Tips to Maximize Septum Life, Minimize Septum Leaks

- Use Agilent Gold Standard, HP Point, 23-26 gauge taper syringes. The point style cores septa significantly less when used with CenterGuide Septa. Taper minimizes septum coring/wear.



HP-Point Style

- Use Agilent CenterGuide Septa. The molded hole minimizes septa coring, counter-intuitive, but true.

Solid Septum

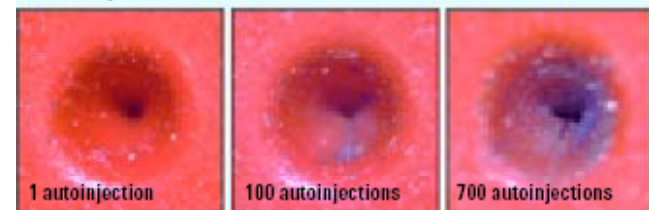
CenterGuide Septum



High-Temperature Septa Without CenterGuide: Major Coring Before 100 Autoinjections

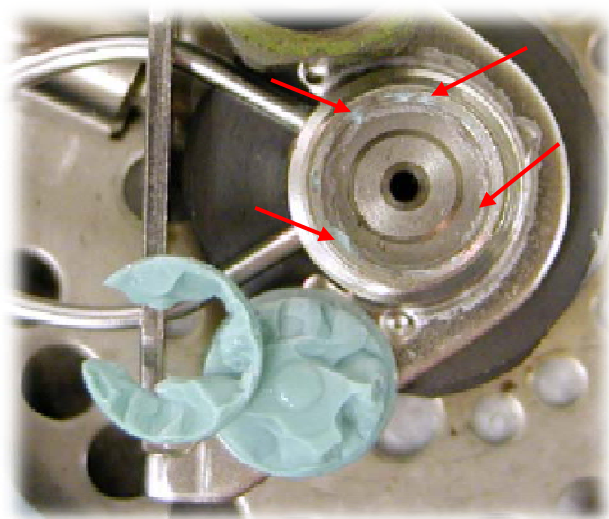


Agilent BTO Septa With CenterGuide: Very Little Coring Even After 700 Autoinjections

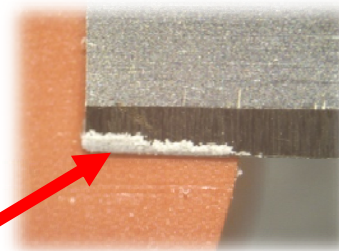


Tips to Maximize Septum Life, Minimize Septum Leaks

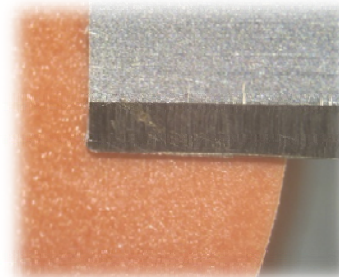
- Use Non-Stick septa, especially Agilent's Centerguide Septa with Proprietary Plasma Treatment



Their's
Talcum Powder!



Our's



- Stuck septa particles can cause sealing problems on next septum installation. Talc can cause activity/trap plugging problems



Agilent Technologies

Tips to Maximize Septum Life, Minimize Septum Leaks



Other Benefits of Agilent's Septa

- Chromatographically “ready” – no need to bake in oven
- Packaging eliminates contamination of septa,
 - “first is as good as the last”
- Less Strain on Syringe compared to solid septa
- Bleed/Temperature Optimized, (to 400C, [trace analysis](#)), p/n 5183-4757
- Advanced Green, (to 350C, [good for general purpose](#)), p/n 5183-4759
- Long Life, (to 350C, [more injections before failure](#)), p/n 5183-4761
- Above are 50 packs, 100 packs also available.



Septa vs GC Column Costs

- Typical cost of 1 Premium Septum (list), \$1.25
- Typical cost of 1 GC Column, 30 m x 0.25 mm ID, \$450.
- No accurate leak rate detector at sub 1 mL/min flow rates.
- “Don’t step over a dollar to pick up a dime!”
- Proactively change inlet septa.



Or Go Septumless! – Merlin Microseal

Low bleed, longer life alternative to standard septa for split/splitless injection

More than 2000 injections, depending on samples and operating conditions

Almost zero downtime for septa changes and injection port liner changes due to septa particulates

Double O-ring type seal around the syringe needle

Spring assisted duckbill to seal the injection port



Agilent Technologies

So, Why Do I Have To Do Inlet Maintenance?

1. Things wear out – septa, syringes, nuts, ferrules, o-rings, etc.
2. Things get dirty – liners, column, gas lines, traps, etc.



The BIGGEST Problem in GC is...

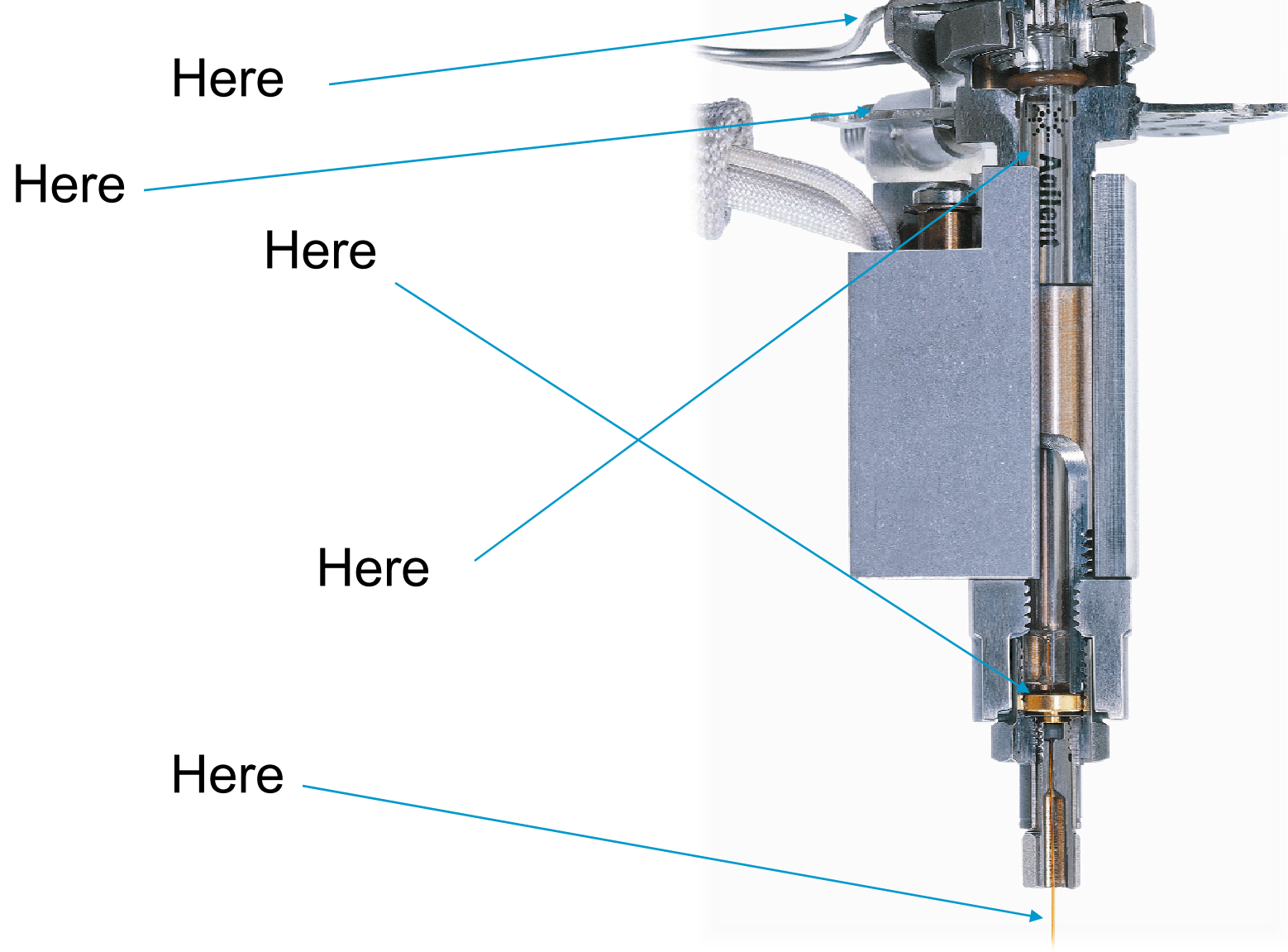
There are more things that DON'T go through a GC than DO!

....therefore, don't inject anything and you'll never have problems.

OK, inject, but realize that everything just got dirty...deal with it!



Where Does it Get Dirty?

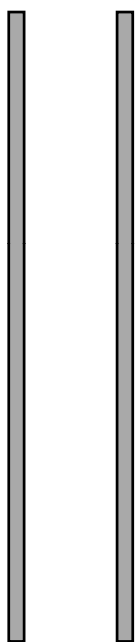


Liner Maintenance

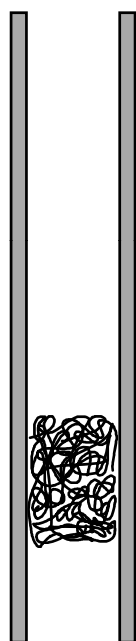
- Liners become contaminated with use, collecting non-volatiles, salts, excess reagents, etc., or become damaged/cracked.
- Should inspect and replace liners often.
- Handle with gloves and forceps.
- Insert into or remove liners only from cool injection ports.
- Replacing with a new liner is recommended to ensure reproducibility



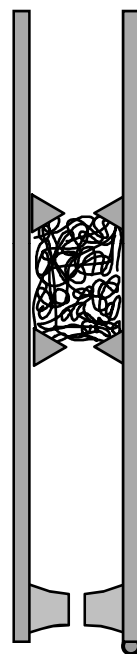
Split Liners – What's What?



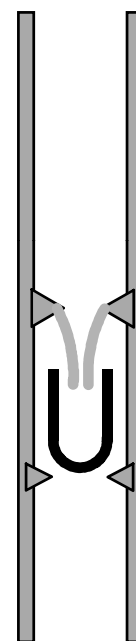
Straight
tube



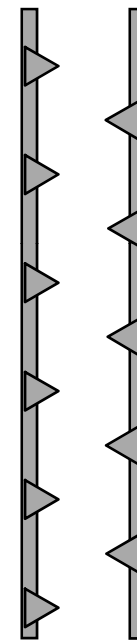
Straight
tube with
glass wool



Fixed glass
wool



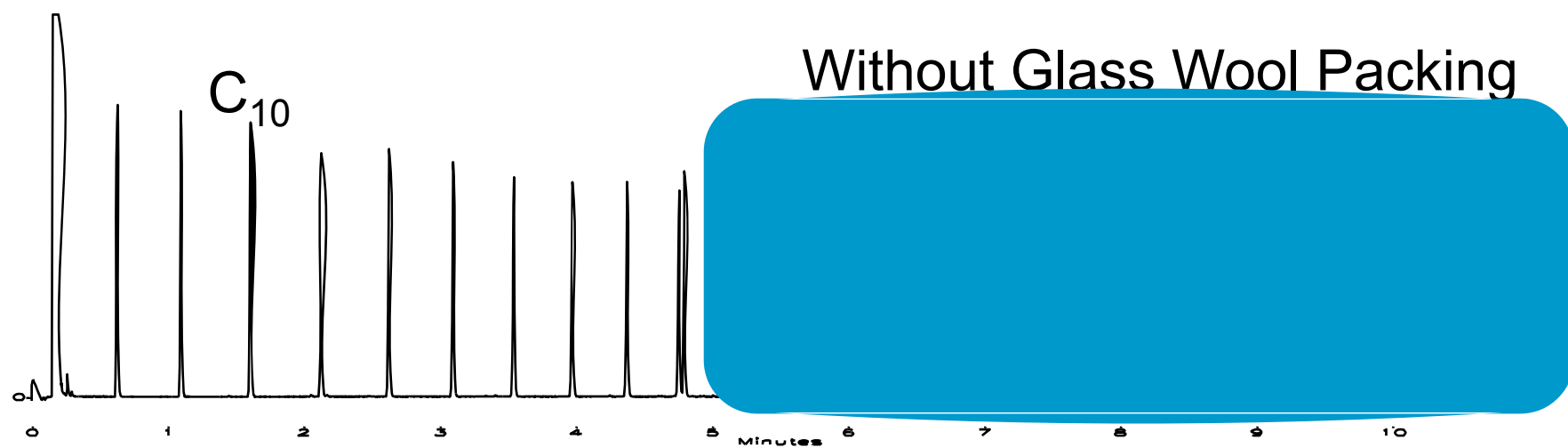
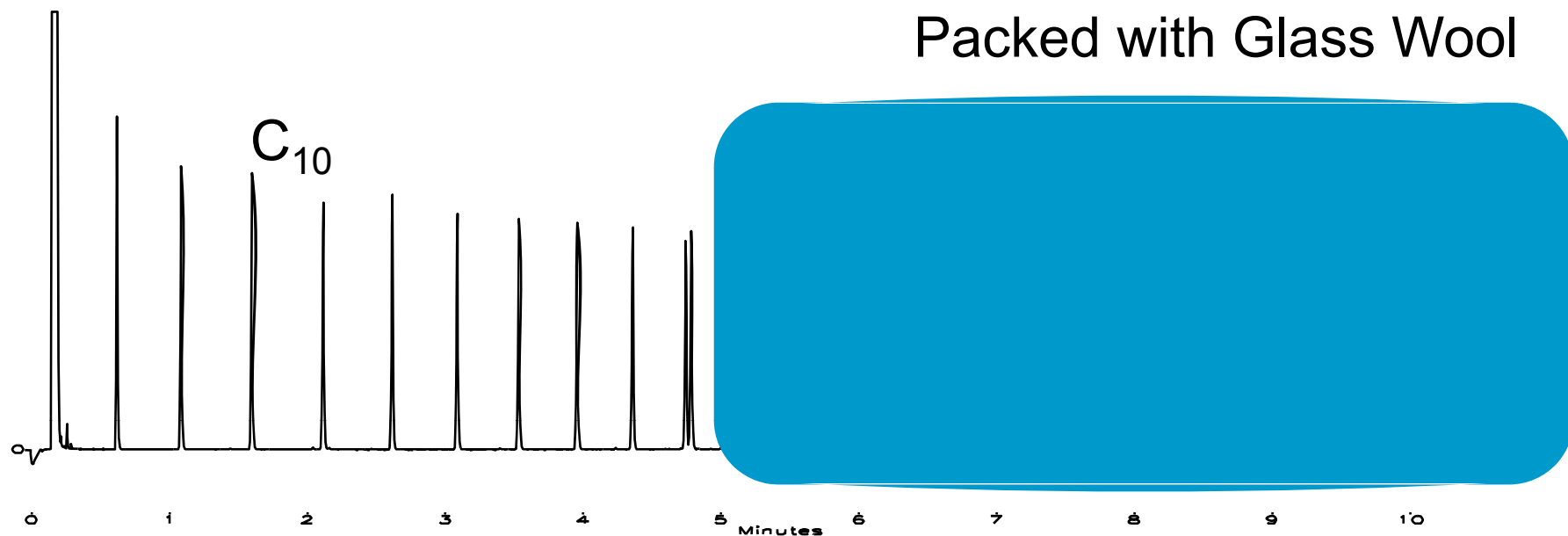
Inverted
cup



Baffle

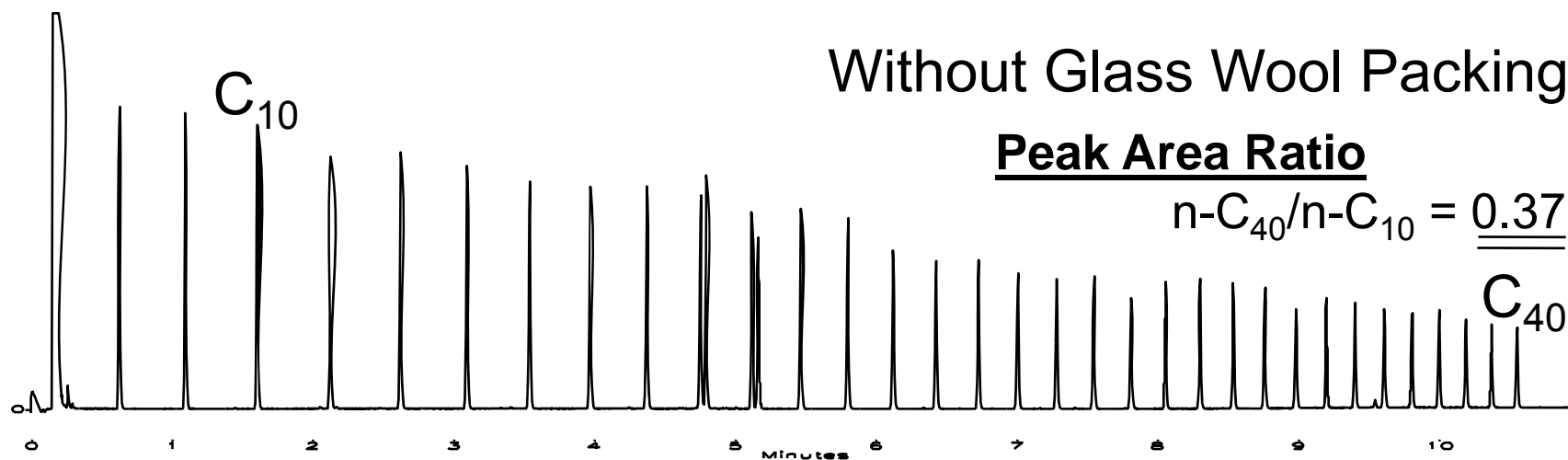
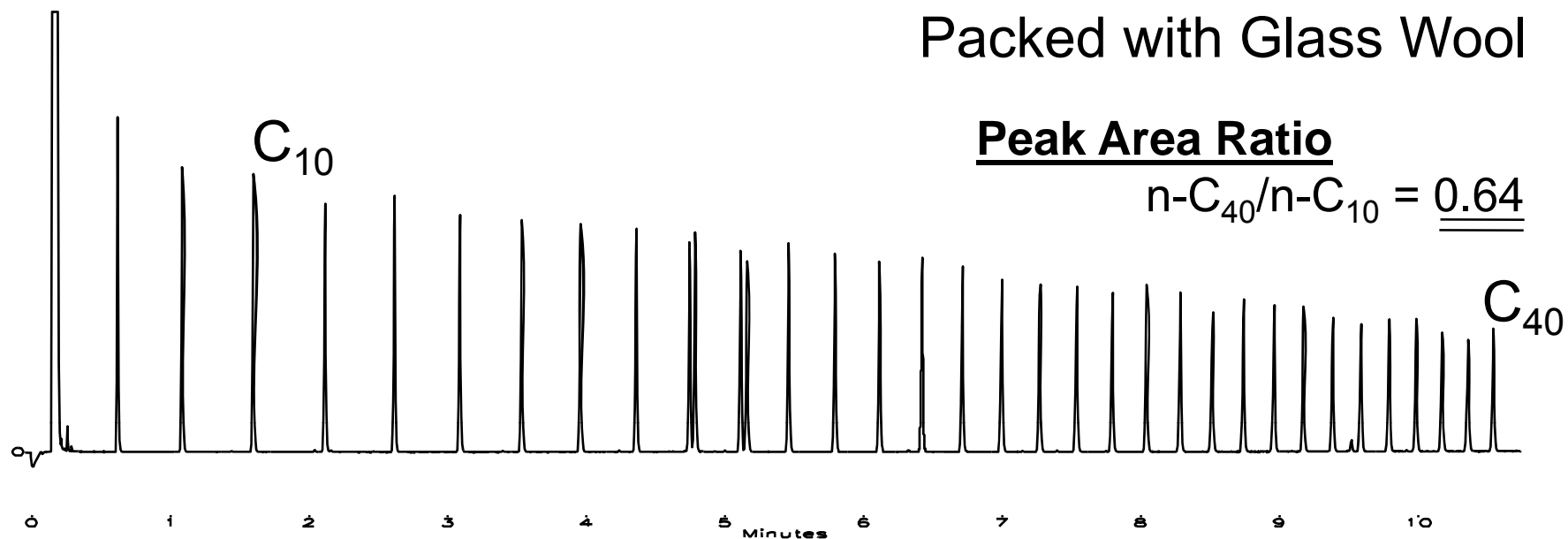


Split Liner

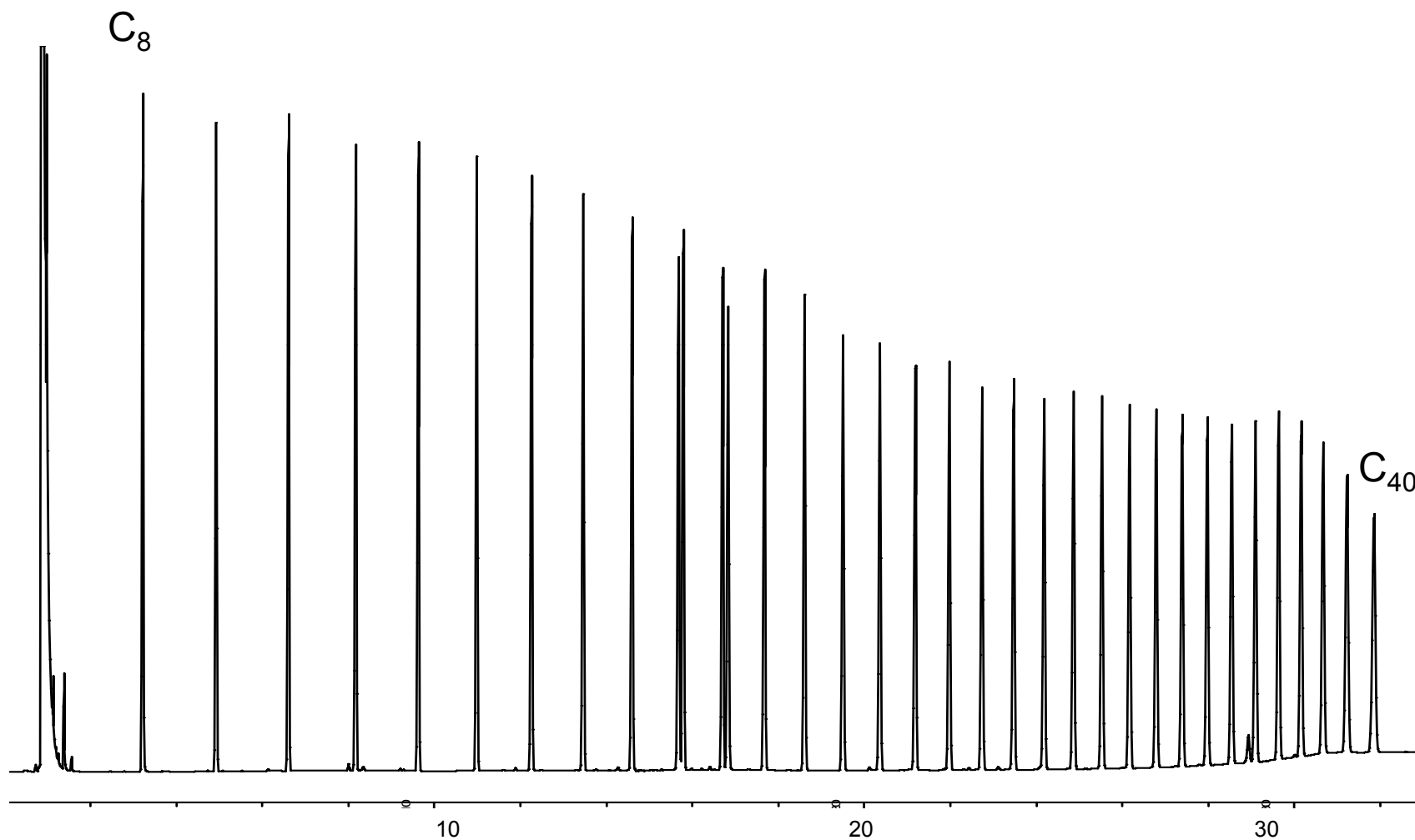


Agilent Technologies

Split Liner



Larger Plug of Glass Wool in the Liner



Oven: 35°C for 4 min, 35-320°C at 10°/min, 320°C for 5 min

Carrier Gas: Helium at 9.5 mL/min



Agilent Technologies

GLASS WOOL

Placement in Liner

Near top of liner:

- Wipes syringe needle of sample
- Can improve injector precision
- Helps to prevent backflash

Near bottom of liner:

- Helps in volatilization of high MW components
- Increases mixing






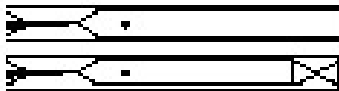
GLASS WOOL

Liner Packing Recommendations

- Amount, size and placement must be consistent for consistent results
- Can be broken upon installation into the liner, exposing active sites
- Liner deactivation with glass wool plug in place is ideal



Splitless Injection Liners

Liner	Part No.	Comments
	5181-3316	Single taper, deactivated, 900 μ L volume. Taper isolates sample from metal seal, reducing breakdown of compounds that are active with metals. For trace samples, general application.
	5062-3587	Single taper, deactivated, with glass wool, 900 μ L volume. Glass wool aides volatilization and protects column. For trace (dirty) samples.
	5181-3315	Double taper, deactivated, 800 μ L volume. Taper on inlet reduces chance for backflash into carrier gas lines. High efficiency liner for trace, active samples.
 Side hole	G1544-80730 G1544-80700	Direct connect liners, single and dual taper, deactivated. Capillary column press fits into liner end, eliminating sample exposure to inlet. Ultimate protection for trace, active samples. Side hole permits use with EPC.



Do liner types really matter?

They do, especially for active compounds like:

- ✉ phenols
- ✉ organic acids
- ✉ pesticides
- ✉ amines
- ✉ drugs of abuse, etc.



Phenols, for example....in a separation of EPA method 8270 compounds



Agilent Technologies

Agilent MS Certified Liners

Consistent quality. FID and MSD tested for Acid/Base deactivation, response linearity, peak symmetry, bleed and background noise

Reproducibility. Tight tolerance in glass dimensions—inner and outer diameters, length, and placement of restrictions and/or glass wool

Assured cleanliness. Manufactured in a clean environment and packaged individually to maintain purity

Lot-tested with FID and MSD for quality assurance

Traceable—with a lot number silk screened on each liner – not just the package

Easy identification of liner type—with a part number on each liner to avoid errors when different liner types look similar

Resistance to endrin/DDT decomposition with **many**, not just a few, injections



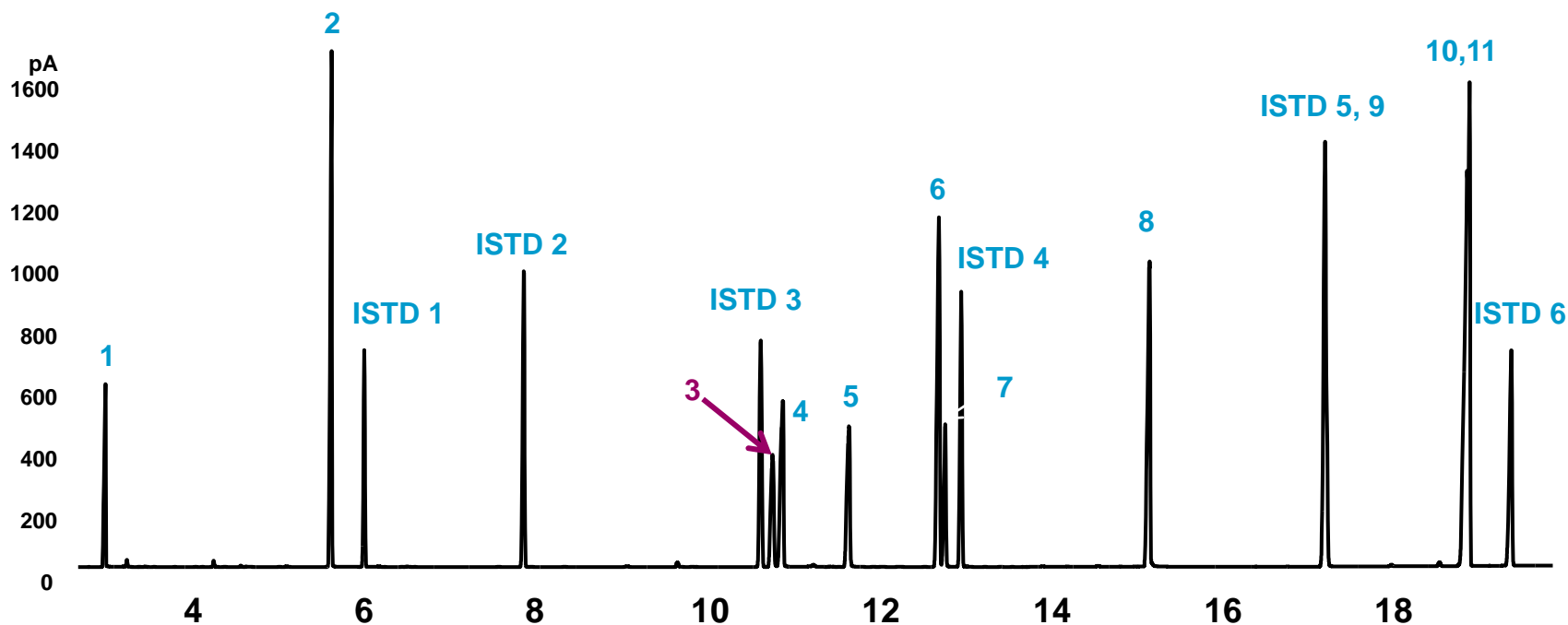
Cool On-Column-FID Injection of 11 Analyte Test Mix

From "Improvements in the
Agilent 6890/5973 GC/MSD
System for Use with USEPA
Method 8270", Agilent Application
Note 5988-3072EN

- 1 N-Nitrosodimethylamine
- 2 Aniline
- 3 2,4-Dinitrophenol
- 4 4-Nitrophenol
- 5 4,6-Dinitro-2-methylphenol
- 6 4-Aminobiphenyl

- 7 Pentachlorophenol
- 8 Benzidine
- 9 3,3-Dichlorobenzidine
- 10 Benzo(b)fluoranthene
- 11 Benzo(k)fluoranthene

- ISTD 1 Dichlorobenzene-d4
- ISTD 2 Naphthalene-d8
- ISTD 3 Acenaphthene-d10
- ISTD 4 Phenanthrene-d10
- ISTD 5 Chrysene-d12
- ISTD 6 Perylene-d12



Agilent Technologies

Splitless Inlet Liners Tested



5062-3587 Single-taper, deactivated, with glass wool

5181-3316 Single-taper, deactivated (open top)

5181-3315 Dual-taper, deactivated (closed top)

G1544-80730 Direct Connect, single-taper, deactivated

G1544-80700 Direct Connect, Dual-taper, deactivated

Vendor X Unknown proprietary deactivation

Hole for EPC



Agilent Technologies

Liner Comparison

2,4-Dinitrophenol Response Factors

160
 80
 20
 5
 ng injected

Experimental:

Agilent 6890 with FID

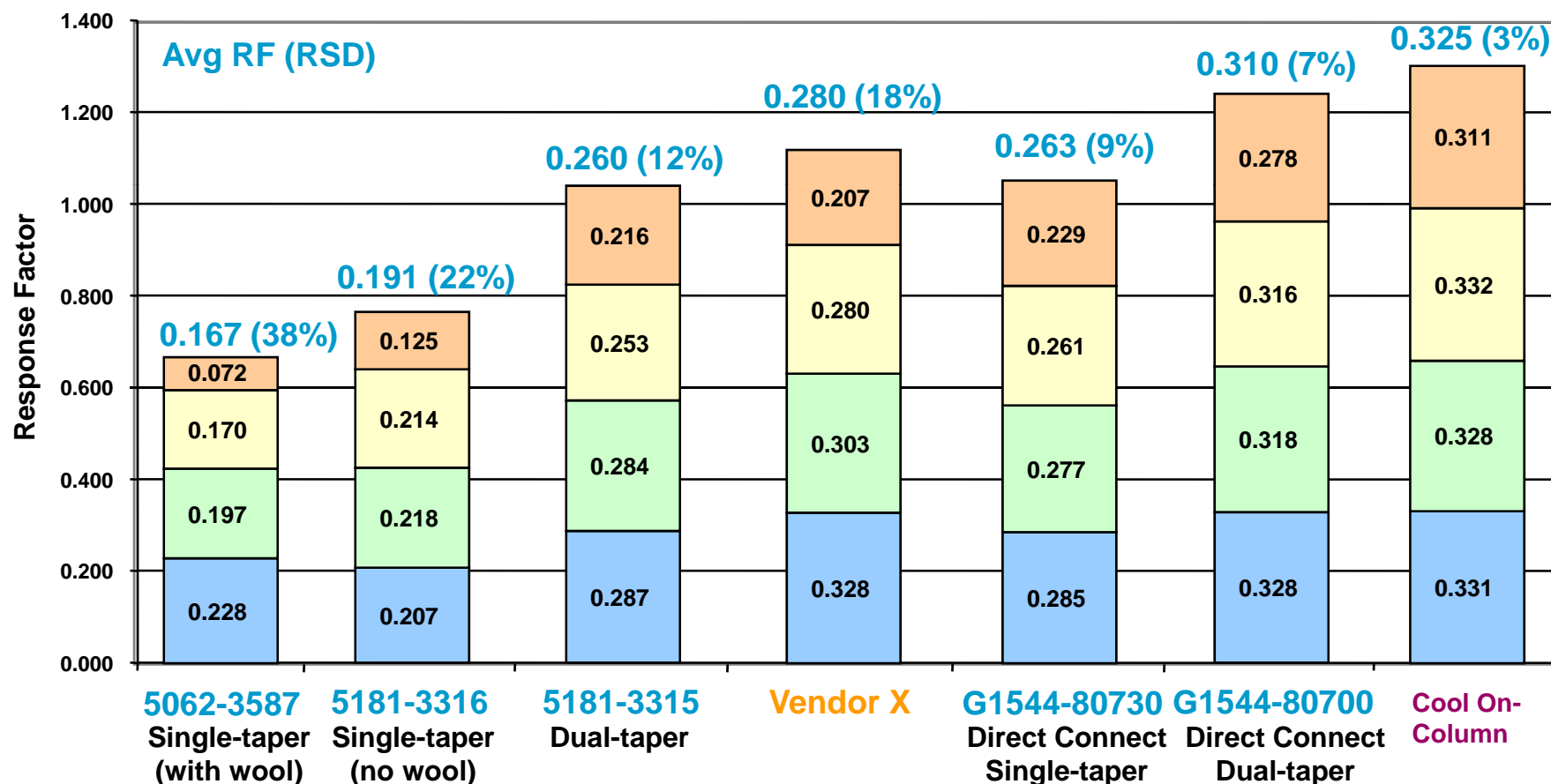
Column = HP-5MS 30m x 0.25mm x 0.5 μ m

Compared COC to various liners

0.75 min Splitless time, 3mL/min column flow

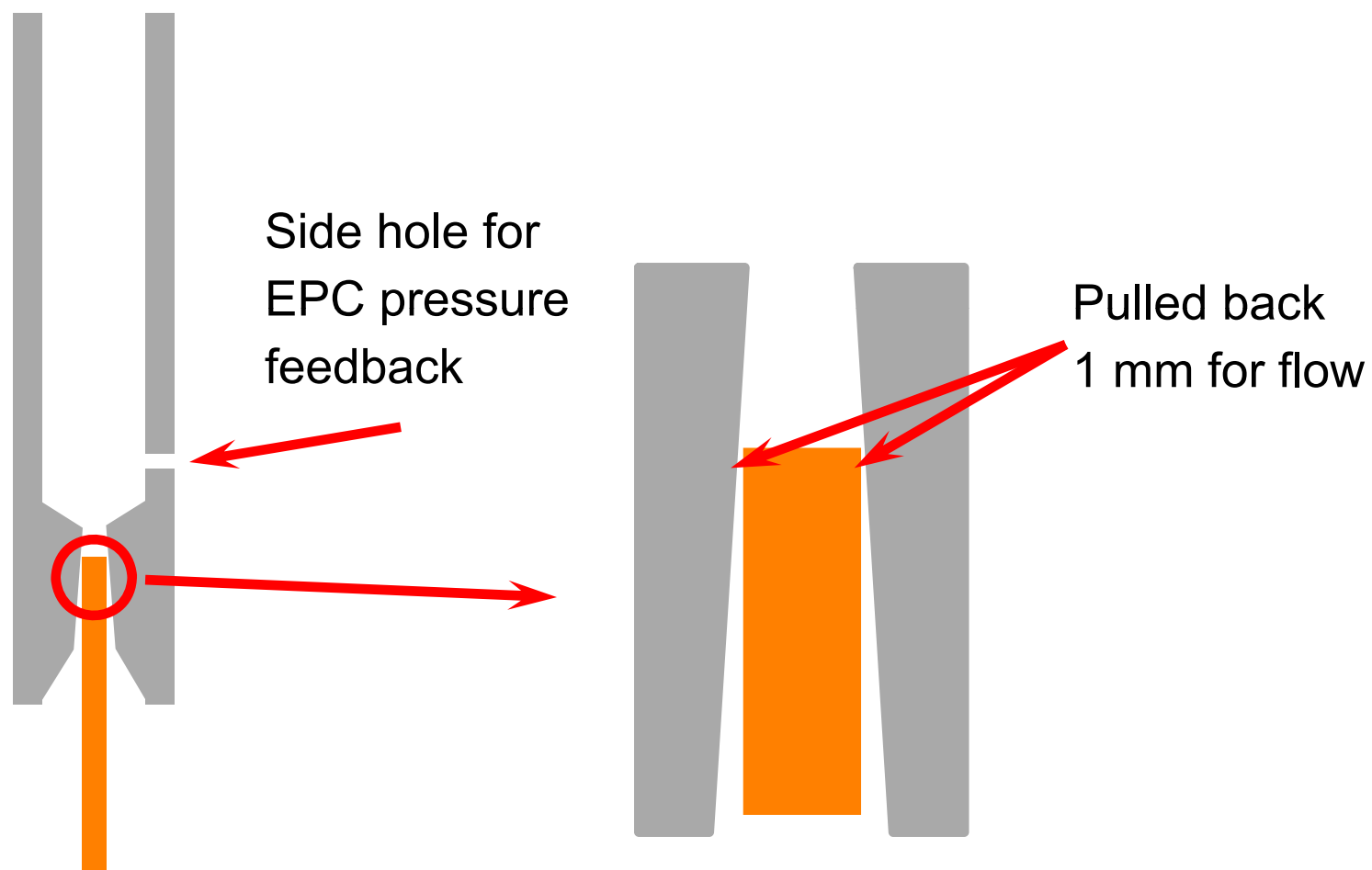
Oven: Temp programmed per 8270 method

Inj. 250°C, Det. 300°C, Sample: 1 μ L 8270 mix



DIRECT CONNECT

Vaporization Liner




Agilent Technologies

Pressure Flow Calculator

<http://www.chem.agilent.com/en-US/Support/Downloads/Utilities/Pages/GcPressureFlow.aspx>

Solvent Vapor Volume Calculator

Approximate vapor volume(ul): **141 ul**  **14 %**

Injection Volume (ul): **1.0**

Inlet Temp (C): **175**

Inlet Pressure: **101**


Pressure Units: ☒ KPa ☐ psi ☐ bar

Print Help OK

Solvent Properties

Hexane


Boiling Pt (C): 68.7
Density (g/cm3): 0.659
Mol Wt. (amu): 86.2

 Solvents

Injection Liner Volume (ul): **19251-60540 strain** 990

Edit Liner list Capacity limits (%) 75 100

Solvent Vapor Volume Calculator

Approximate vapor volume(ul): **1192 ul**  **120%**

Injection Volume (ul): **1.0**

Inlet Temp (C): **250**

Inlet Pressure: **14.6**


Pressure Units: ☐ KPa ☒ psi ☐ bar

Print Help OK

Solvent Properties

Water

Boiling Pt (C): 100
Density (g/cm3): 0.998
Mol Wt. (amu): 18.02

 Solvents

Injection Liner Volume (ul): **19251-60540 strain** 990

Edit Liner list Capacity limits (%) 75 100



Agilent Technologies

For Easy Liner Maintenance on 5890/6890/6850

Flip Top for Split/Splitless injection ports

- 30 sec liner change out
- No more hunting for that “funny looking” wrench!
- Saves fingers from getting burned
- Increases instrument up time



For a limited time only – Send an email to abby_folk@agilent.com before May 1st. and get your Flip Top for \$180 (a 64% discount)



Agilent Technologies

Turn Top Inlet Sealing System on NEW 7890



- Fast/Easy Split/Splitless Inlet Maintenance

... changing liners has never been easier



Agilent Technologies

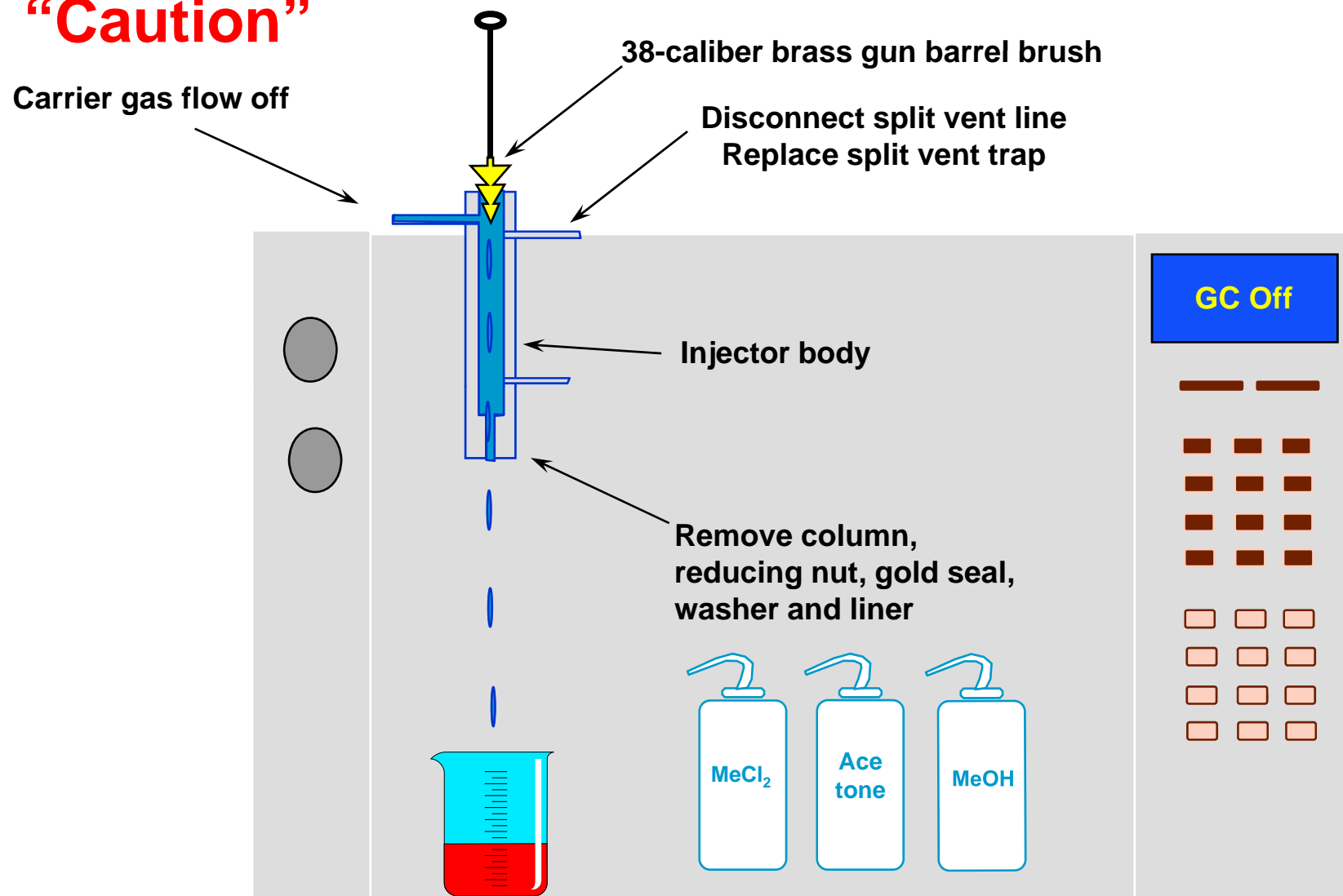
Common Care and Maintenance Scheme for GC Columns

1. Bake out the column for no more than 2 hours.
2. Cut off 6"-1ft of the inlet end of the column.
3. Cut off more column. (repeat as necessary)



Cleaning the Split/Splitless Injector

“Caution”



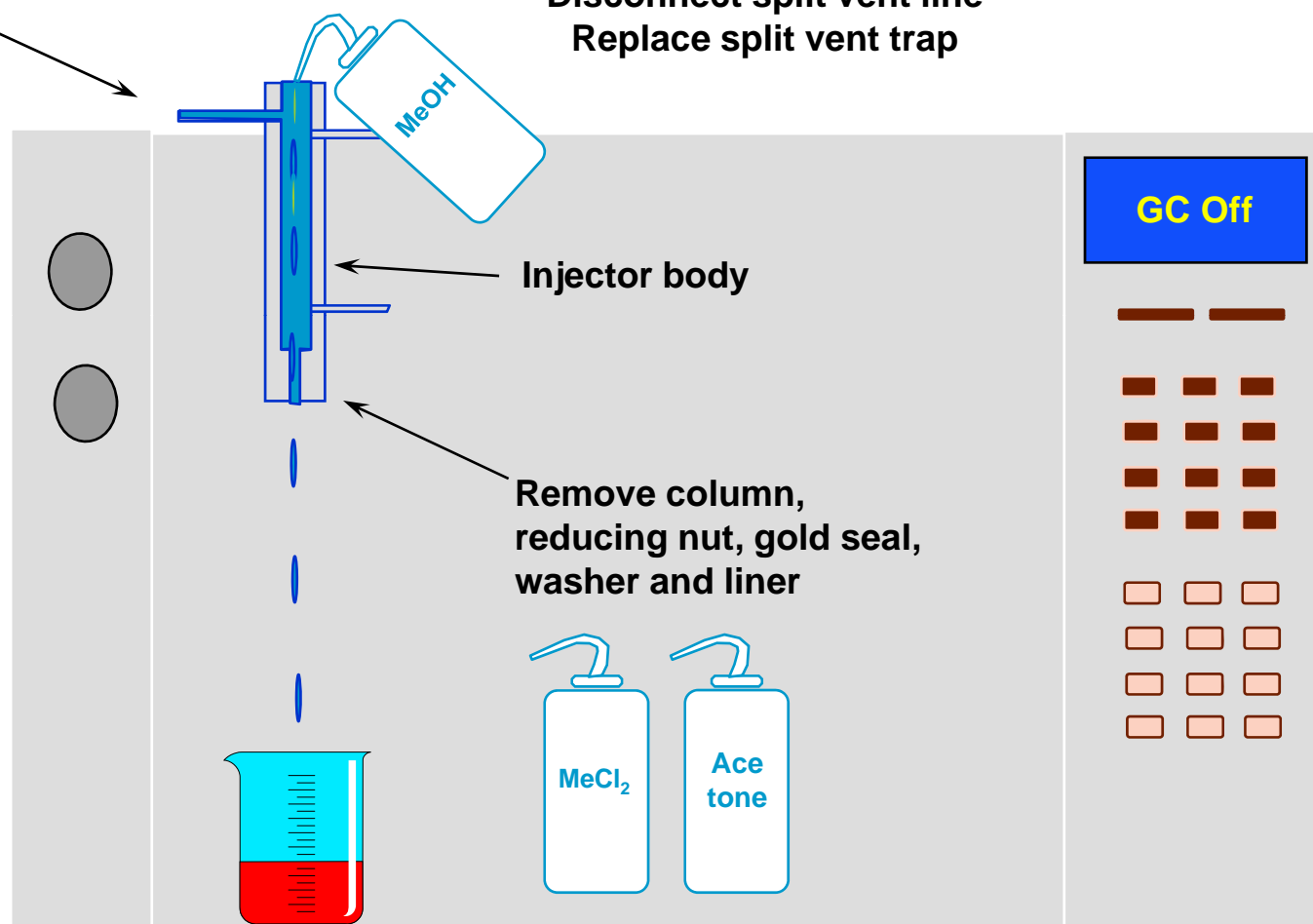
Agilent Technologies

Cleaning the Split/Splitless Injector

“Caution”

Carrier gas flow off

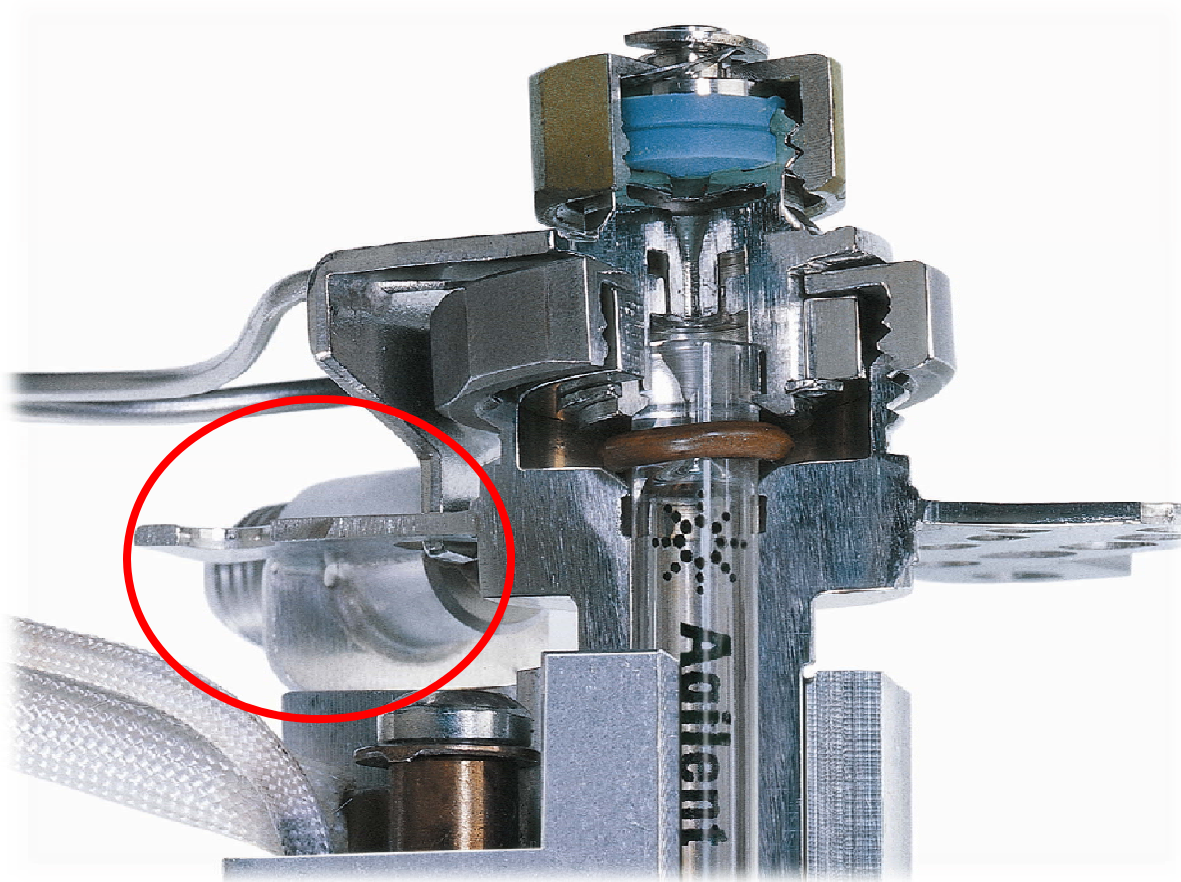
Disconnect split vent line
Replace split vent trap



Agilent Technologies

Finding the Split Vent Trap

Follow the split vent line back to the EPC



Agilent Technologies

Finding the Split Vent Trap

Remove cover at Split Vent



Agilent Technologies

Replacing the Split Vent Trap

Finger Tight Knurled Nut



G1544-80530



5188-6495



Agilent Technologies

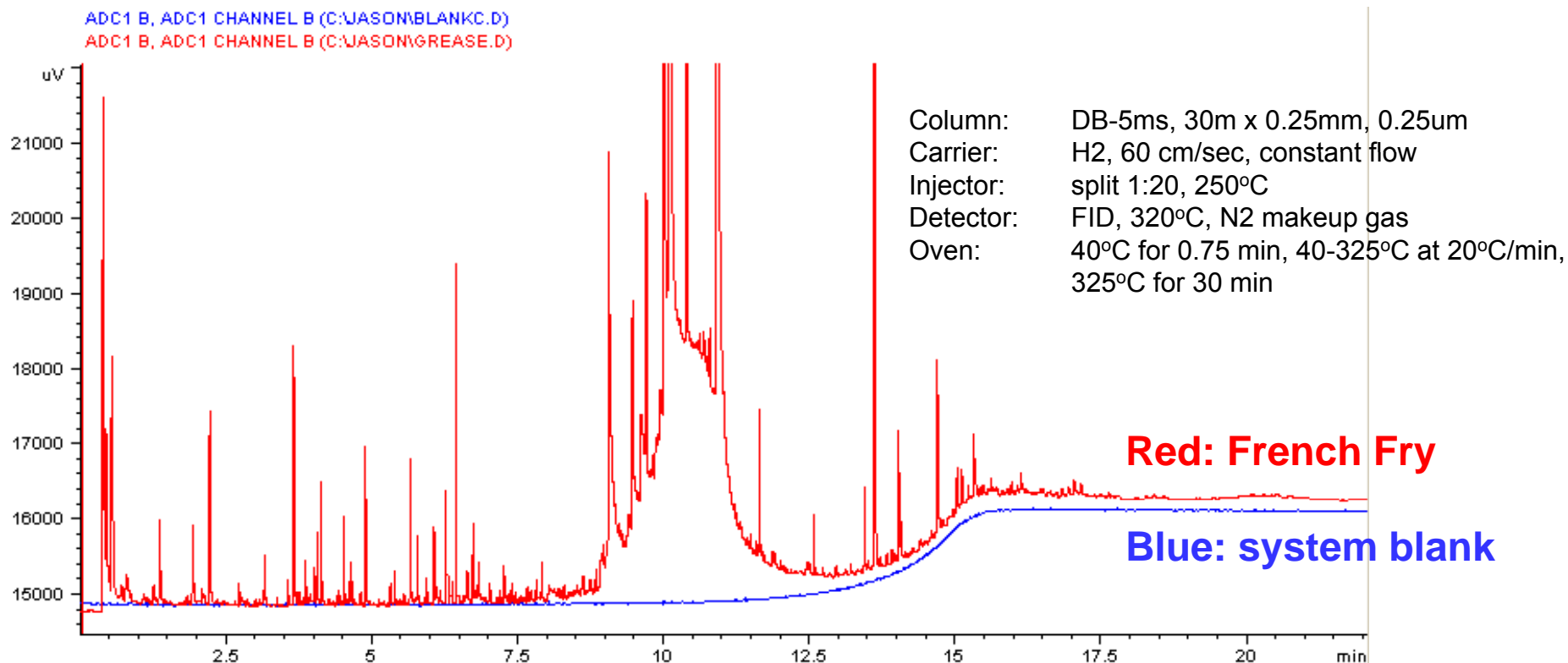
Be Careful When Doing Maintenance...

You may be the CONTAMINATOR!



Agilent Technologies

Contamination of system by residue on fingers during column installation



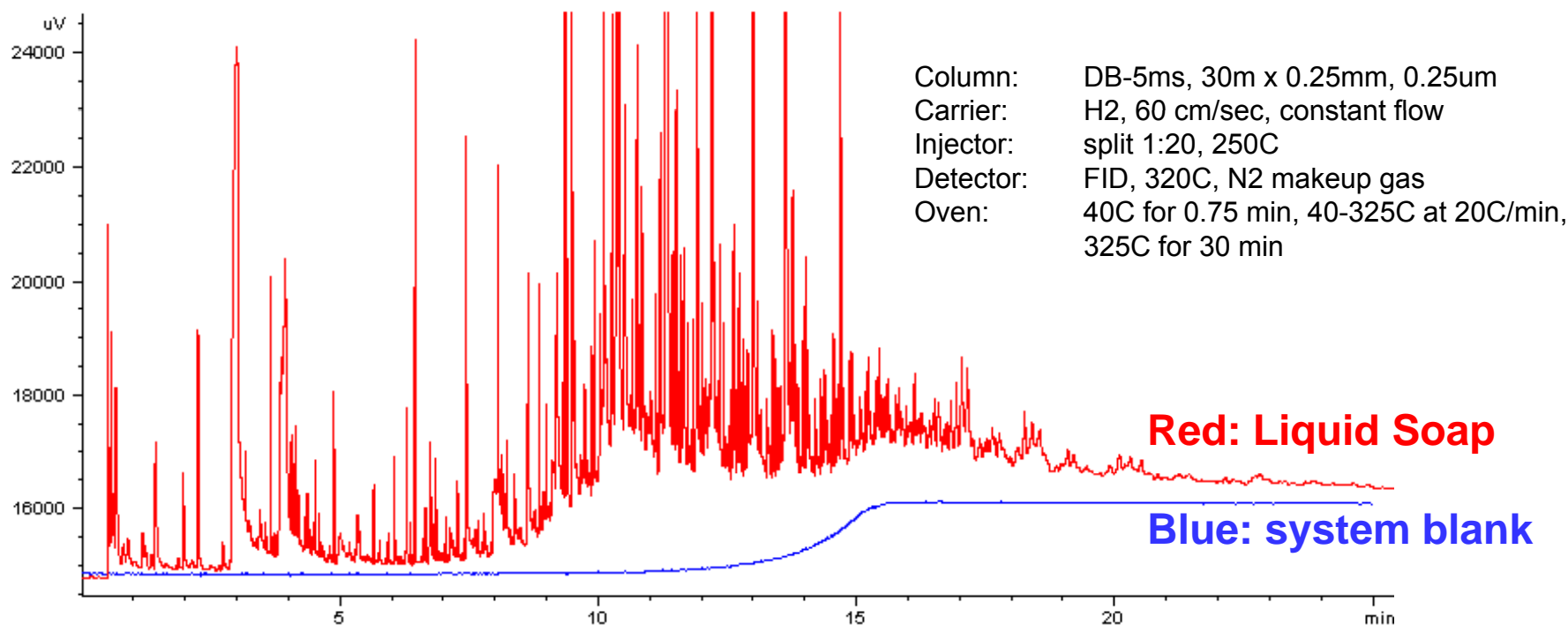
Procedure:

- (1) Held French fry for 5 seconds.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40°C.
- (6) Started oven temperature program as soon as oven reached 40°C.



Agilent Technologies

Contamination from Liquid Soap



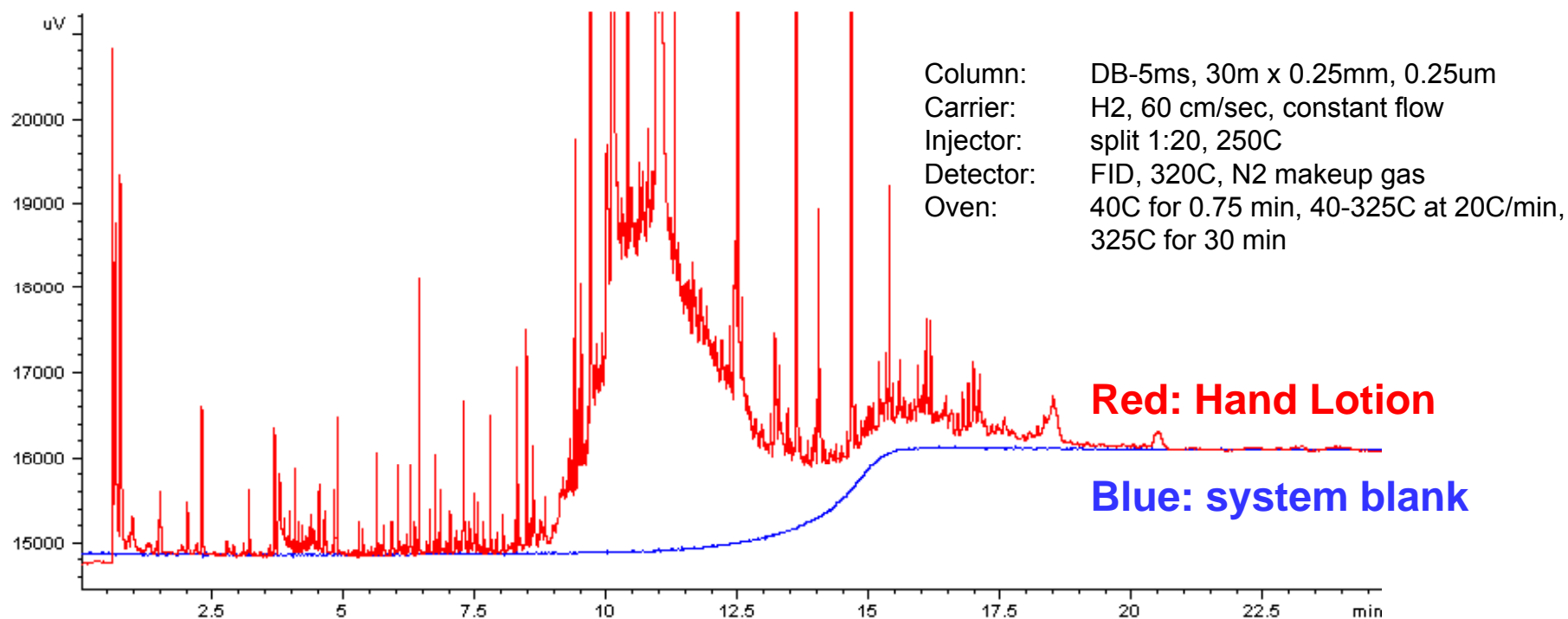
Procedure:

- (1) One very small drop of liquid soap placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.



Agilent Technologies

Contamination from Hand Lotion



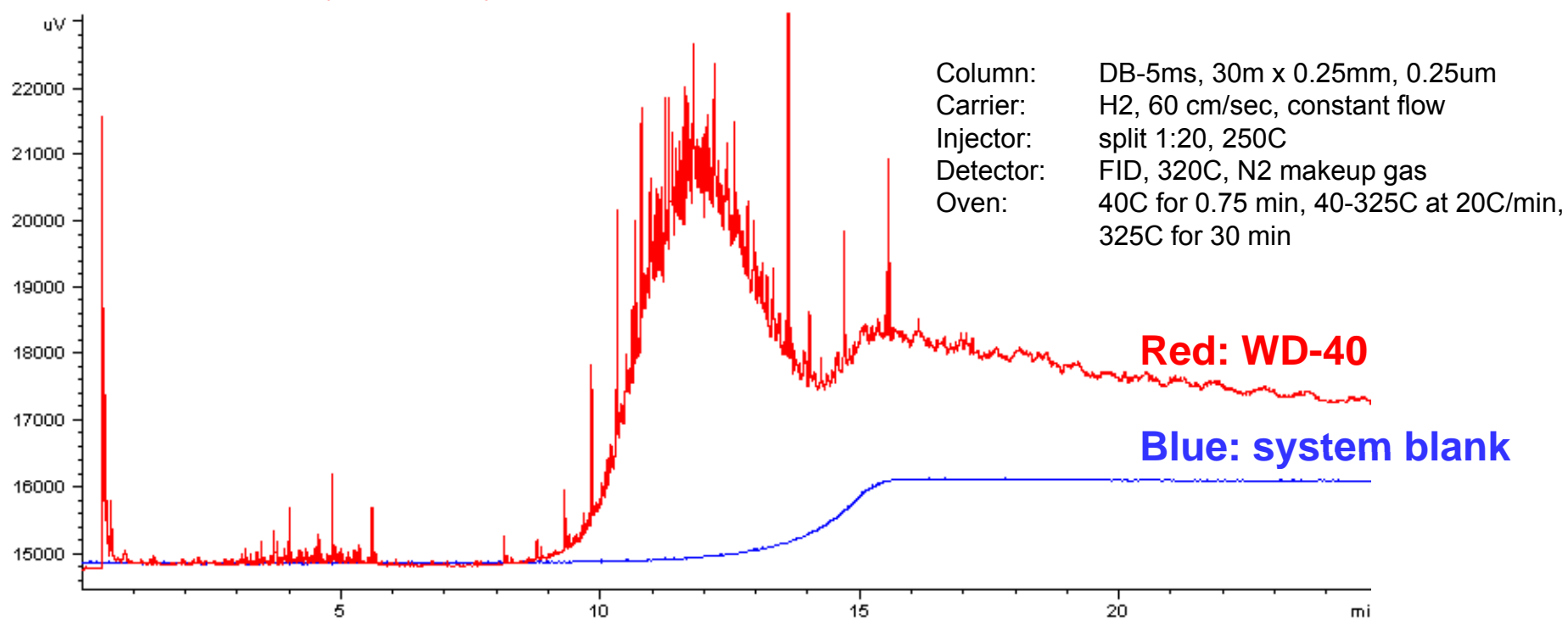
Procedure:

- (1) One very small drop of hand lotion placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.



Agilent Technologies

Contamination from Lubricant



Procedure:

- (1) One very small drop of WD-40 liquid placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.



Agilent Technologies

Root Causes of Inlet Performance Degradation, and Consequences

Accumulation of Sample Residues

- Loss of response, tailing on active analytes, split vent trap fouling and inaccurate EPC flow control

Accumulation of consumables wear particles

- Same as above, plus “bleed peaks”

Leak in Septum Nut, Septum

- Damage to O₂ sensitive detectors, irreversible damage to column

Non-Optimized Set-up

- O-ring, Gold Seal, Ferrules, Column Nuts
- Faster inlet performance degradation between maintenance sessions



Real-Time Monitoring



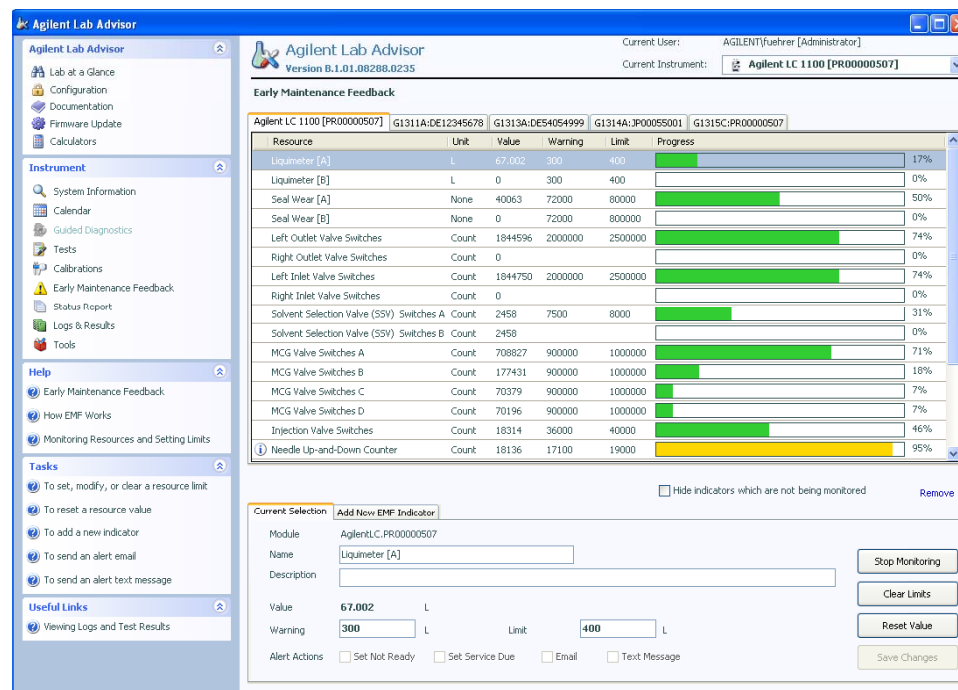
Maximize instrument performance with Maintenance and EMF indicators

Instant visual indication

- Resource counters
- Progress bars
- EMF indicators
- Real-time indicators and alerts of preventive maintenance needs
- Alerts possibilities via mail or text message when limits are reached

Notifies users when it's time to change out consumables or perform basic upkeep tasks

To be sure getting the maximum performance from all instruments



Agilent J&W Scientific Technical Support

- 800-227-9770 (phone: US & Canada)*
 - *Select option 3, then 3, then 1.*
- www.agilent.com/chem
- gc-column-support@agilent.com

