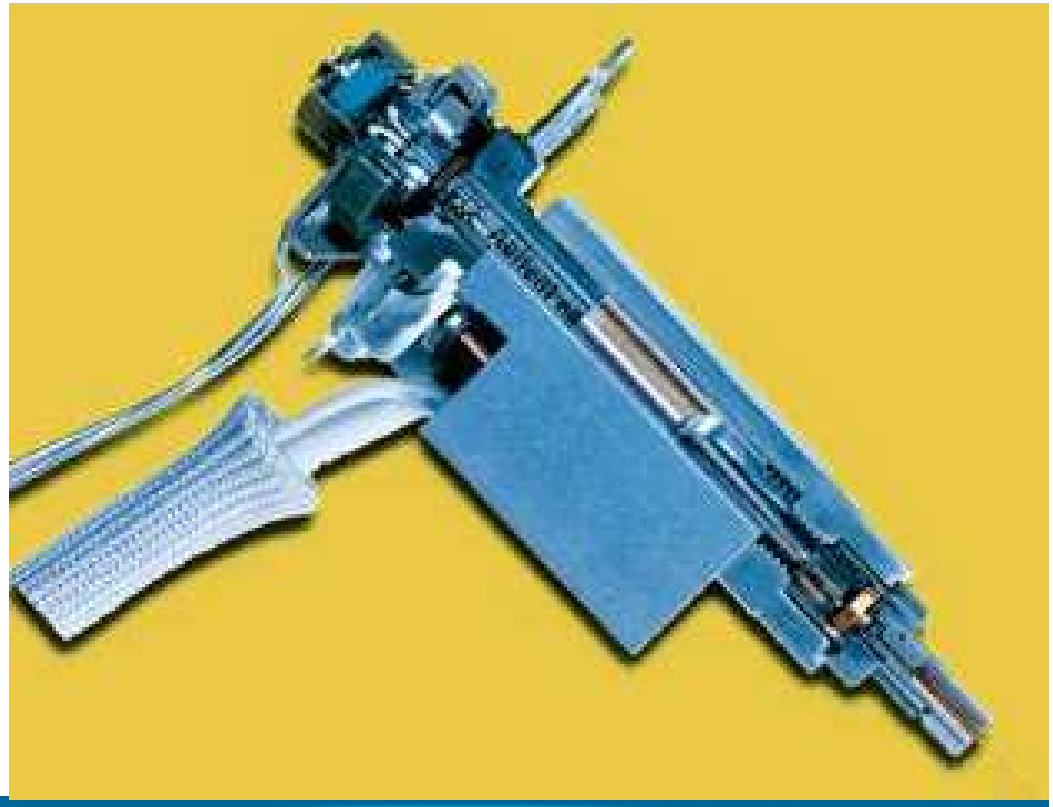
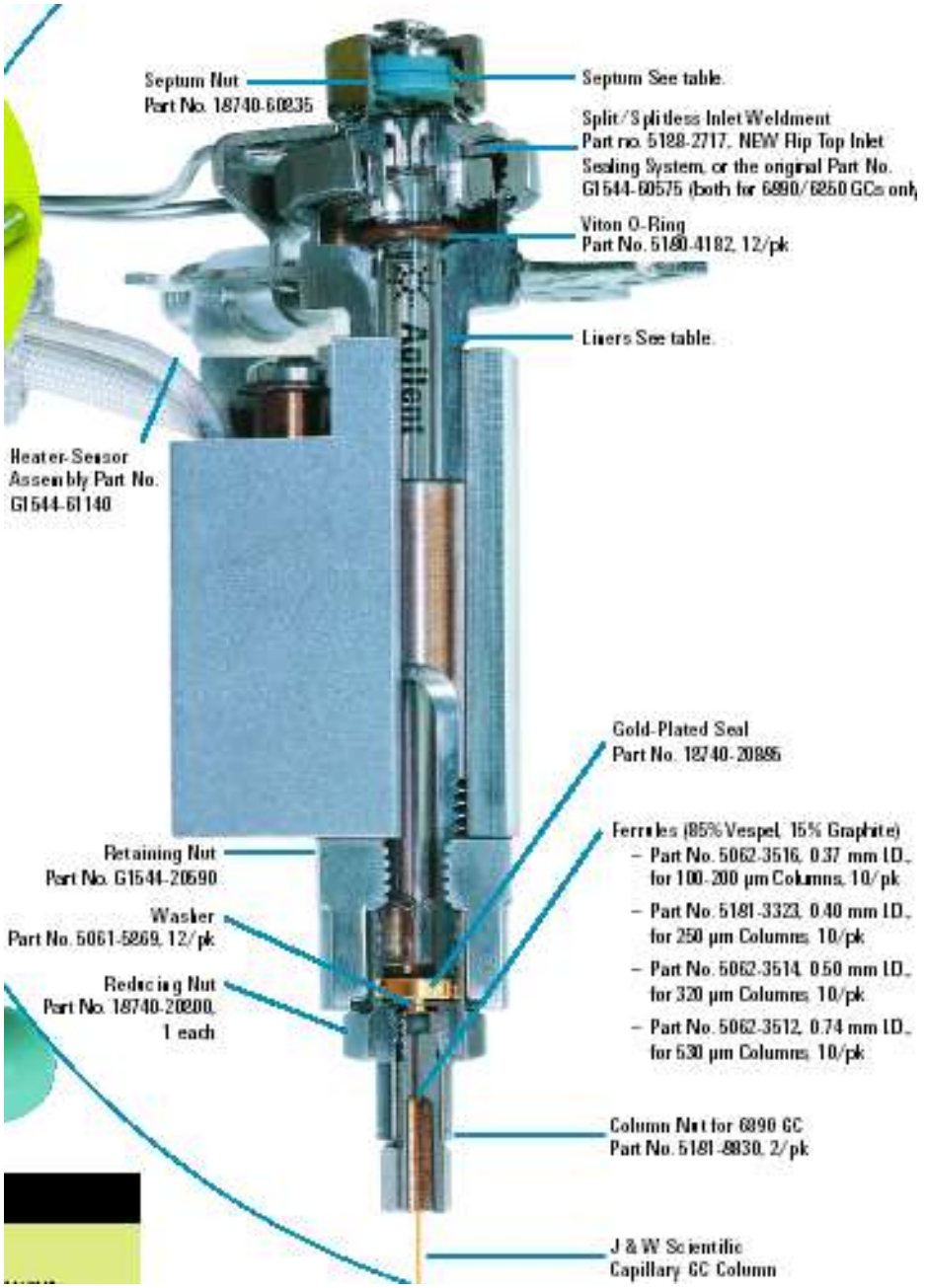
Maintaining/Optimizing Your Agilent GC's Flow Path



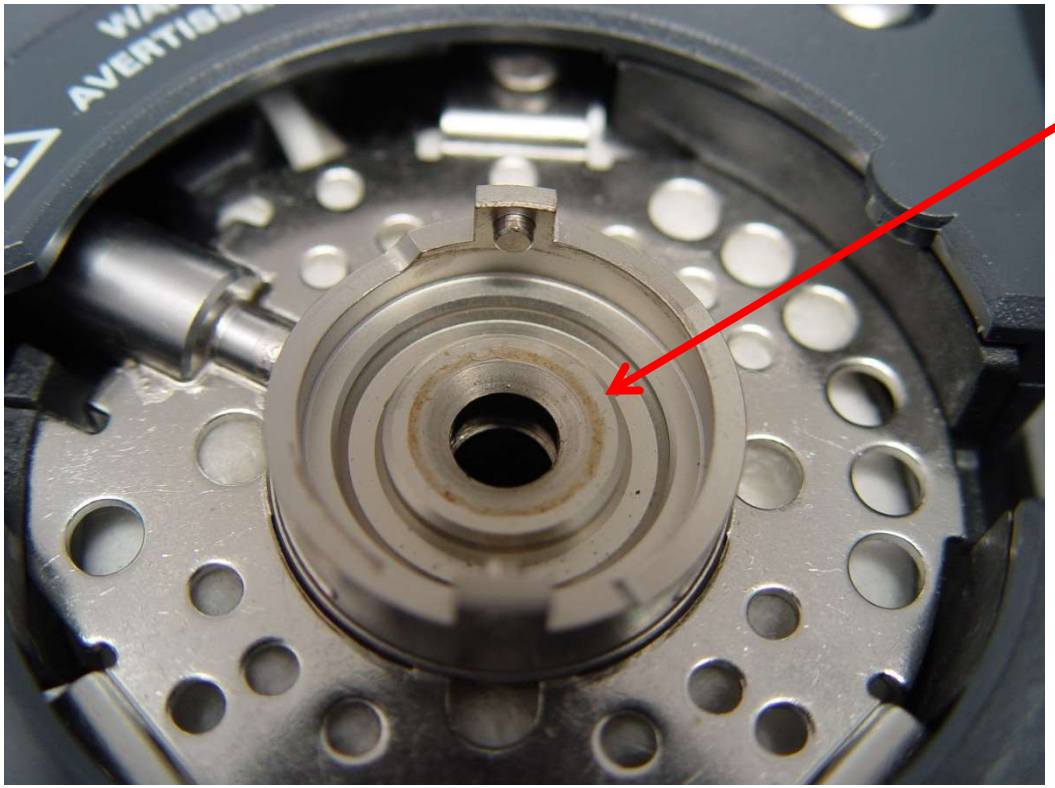
Agilent Technologies

Split/Splitless Injector Parts



More “Off-Brand” O-Ring Issues

Controlled Substances Analysis, H2 Carrier

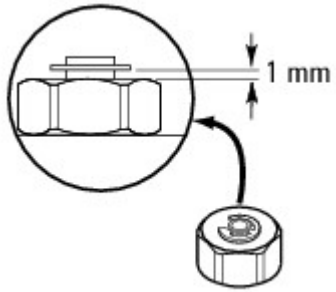


- Residue on top of
- inlet weldment

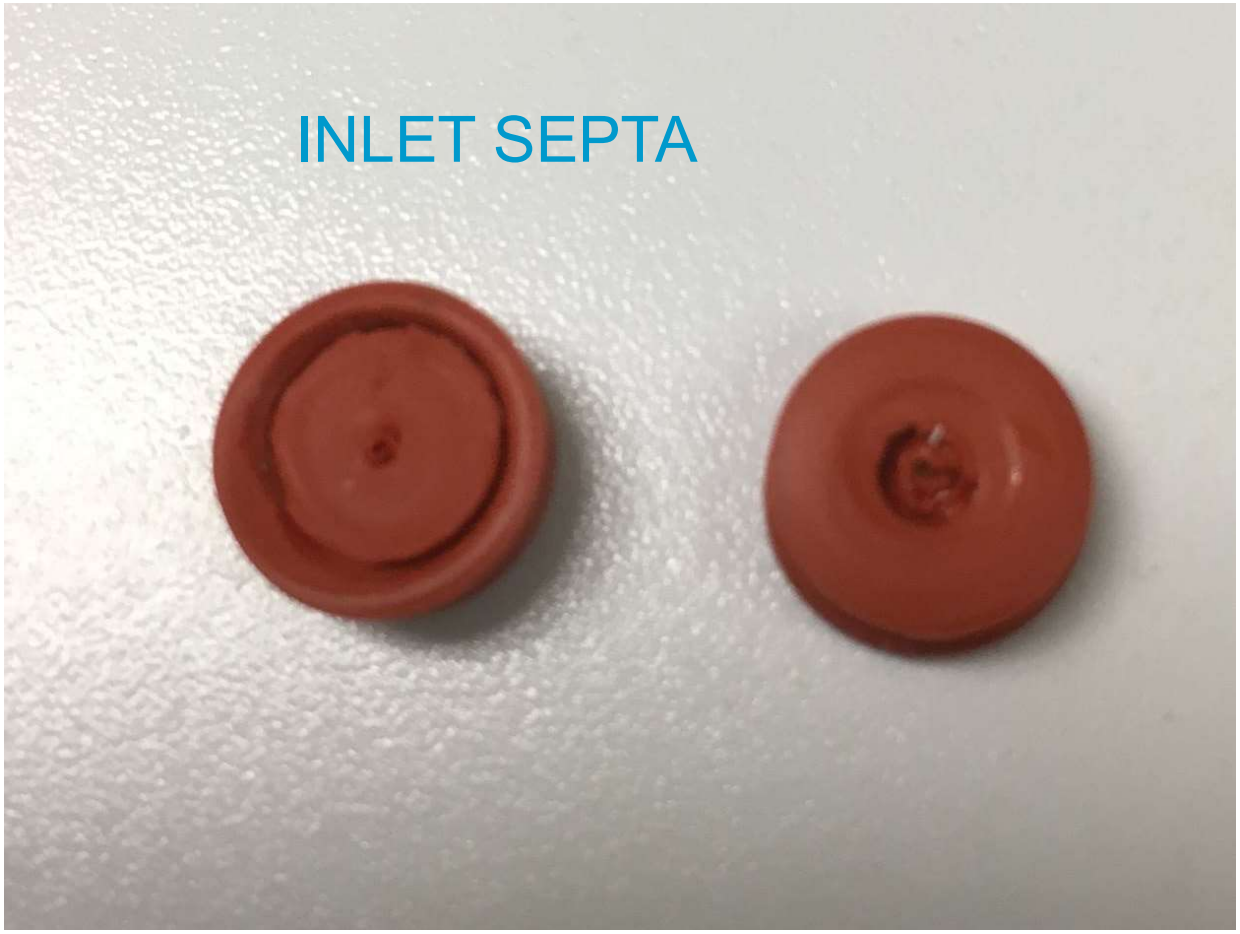
Problem Resolution:
Agilent Non-Stick Liner O-Ring
p/n 5188-5365, 10PK

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 ½ to ¾ 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.

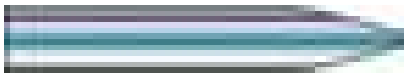


Result of Over-tightened Septa Nuts



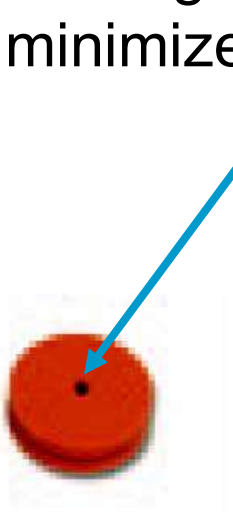
Tips to Maximize Septum Life, Minimize Septum Leaks

– 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.



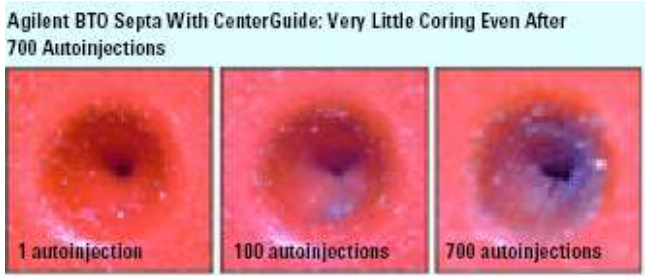
HP-Point Style

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



Solid Septum

CenterGuide Septum



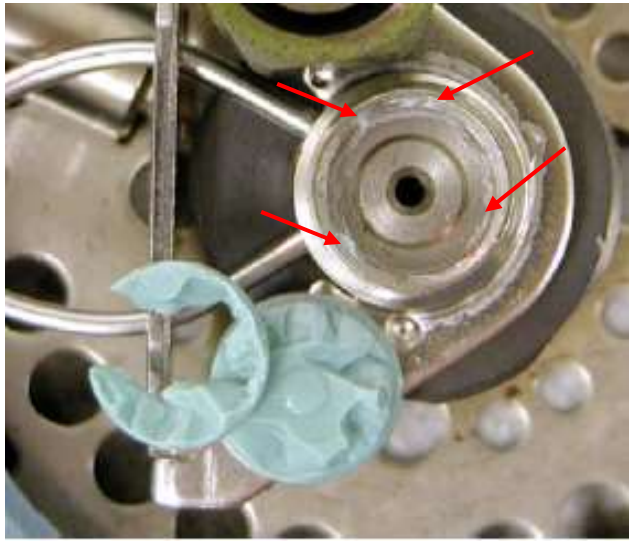
Turn Top Inlet Sealing System on 7890

- Fast/Easy Split/Splitless Inlet Maintenance

… changing liners has never been easier

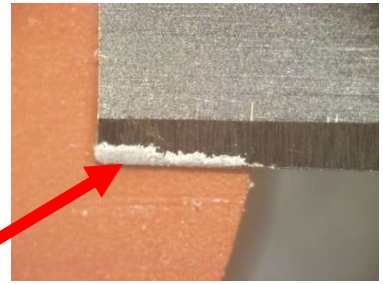
Tips to Maximize Septum Life, Minimize Septum Leaks

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



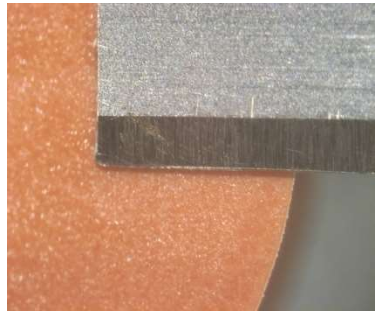
Their's

Talcum Powder!



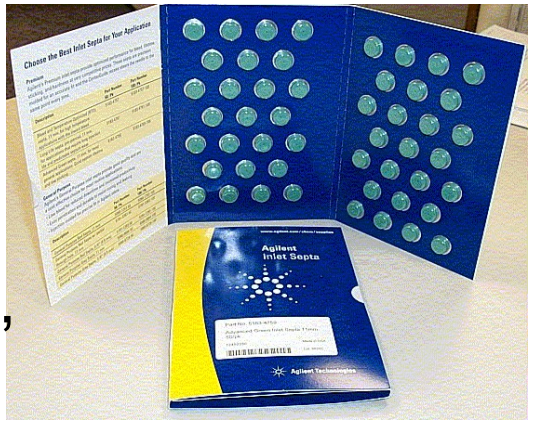
NEW!

Our's



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

Other Benefits of Agilent's "Centerguide" Septa



- 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

What if you …

- ✓ Could install the column easily
- ✓ Finger tighten, without tools that cause over tightening
- ✓ And make leak free connections
- ✓ Which don’t need to be retightened
- ✓ Every time?

Self-Tightening Column Nuts

- **Reliable performance:** Innovative spring-driven piston continuously presses against ferrule – maintaining a leak-free seal

- **Less wasted time:** No retightening needed after repeated thermal cycles

- **Ease of use:** Finger-tight, consistent connections *without tools*

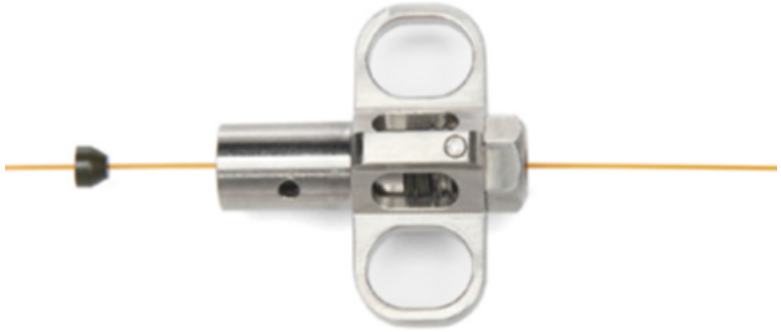
- **Faster maintenance:** Low-torque seal prevents sticking or crumbling during removal

- **Leak Free = Lower column bleed:** Longer column life

Video at **agilent.com/chem/STnutvideo**

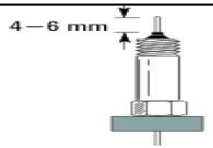
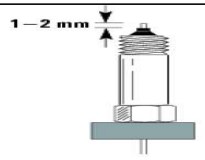
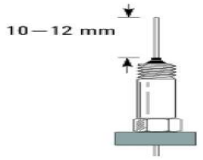
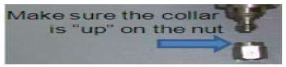
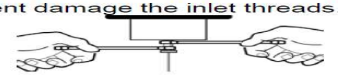
Self Tightening Column Nuts

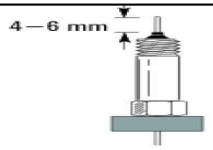
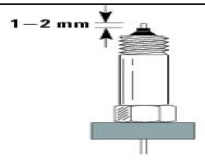
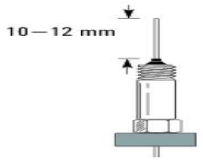
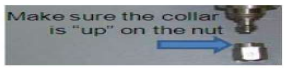
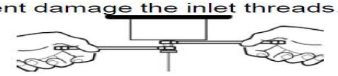
For inlet or detector
p/n 5190-6194



For mass spec transfer line
p/n 5190-5233

Installation Distance Matters

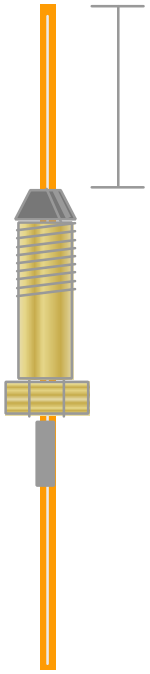
Inlet	Diagram	Procedure
Split/Splitless		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 4-6 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Purged Packed		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 1-2 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Multimode		**NOTE:** Make sure the column adapter nut on the inlet base is ***fully threaded on*** and ***spinning freely – Collar Up!***  **Tighten with two wrenches** - ¼" and 5/16" To prevent damage the inlet threads. 

Inlet	Diagram	Procedure
Split/Splitless		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 4-6 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Purged Packed		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 1-2 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Multimode		**NOTE:** Make sure the column adapter nut on the inlet base is ***fully threaded on*** and ***spinning freely – Collar Up!***  **Tighten with two wrenches** - ¼" and 5/16" To prevent damage the inlet threads. 

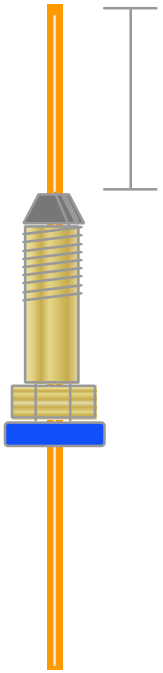
Column Installation

Measuring the right distance

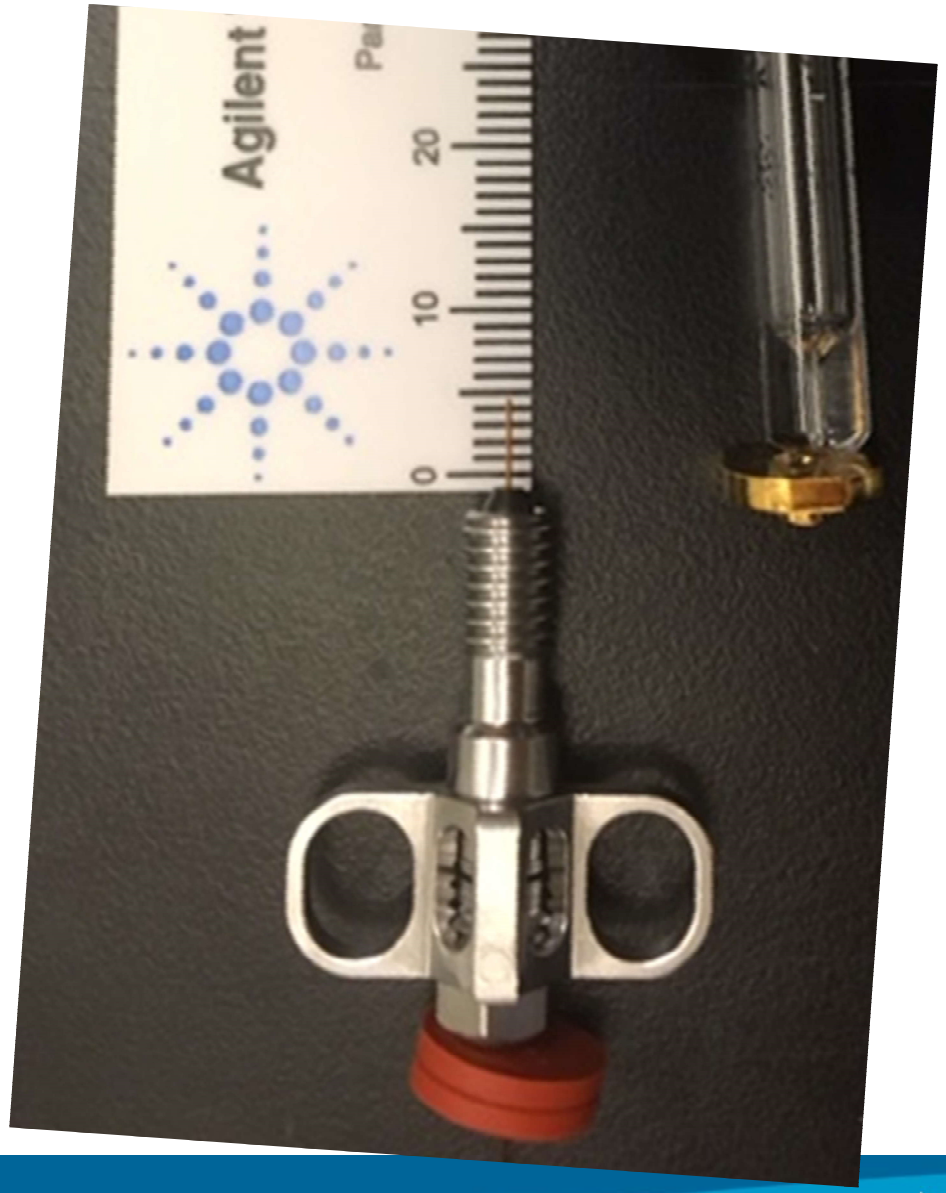
White out



Septa

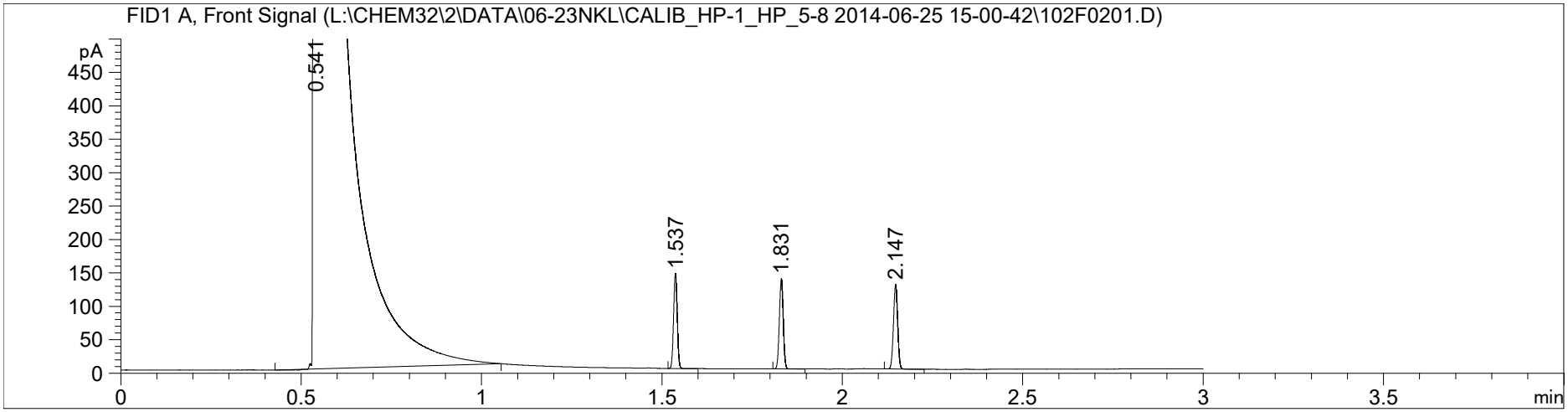


Why Does Distance Matter?

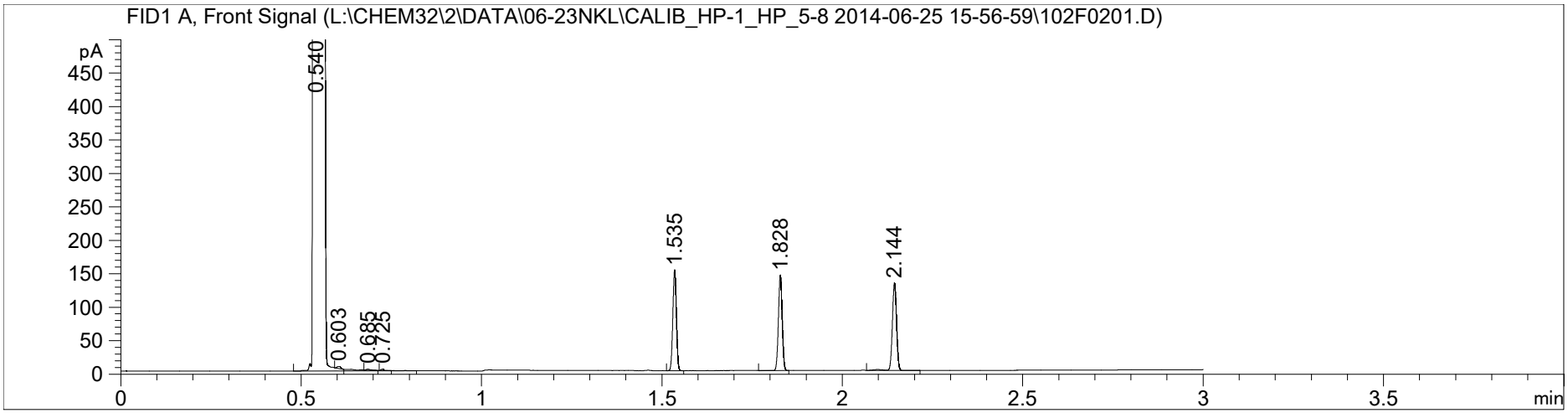


Tailing – Poor Installation

MMI Inlet 8 mm Depth of column

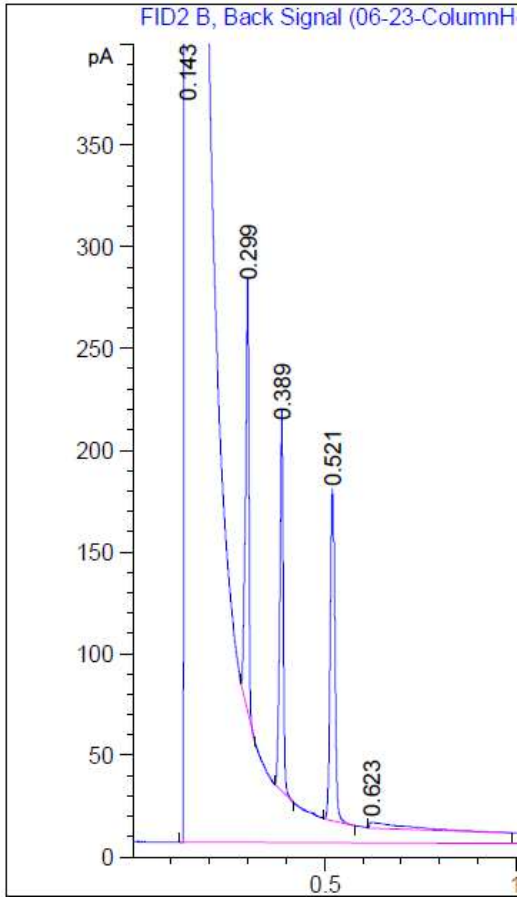


MMI Inlet 9 mm Depth of column

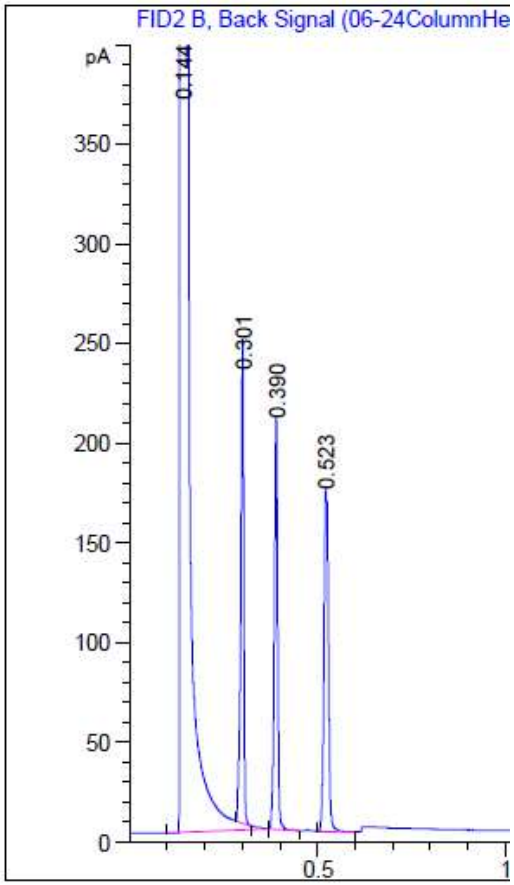


Importance of Measuring is Sample Dependant too!

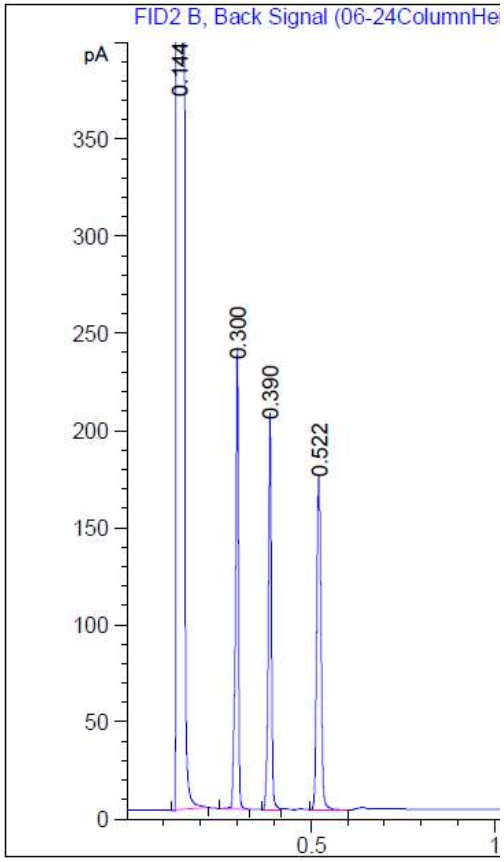
10 mm



10.5 mm



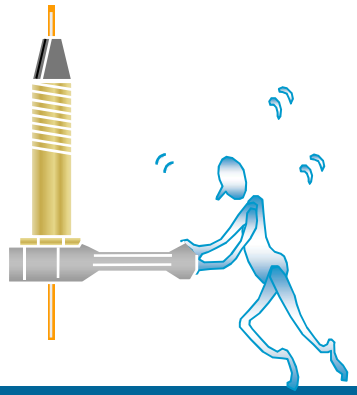
11 mm



Replace Because…Most Common Causes of Leaks

Re-use and mis-installation.

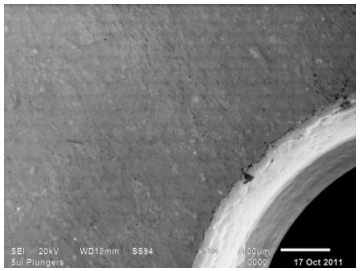
- Leak from O-ring, Gold Seal, ferrules, column nuts
- O-rings are elastomer compression fittings designed for one use, not perfectly elastic.
- Gold seals are designed for one use, knife edge cuts into gold layer giving leak tight seal w/o shrinkage or potential organic contaminants from polyimide out-gassing/degradation.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



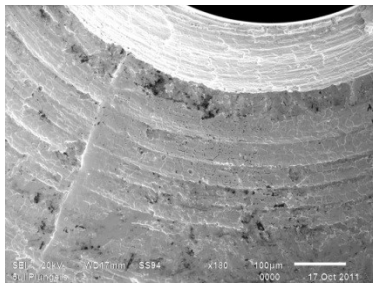
Certified gold inlet seal, 5188-5367

Agilent UI Gold Seal: Deactivated Gold Surface

- Soft gold plating is essential for proper sealing
- Ultra Inert chemistry blocks active sites (gold is NOT inert)
- Smooth surface doesn't leak



Agilent MIM seal



Competitor's machined seal

Reliable ppb and ppt measurements require attention to the little things!

Ferrule Pre-swaging & MS Interface Installation Tools

- Ensures proper length of column into the fittings, every time
- For graphite or metal ferrules

Metal ferrule tool
G3440-80218

Graphite ferrule tool
G3440-80217

Pre-swaging Tool for CFT Devices

Graphite Ferrules Have a Down Side Too!

Pros
- High temperature range (450C)
- Low Cost
- Soft, easily conforms

Con
- Can flake, fall apart or extrude
- Permeable

NOT recommended with MS or ECD

Column Installation Procedure

- Install the column
- Leak and installation check
- Column conditioning
- Bleed profile
- Test mix

Cutting The Column

Gently scribe through the polyimide coating.
Do not attempt to cut the glass.

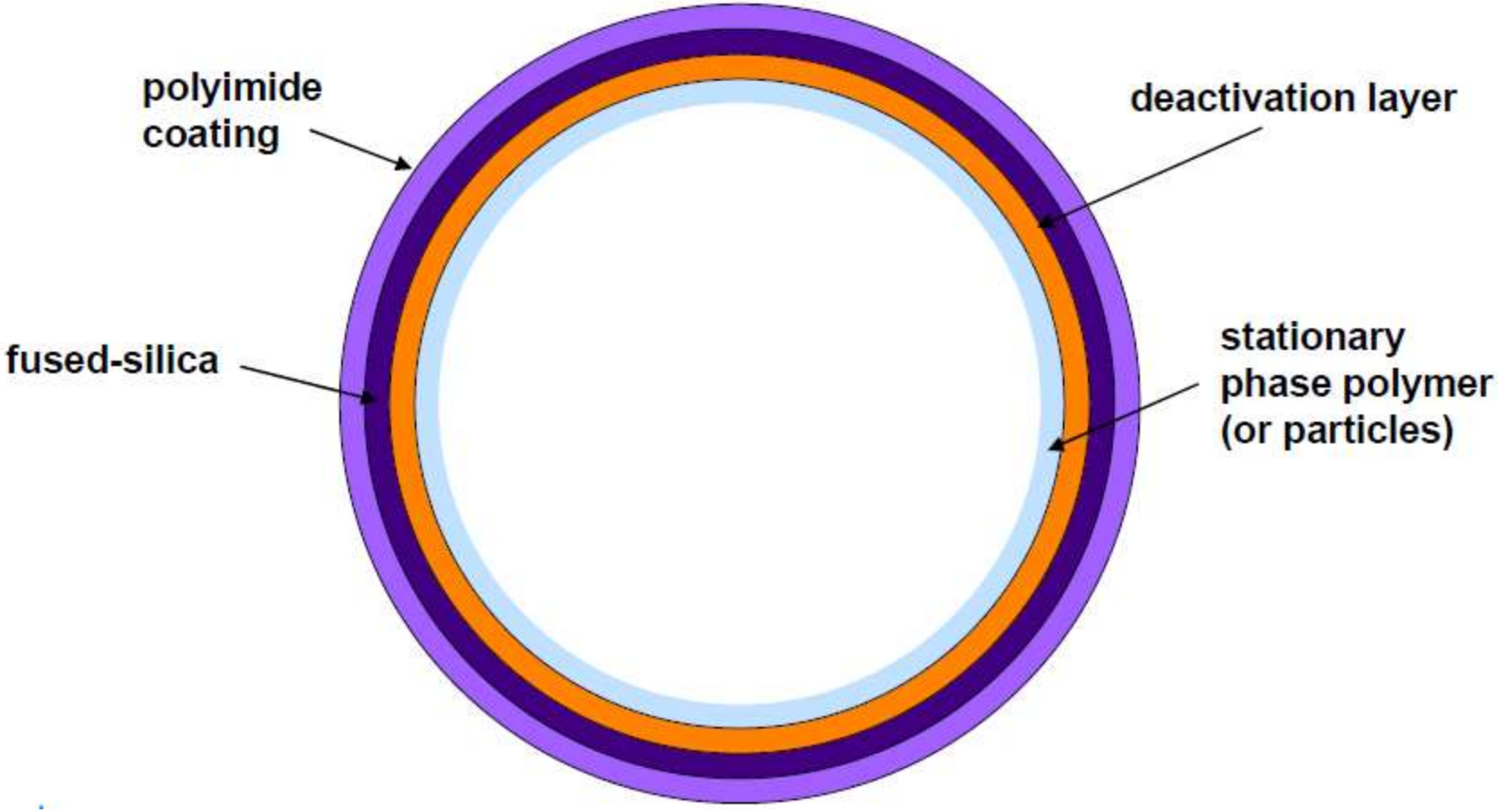
Recommended tools:

Diamond or carbide tipped pencil; or sapphire cleaving tool, ceramic wafer
Ocular

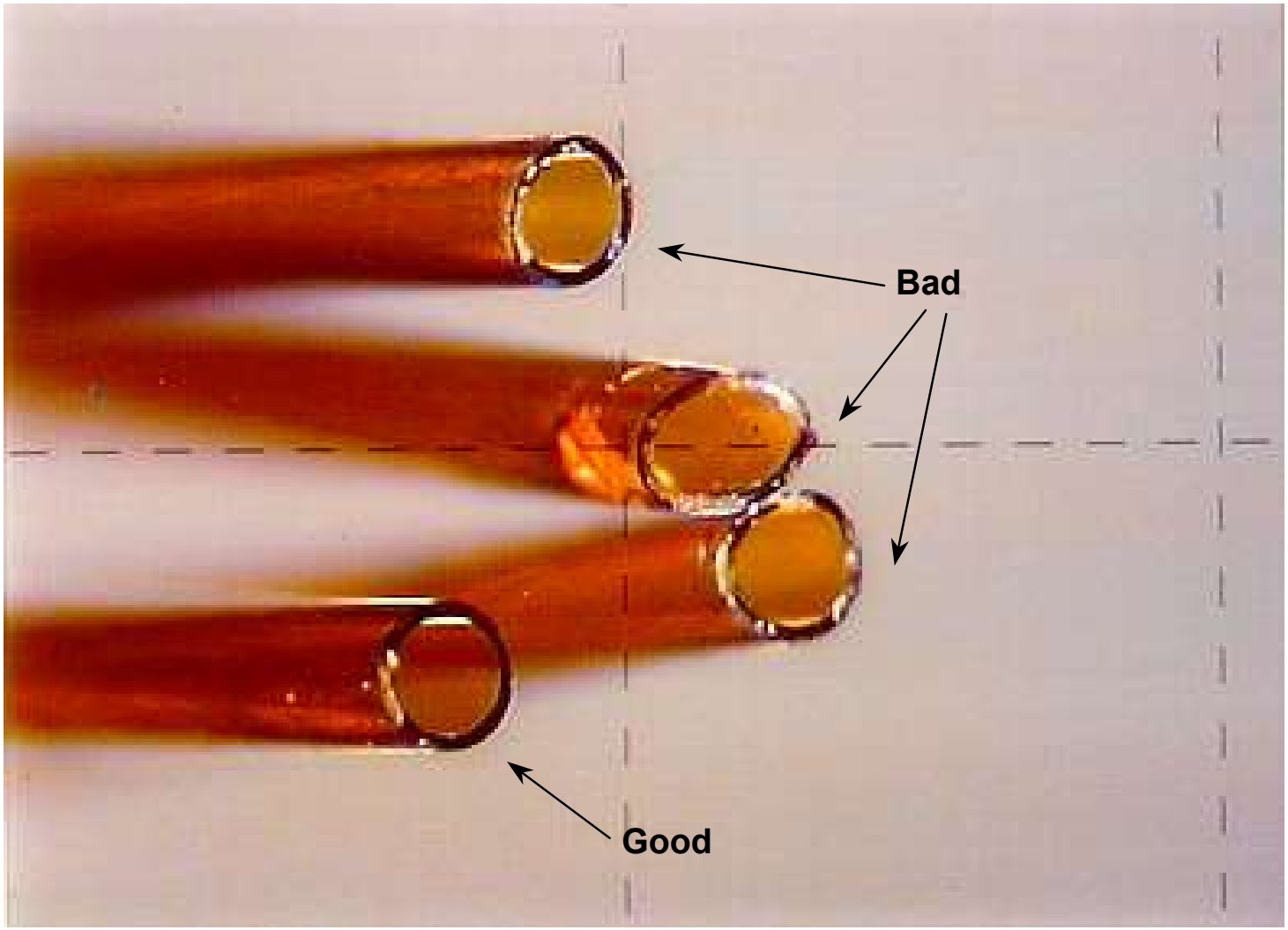
Do not use:

Scissors, file, etc.

Capillary Column Cross Section

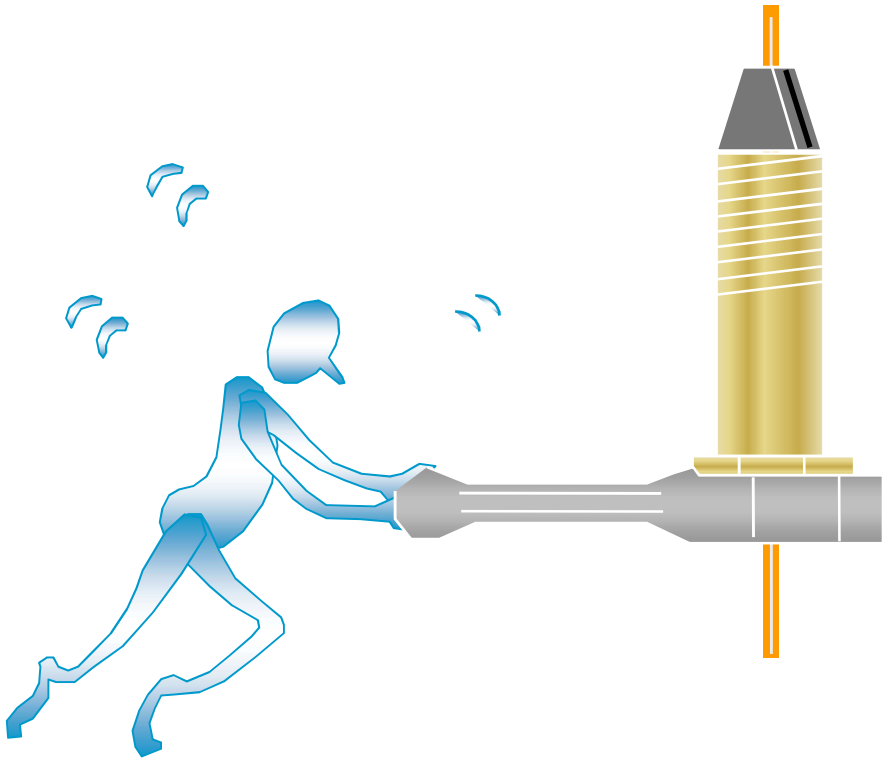


Examples of Column Cuts

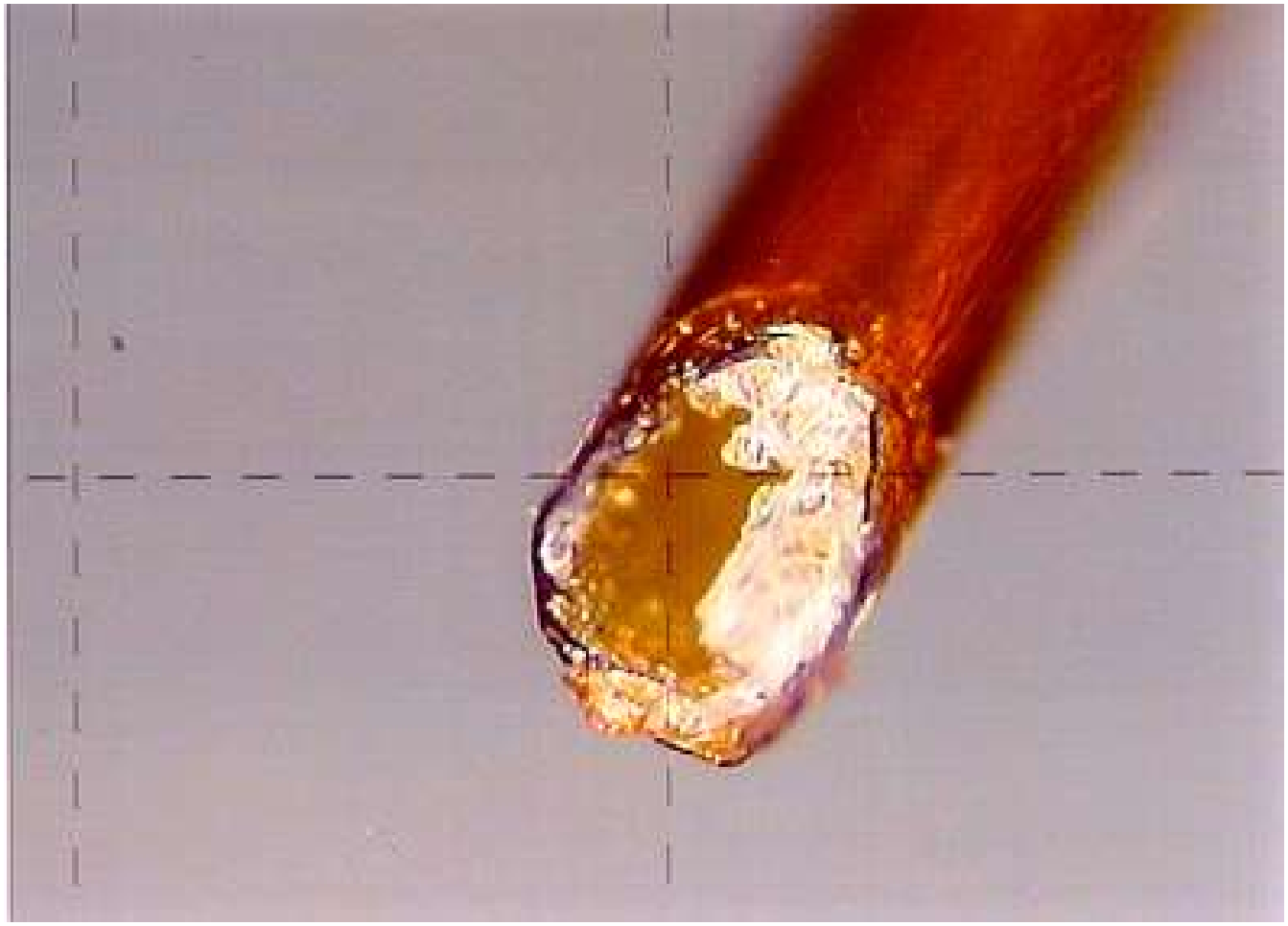


Column Installation

How tight is tight?



Overtightened Ferrule



Column Installation
Leak Check

DO NOT USE SNOOP

Electronic leak detector

IPA/Water (50:50)

Inject a non-retained peak

Column Conditioning

System must be leak free before conditioning column

Heat the column to the lower of:

- **Isothermal maximum temperature OR**
- **20° to 30°C above highest operation temperature**
- **Temperature programming is not necessary**

Stop conditioning when the stable baseline is obtained:

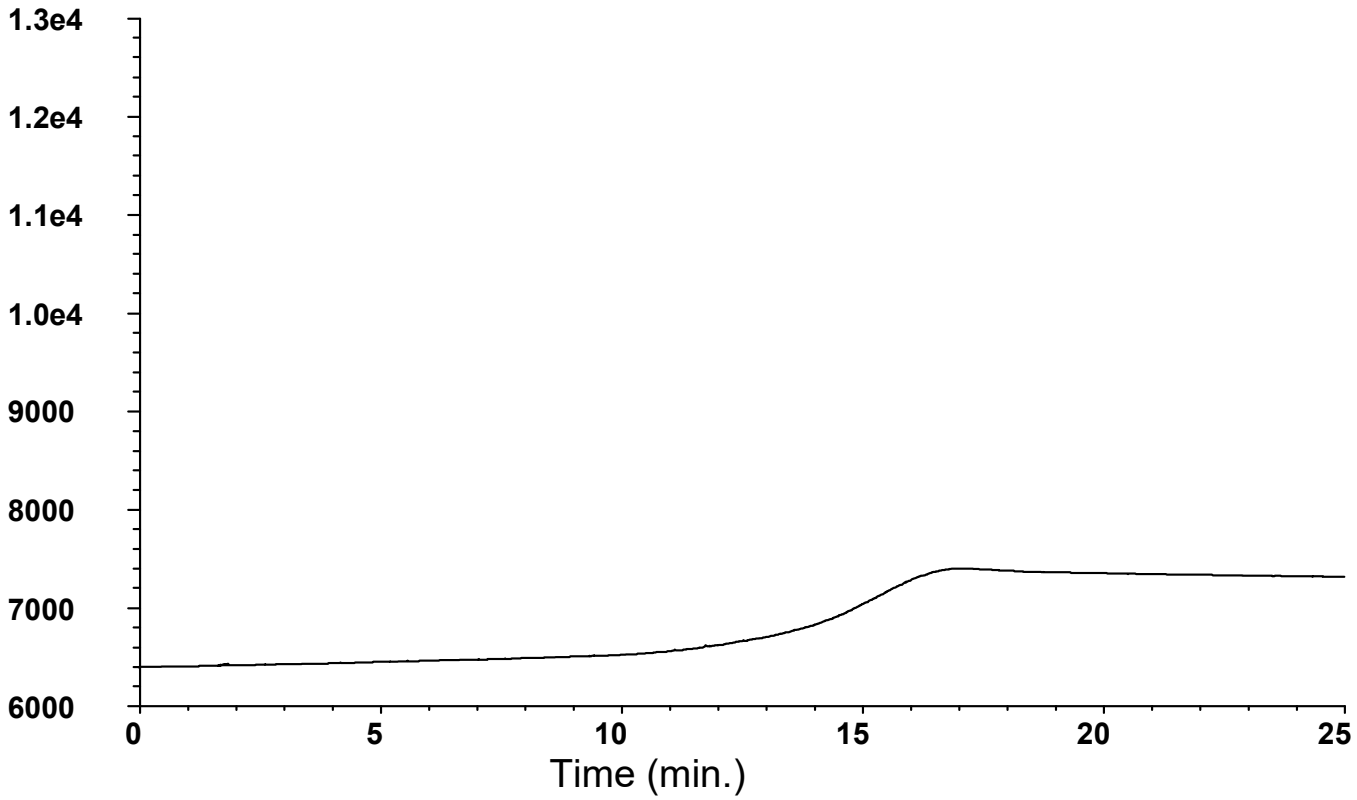
- **1 to 2 hours in most cases**

What about “Baking Out” Columns?

- SURE! But…
- Only removes semi-volatile residues
- Limit to 1-2 hours
- May polymerize some contaminants
- Reduces column life

Generating a Bleed Profile

Temperature program the column without an injection*



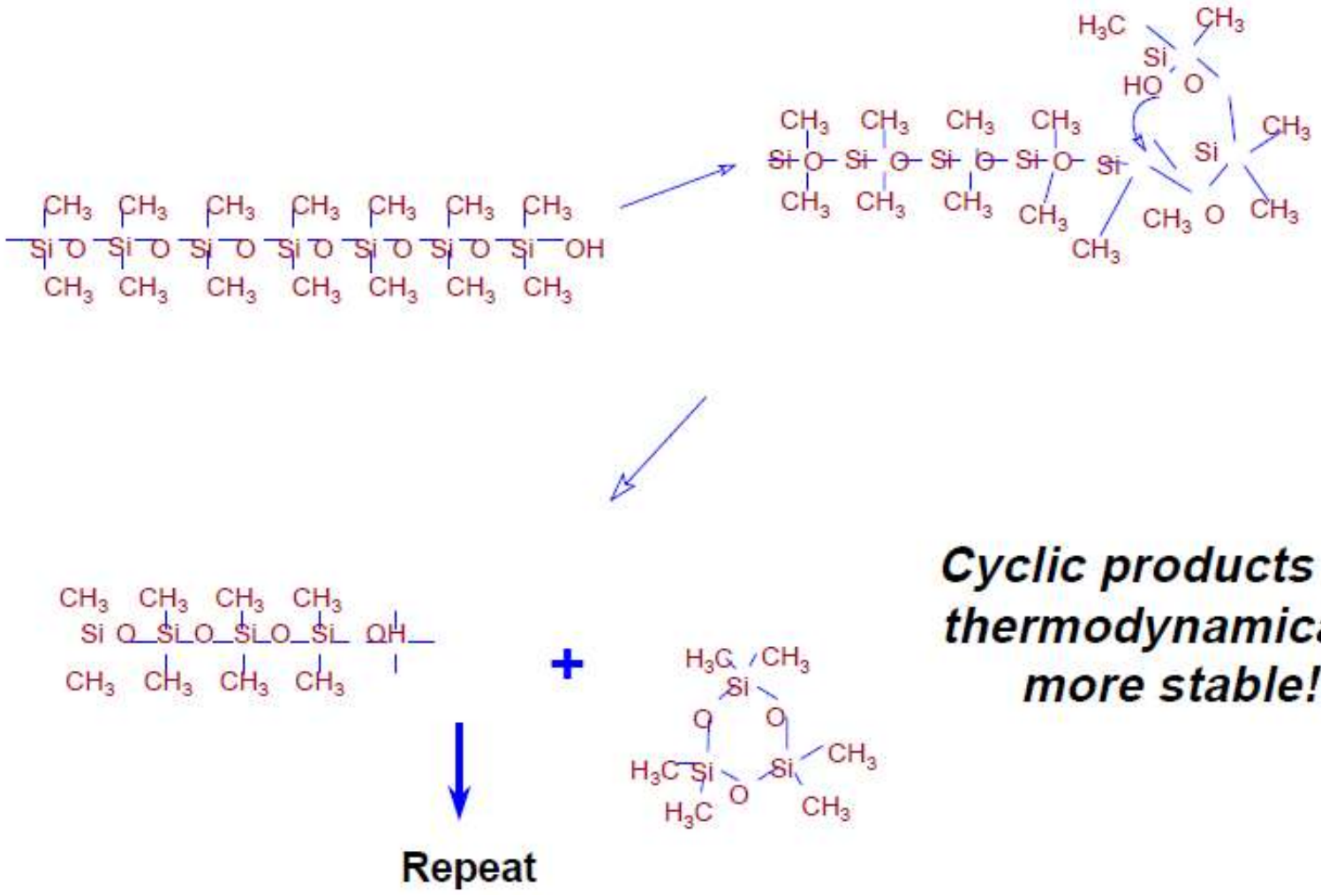
*DB-1 30m x .32mm I.D., .25µm
Temperature program // 40°C, hold 1 min // 20°/min to 320°C, hold 10 min.

What is Bleed?

- thermodynamic equilibrium process occurs to some degree in all columns
- polysiloxane backbone releases low molecular weight, cyclic fragments
- occurs at low level in low temperature, O2-free, clean system
- increased by increased temperature, oxygen exposure or chemical contamination

Bleed: Why Does It Happen?

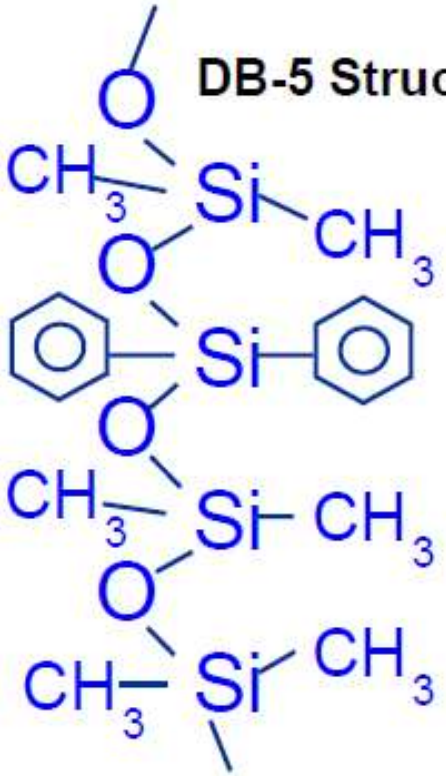
"Back Biting" Mechanism of Product Formation



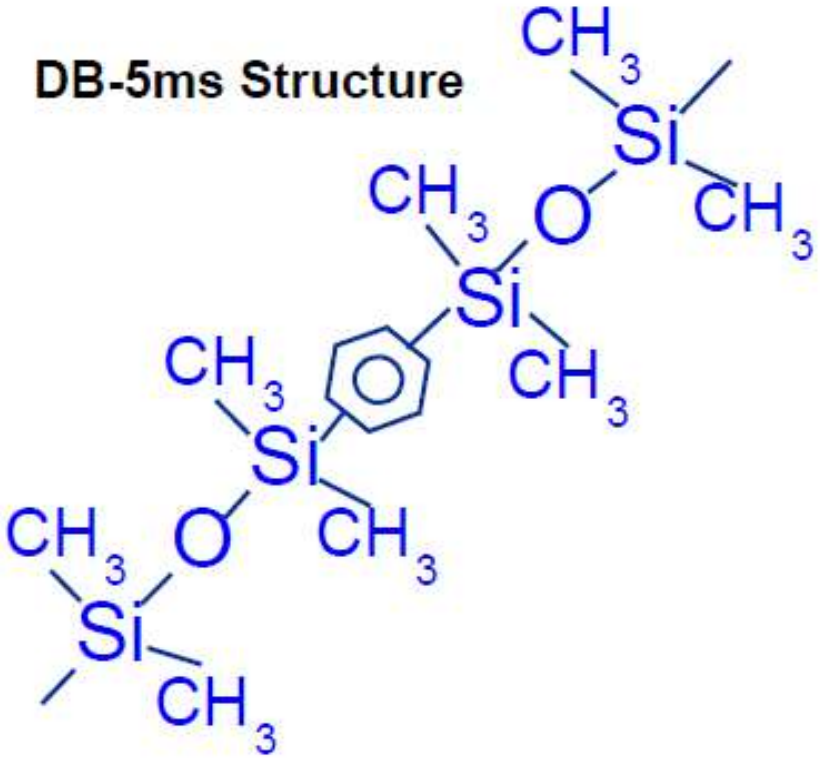
Cyclic products are thermodynamically more stable!

DB-5MS Structure

DB-5 Structure



DB-5ms Structure



DB-5
5% Phenyl

DB-5ms
1. Increased stability
2. Different selectivity
3. Optimized to match DB-5

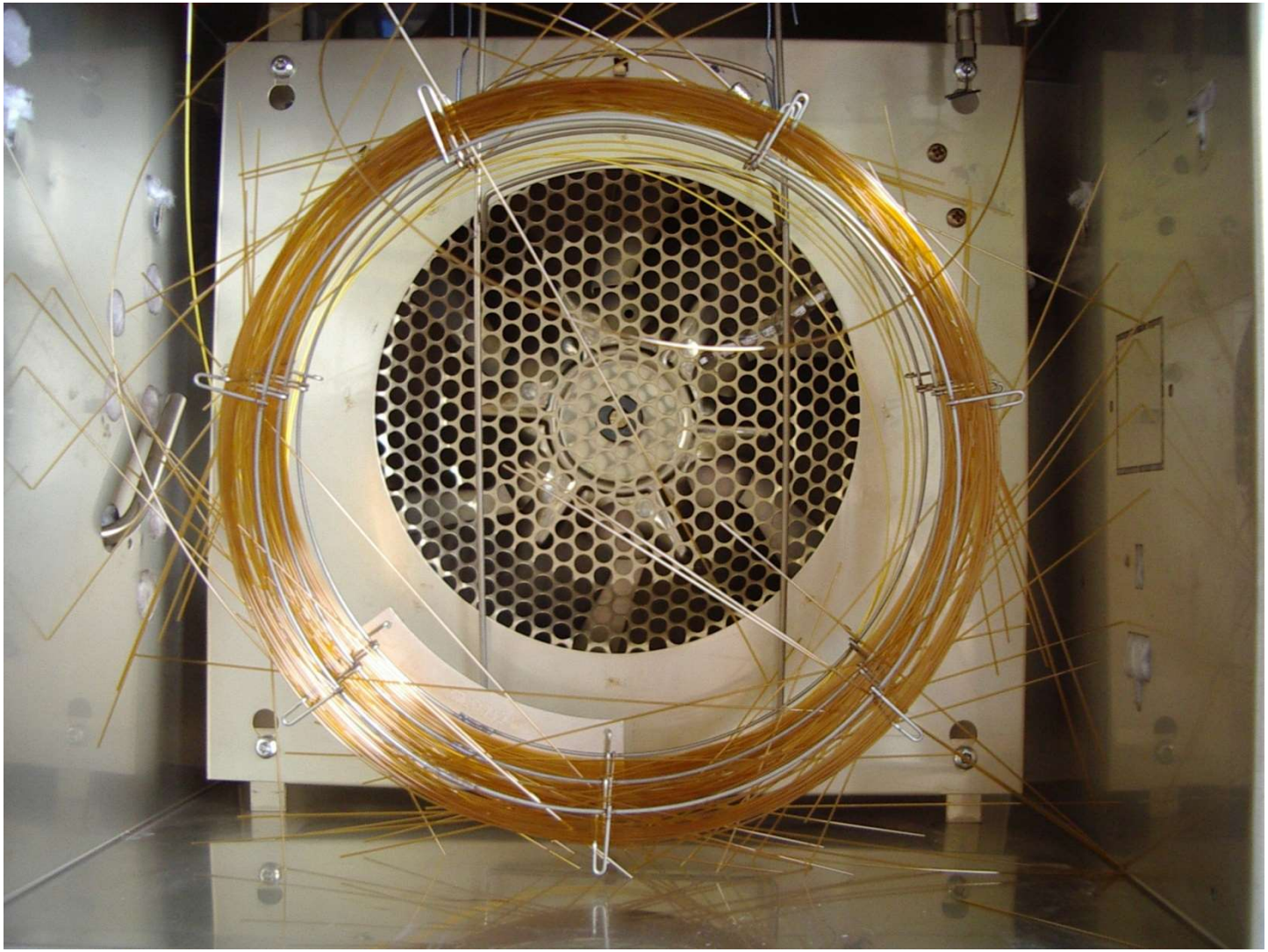
Common Causes of Column Performance Degradation and or Short Column Life

- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O_2 damage)
- Chemical damage by samples
- Contamination

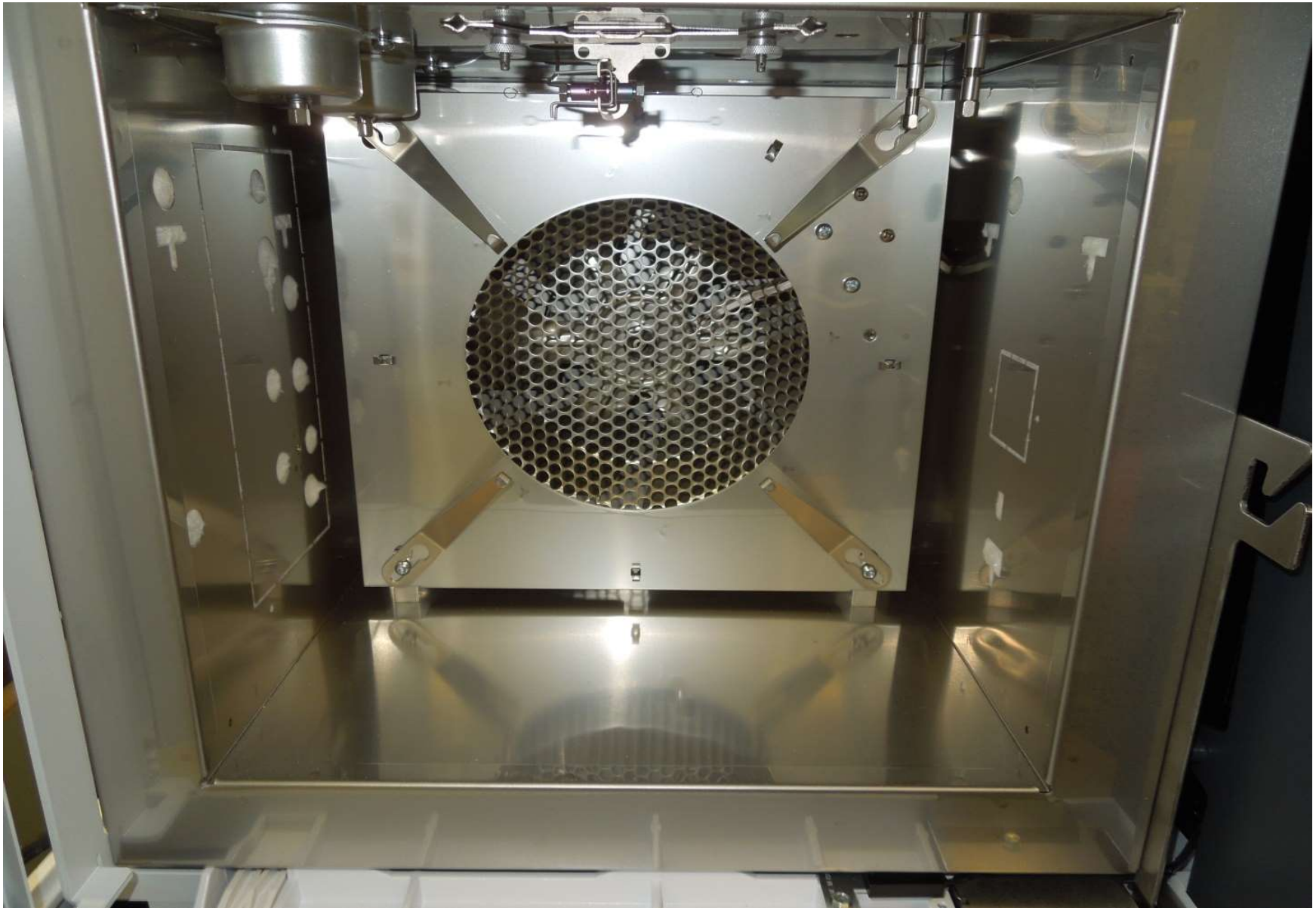
Physical Damage to The Polyimide Coating

- **The smaller the tubing diameter, the more flexible it is.**
- **Avoid scratches and abrasions**
- **Immediate breakage does not always occur upon physical damage**

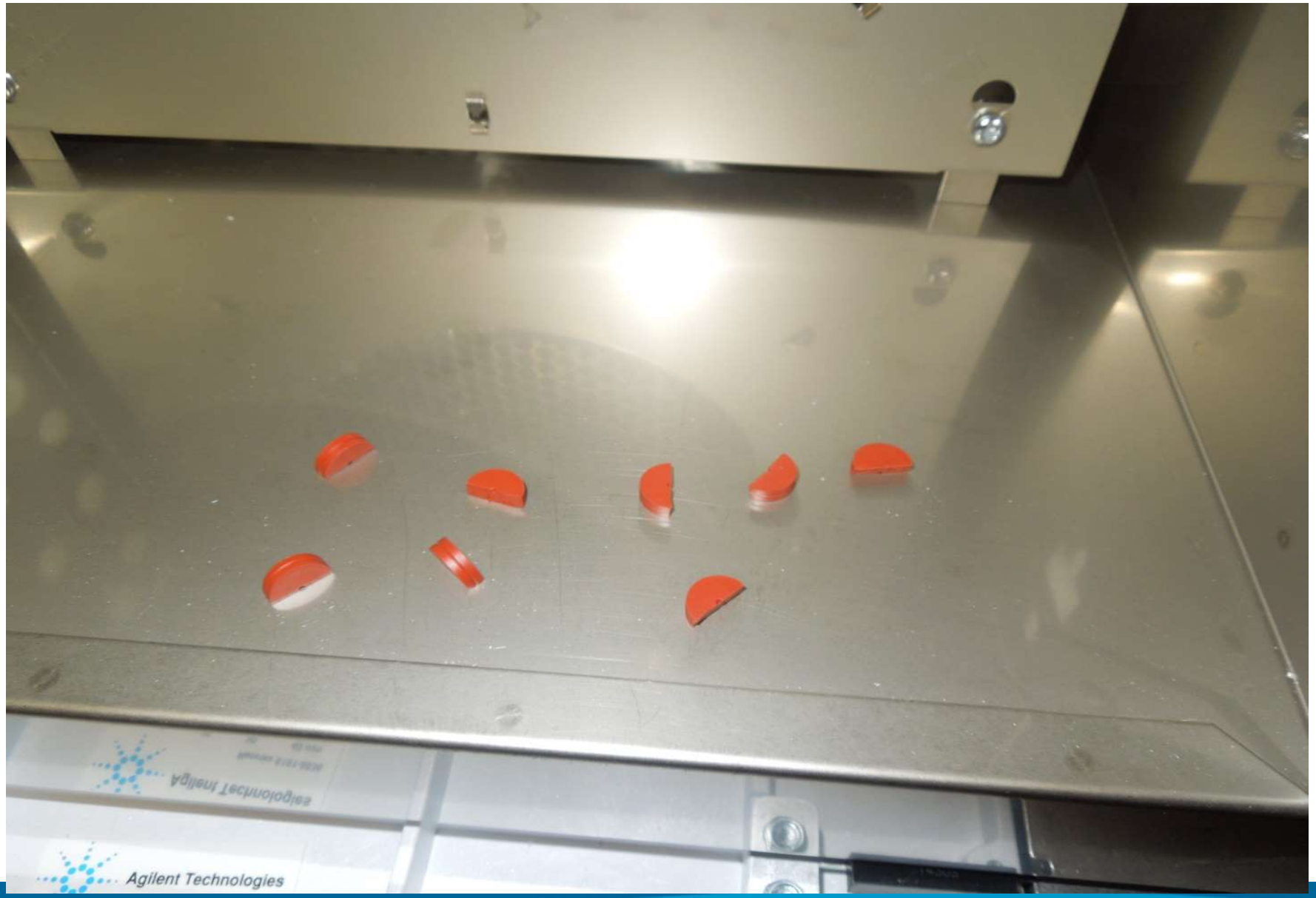
NOT what you want your column to look like!



Remove Column Clips

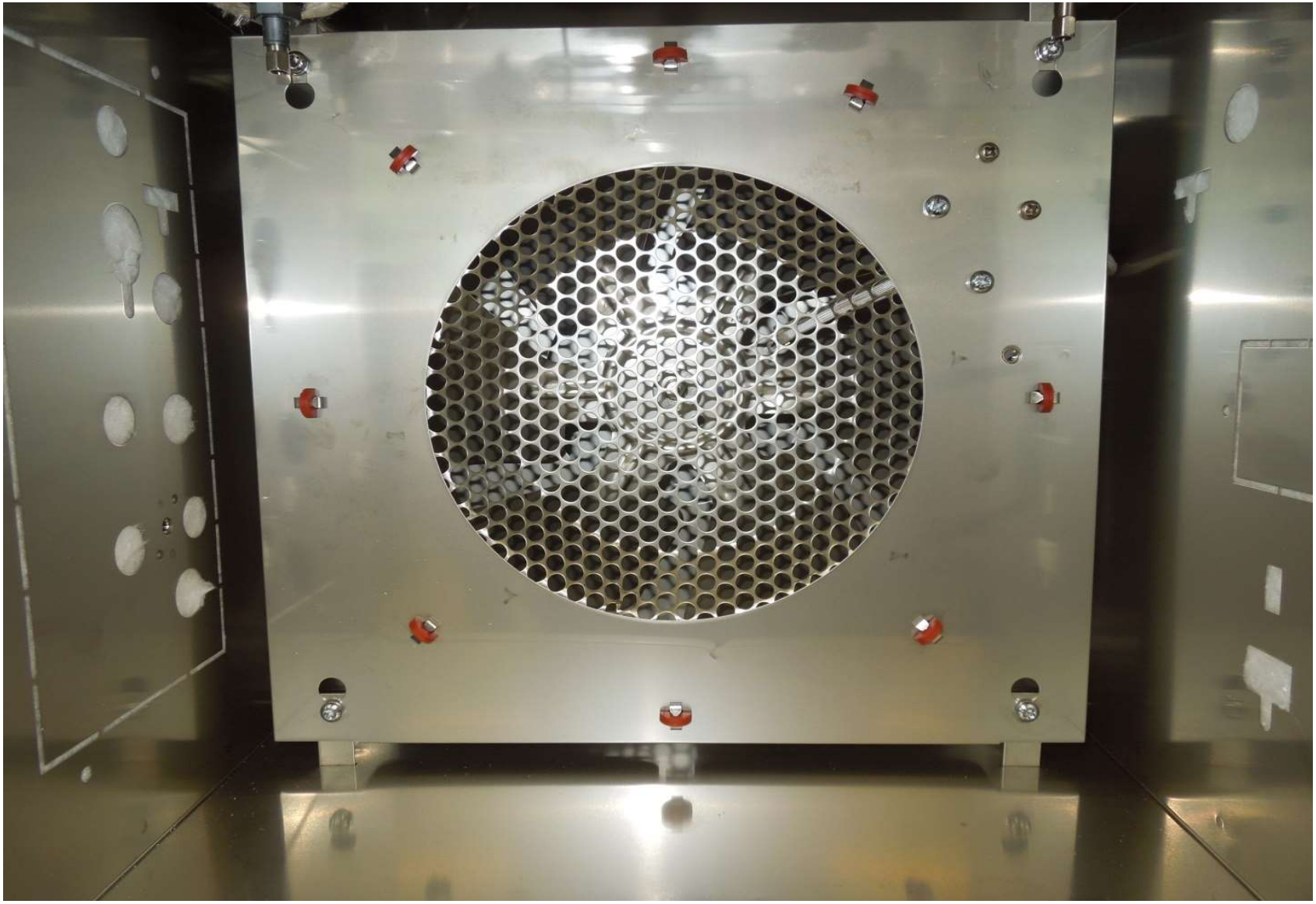


Cut Septa



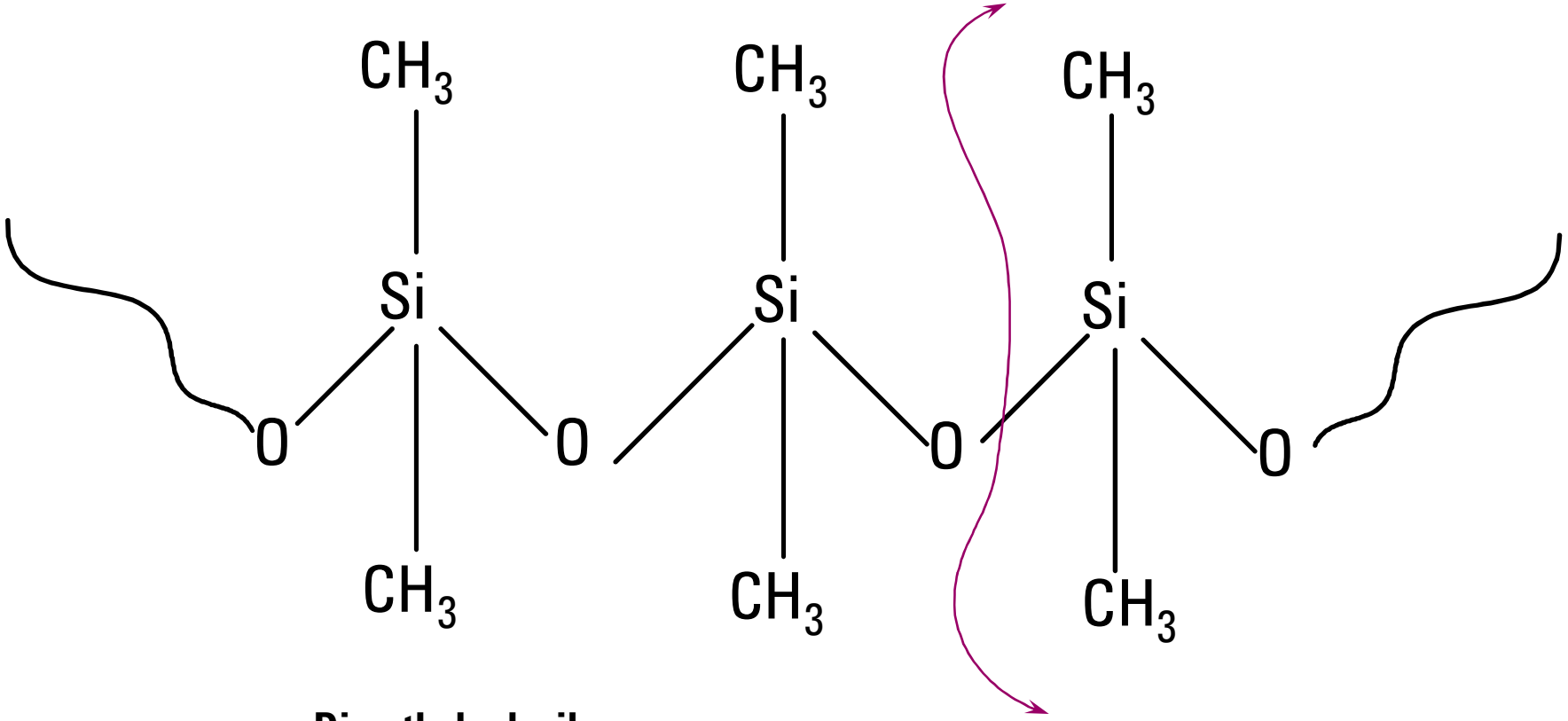
Install Septa

All Septa Installed



Thermal Damage

Degradation of the stationary phase is increased at higher temperatures. Breakage along the polymer backbone.



Dimethylpolysiloxane

Thermal Damage
What To Do If It Happens

- Disconnect column from detector
- “Bake out” overnight at isothermal limit
- Remove 10-15 cm from column end

Thermal Damage

- **Rapid degradation of the stationary phase caused by excessively high temperatures**

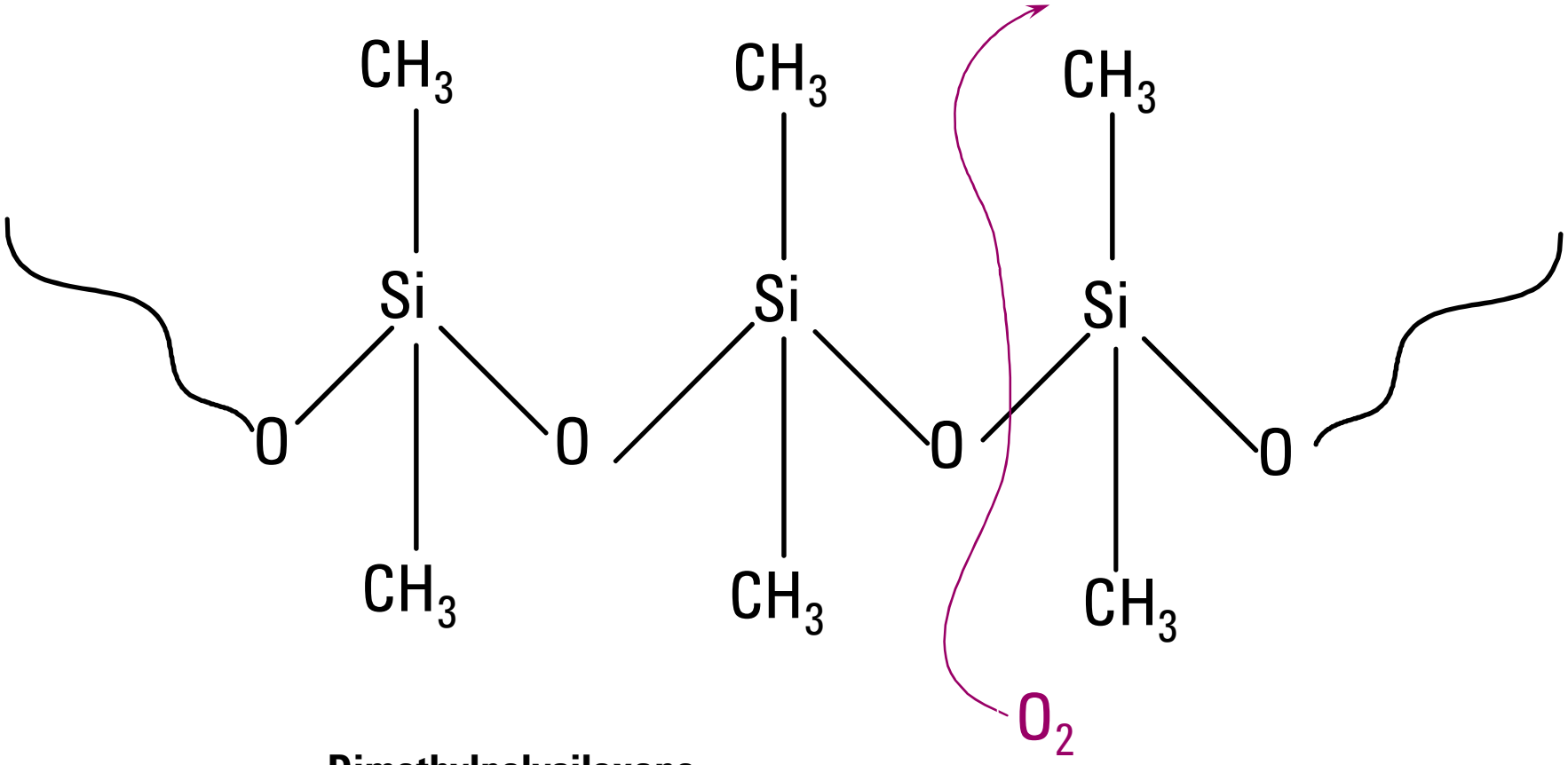
  Isothermal limit = Indefinite time

  Programmed limit = 5-10 minutes

- **Temporary "column failure" below lower temperature limit**

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.



Dimethylpolysiloxane

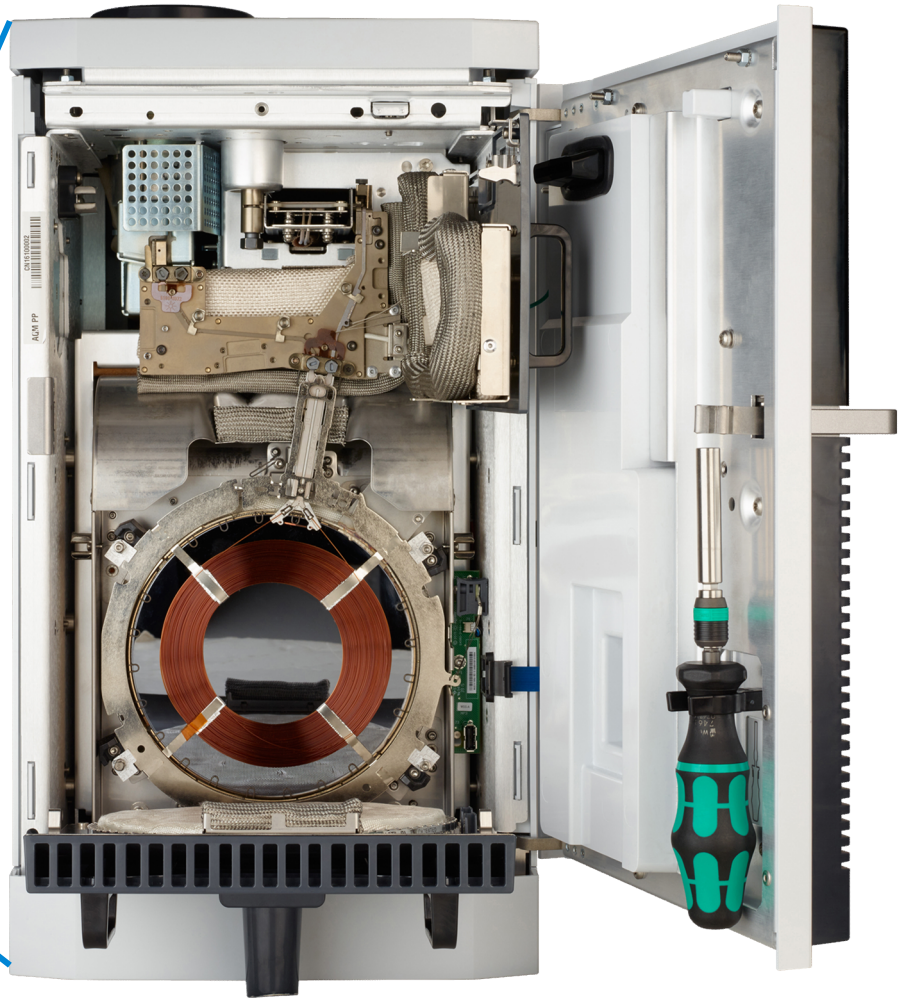
Oxygen Damage
What To Do If It Happens

- Rapid damage to the column
- Usually results in irreversible column damage

How to Prevent Column Damage by Oxygen

- High quality carrier gas (4 nine's or greater)
- Leak free injector and carrier lines
  - Change septa
  - Maintain gas regulator fittings
- Appropriate impurity traps

A New GC – Intuvo 9000



A New Portfolio of GC Consumables

UI Inlet Liners

Guard Chip

Intuvo Gaskets

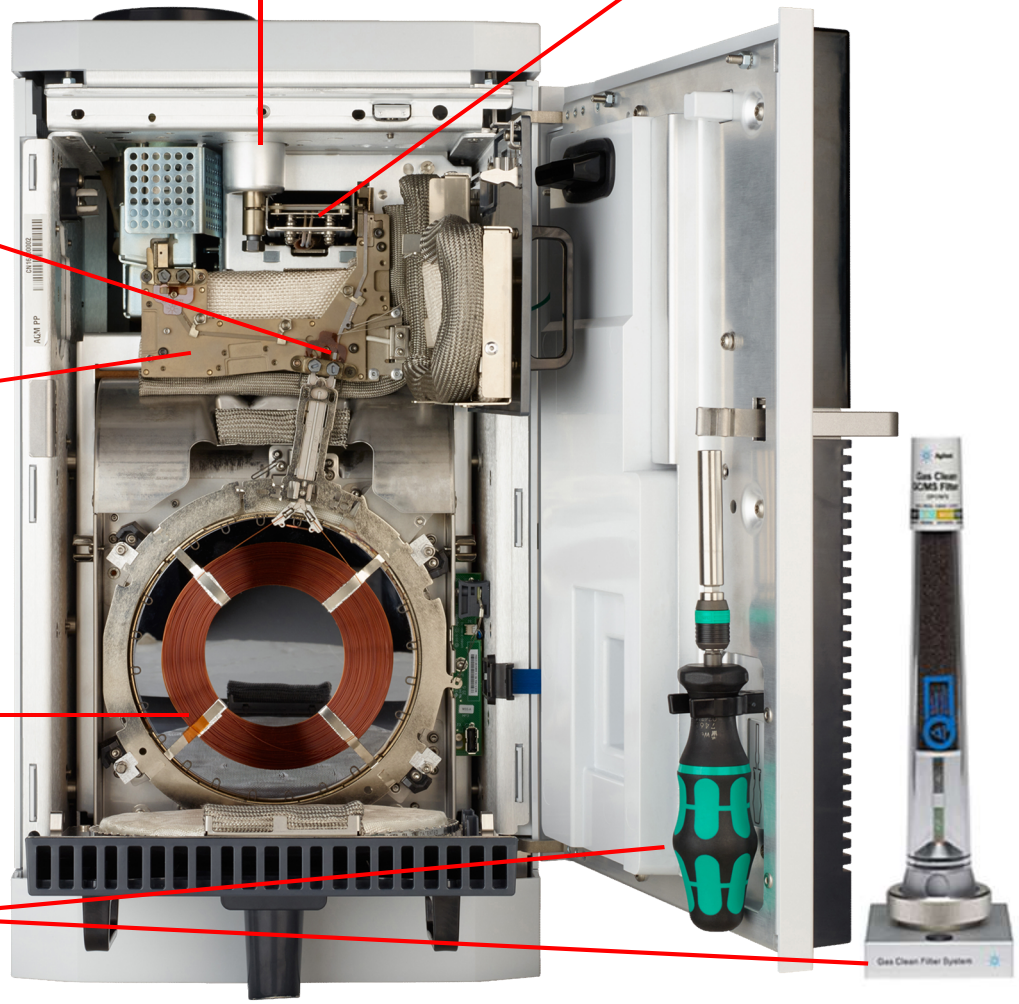
5190-9072
≤ 350 °C

5190-9073
Nickel. ≤ 450 °C

5190-9074
No hole. ≤ 350 °C

Smart Keys

Intuvo Flow Chips



No-trim column

Tools & Accessories

Where Does it Get Dirty?

Here

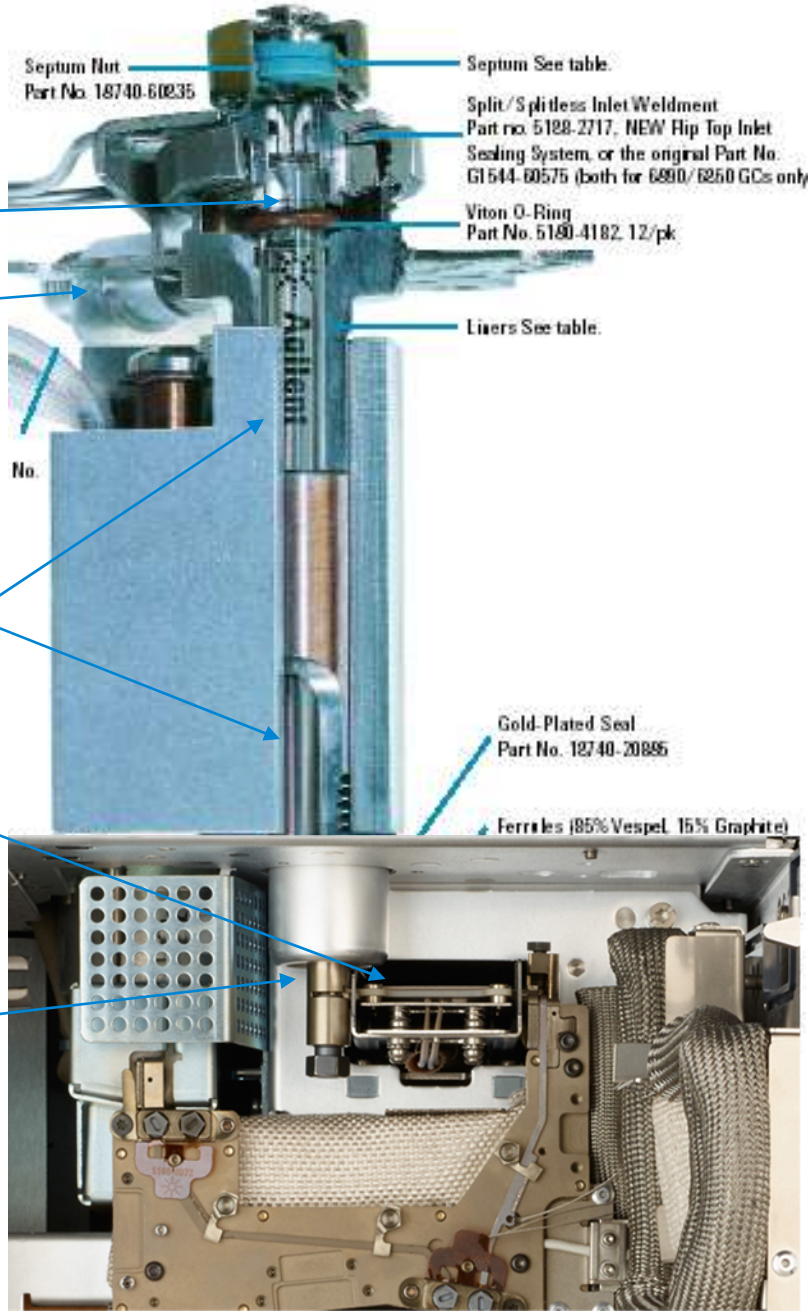
Here

Here

Here

Here

Here



Not the Column!

A New Way to do Maintenance

Guard Chip

Septum

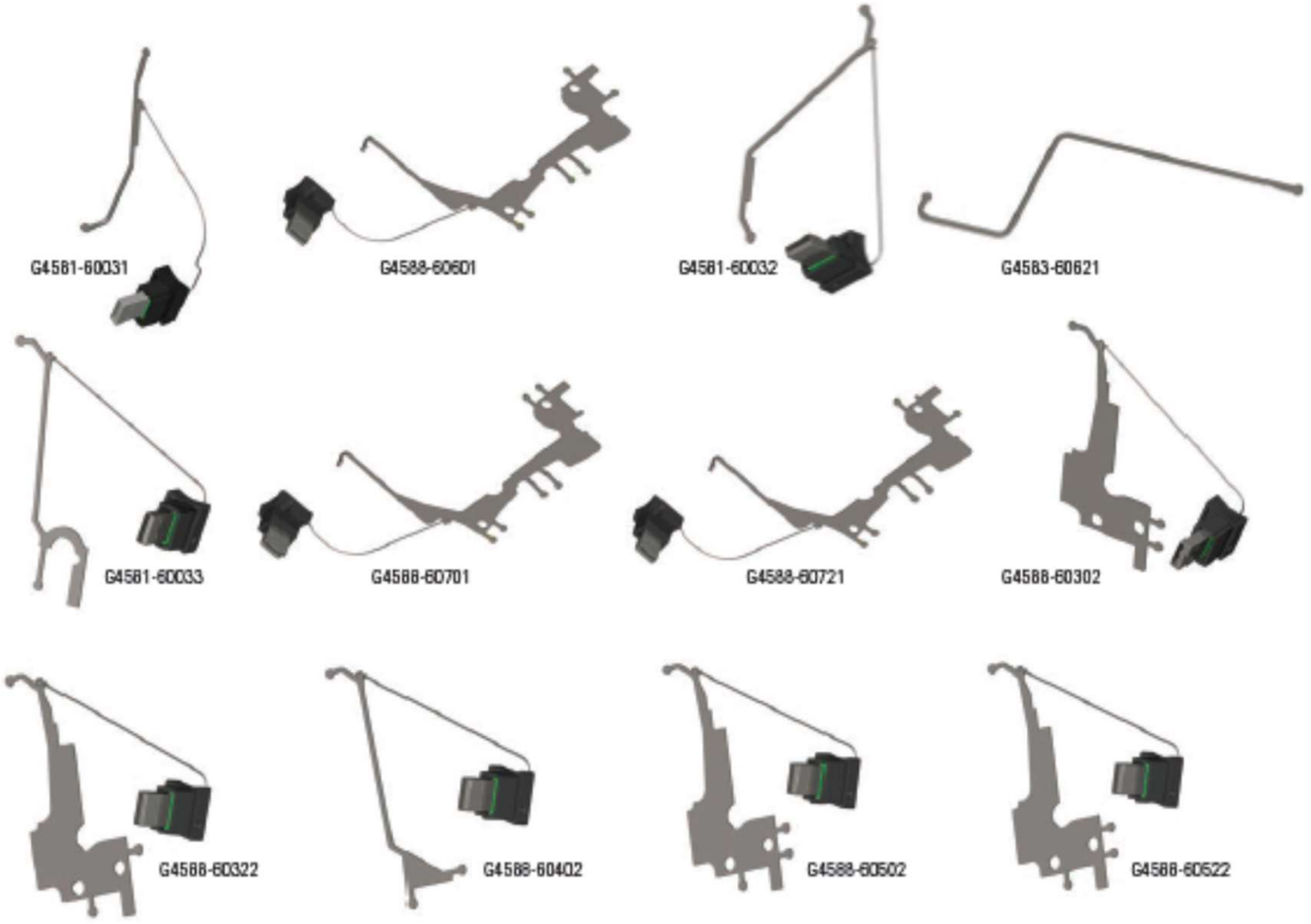
UI Inlet Liner

Gasket

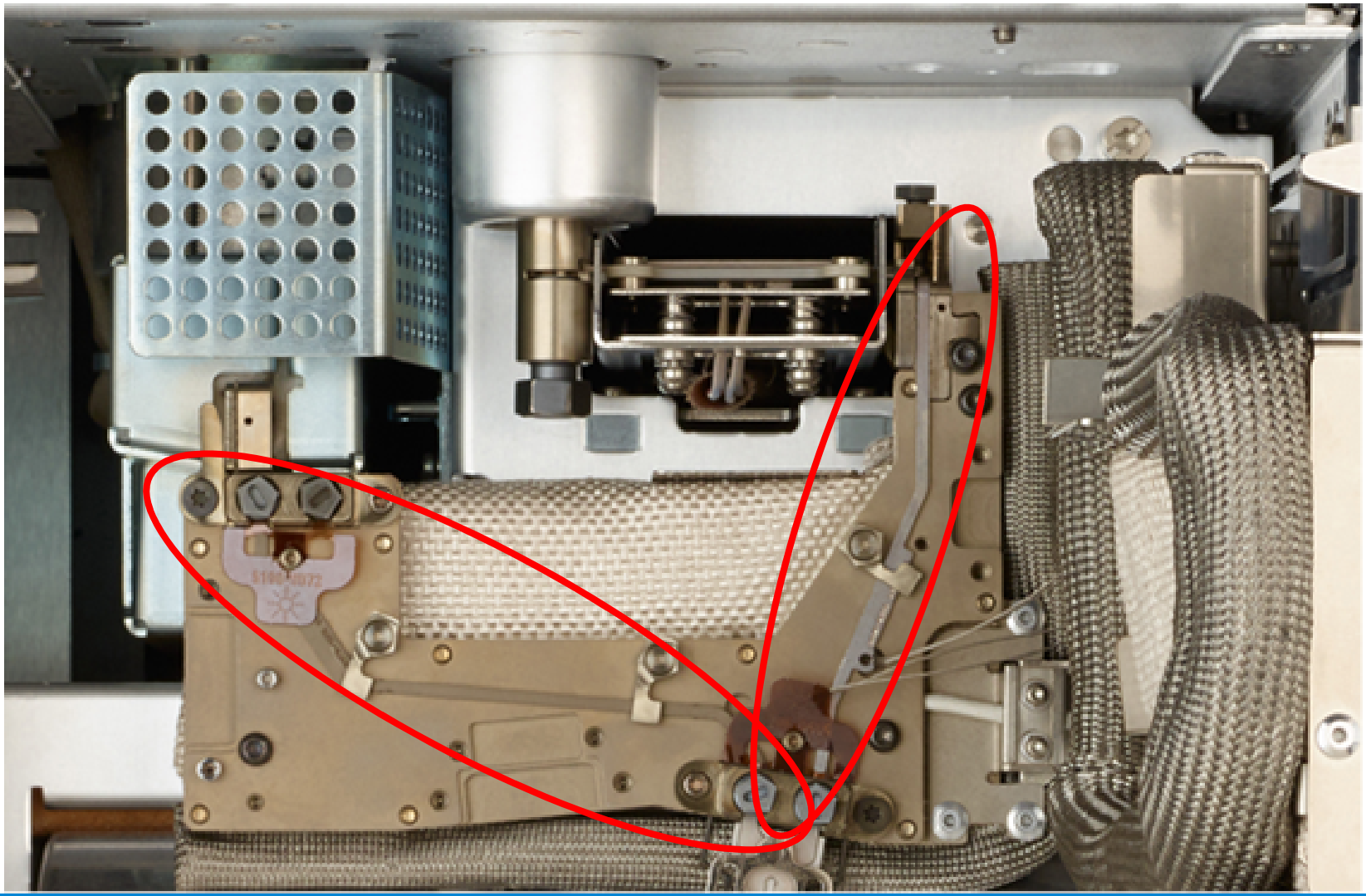
No-trim column

Tool

Intuvo Flow Chips – UI Fixed Length Flow Paths

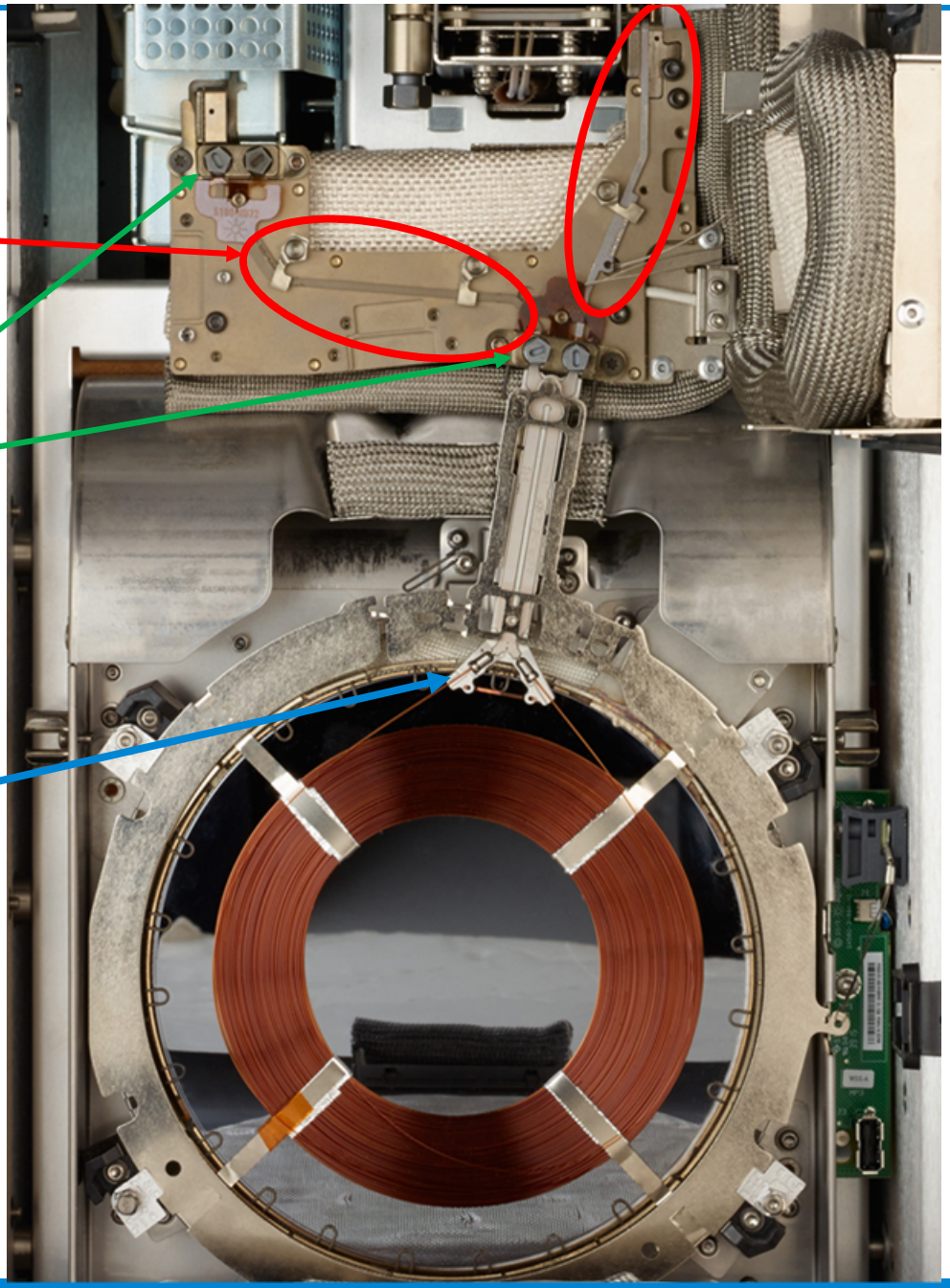


Intuvo Flow Chips – Installation distance perfect always

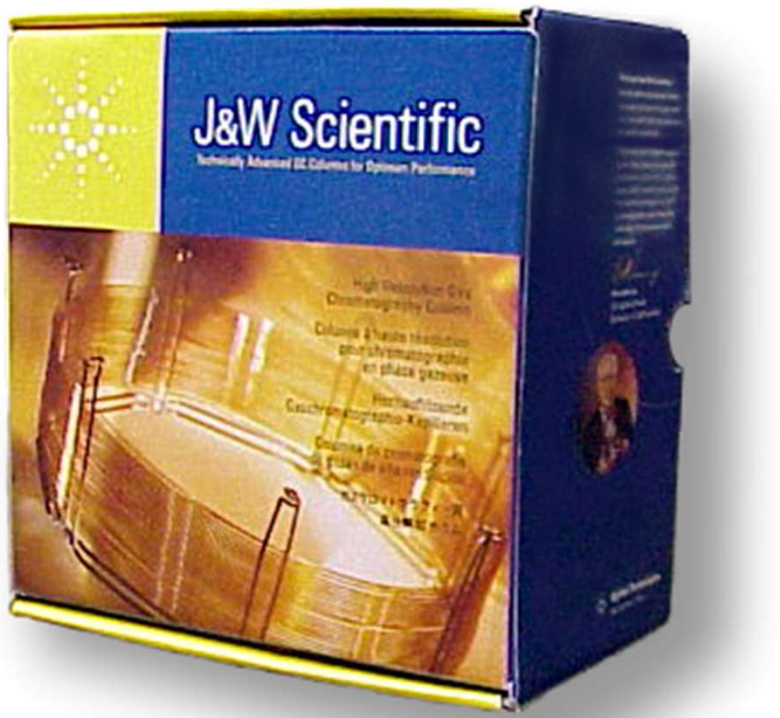


No More
- measuring
- over-tightening

- trimming

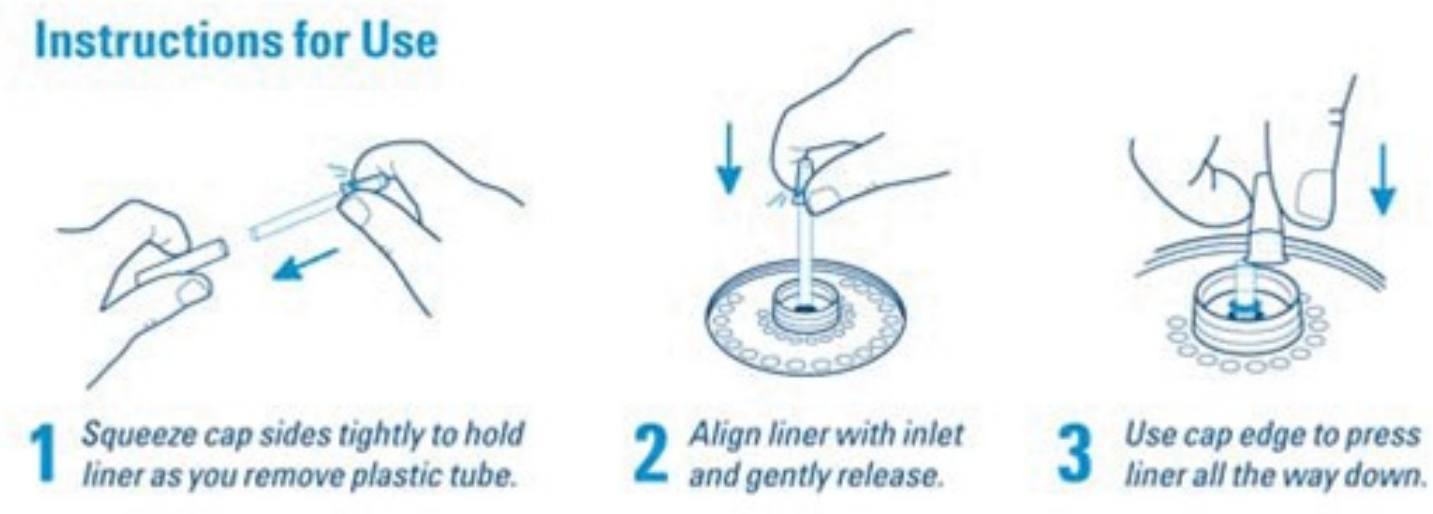


Let’s talk about Liner type

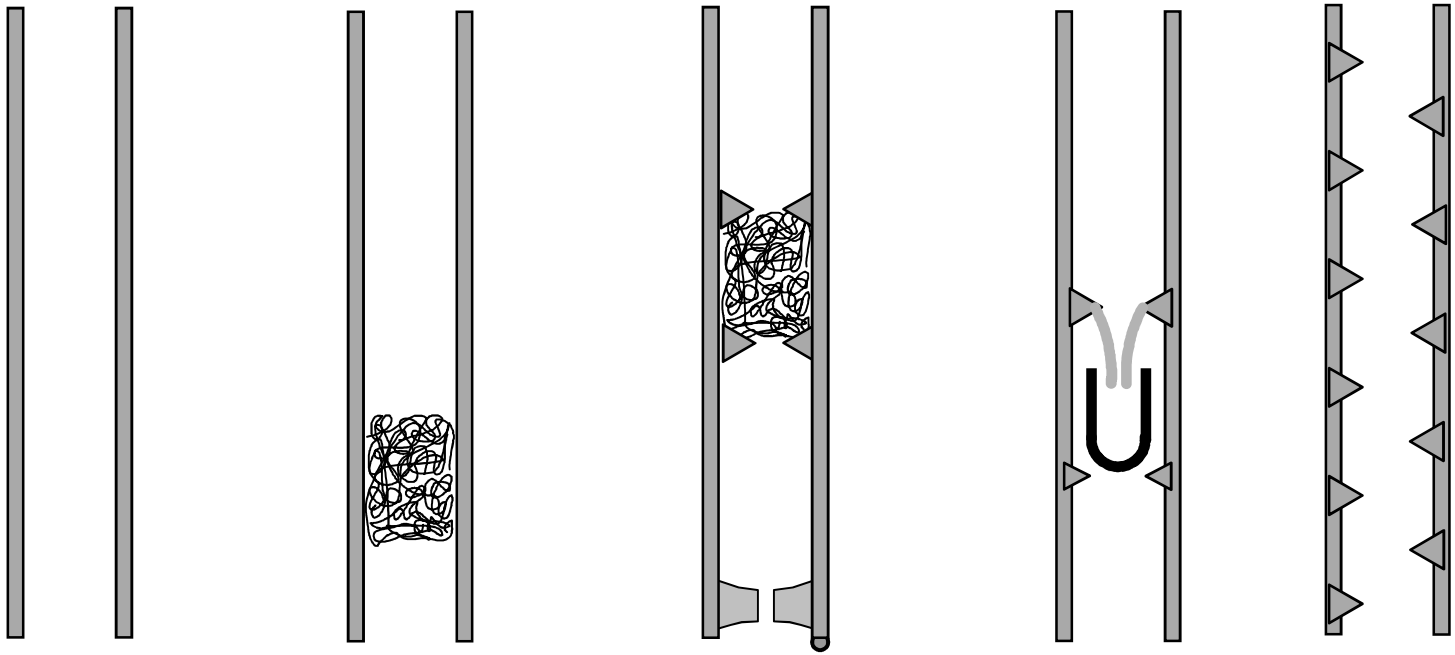


Ultra Inert Liners

- **Touchless packaging**
- Easy installation of new, clean liner
- without risk of contamination from touching
- Includes non-stick plasma treated O-ring



Split Liners – What's What?



Straight tube

Straight tube with glass wool

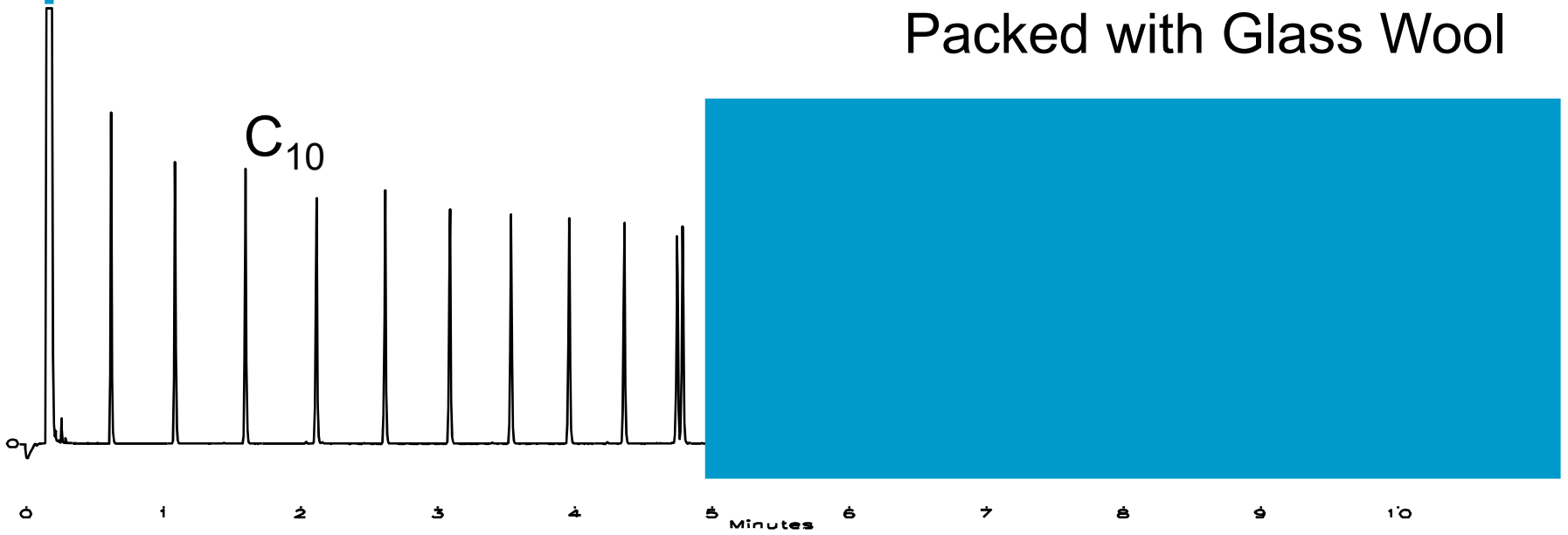
Fixed glass wool

Inverted cup

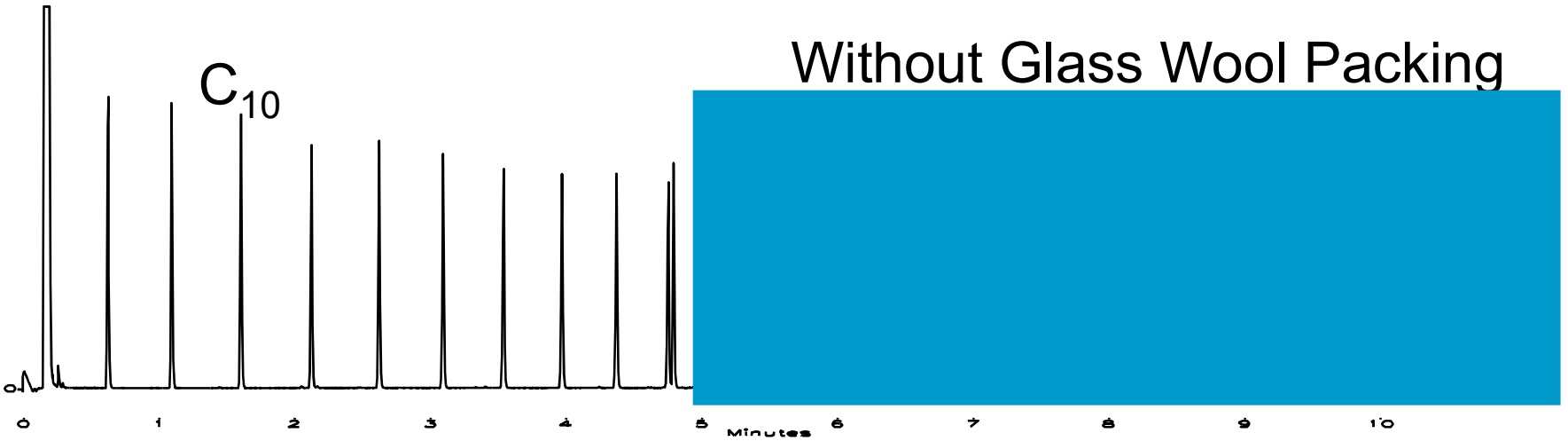
Baffle

Split Liner

Packed with Glass Wool

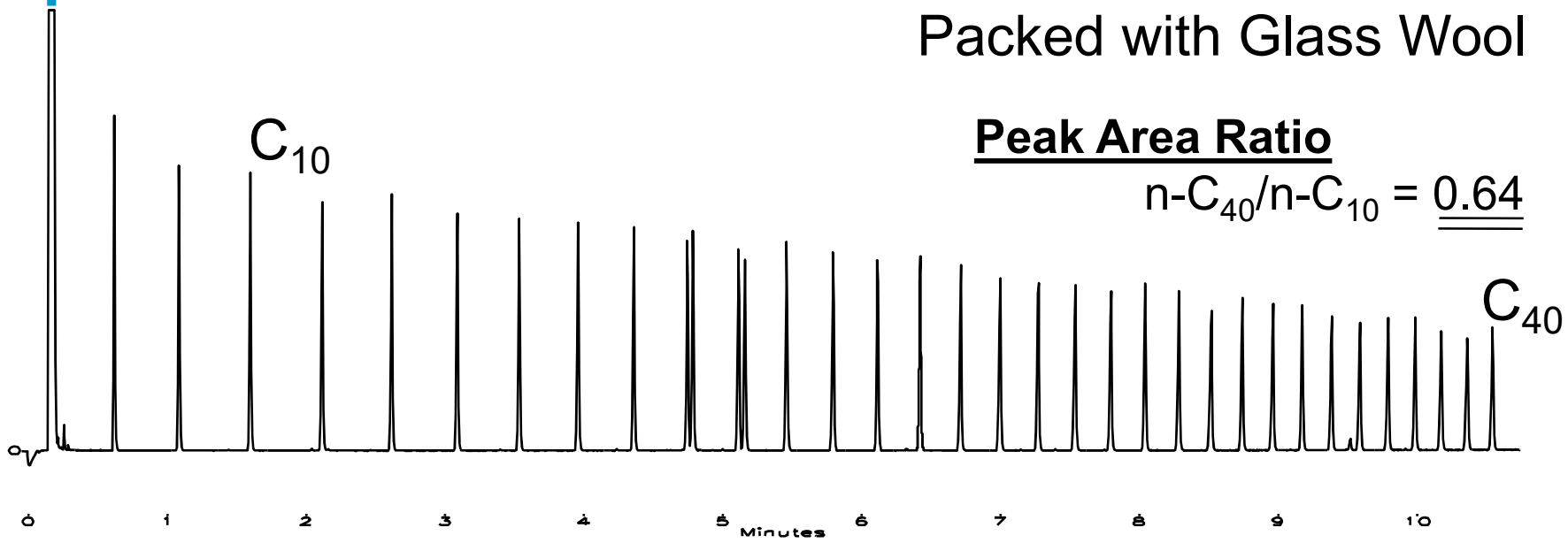


Without Glass Wool Packing



Split Liner

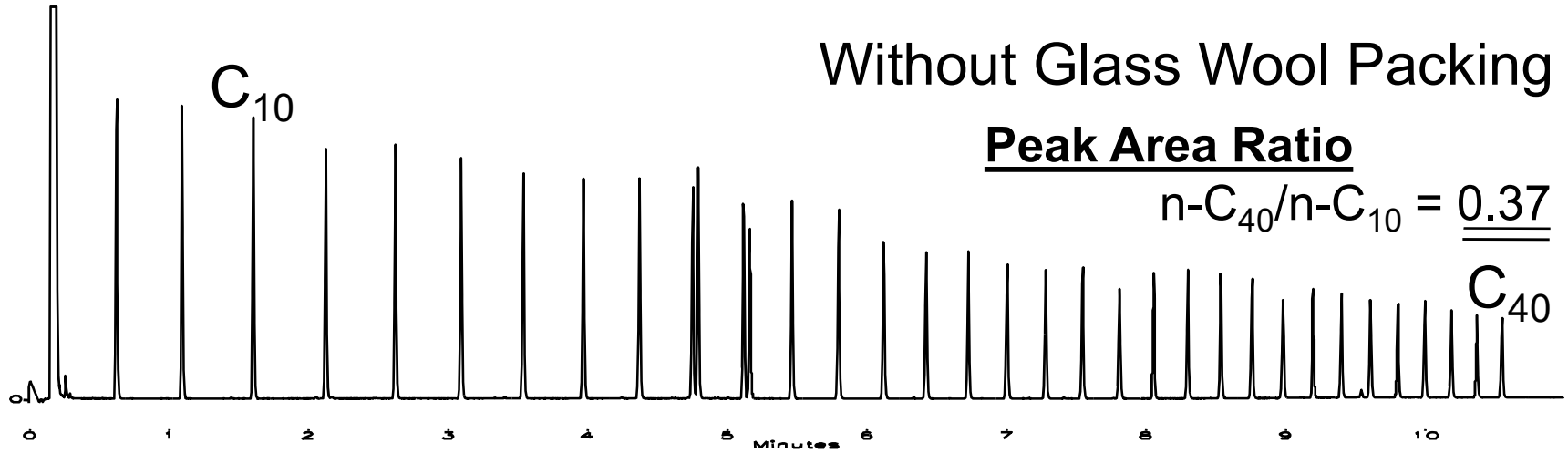
Packed with Glass Wool



Peak Area Ratio

$n\text{-}C_{40}/n\text{-}C_{10}$ = 0.64

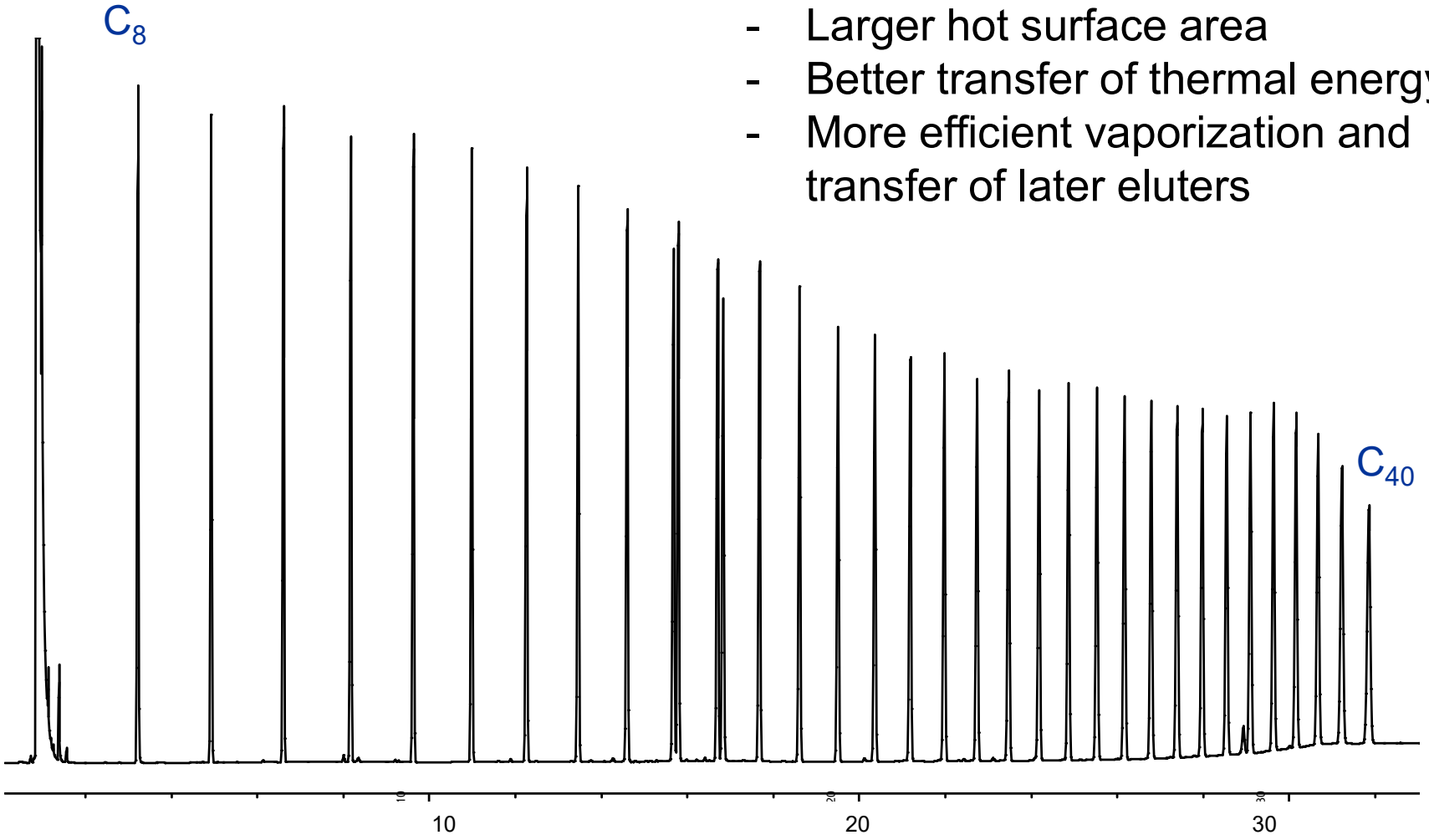
Without Glass Wool Packing



Peak Area Ratio

$n\text{-}C_{40}/n\text{-}C_{10}$ = 0.37

Larger Glass Wool Plug Added to Liner



- Larger hot surface area
- Better transfer of thermal energy
- More efficient vaporization and transfer of later eluters

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

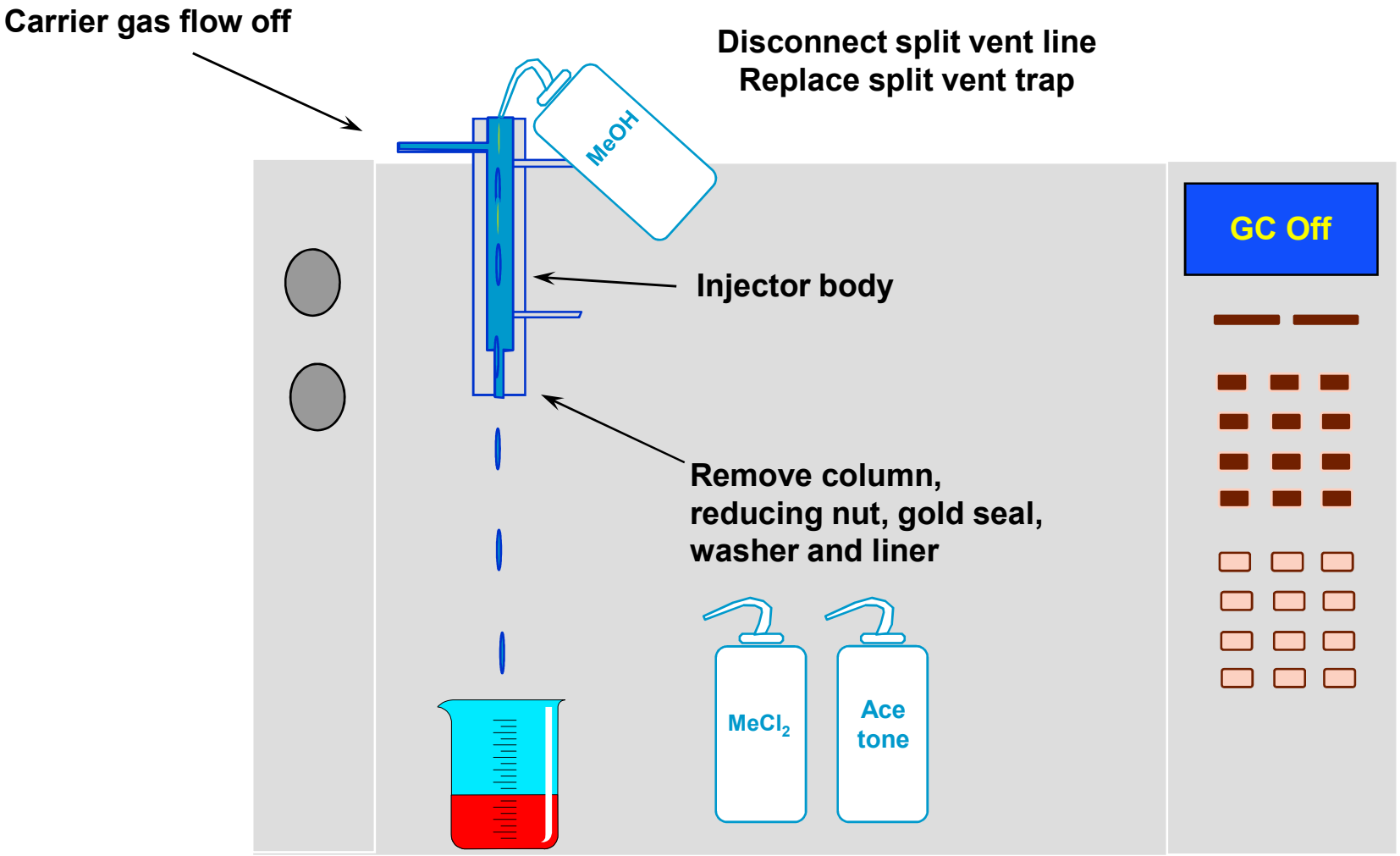
• Can be broken upon installation into the liner, exposing active sites

•Liner deactivation with glass wool plug in place is ideal

Splitless Injection Liners

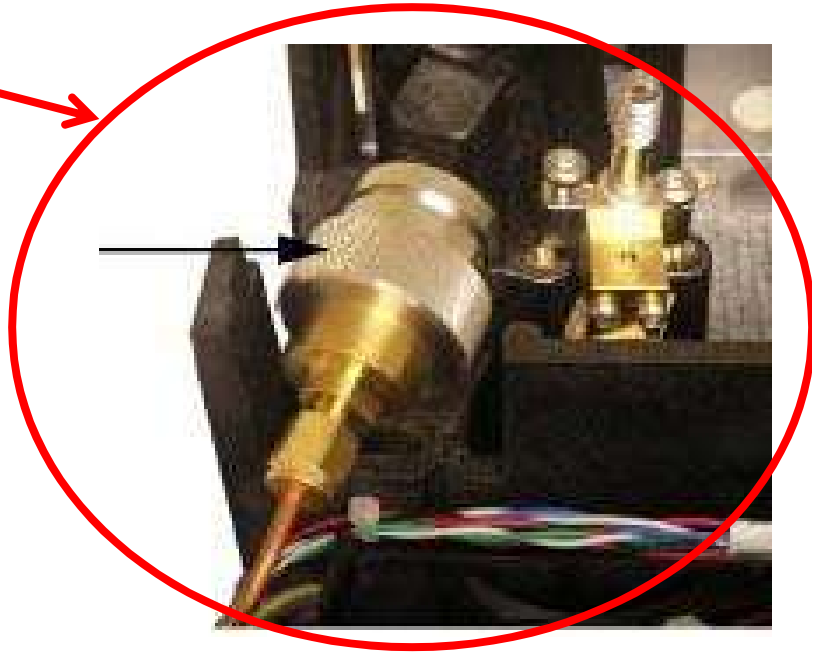
Liner	Part No.	Comments
	5181-3316	Single taper, deactivated, 900µL volume. Taper isolates sample from metal seal, reducing breakdown of compounds that are active with metals. For trace samples, general application.
	5062-3587	Single taper, deactivated, with glass wool, 900µL volume. Glass wool aides volatilization and protects column. For trace (dirty) samples.
	5181-3315	Double taper, deactivated, 800µL volume. Taper on inlet reduces chance for backflash into carrier gas lines. High efficiency liner for trace, active samples.
	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.

Cleaning the Split/Splitless Injector



Finding the Split Vent Trap

Remove cover at Split Vent

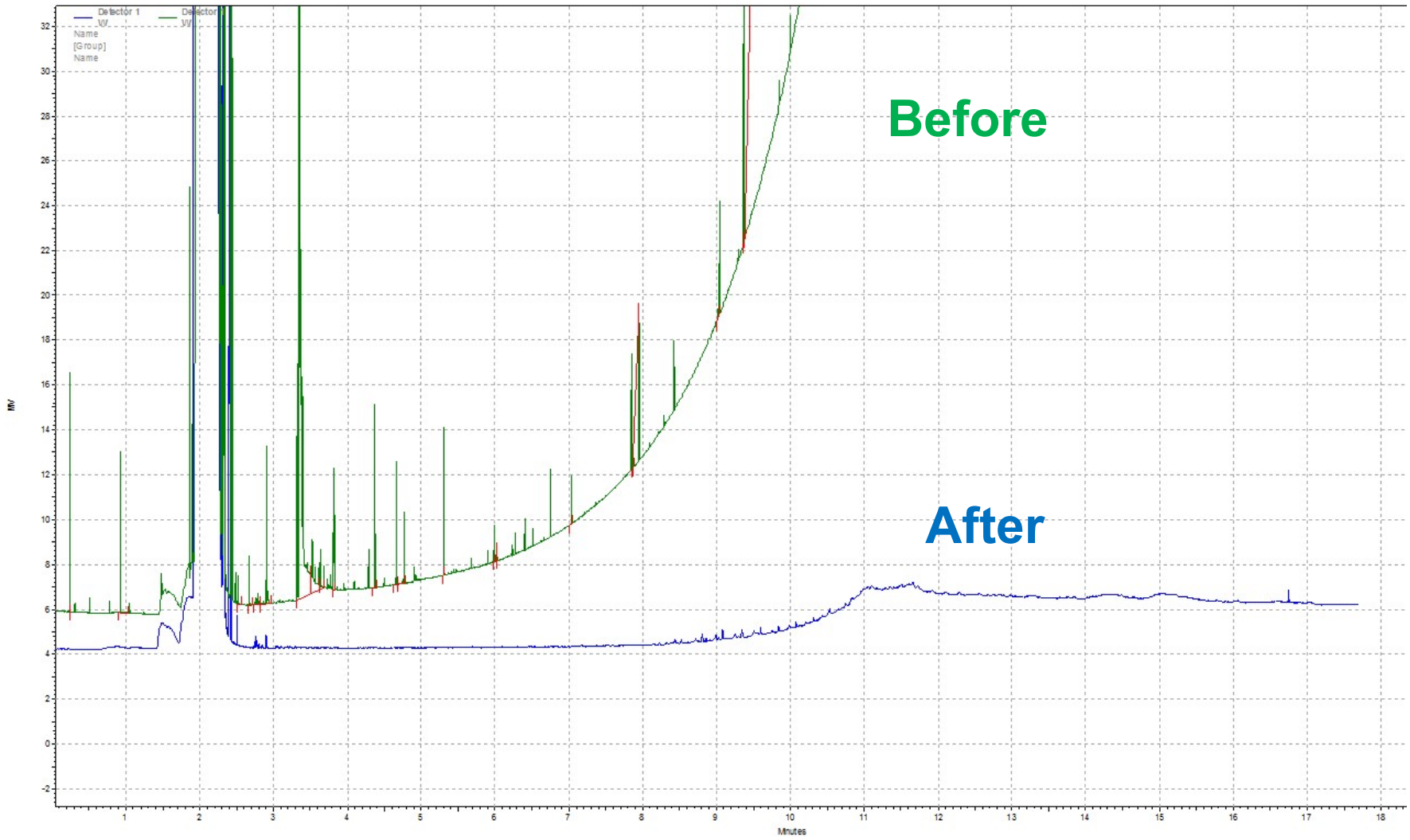


Replacing the Split Vent Trap

Finger Tight Knurled Nut

G1544-80530

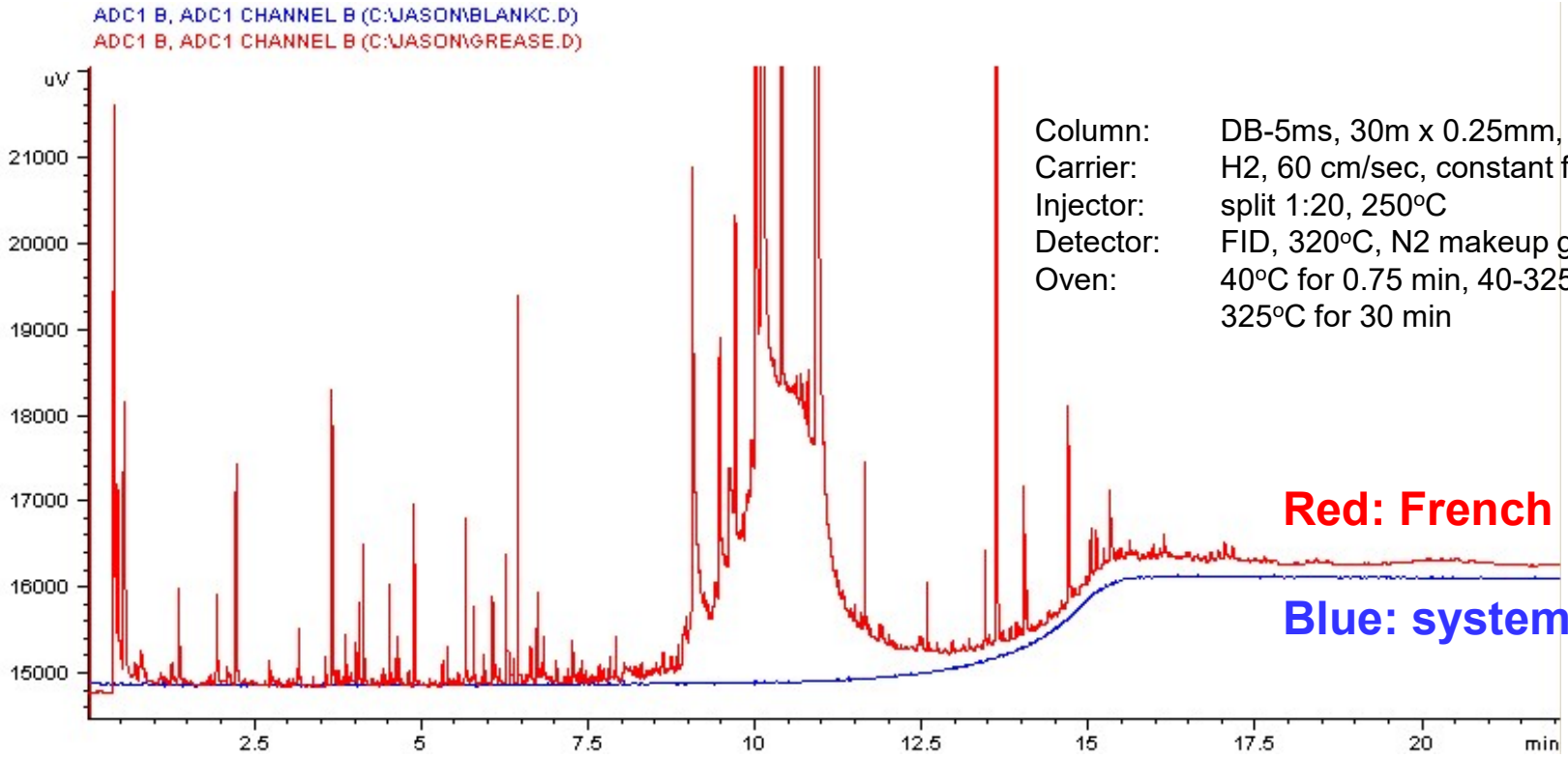
Split Vent Trap Changed (Column Bleed?!?)



Be Careful When Doing Maintenance…

You may be the CONTAMINATOR!

Contamination of system by residue on fingers during column installation



Column: DB-5ms, 30m x 0.25mm, 0.25um
Carrier: H2, 60 cm/sec, constant flow
Injector: split 1:20, 250°C
Detector: FID, 320°C, N2 makeup gas
Oven: 40°C for 0.75 min, 40-325°C at 20°C/min, 325°C for 30 min

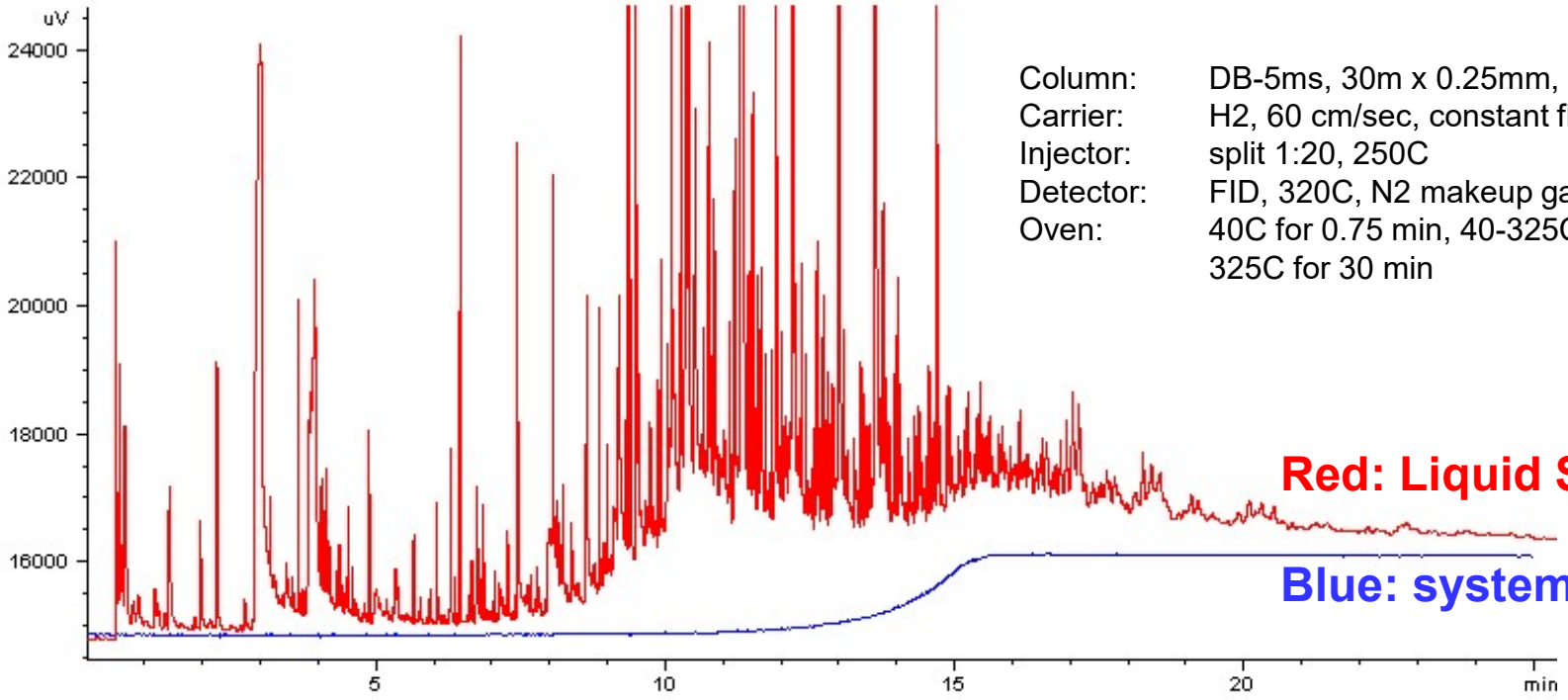
Red: French Fry

Blue: system blank

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.

Contamination from Liquid Soap



Column:	DB-5ms, 30m x 0.25mm, 0.25um
Carrier:	H2, 60 cm/sec, constant flow
Injector:	split 1:20, 250C
Detector:	FID, 320C, N2 makeup gas
Oven:	40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

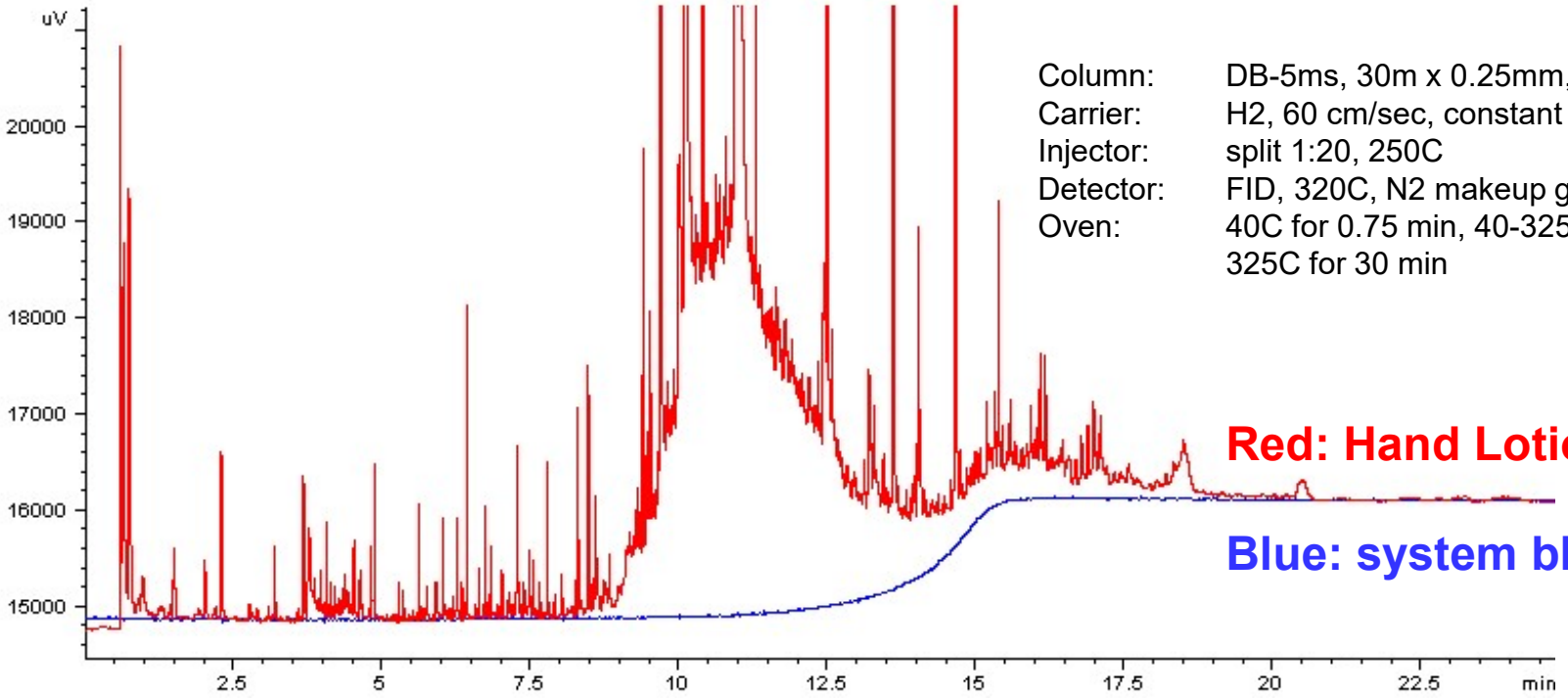
Red: Liquid Soap

Blue: system blank

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.

Contamination from Hand Lotion



Column:	DB-5ms, 30m x 0.25mm, 0.25um
Carrier:	H2, 60 cm/sec, constant flow
Injector:	split 1:20, 250C
Detector:	FID, 320C, N2 makeup gas
Oven:	40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

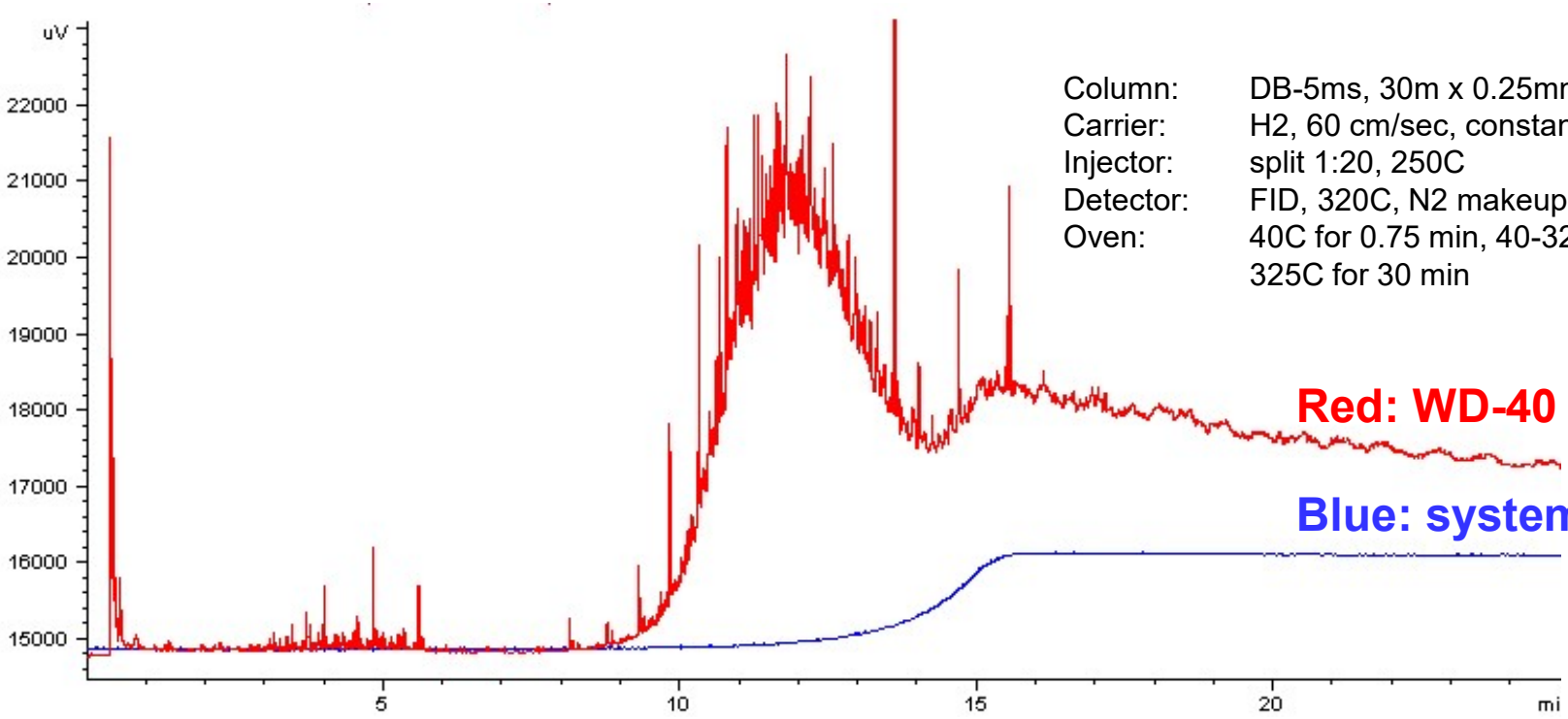
Red: Hand Lotion

Blue: system blank

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.

Contamination from Lubricant



Column:	DB-5ms, 30m x 0.25mm, 0.25um
Carrier:	H2, 60 cm/sec, constant flow
Injector:	split 1:20, 250C
Detector:	FID, 320C, N2 makeup gas
Oven:	40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

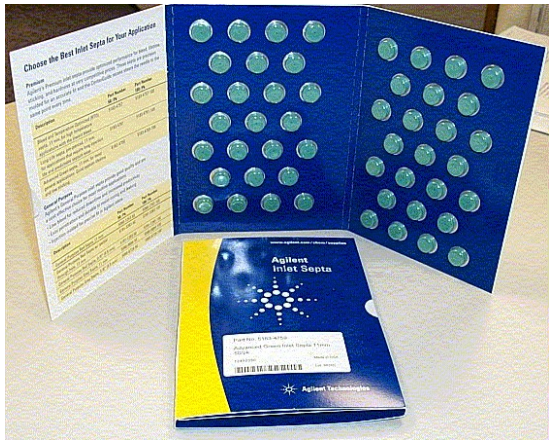
Red: WD-40

Blue: system blank

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.

More and More “Touchless” Packaging



TECHNICAL SUPPORT

1-800-227-9770, #3,3,1

gc-column-support@agilent.com

VIDEOS!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Agilent Multimode Inlet
(MMI)
0:03 / 7:52
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AgilentChem
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This video describes proper maintenance procedures for the Agilent Multimode Inlet (MMI) for GC, including changing the liner, installing the
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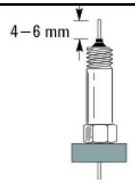
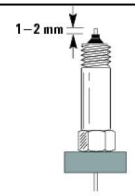
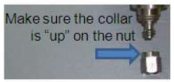
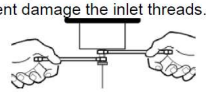
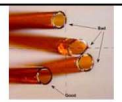
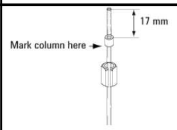
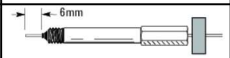
Reference Guides

Agilent GC Support - Quick Reference Guide

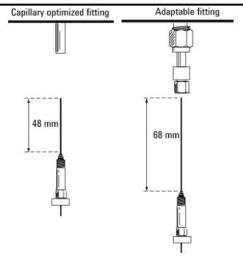
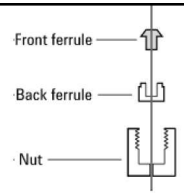
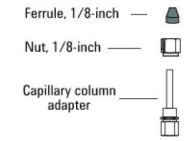
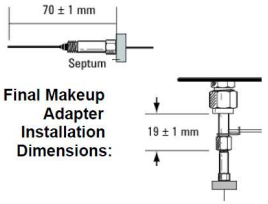
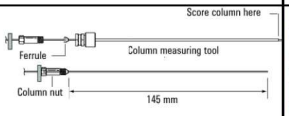
GC Column Installation Quick Reference Guide - Inlets

Inlet	Diagram	Procedure
Split/Splitless		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 4-6 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Purged Packed		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Pull the column back so that 1-2 mm of column is extending past the end of the ferrule. Thread the column nut and column into the inlet and tighten slightly past where the column grabs – retighten after heating.
Multimode		**NOTE:** Make sure the column adapter nut on the inlet base is ***fully threaded on*** and ***spinning freely – Collar Up!***  **Tighten with two wrenches** - ¼" and 5/16" To prevent damage the inlet threads. 
Cool On Column		Insert the column all the way into the inlet until you feel the spring tension – do not withdraw. ***The column cut is critical***. **Tighten with two wrenches** - ¼" and 5/16" to avoid damaging the inlet.
PTV		There should be 17mm of column above the graphpak ferrule – the graphpak ferrule should be installed with the graphite end towards the inlet base. The column nut is slotted. Use a 5 mm wrench to tighten the fitting.
Volatiles Interface		There is a longer column nut for the VI so that you don't have to remove the inlet block. Part Number - G3504-20504

Agilent Technologies

Agilent GC Support - Quick Reference Guide

GC Column Installation Quick Reference Guide - Detectors

Detector	Diagram	Procedure
FID/NPD		Place a septum over the column, then the column nut and ferrule. Trim the end of the column with a column cutter. Thread the column nut and column into the detector base. Insert the column all the way into the detector jet until it stops, then withdraw 1-2 mm before tightening the nut. The dimensions shown of 48 and 68 mm are provided for very narrow columns (<100um) which may go all the way through the jet.
TCD – Using the standard Low Leakage Ferrules		Place a 1/8" Swagelok nut and the Back and Front ferrules over the column. (Be sure to select the front ferrule size to match the column) Trim the end of the column with a column cutter. Thread the column nut and column into the detector base. Insert the column all the way into the detector jet until it stops, then withdraw 1-2 mm before tightening the nut. Re-tighten after heating.
TCD – Original Design Column Adapter		Install the column adapter to the 1/8" Swagelok TCD base using a graphite/vespel ferrule. Thread the column nut and column into the adapter. Insert the column all the way into the detector jet until it stops, then withdraw 1-2 mm before tightening the nut.
uECD		Be sure that the Makeup gas adapter is installed with the 19 mm dimension shown. Thread the column nut with column into the make-up gas adapter. Insert the column all the way into the mixing liner until it stops, then withdraw 1-2 mm - Use 2 wrenches to tighten the nut.Re-tighten after heating. The 70 mm dimension shown is provided for very narrow columns (<100um) which may go all the way through the mixing liner.
FPD		Install the column into the Column Measuring Tool Provided. Trim the column such that 1 mm extends from the end of the tool. Tighten the column nut and mark the position of the column at the back of the nut. Install into the detector base.

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Questions…

Thank you for your attention