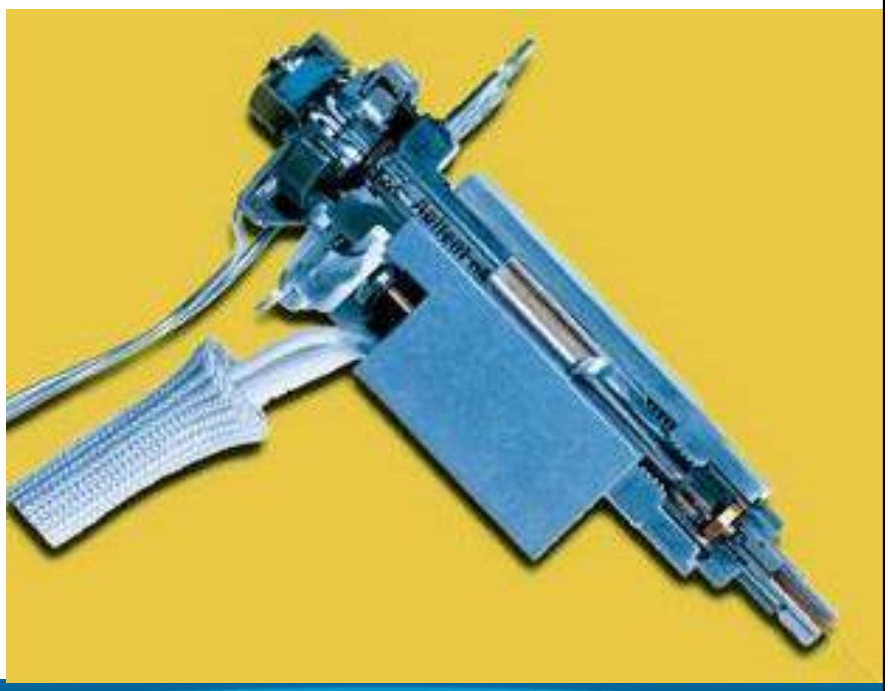
Maintaining Your

Split/Splitless Injection Port



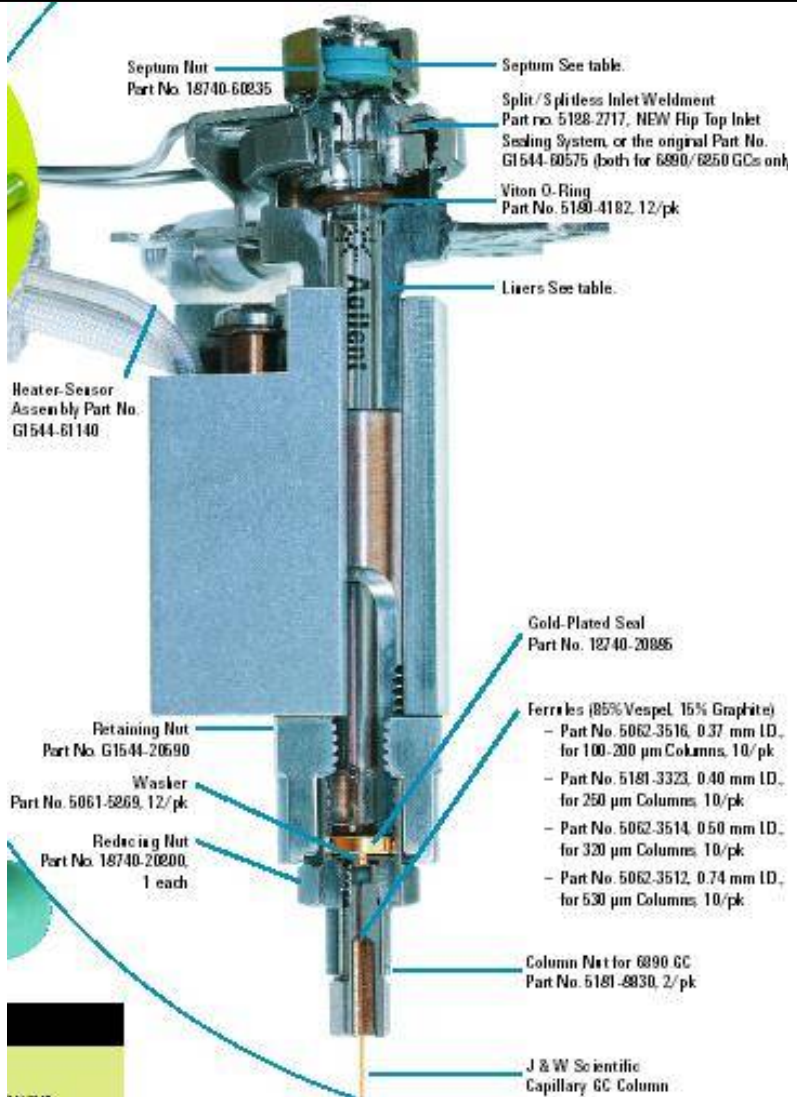
Agilent Technologies

So, Why Do I Have To Do Maintenance?

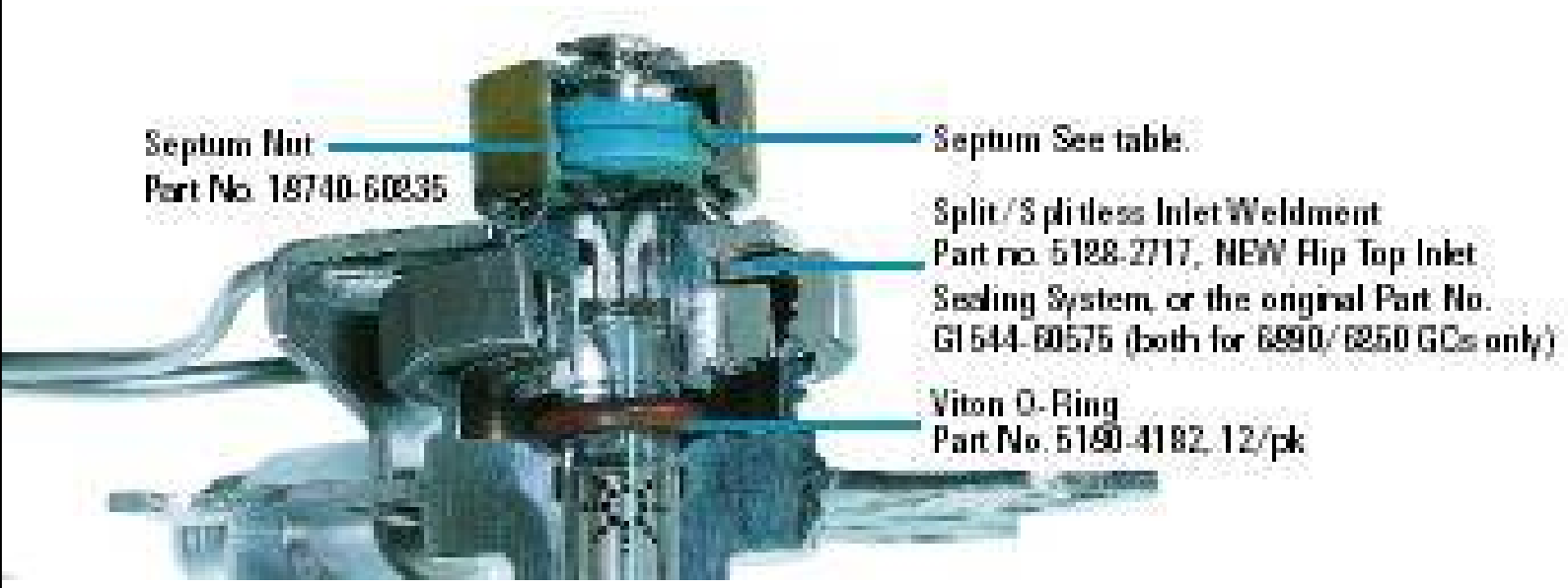
1. Things get dirty liners, column, gas lines, traps, etc.

2. Things wear out septa, syringes, nuts, ferrules, o-rings, etc.

Split/Splitless Injector Parts



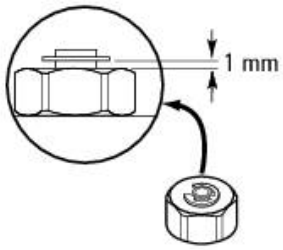
Septum & Septum Nut



Viton O-Ring & Weldment Nut (or Flip-Top)

Leaks Due to Septum Nut

- With repeated use, conical needle guide gets worn, out of round, and needs replacement as 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.
- non-Agilent Autosamplers (ours are precisely aligned), manual injection, larger gauge syringes
- Replace septum nut annually for peace of mind.

Turn Top Inlet Sealing System on NEW 7890

- Fast/Easy Split/Splitless Inlet Maintenance

For Easy Liner Maintenance on 5890/6890

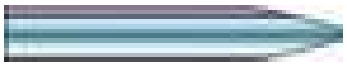
NEW!!

Flip Top for Split/Splitless injection ports

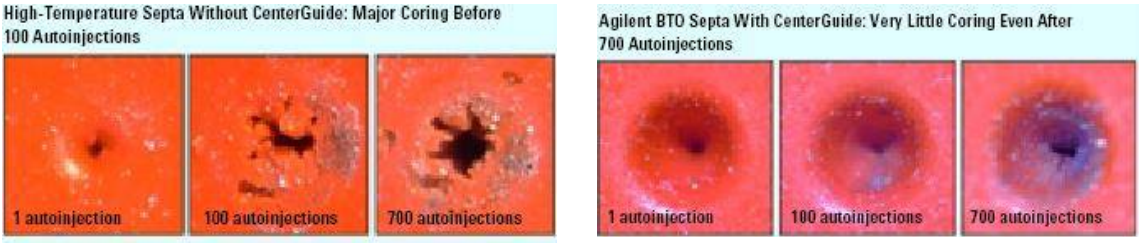
- 30 sec liner change out
- No more hunting for that
- Saves fingers from getting burned
- Increases instrument up time

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.

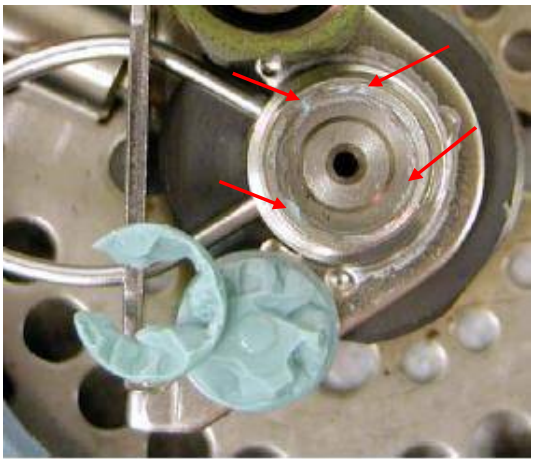


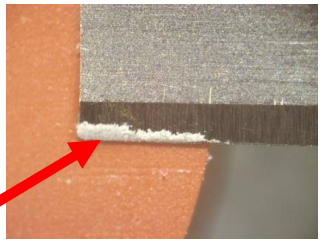
- Use Agilent CenterGuide Septa. The molded hole minimizes septa coring, counter-intuitive, but true.



Tips to Maximize Septum Life, Minimize Septum Leaks

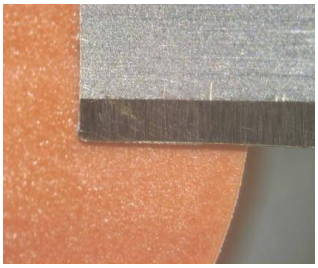
- Use Non-Septa with Proprietary Plasma Treatment



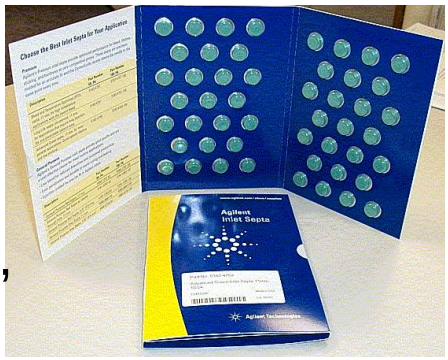


Talcum Powder!

NEW!



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



- Packaging eliminates contamination of septa,
- 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.

- Proactively change inlet septa.

Or Go Septumless! Merlin Microseal

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

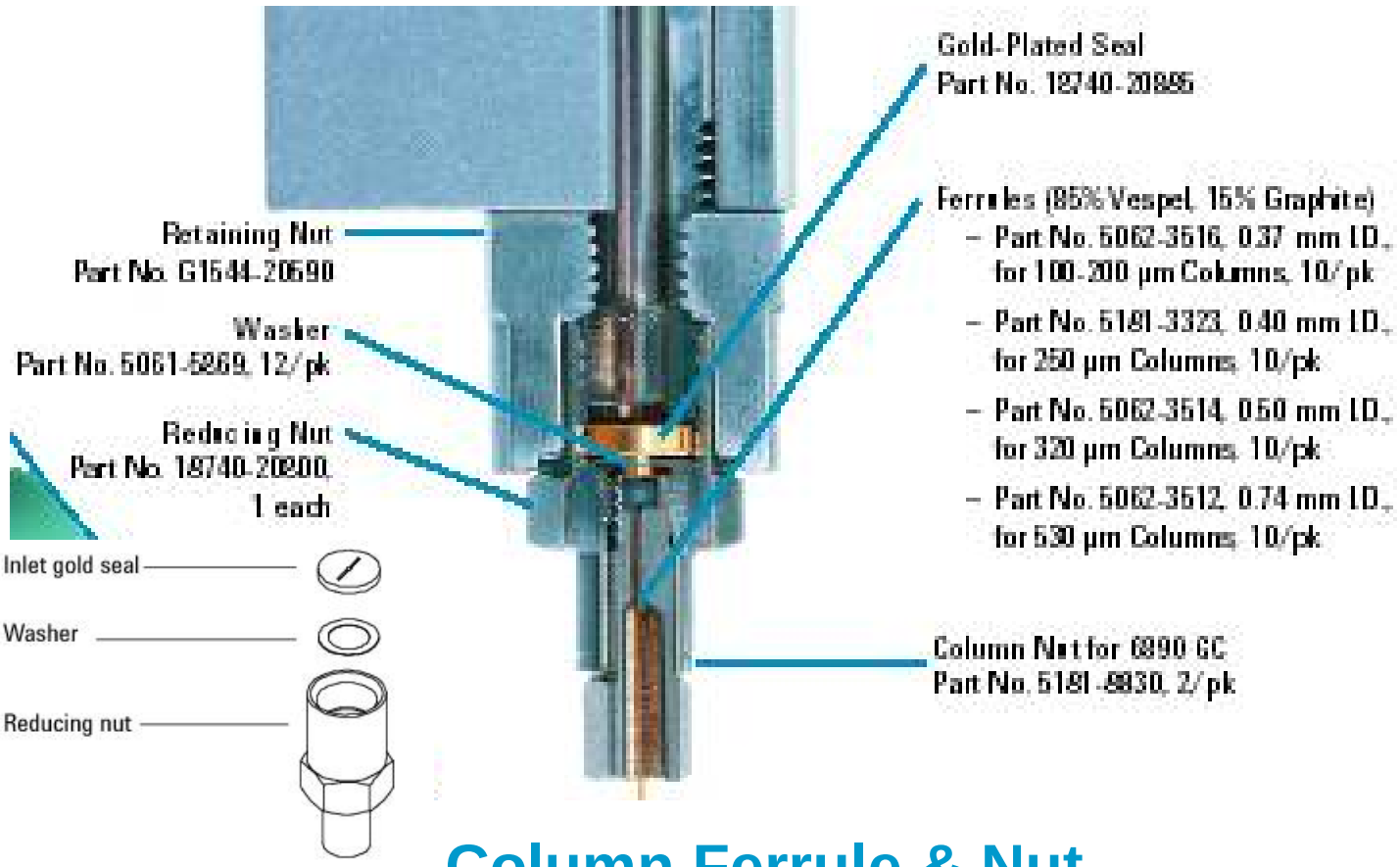
More than 2000 injections, depending on samples and operating conditions

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

Gold Seal & Washer

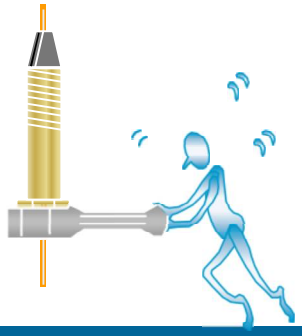


Column Ferrule & Nut

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.

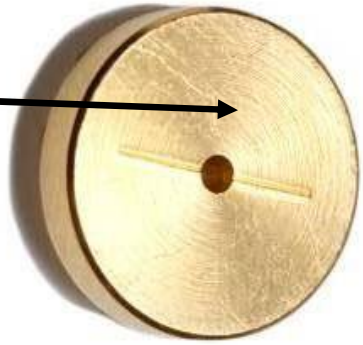
- Gold seals are designed for one use, knife edge cuts into gold layer giving leak tight seal w/o shrinkage or potential organic contaminants from polyimide out-gassing/degradation.
- Re-using could result in overlap in seal rings, resulting in a leak.
- Over-tightening of fittings

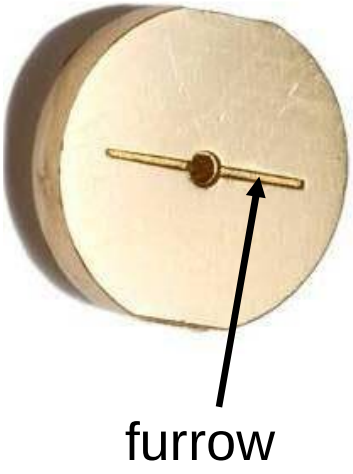


Certified gold inlet seal, 5188-5367

Gold Seal

- New Agilent Gold Plated Seal
  - Metal Injection Molded with consistently smooth surface no radial grooves with reproducible furrow.
- New Gold Seal Kit 5188-5367
  - Includes the washer.





Ferrule Pre-swaging & MS Interface Installation Tools

- [illegible]ength of column into the fittings, every time
- For graphite or metal ferrules

Metal ferrule tool
G3440-80218

Graphite ferrule tool
G3440-80217

Agilent Technologies

Graphite Ferrules Have a Down Side Too!

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

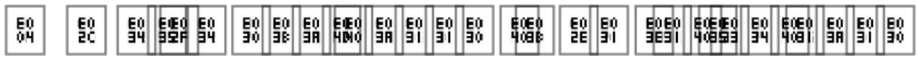
Con
- Can flake, fall apart or extrude
- Permeable

NOT recommended with MS or ECD

Leak Test

- Pressure Test
- MS Air/Water
- Leak Detector
  - only 310g/11oz
  - 1 second
  - for hydrogen and helium
  - hours of life

- Could install the column easily
- Finger tighten, without tools that cause over tightening
- And make leak free connections



- Every time?

Self-Tightening Column Nuts

Innovative spring-driven piston continuously presses against ferrule maintaining a leak-free seal

No retightening needed after repeated thermal cycles

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

Low-torque seal prevents sticking or crumbling during removal

Leak Free = Lower column bleed: Longer column life

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

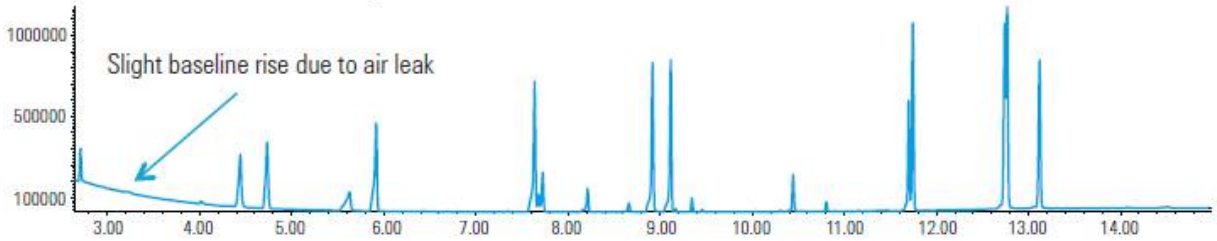
Self Tightening Column Nuts

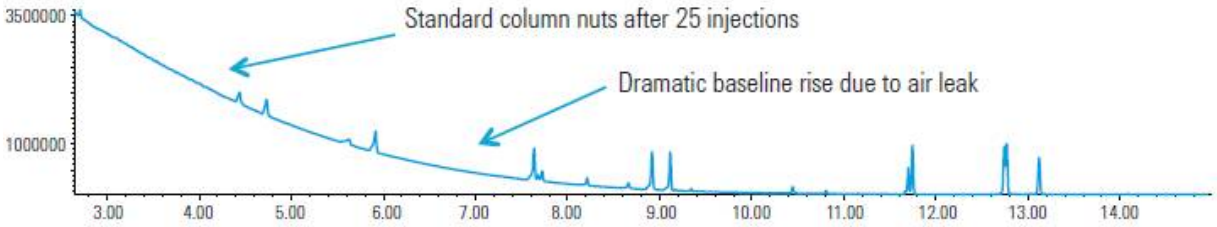
For inlet or detector
p/n 5190-6194

For mass spec transfer line
p/n 5190-5233

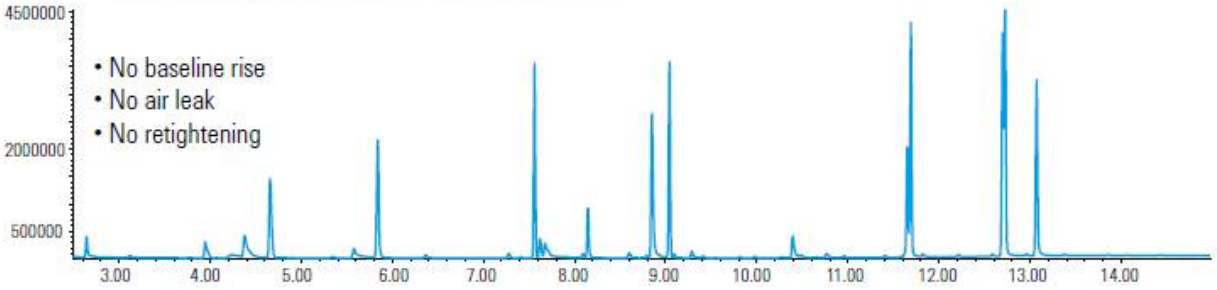
Self-Tightening Column Nuts

Standard column nuts new fitting





Agilent Self Tightening Column Nuts after 400 injections



problems.

Where Does it Get Dirty?

Here

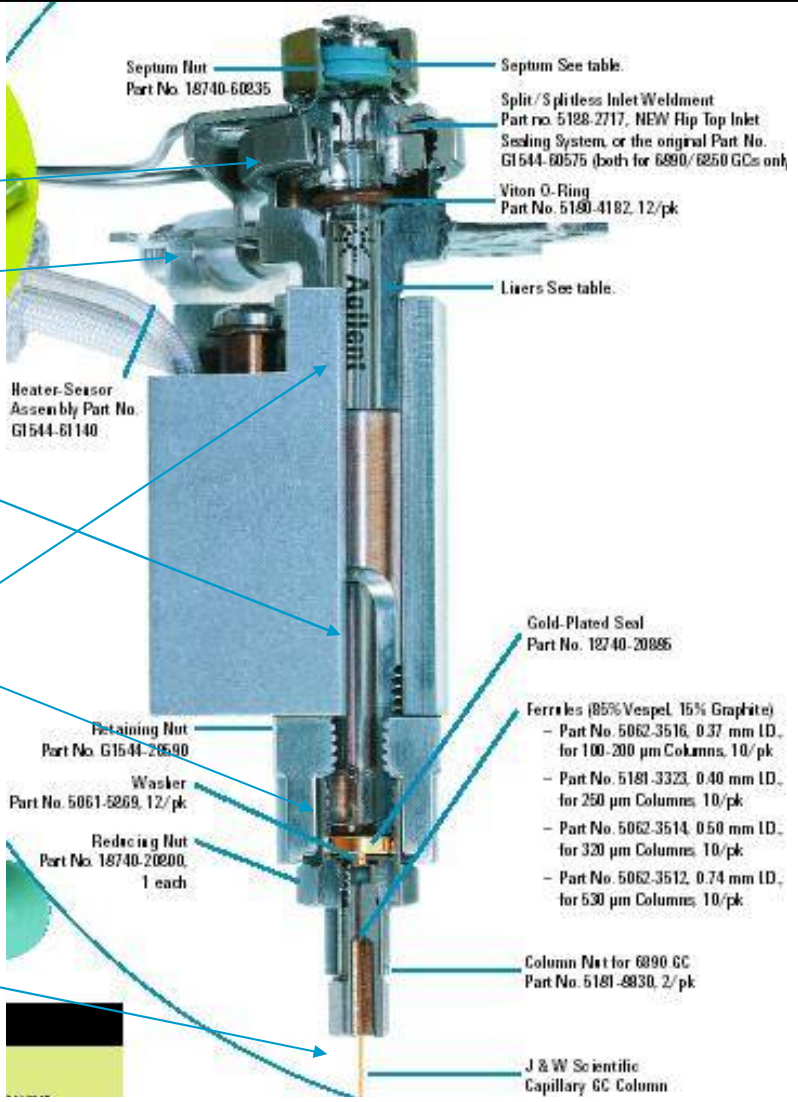
Here

Here

Here

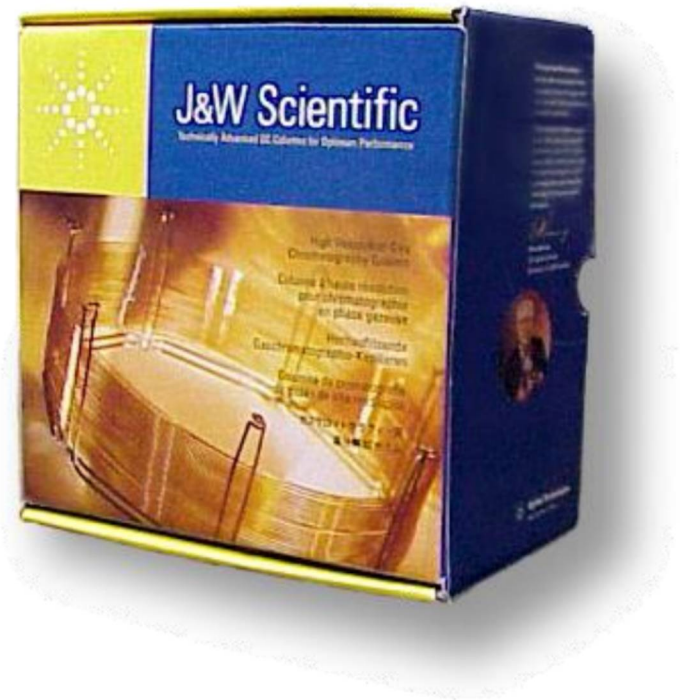
Here

Here



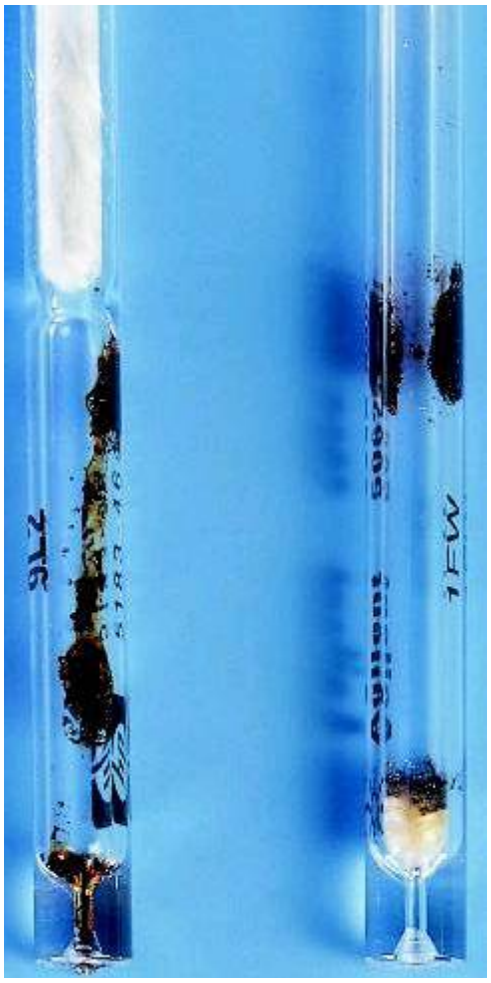
What Are You Injecting!?

Time to talk about Liner & Column



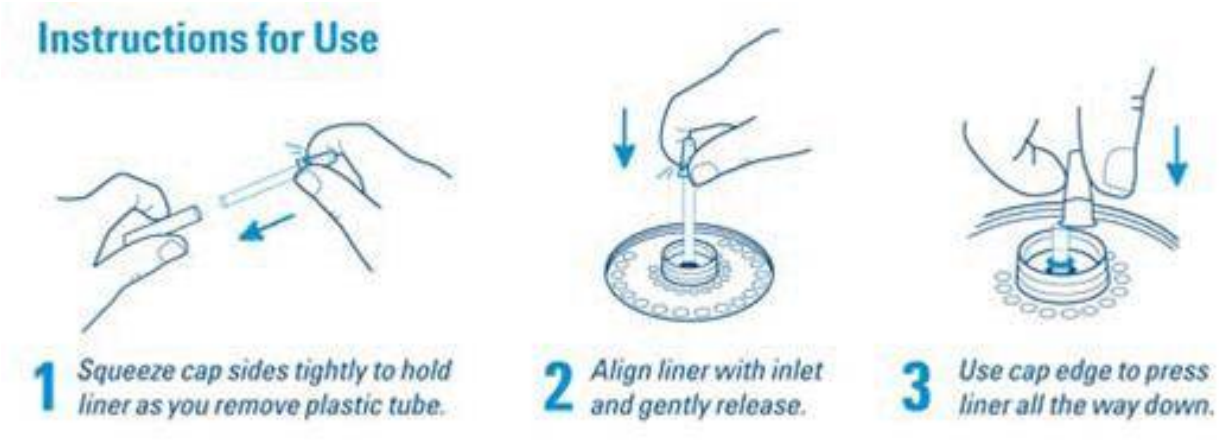
Liner Maintenance

- 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

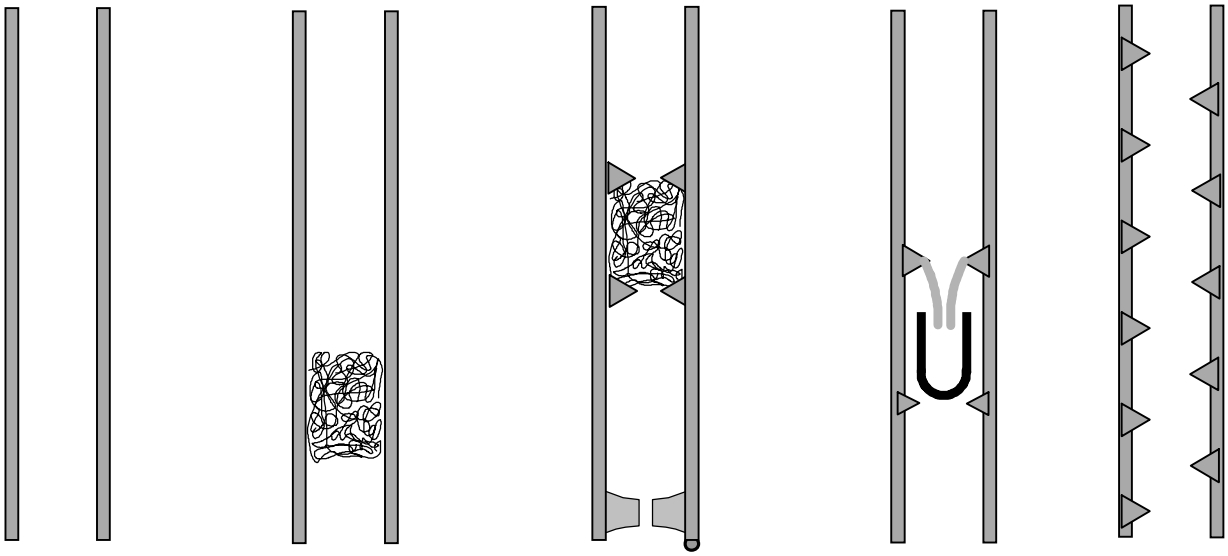


AND Ultra Inert Liners

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



Split Liners



Straight tube

Straight tube with glass wool

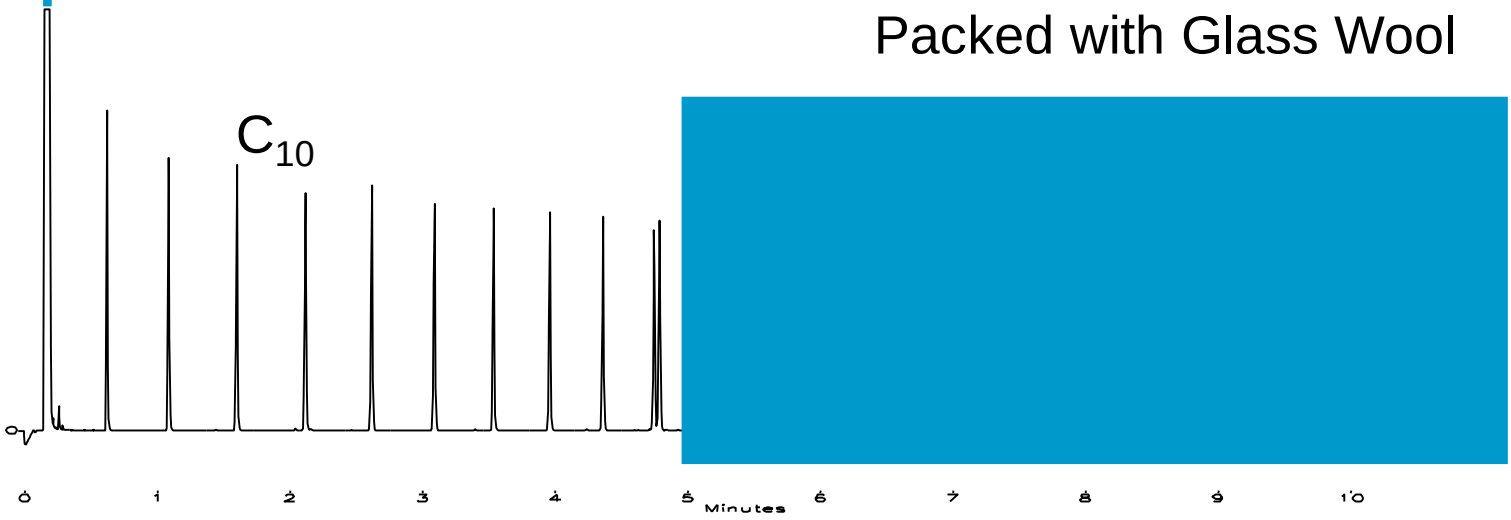
Fixed glass wool

Inverted cup

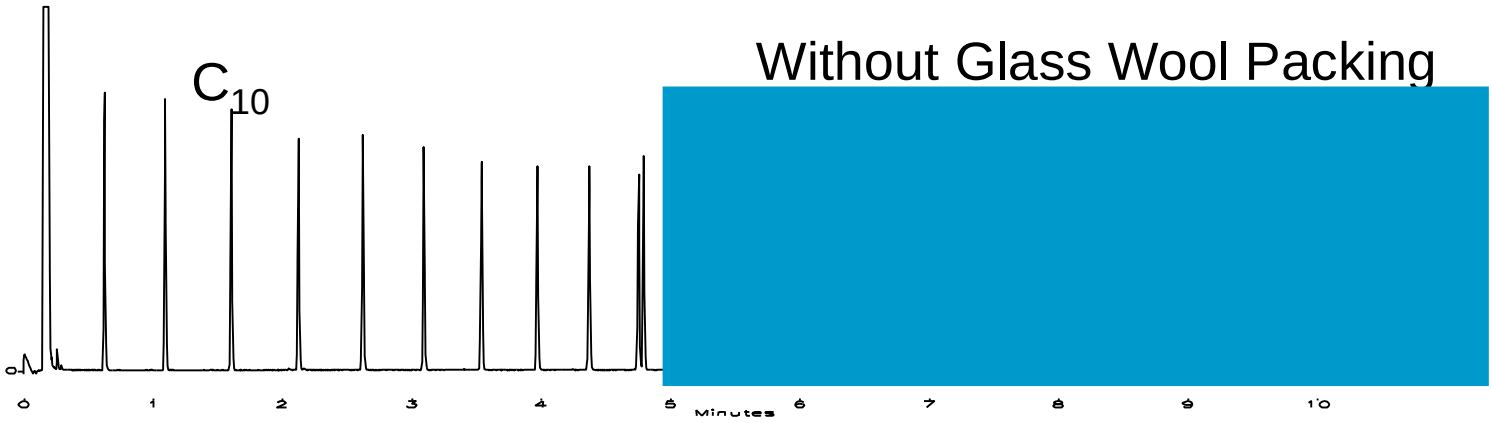
Baffle

Split Liner

Packed with Glass Wool

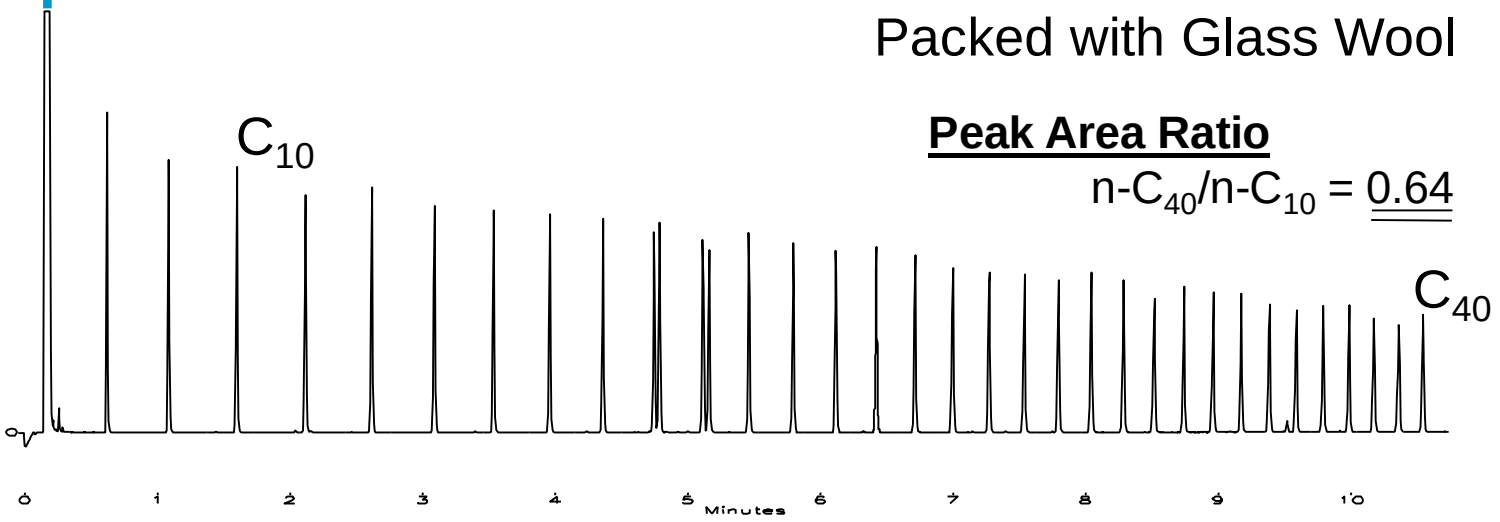


Without Glass Wool Packing



Split Liner

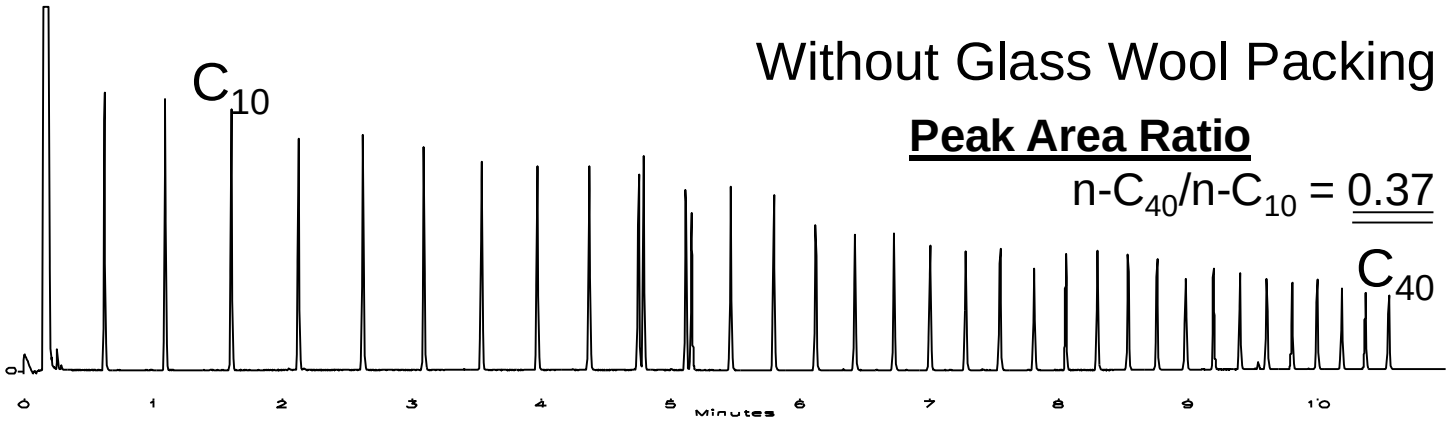
Packed with Glass Wool



Peak Area Ratio

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

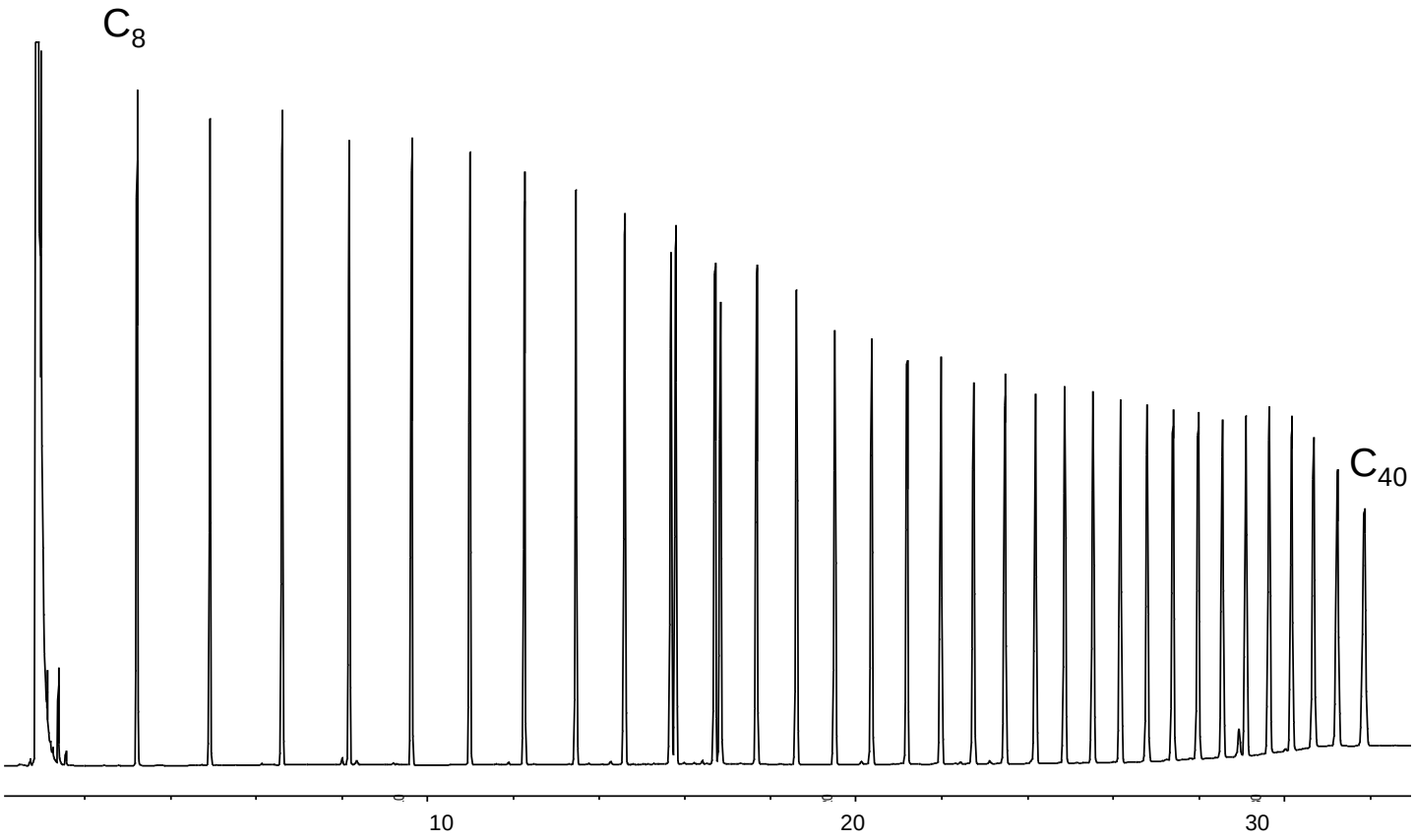
Without Glass Wool Packing



Peak Area Ratio

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

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

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:

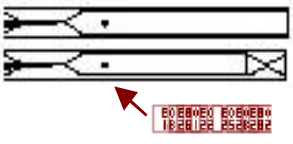
- Helps in volatilization of high MW components
- Increases mixing

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

Causes of Short Column Life

Breakage/damage to the polyimide (rare)

Stationary phase exposed to oxygen

Exceeding upper temperature limits

Chemical damage to the stationary phase

Non-volatile residues (contamination)

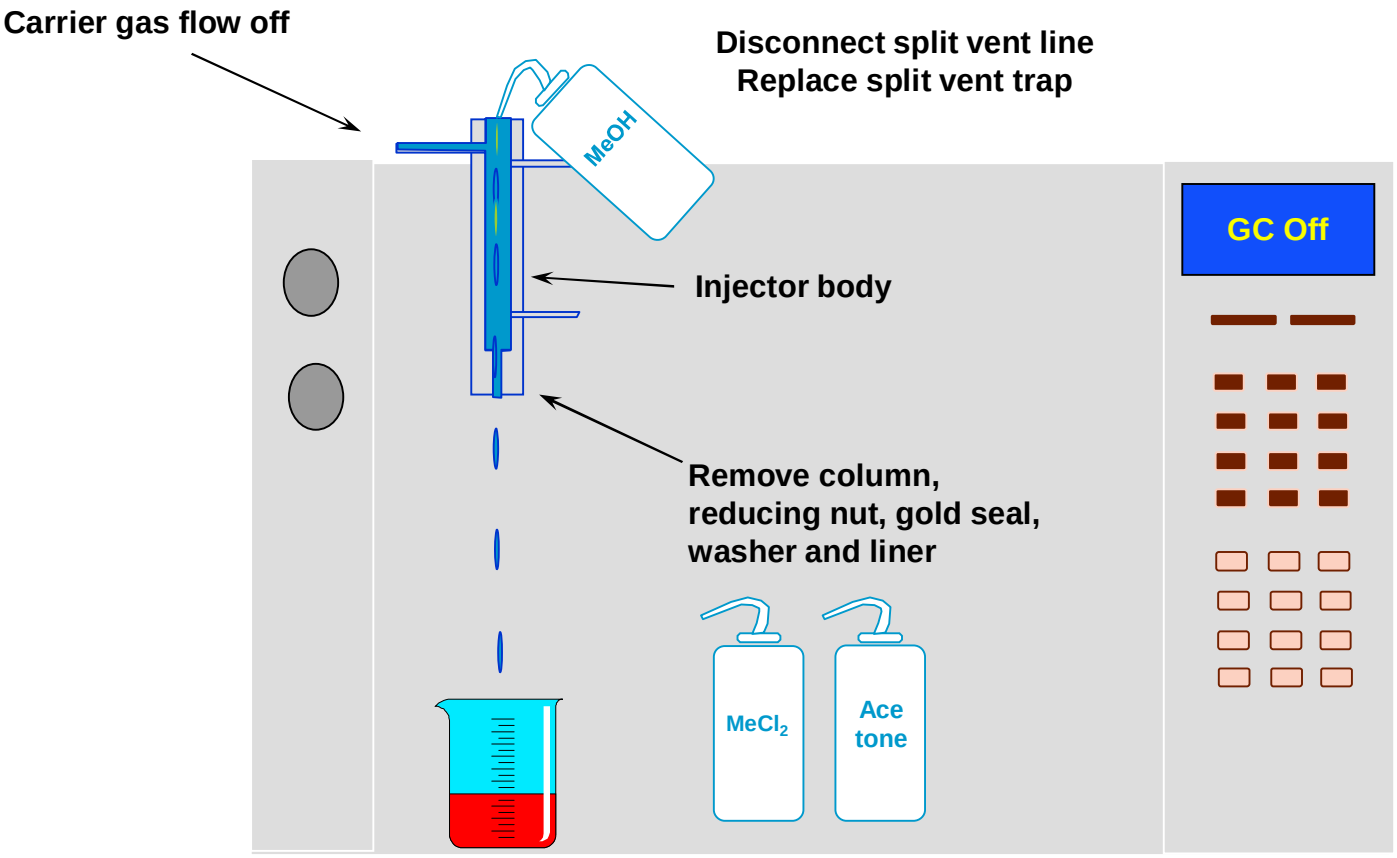
Common Care and Maintenance Scheme for GC Columns

1. Bake out the column for no more than 2 hours.


1ft of the inlet end of the column.

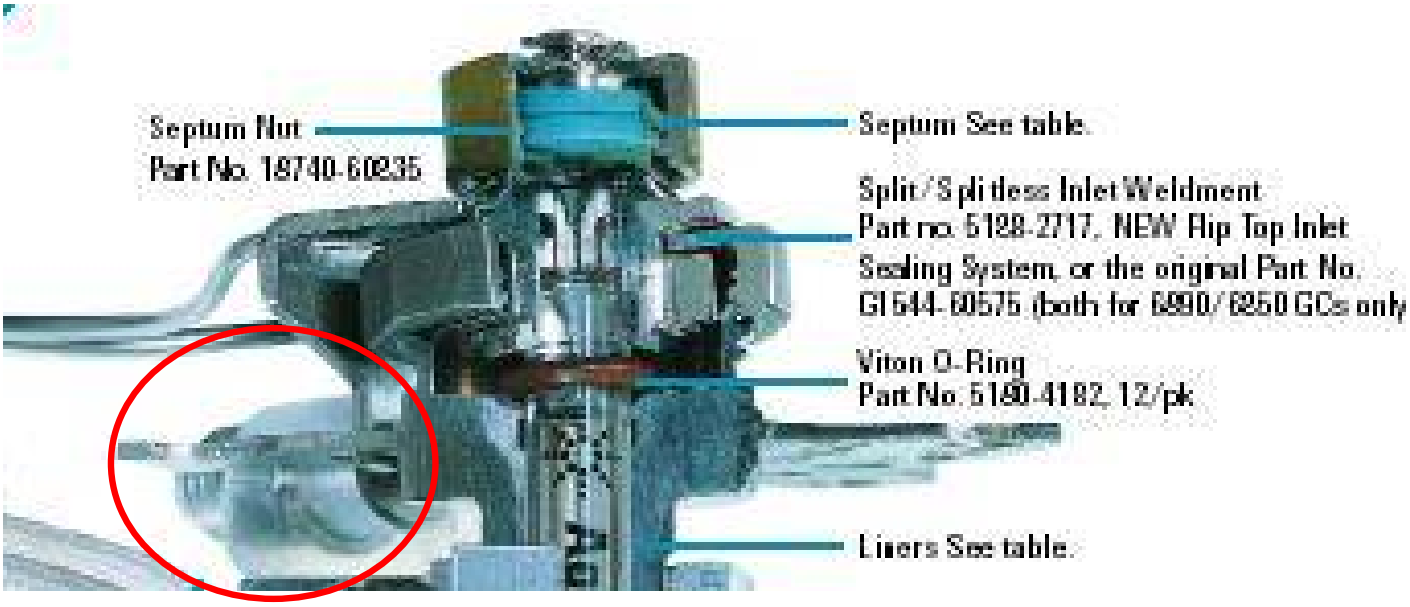
3. Cut off more column. (repeat as necessary)

Cleaning the Split/Splitless Injector



Finding the Split Vent Trap

Follow the split vent line back to the EPC



Finding the Split Vent Trap

Remove cover at Split Vent

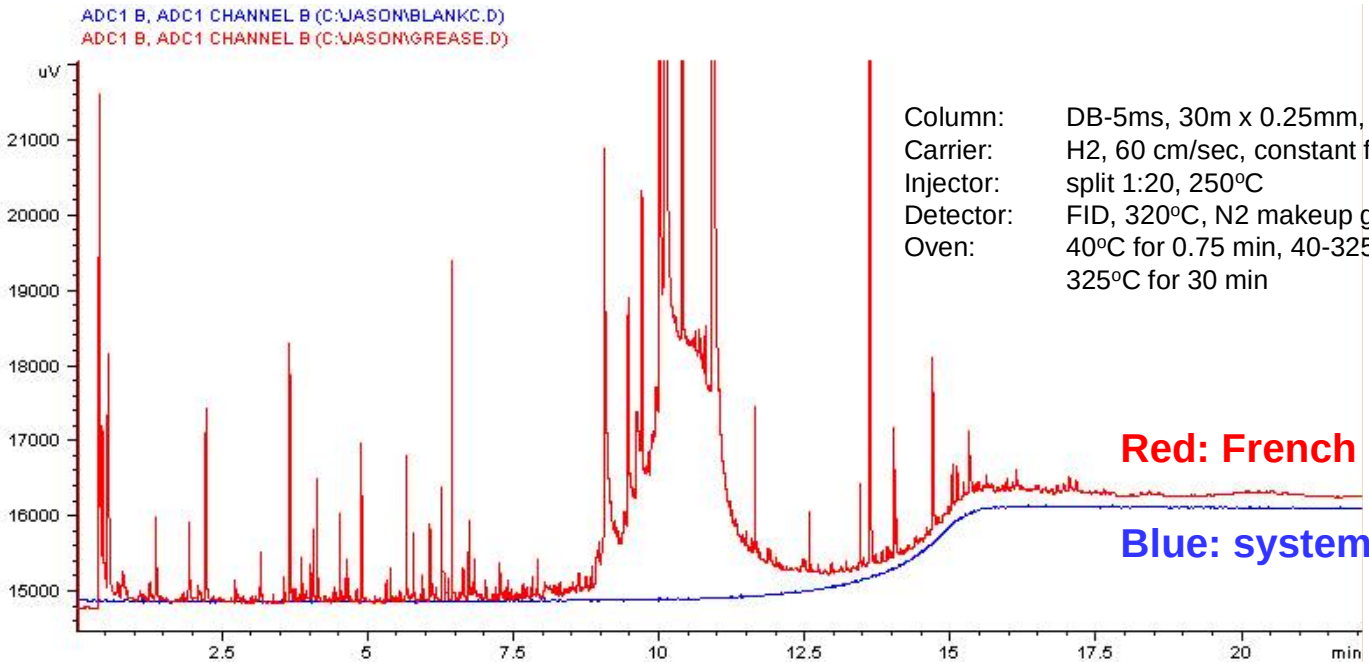
Replacing the Split Vent Trap

Finger Tight Knurled Nut

G1544-80530

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

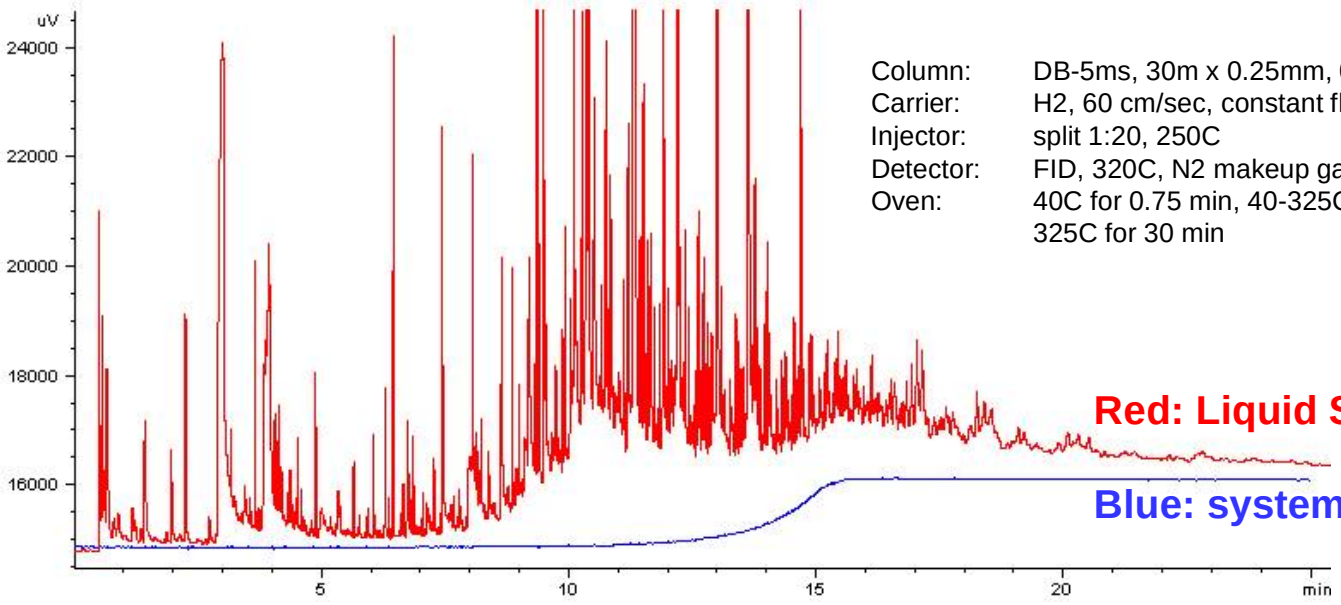
Red: French Fry

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

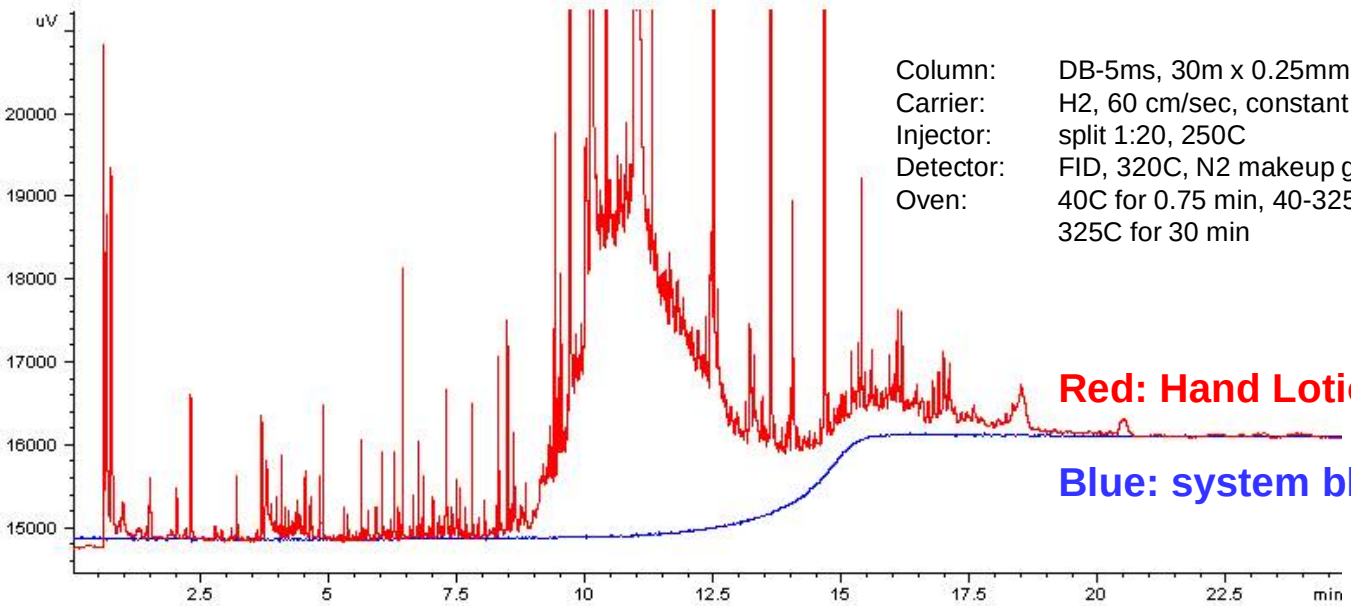
Red: Liquid Soap

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

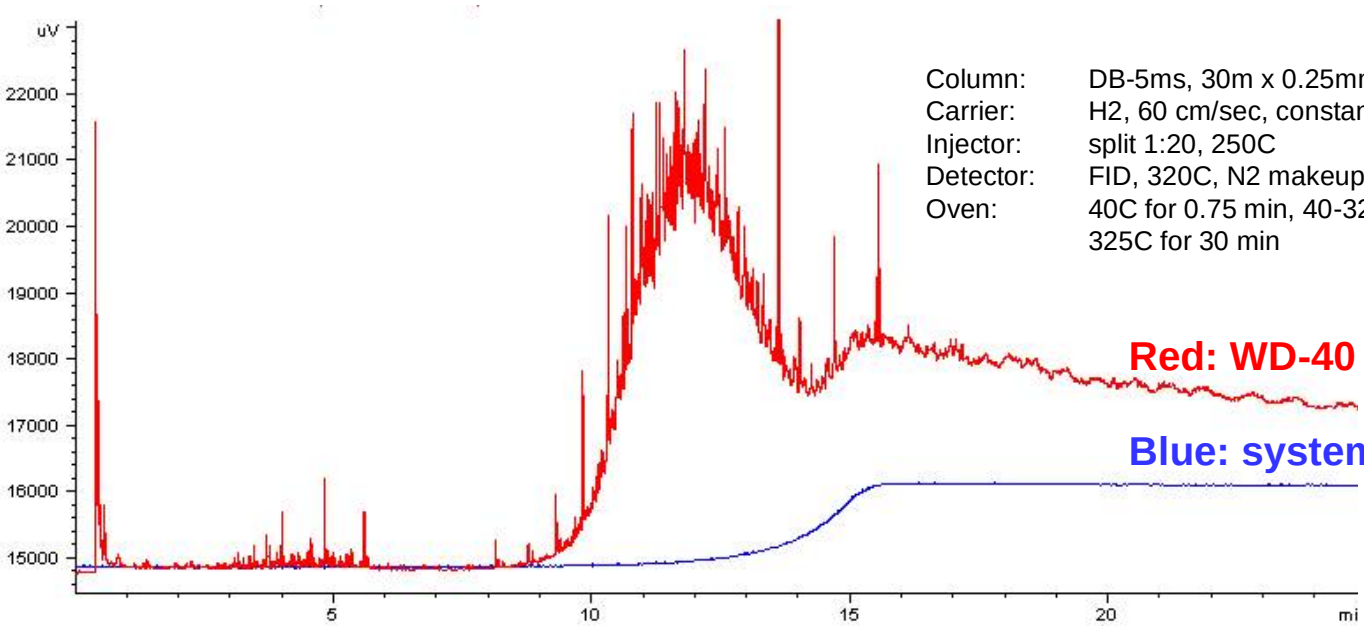
Red: Hand Lotion

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



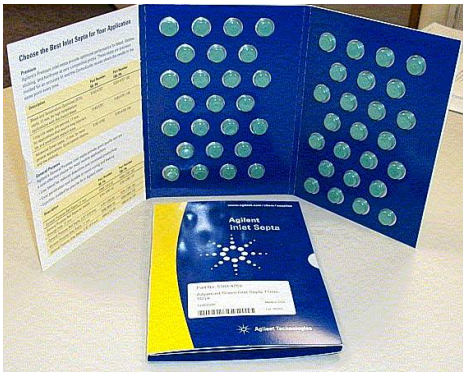
Column:	DB-5ms, 30m x 0.25mm, 0.25um
Carrier:	H2, 60 cm/sec, constant flow
Injector:	split 1:20, 250C
Detector:	FID, 320C, N2 makeup gas
Oven:	40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

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.


Agilent
Inlet Septa


Agilent
Ferrule 0.4mm ID Vespel/Graphite 85%/15%
(short) 10/pk fits Col.ID 100 - 250µm
Part No. 5181-3323
0853195
Made in USA
www.agilent.com/chem/supplies


ON10149


Agilent UltiMetal Plus
Flexi-Metal Ferrules
AGILENT INERT FLOW PATH
Agilent Technologies
Column id
0.10-0.25mm

Conclusions for a Problem Free GC

- Start With High Quality Consumables (UI?)
- Never Have a Leak (no oxygen)
- Never Inject Anything (dirty or reactive)

- Do Routine Inlet Maintenance Often

TECHNICAL SUPPORT

1-800-227-9770, #3

1-972-699-6423 (Daron)

1-866-912-6701 (toll free)

E-mail:
Daron_Decker@Agilent.com