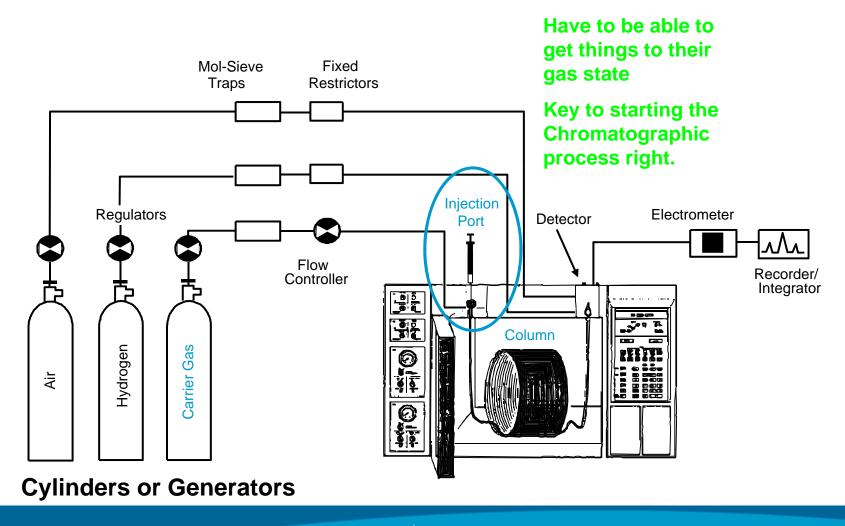
Tips and Tricks of Injector Maintenance

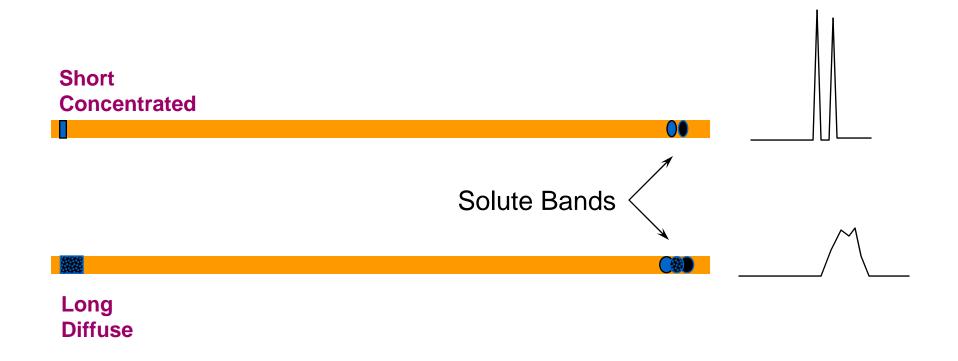
Abby Folk Agilent GC Technical Support August 2009



Typical Gas Chromatographic System



Influence of Injection Efficiency



Same column, same chromatographic conditions

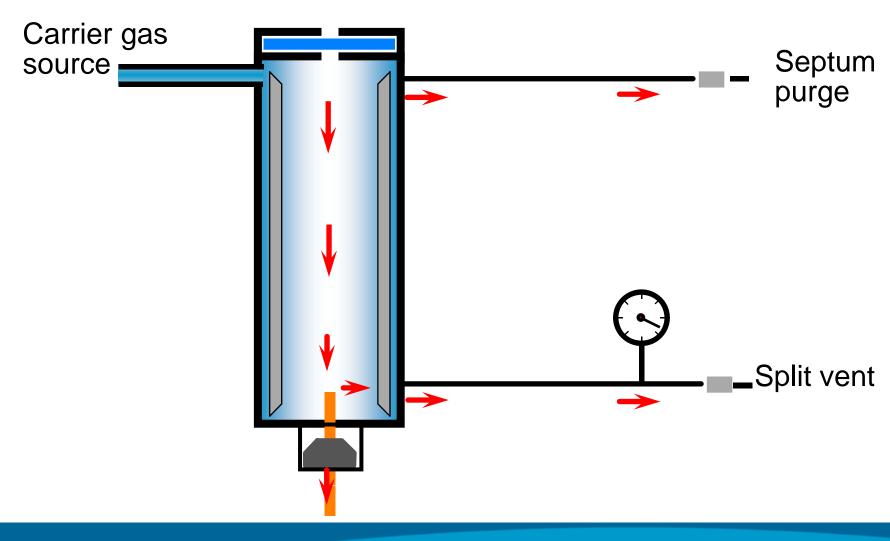
Injectors

Split

Splitless

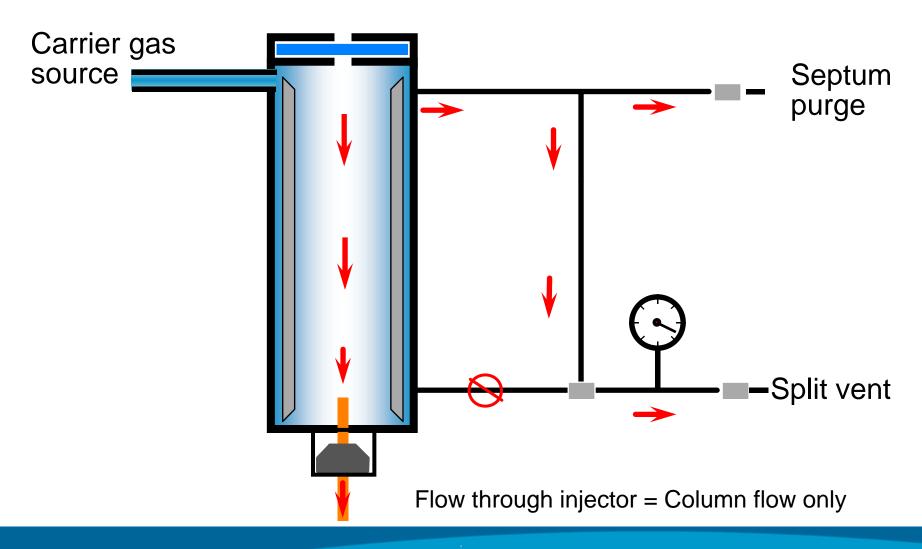
Split Injector

Flow Path



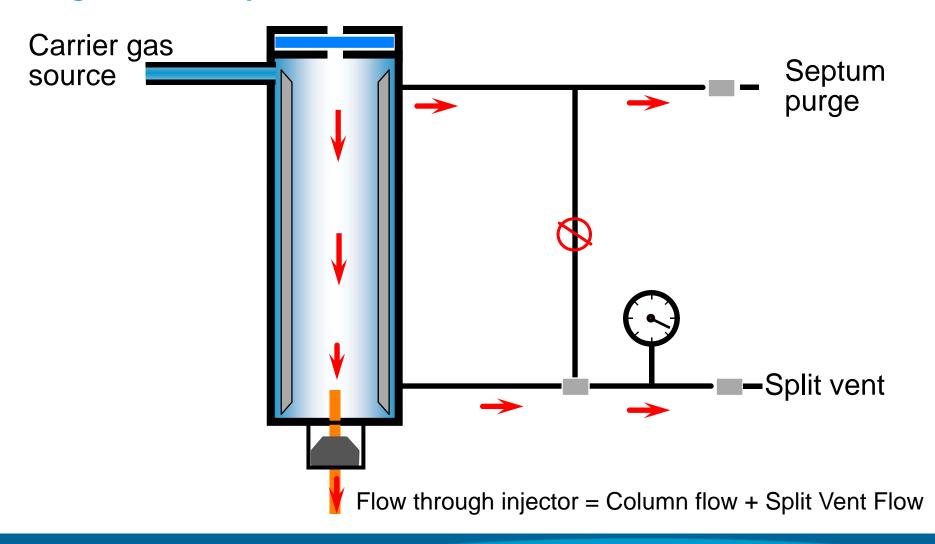
Splitless Injector

Purge Off At Injection

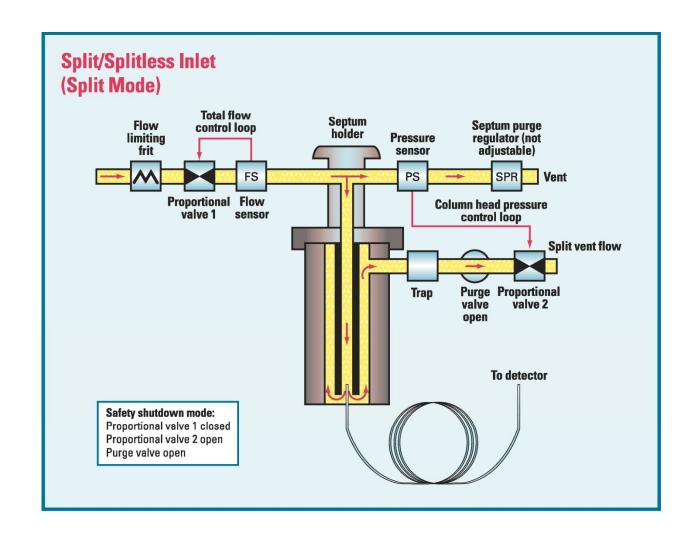


Splitless Injector

Purge On After Injection



Split/Splitless Injector



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 *Gold Standard* GC Autosampler Syringes New syringe Pub #5990-3563EN

Improved Quality



Improved Packaging

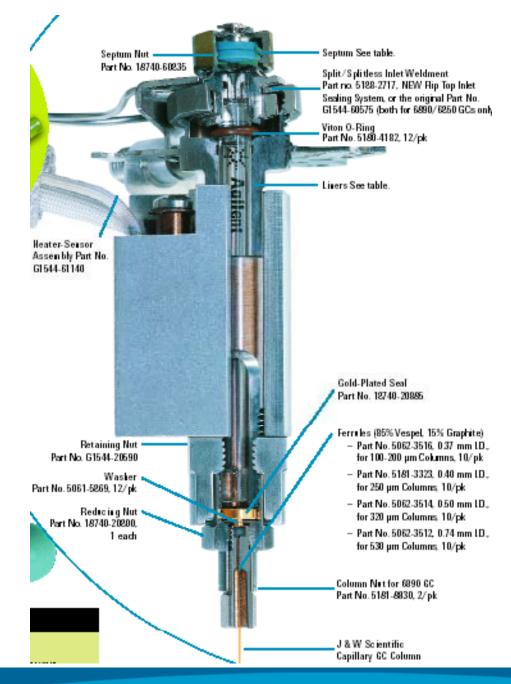




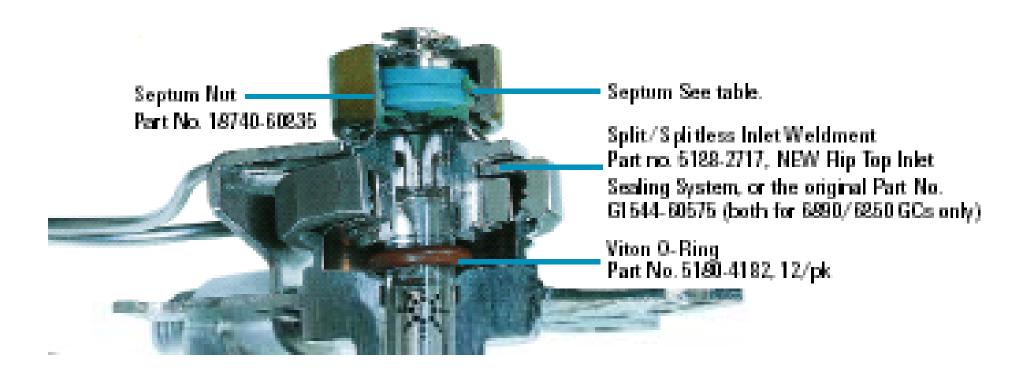


BUT...They Don't Last FOREVER!!

Split/Splitless Injector Parts

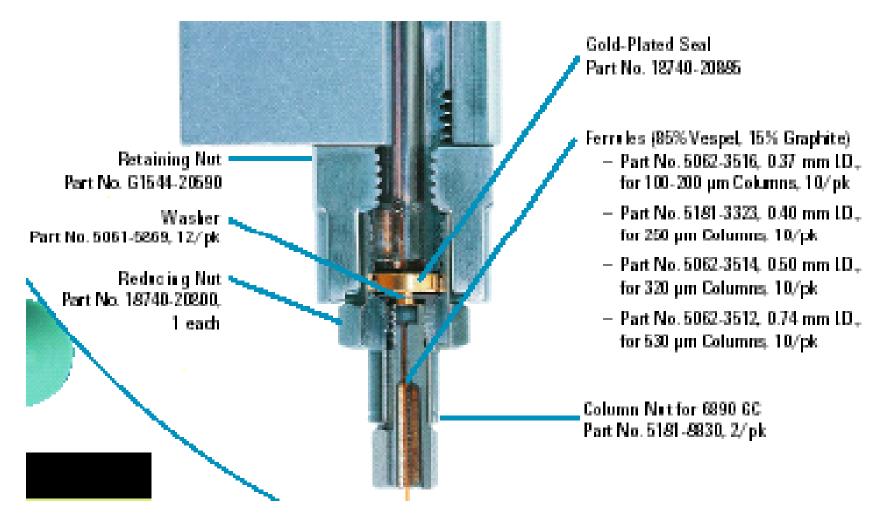


Septum and Septum Nut



Viton O-Ring P/N 5188-5365

Gold Seal & Washer



Column Ferrule & Nut

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 P/N 5188-5365

Inlet O-ring Flip Top 5188-5366



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



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.
- Re-using could result in overlap in seal rings, resulting in a leak.
- Over-tightening of fittings









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



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.



Tips to Maximize Septum Life, Minimize Septum Leaks

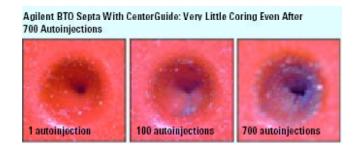
Use Agilent Gold Standard, HP Point, 23-26 gauge taper syringes. The point style cores septa significantly less when used with CenterGuide Septa. Taper minimizes septum coring/wear.
 HP-Point Style

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

Solid Septum

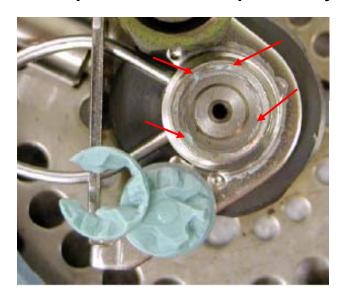
High-Temperature Septa Without CenterGuide: Major Coring Before 100 Autoinjections 1 autoinjection 100 autoinjections 700 autoinjections

CenterGuide Septum



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!





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

Tips to Maximize Septum Life, Minimize Septum Leaks

Other Benefits of Agilent's 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

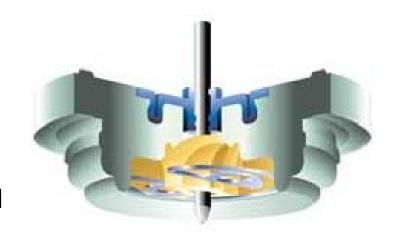
Low bleed, longer life alternative to standard septa for split/splitless injection

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







So, Why Do I Have To Do Maintenance?

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

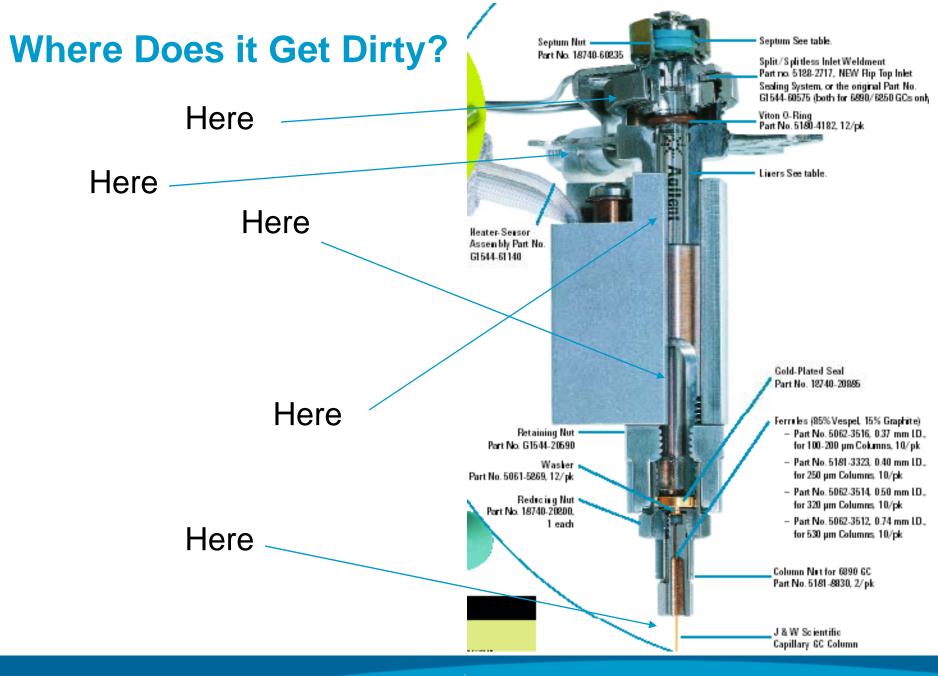
2. Things get dirty – liners, column, gas lines, traps, etc.

The BIGGEST Problem in GC is...

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

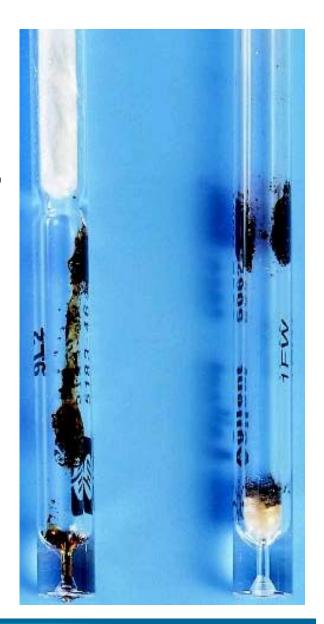
....therefore, don't inject anything and you'll never have problems.

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

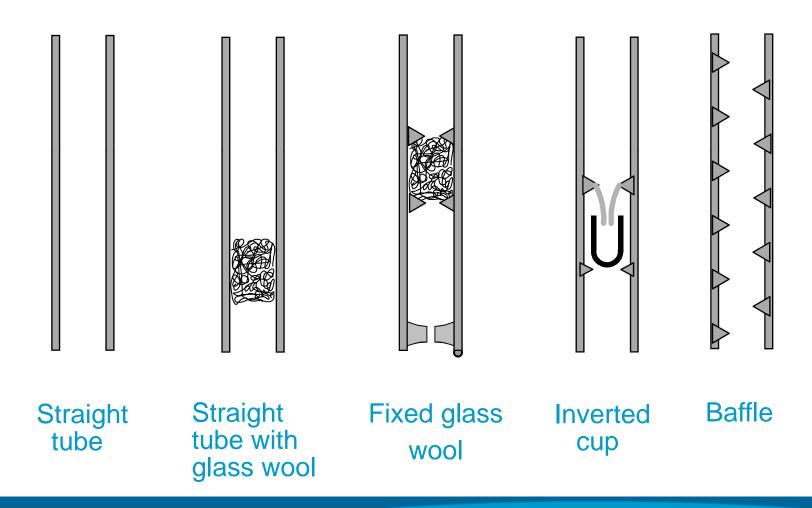


Liner Maintenance

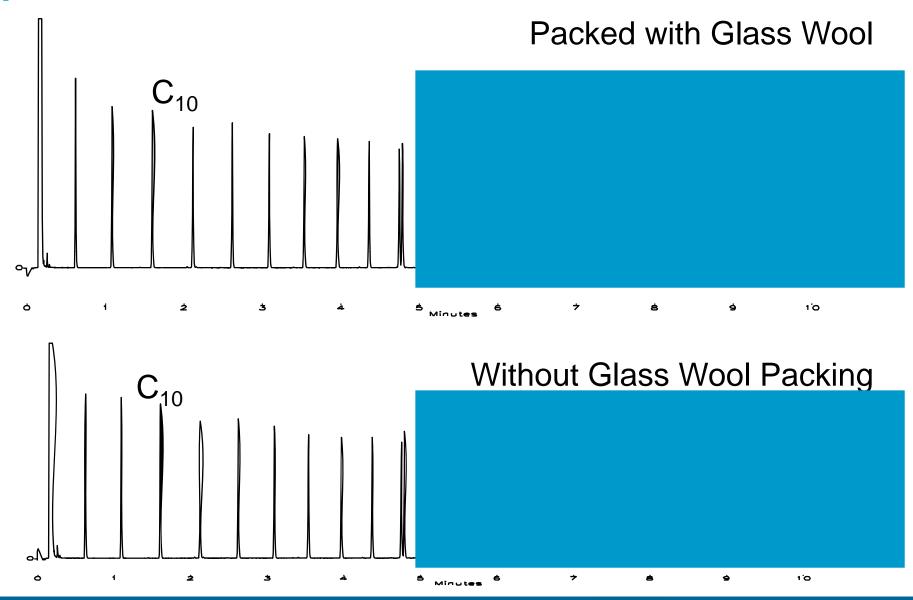
- •Liners become contaminated with use, collecting non-volatiles, salts, excess reagents, etc., or become damaged/cracked.
- •Should inspect and replace liners often.
- Handle with gloves and forceps.
- Insert into or remove liners only from cool injection ports.
- Replacing with a new liner is recommended,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



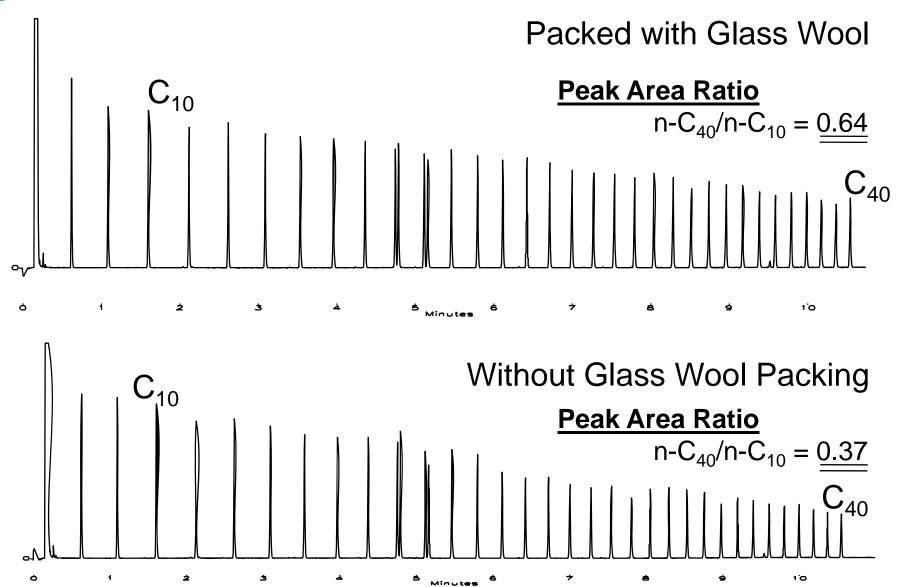
Split Liners – What's What?



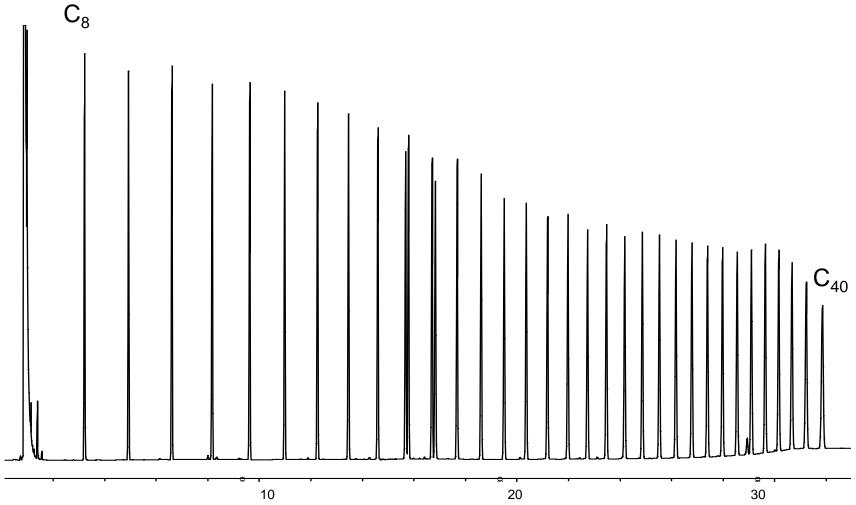
Split Liner



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

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:

- □ pesticides
- ⊠ drugs of abuse, etc.

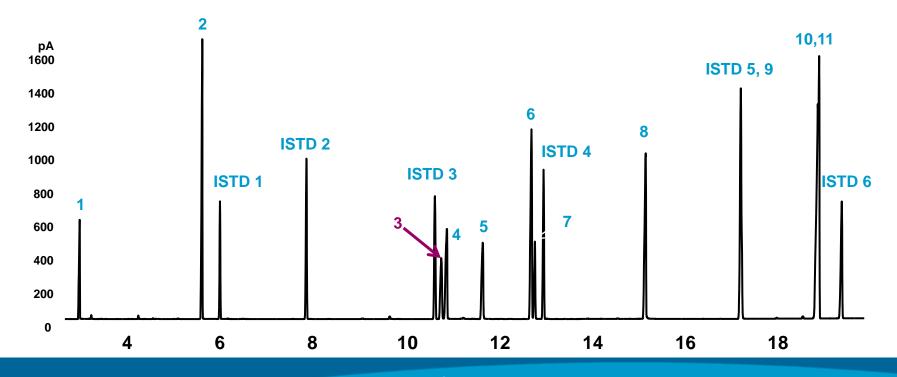


Phenols, for example....in a separation of EPA method 8270 compounds

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

- N-Nitrosodimethylamine
 Aniline
 2,4-Dinitrophenol
 4-Nitrophenol
 4,6-Dinitro-2-methylphenol
 4-Aminobiphenyl
- 7 Pentachlorophenol8 Benzidine9 3,3-Dichlorobenzidine
- 10 Benzo(b)fluoranthene11 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



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

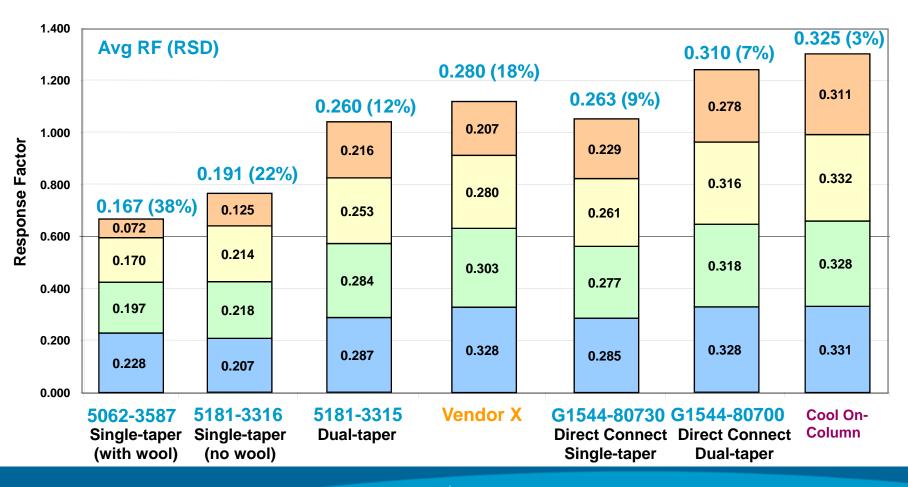
Liner Comparison

2,4-Dinitrophenol Response Factors

■ 160 ■ 80 ■ 20 ■ 5 ng injected

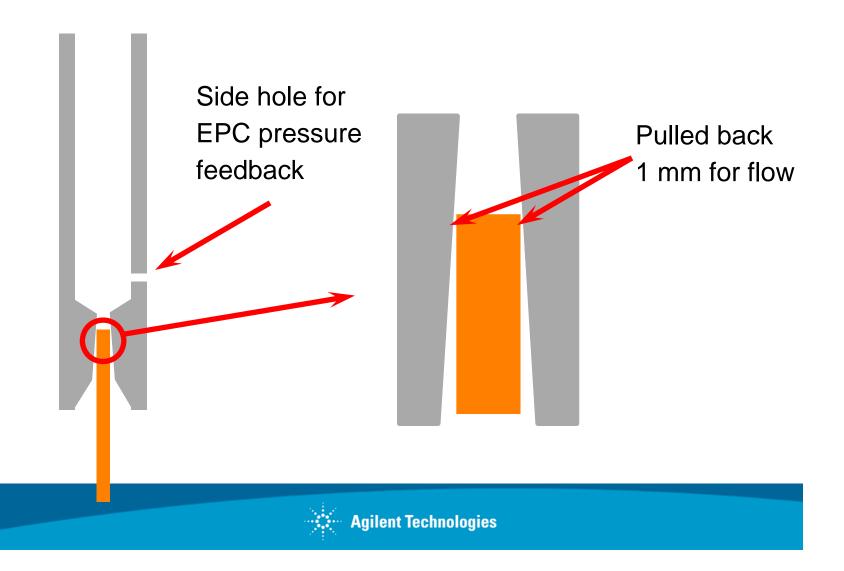
Experimental:

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



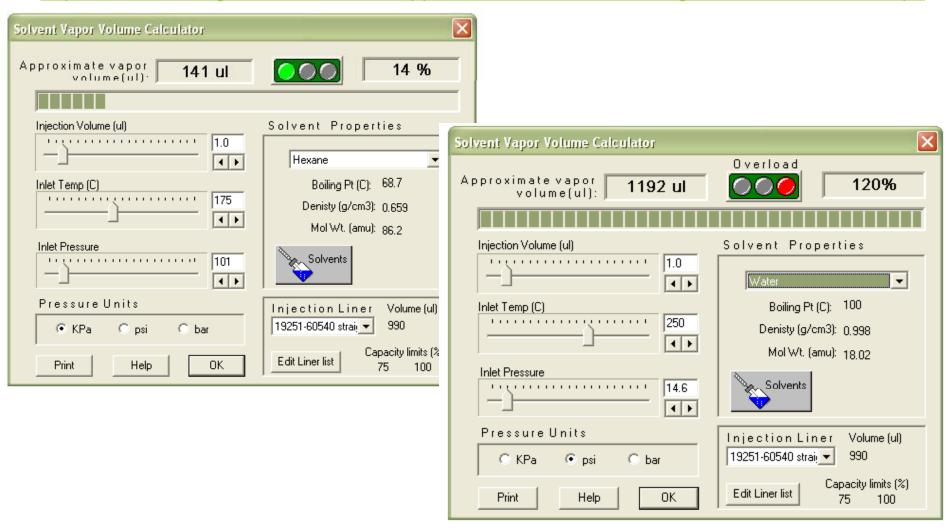
DIRECT CONNECT

Vaporization Liner



Pressure Flow Calculator

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



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 August 31st and get your Flip Top for \$180 (a 64% discount)

Turn Top Inlet Sealing System on NEW 7890



- Fast/Easy Split/Splitless Inlet Maintenance

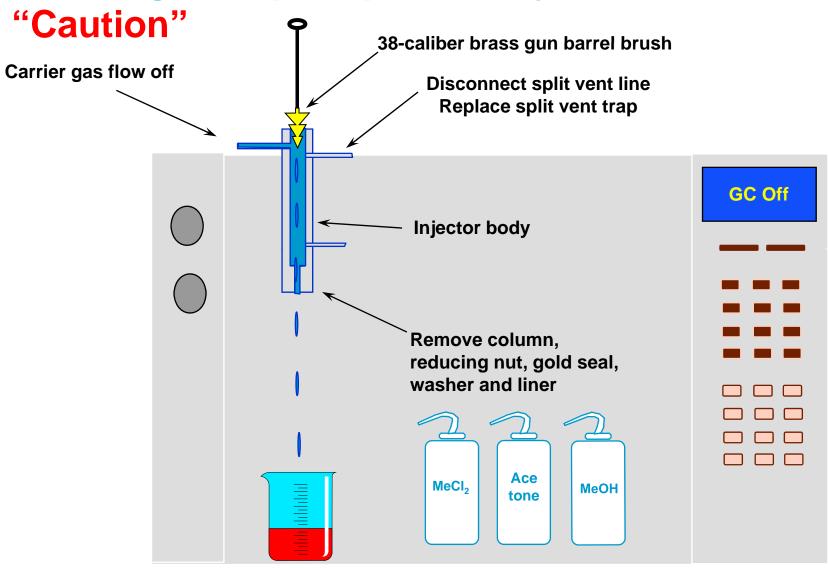
... changing liners has never been easier



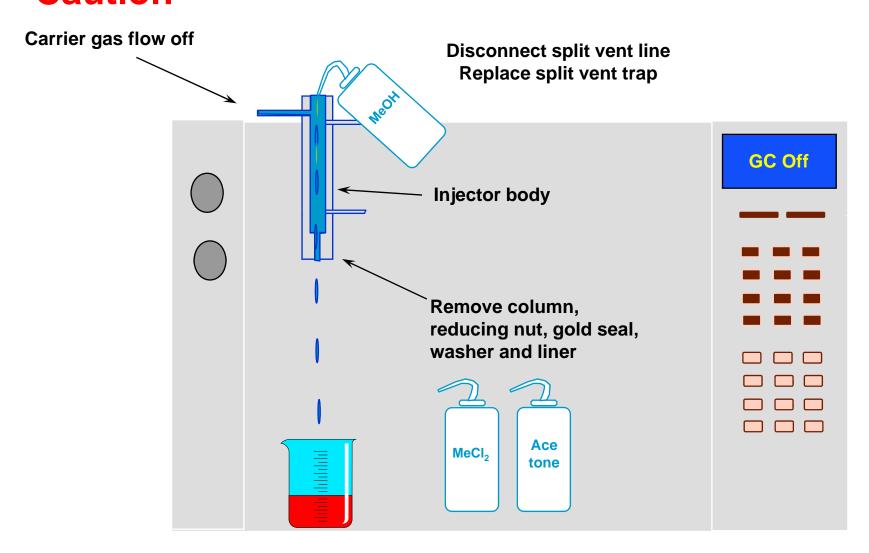
Common Care and Maintenance Scheme for GC Columns

- 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

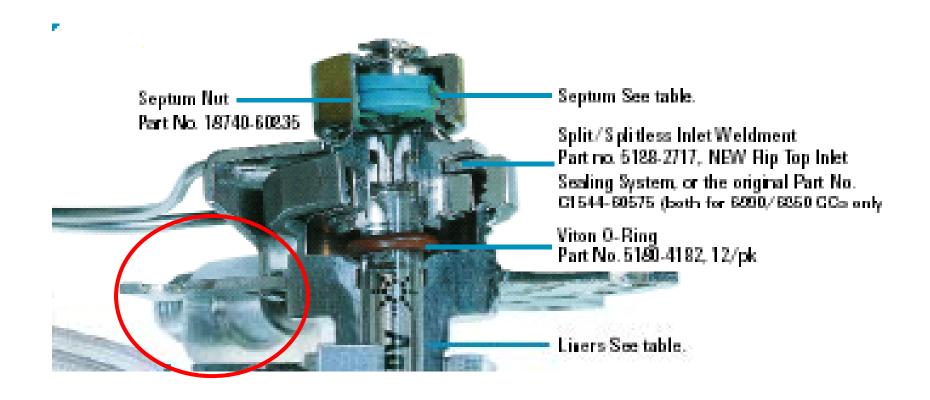


Cleaning the Split/Splitless Injector "Caution"



Finding the Split Vent Trap

Follow the split vent line back to the EPC



Finding the Split Vent Trap

Remove cover at Split Vent

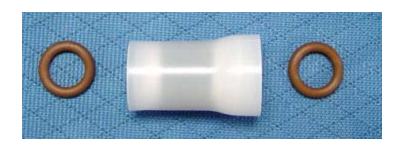


Replacing the Split Vent Trap

Finger Tight Knurled Nut



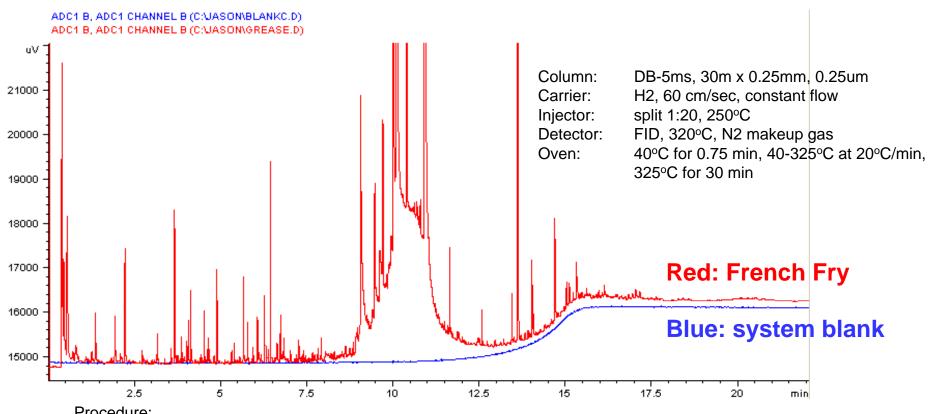
G1544-80530



Be Careful When Doing Maintenance...

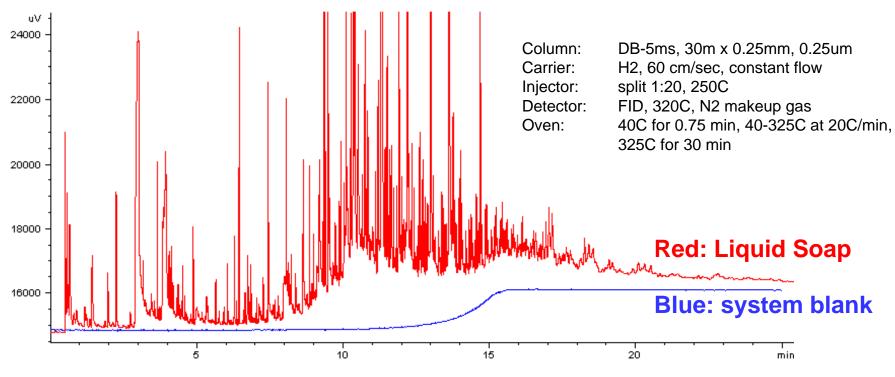
You may be the CONTAMINATOR!

Contamination of system by residue on fingers during column installation



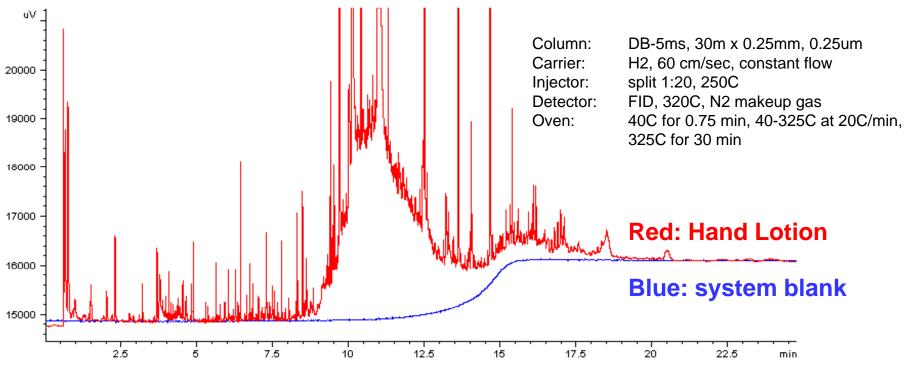
- Held French fry for 5 seconds. (1)
- Fingertip was wiped with paper towel to remove as much of the offending material as possible. (2)
- (3)Lightly touched the part of the column sticking up above the ferrule.
- Installed column into injector.
- (5)Set oven temperature to 40°C.
- Started oven temperature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

Contamination from Liquid Soap



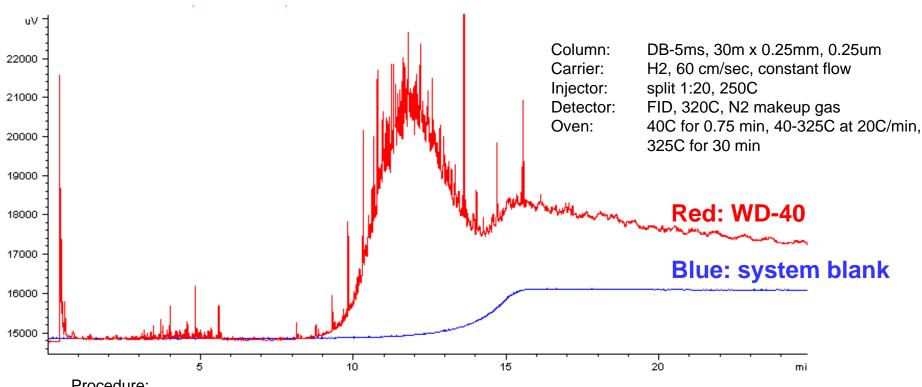
- (1) One very small drop of liquid soap placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature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

Contamination from Hand Lotion



- (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



- 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.
- Installed column into injector. (4)
- (5)Set oven temperature to 40C.
- Started oven temperature program as soon as oven reached 40C.

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

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