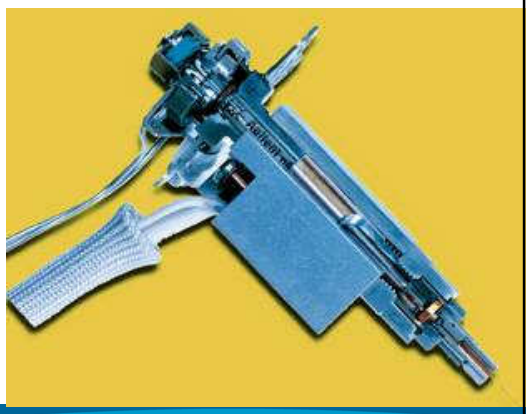
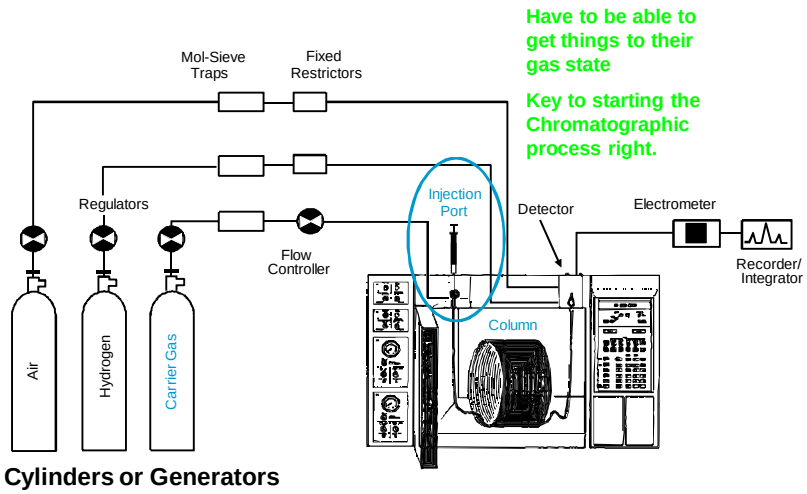
# Maintaining Your Agilent GC's Split/Splitless Injection Port





# Typical Gas Chromatographic System

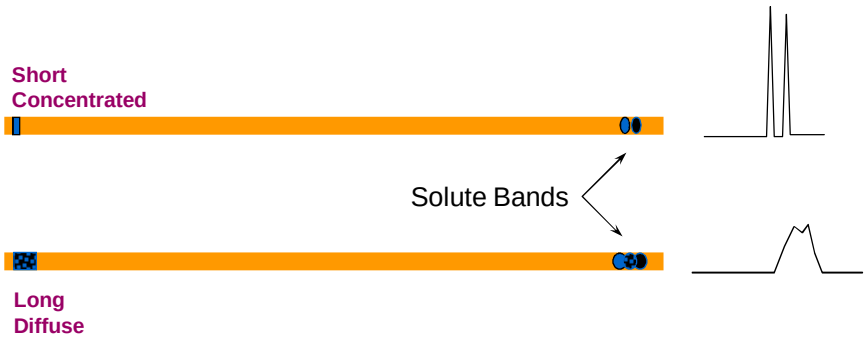



**Have to be able to get things to their gas state**

**Key to starting the Chromatographic process right.**

**Cylinders or Generators**

## Influence of Injection Efficiency




**Same column, same chromatographic conditions**

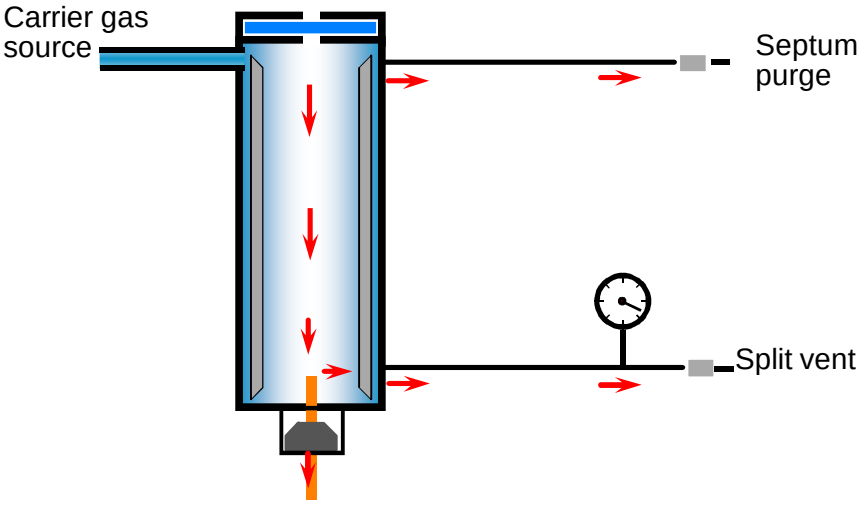


## Injectors

Split

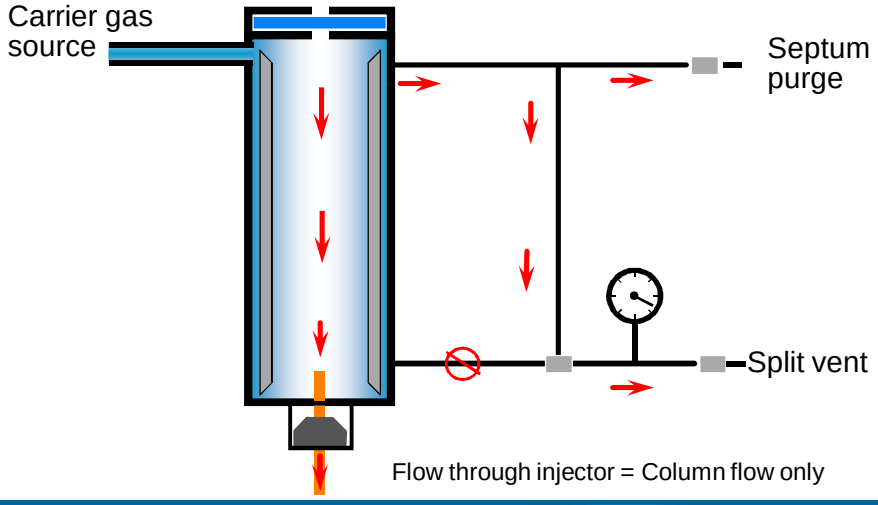
Splitless

## Split Injector
### Flow Path






## Splitless Injector
### Purge Off At Injection



## Splitless Injector
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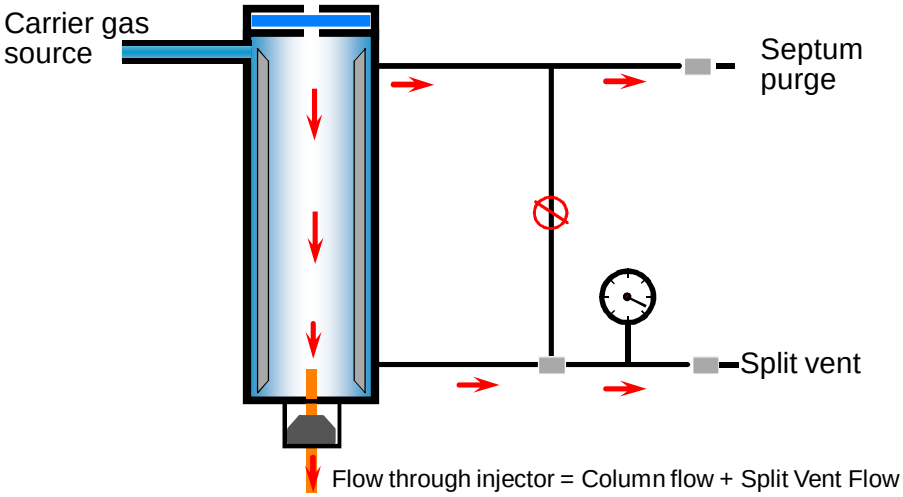
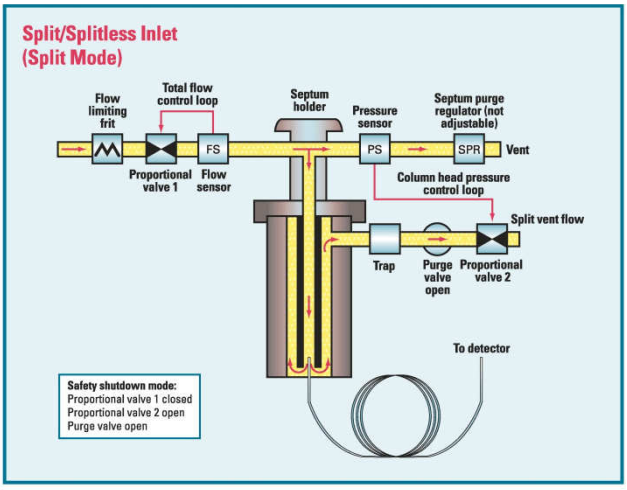





## Split/Splitless Injector



## 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.



## New Agilent's Blue Line GC Autosampler Syringes

**Improved Quality**

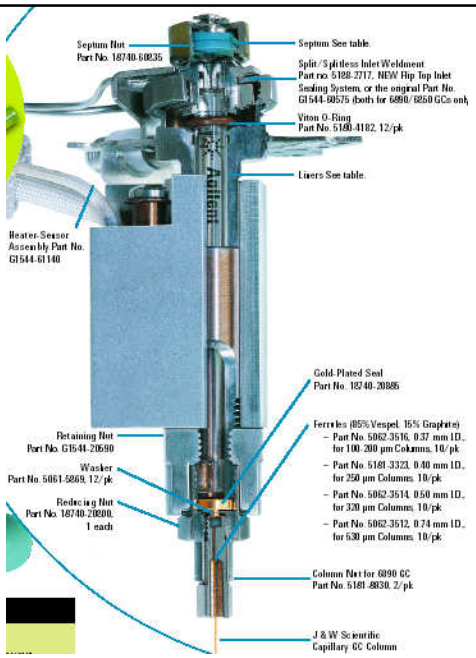
**Improved Packaging**

**More value!**

**And new New color-coded manual syringes**

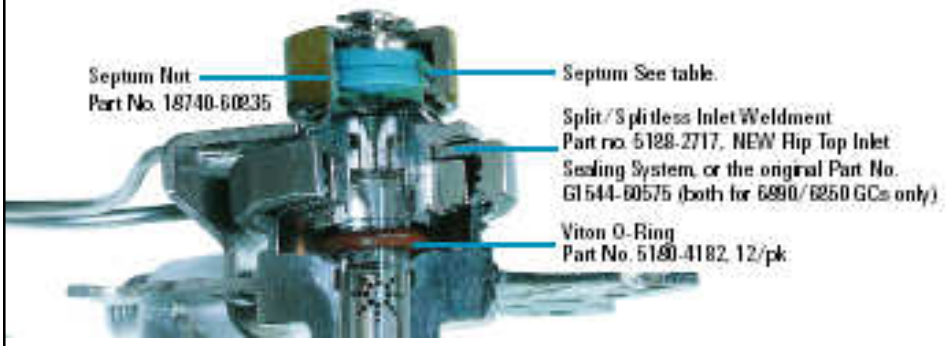
**BUT…They Don't Last FOREVER!!**

## Split/Splitless Injector Parts



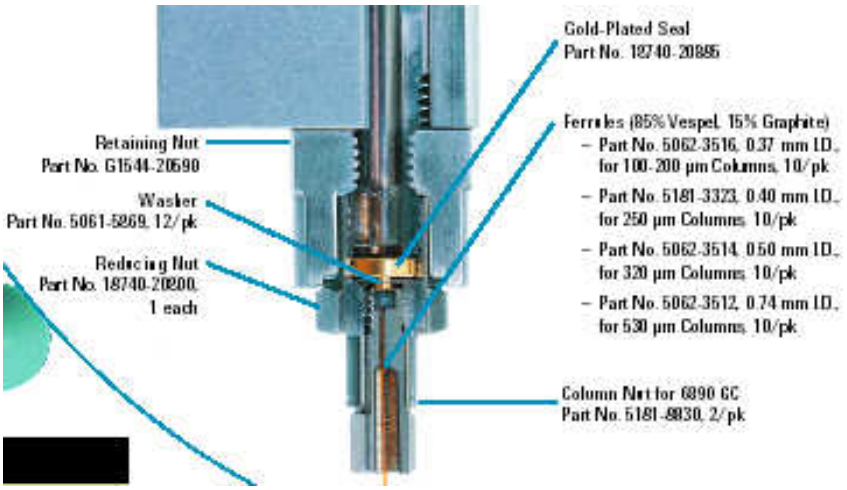



## Septum and Septum Nut




## Viton O-Ring

## 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

## Leak Checking

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

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

Column Installation

Certified gold inlet seal, 5188-5367

## 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

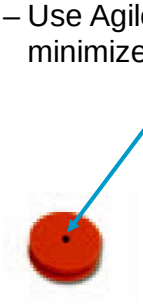
- With repeated use, conical needle guide gets worn, out of round, and needs replacement as septum can begin to "bulge" out, especially with excessive tightening,
- Septa fail faster because needle is not guided with as much precision.
- Under or Over tightening—tighten nut until c-clamp on top stops 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

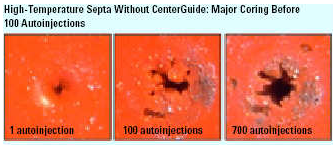
– Use Agilent Gold Standard or Blue line, HP Point, 23-26 gauge taper syringes. The point style cores septa significantly less when used with CenterGuide Septa. Taper minimizes septum coring/wear.

**HP-Point Style**

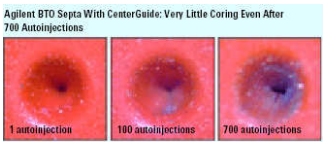
– Use Agilent CenterGuide Septa. The molded hole minimizes septa coring, counter-intuitive, but true.



**Solid Septum**



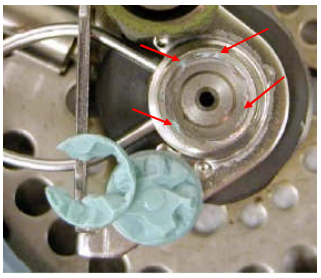

**CenterGuide Septum**





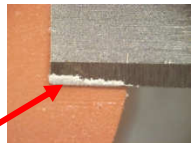

## Tips to Maximize Septum Life, Minimize Septum Leaks

– 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

## 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

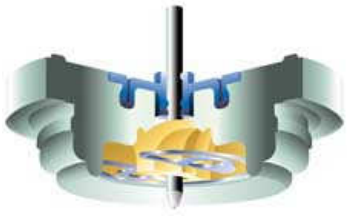
Low bleed, longer life alternative to standard septa for split/splitless injection

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!



## Where Does it Get Dirty?

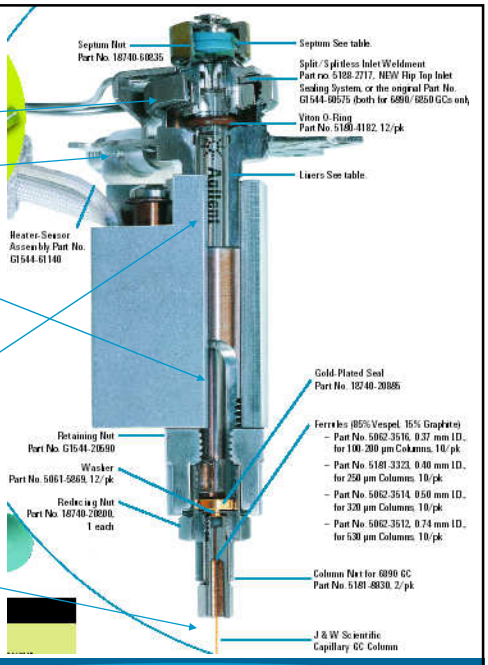
Here

Here

Here

Here

Here

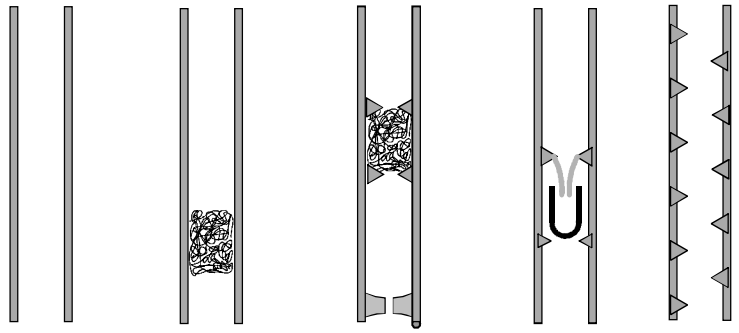


## Liner Maintenance

- 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



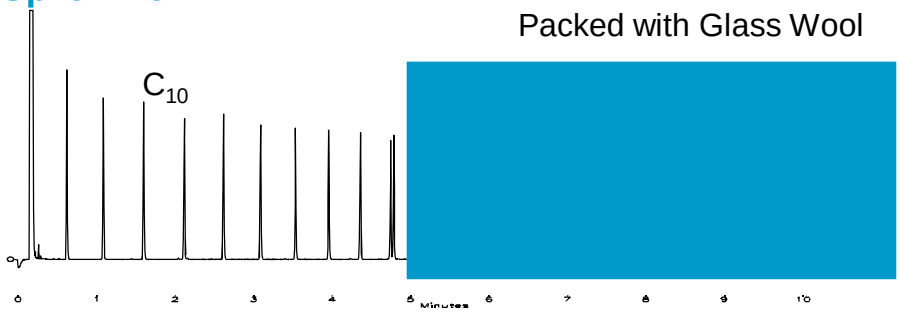
## Split Liners – What's What?



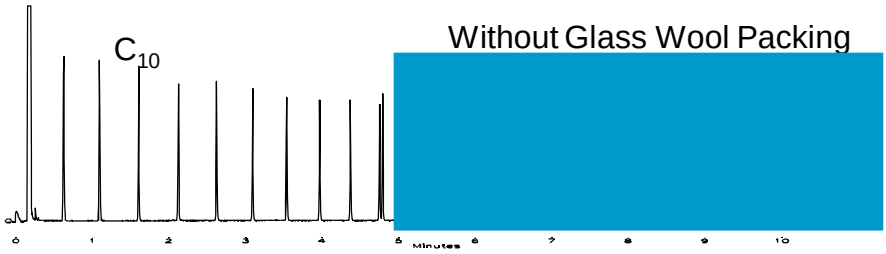
Straight tube | Straight tube with glass wool | Fixed glass wool | Inverted cup | Baffle

## Split Liner

Packed with Glass Wool



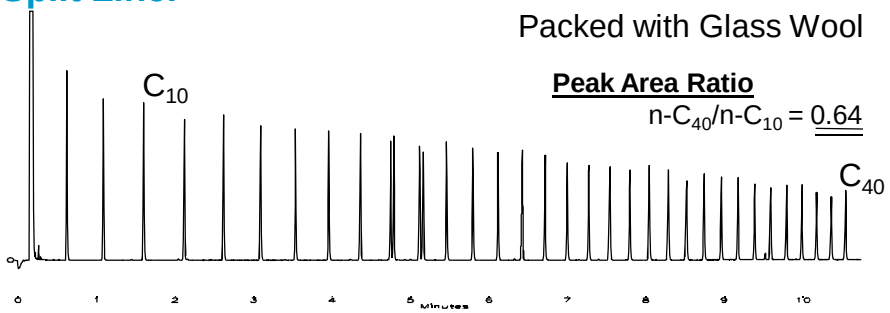

Without Glass Wool Packing






## Split Liner

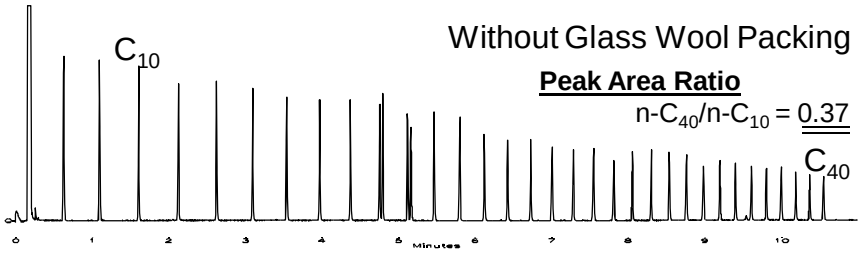
Packed with Glass Wool




**Peak Area Ratio**

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

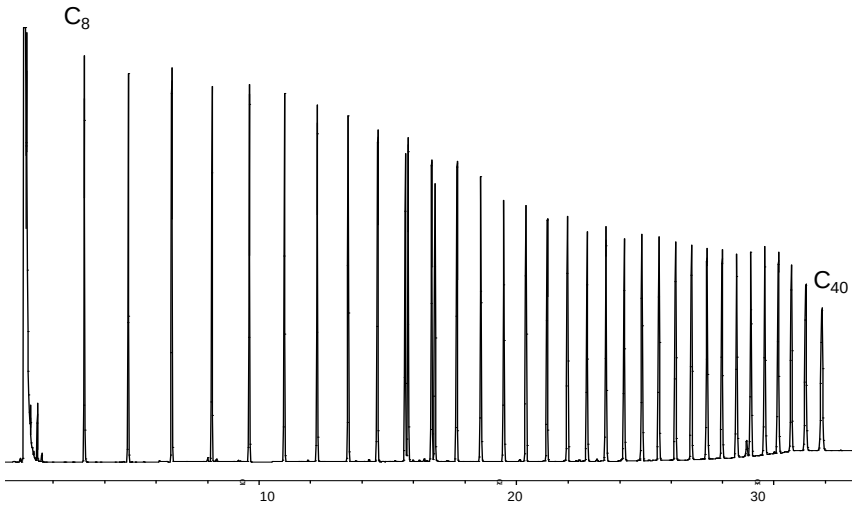
Without Glass Wool Packing




**Peak Area Ratio**

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

## Larger Plug of Glass Wool in the Liner




**Oven: 35℃ for 4 min, 35-320℃ at 10℃/min, 320℃ fo r 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

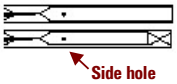
•Amount, size and placement must be consistent for consistent results

• 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, 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<br>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.

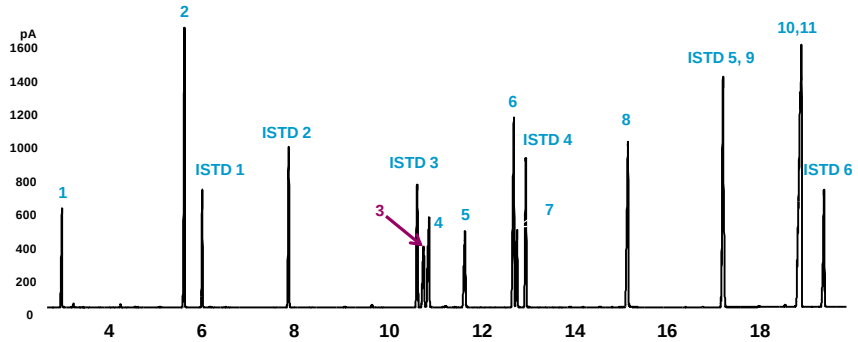
Phenols, for example….in a separation of EPA method 8270 compounds



## 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 | 7 | Pentachlorophenol | ISTD 1 | Dichlorobenzene-d4 |
| 2 | Aniline | 8 | Benzidine | ISTD 2 | Naphthalene-d8 |
| 3 | 2,4-Dinitrophenol | 9 | 3,3-Dichlorobenzidine | ISTD 3 | Acenaphthene-d10 |
| 4 | 4-Nitrophenol | 10 | Benzo(b)fluoranthene | ISTD 4 | Phenanthrene-d10 |
| 5 | 4,6-Dinitro-2-methylphenol | 11 | Benzo(k)fluoranthene | ISTD 5 | Chrysene-d12 |
| 6 | 4-Aminobiphenyl | | | 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

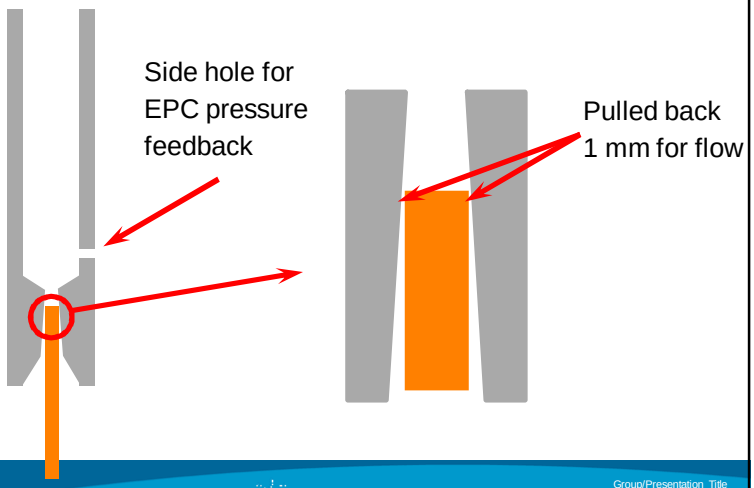
G1544-80700 Direct Connect, Dual-taper, deactivated

Vendor X Unknown proprietary deactivation



## DIRECT CONNECT

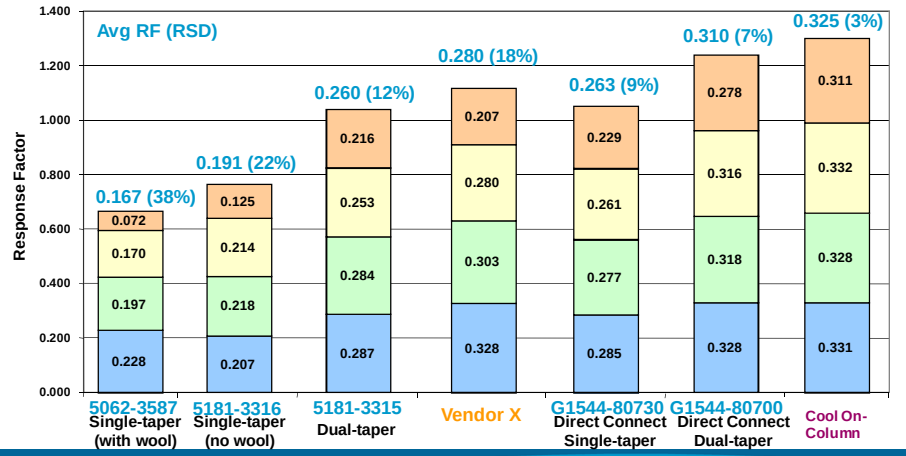
### Vaporization Liner



## Liner Comparison

**Experimental:**
**Agilent 6890 with FID**
**Column = HP-5MS 30m x 0.25mm x 0.5µm**
**Compared COC to various liners**
**0.75 min Splitless time, 3mL/min column flow**
**Oven: Temp programmed per 8270 method**
**Inj. 250℃, Det. 300℃, Sample: 1 µL 8270 mix**

### 2,4-Dinitrophenol Response Factors






## For Easy Liner Maintenance on 5890/6890

**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**

## Turn Top Inlet Sealing System on NEW 7890

**- Fast/Easy Split/Splitless Inlet Maintenance**

**… changing liners has never been easier**



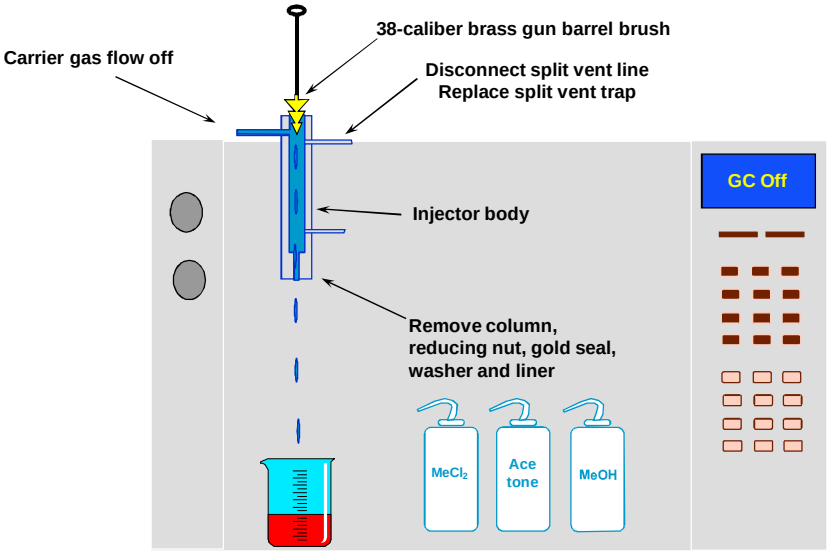
## Common Care and Maintenance Scheme for GC Columns

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

2. Cut off 6”-1ft of the inlet end of the column.

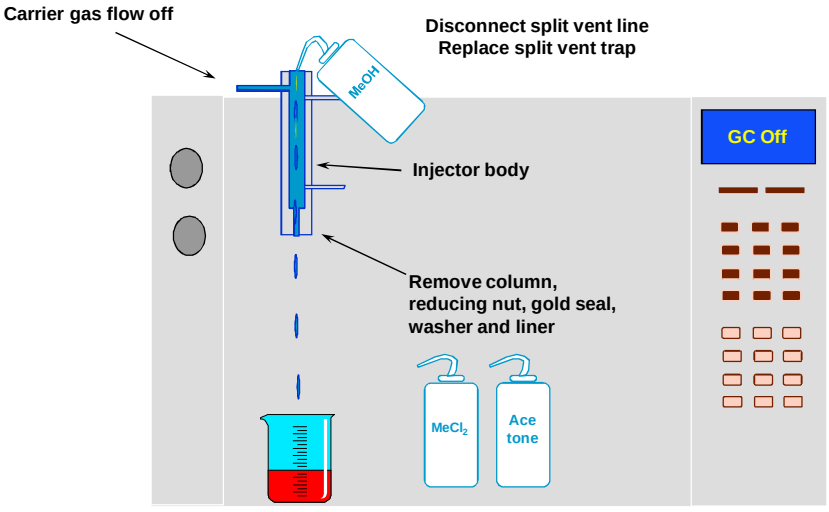
3. Cut off more column. (repeat as necessary)

## Cleaning the Split/Splitless Injector



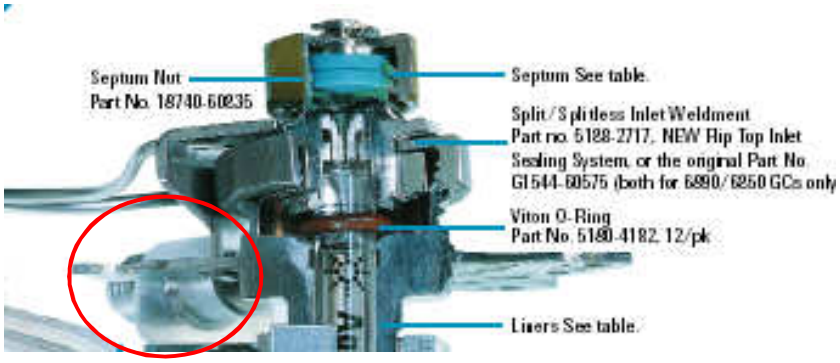



## Cleaning the Split/Splitless Injector



## 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

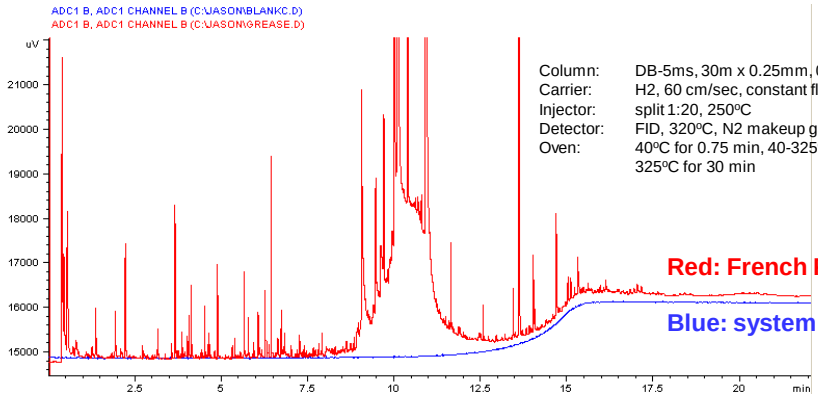
G1544-80530



## Be Careful When Doing Maintenance…

**You may be the CONTAMINATOR!**

# Contamination of system by residue on fingers during column installation




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

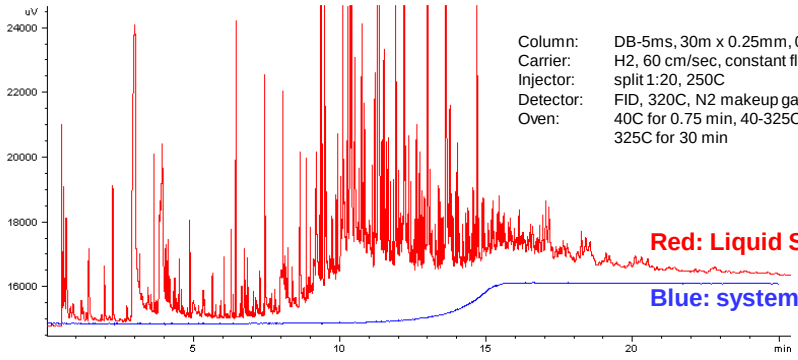
**Red: French Fry**

**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 40ºC.
(6) Started oven temperature program as soon as oven reached 40ºC.



# Contamination from Liquid Soap



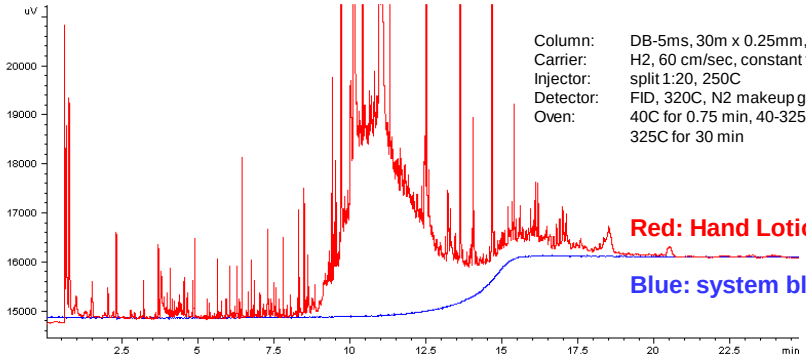

Column: DB-5ms, 30m x 0.25mm, 0.25um
Carrier: H2, 60 cm/sec, constant flow
Injector: split 1:20, 250C
Detector: FID, 320C, N2 makeup gas
Oven: 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

**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

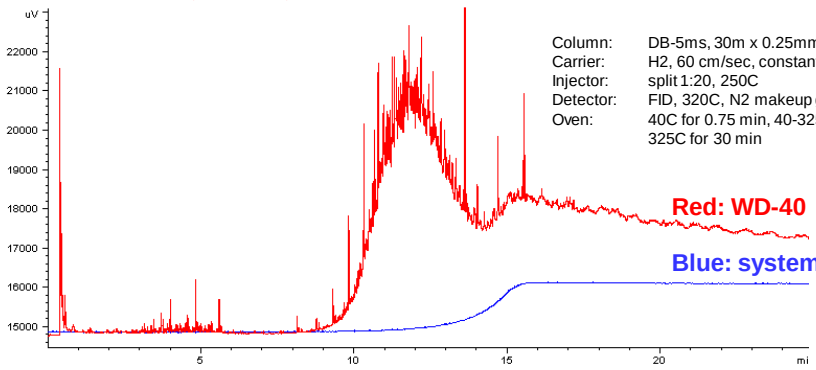
**Red: Hand Lotion**

**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.

## 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 $O_2$ sensitive detectors, irreversible damage to column

Non-Optimized Set-up

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