



Recent Advances to Ensure Simple, Leak Free GC Column Connections

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GC column installation is a challenge...

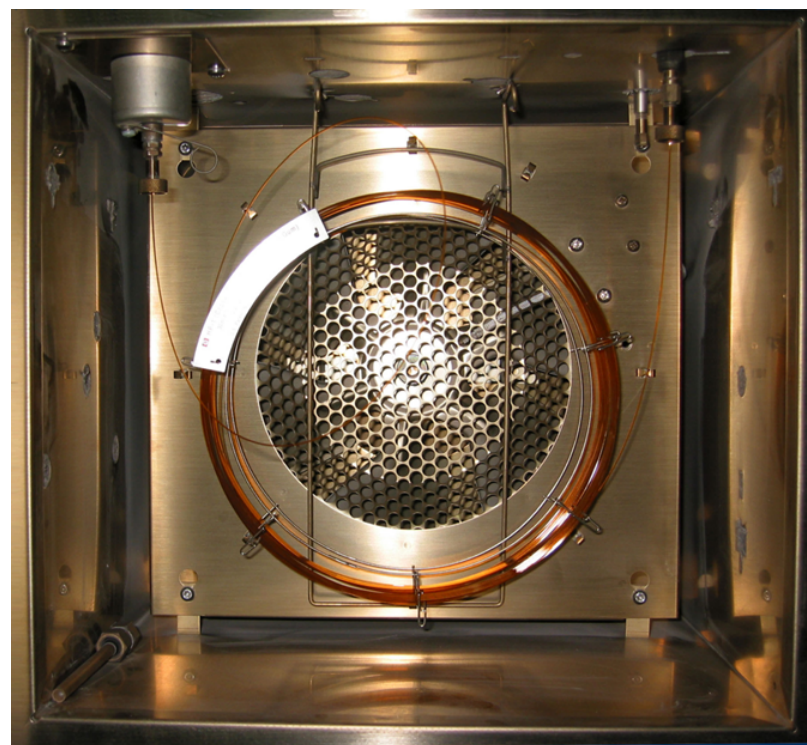
A routine, basic procedure but not easy to do

- Very small parts
- Wrenches in cramped, dark space

2 chances to get it wrong:
at the inlet and the detector

What can go wrong?

- Height into the fitting
- Leaks
- Activity



How do you make better GC column connections?

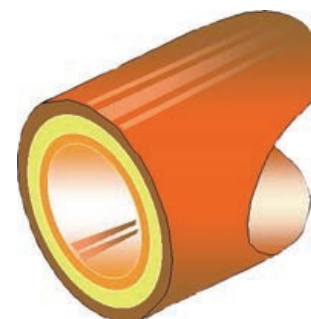
1. Start with the right tools and supplies

- Column nuts
- Material : stainless – but need brass for MS interface
- Choice of right ferrules

2. Proper assembly process

- Make a good clean column cut – every time
- After ferrule is installed
- Fused silica tubing cutters : ceramic
- Magnifier to inspect the cut - cracked fused silica and flaps of polyimide are active sites that ruin chromatography

3. Ensure the proper and consistent length of column into the fitting



Supply Selection: Which Capillary Ferrules to use?



polyimide



polyimide/graphite



graphite



flexible metal

Composition	Re-use	Max T	Use	Limitation
Polyimide (Vespel)	yes	280	Easy seal	Shrink after heating causing leaks after thermal cycle; isothermal only
Graphite	yes	450	FID, NPD Inlets	Contamination, permeable to air – not for oxygen sensitive detectors
Polyimide/graphite (85% / 15%)	limited	350	MS, ECD, Inlets	Still shrink after thermal cycles creating leaks; need to retighten regularly
Flexible metal	no	450	Capillary Flow Technology (backflush, splitters, ...)	May not seal well with damaged fittings or rough surfaces



“Short” ferrules for inlet and detector configurations on Agilent GC’s



“Long” ferrules for MS transfer lines and MS interface nut



Supply selection: Graphite Ferrules

Often selected because

- high temperature range (450C)
- the least expensive capillary ferrules
- Soft, easily conform to fitting shape and size

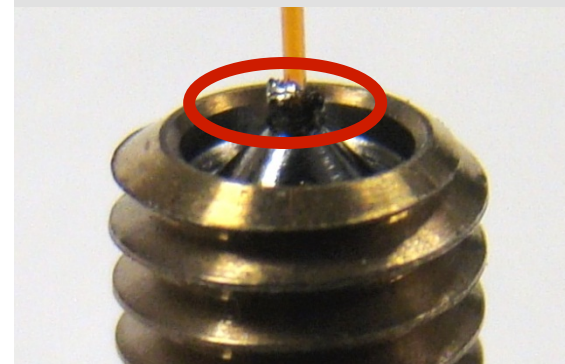
But do not over tighten!

Over tightening of ferrules

- Causes ferrule material to extrude into the fitting
- Creates active sites if in the flow path
- Can flake or fall apart, blocking flow path and requiring extensive maintenance

Remember Graphite is NOT recommended with MS, ECDs or for use with column stationary phases sensitive to air because it is permeable to air

Active sites in the flow path



Extensive Inlet maintenance needed



Supply Selection: Graphite/polyimide blend ferrules

Graphite / polyimide blend ferrules are very popular

- Recommend
 - Long style for Mass Spec
 - Short for Inlets, and other oxygen sensitive detectors, like ECD
- Soft enough to make seal against rough surfaces
- Fairly high temperature stability (350C)
- Don't extrude beyond the fitting when tightened (like graphite)
- Lower air permeability than pure graphite

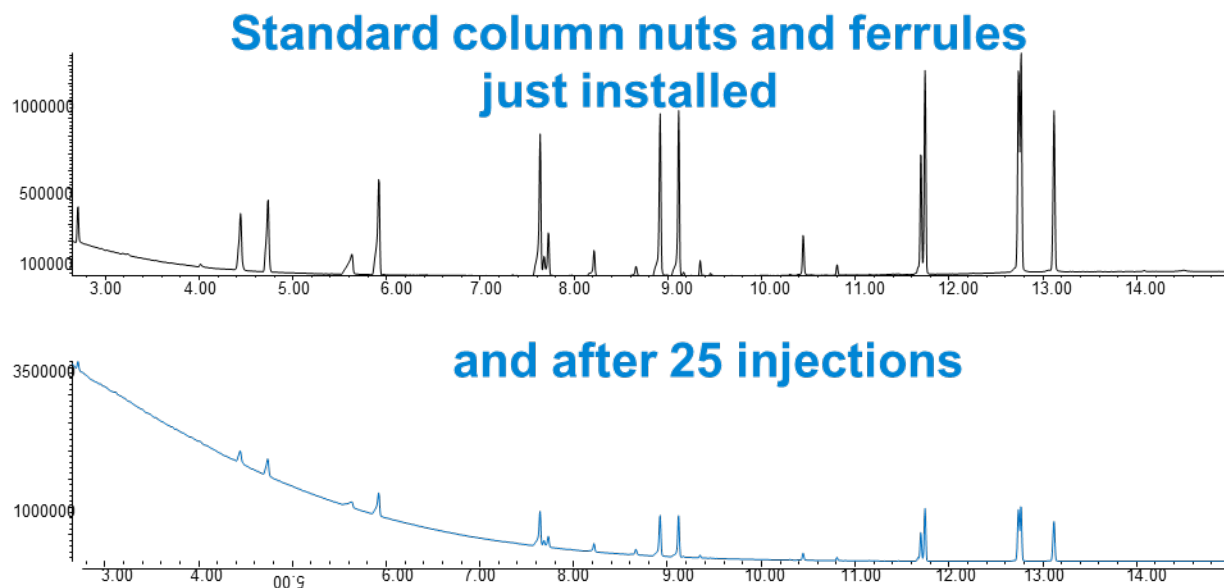


Graphite / Polyimide blend capillary ferrules

Unfortunately ... leak following normal temperature program runs

Studies show the leaking continues with use of the ferrules -

Not just after the first one or two runs [Application Note 5991-3612EN](#)



Frequent re-tightening of the fitting is needed to maintain a leak-free seal – and system performance and productivity

Supply Selection: capillary column nuts

Column nuts are determined by the instrument fitting

Basic mechanical fittings with little enhancements

- Brass for the MS
- Some finger tight designs for ease of use

New design addresses ease of use and productivity issues of leaking and over tightening



Better Connections: Agilent Self Tightening Column Nuts

Designed for use with *short* graphite/polyimide blend ferrules – both at the inlet and the MS interface – so only one type of ferrule needed for both ends of the column!



For inlet or detector



For mass spec transfer line

Short ferrule exposes more thread of the fitting for better sealing

How do Self Tightening column nuts work?

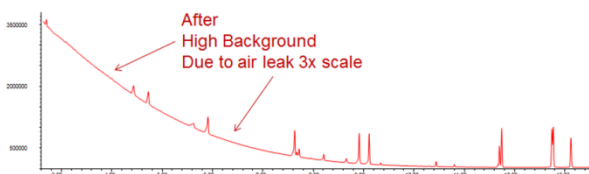
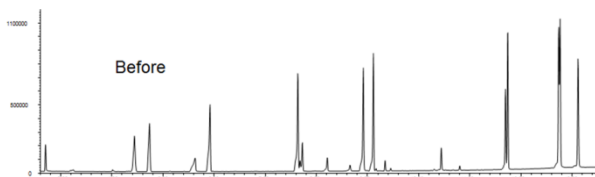
- Ease of use – install in dark, small space in GC oven without wrenches
- Wing design for finger tight installation with graphite/polyimide blend ferrules
- No tools dramatically reduces force preventing over tightening or damage
- Robust stainless steel construction

Plus....

- Novel **spring driven piston** design that continuously presses against the ferrule to **maintain a leak-free fitting** even when the ferrule shrinks during temperature program!



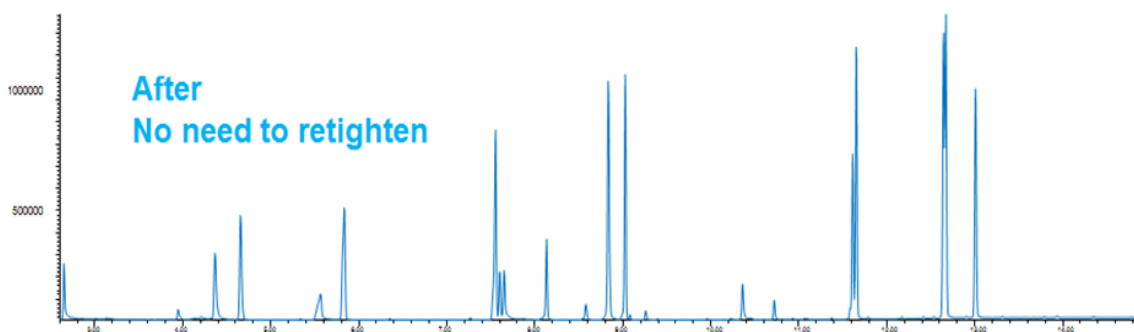
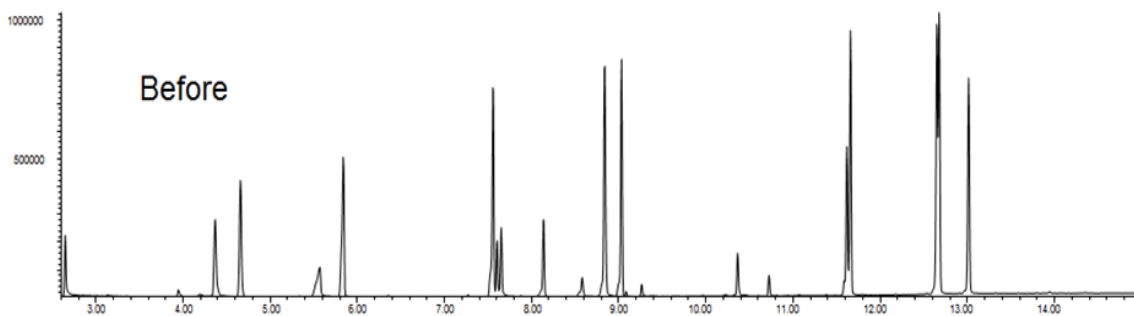
Benefit of Self-Tightening Column nuts



Take you from this....

.... to this!

Without retightening, the baseline remains flat after 400 runs with no indication of leaks when using the Self Tightening column nuts



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How do you make better GC column connections?

✓ 1. Start with the right tools and supplies

- Choice of right ferrules = short graphite/polyimide
- Column nuts = Self Tightening

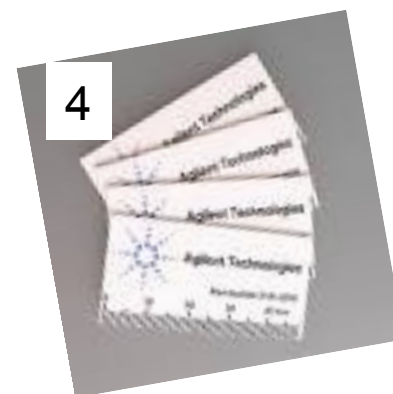
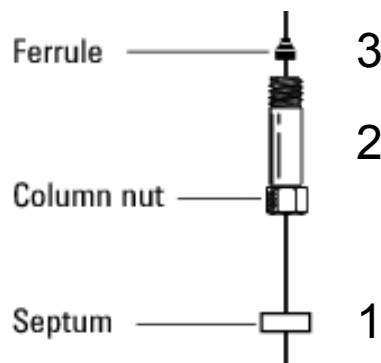
2. **Proper assembly process**

- Make a good clean column cut – every time
 - After ferrule is installed
 - Fused silica tubing cutters : ceramic
 - Magnifier to inspect the cut - cracked fused silica and flaps of polyimide are active sites that ruin chromatography
- ## 3. Ensure the proper and consistent length of column into the fitting



Column installation assembly process

1. Thread through an inlet septum
2. Pass column through the column nut
3. Install ferrule onto the column tubing
4. THEN make a fresh cut
5. Inspect the cut; repeat cut if any jagged or rough edges



good cut



bad cut



bad cut



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Ensure the proper length ... Column Installation Tools

Follow the manufacturer's recommended procedure

- Optimized GC performance and reproducibility requires ensuring the proper length of column into the fittings, every time
- Column Installation pre-swaging tools for the Split/Splitless inlet
Available for Agilent GC models 7890 and 6890, for graphite or metal ferrules
PreSwaging tool for the MS interface



G1099-20030



G3440-80218 / G3440-80217



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- ✓ 1. Start with the right tools and supplies
 - Column nuts = Self Tightening
 - Choice of right ferrules = short graphite/ polyimide
- ✓ 2. Proper assembly process
 - Make a good clean column cut – every time
 - After ferrule is installed
 - Inspect the cut
- ✓ 3. Ensure the proper and consistent length of column into the fitting



Better connections: Beyond the basics

Effluent manipulation for productivity:

- Split effluent from a single column to 2 even 3 detectors on the same GC.
- Backflush - replaces a “bakeout” period for high-boiling analytes that collect in the column then interfere with subsequent analyses

GC x GC or 2-dimensional GC

- Powerful tool used in environmental, food and flavor analysis, and hydrocarbon processing industries
- Separates complex mixtures using two independent columns with different stationary phase selectivity
- Columns are connected in series and separated by a modulator

Limitations to Adoption of :

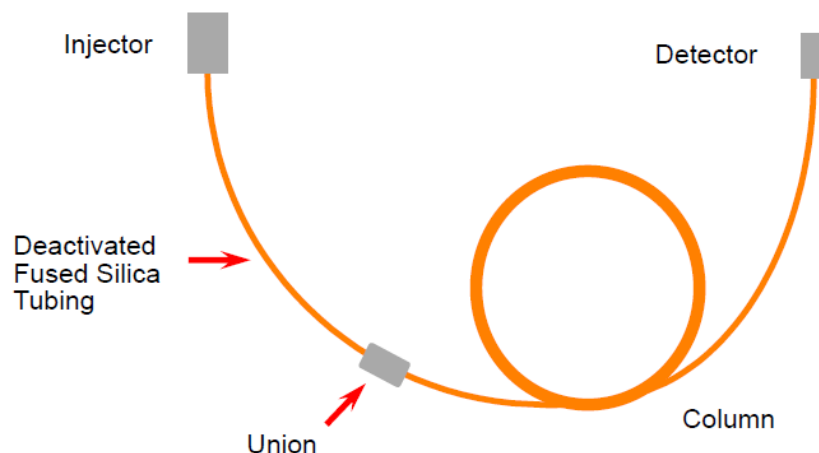
All column connections must:

- be inert, not adsorbing or decomposing analytes
- have low thermal mass and low dead volume to maintain sharp peak shapes
- be leak free – and stay so
- not outgas from materials used to make the seal
- withstanding the temperatures used in the GC analysis
- not be technique dependent – must be easy to do

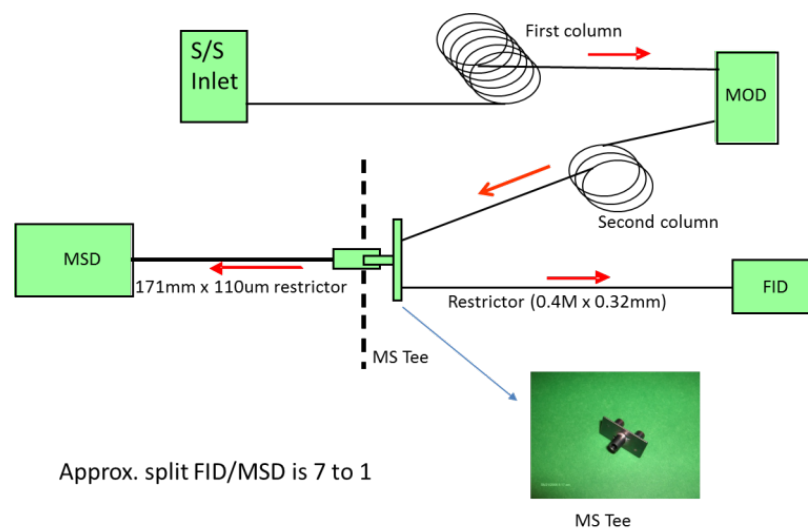


Advanced techniques increase the number of connections

Simple addition of a retention gap or guard column doubles the column connections



Powerful advanced GCxGC systems with many connections



Better Connections: Glass column connectors

Ultra Inert Press Fits

Join retention gap or guard column to analytical, or split effluent

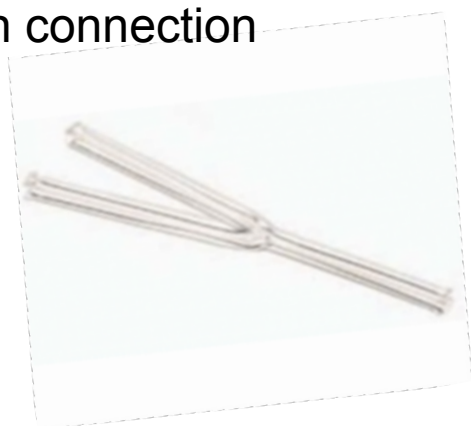
Dependable inertness performance at a lower cost

Improved robustness, holding strength

Batch certified inertness

Improved packaging and installation instructions

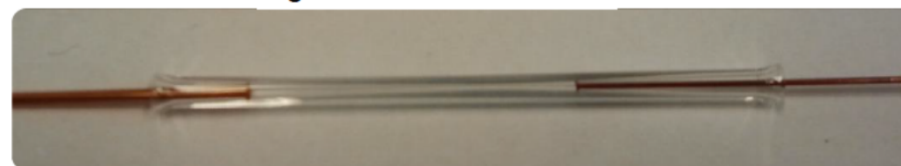
Easier to use - transparent deactivation gives visibility of the column connection



Competitive Press-Tight



Agilent Ultra Inert Press Fit



Better Connections: Capillary Flow Technology Devices

UltiMetal Plus Ultimate Union/UltiMetal Plus Tee

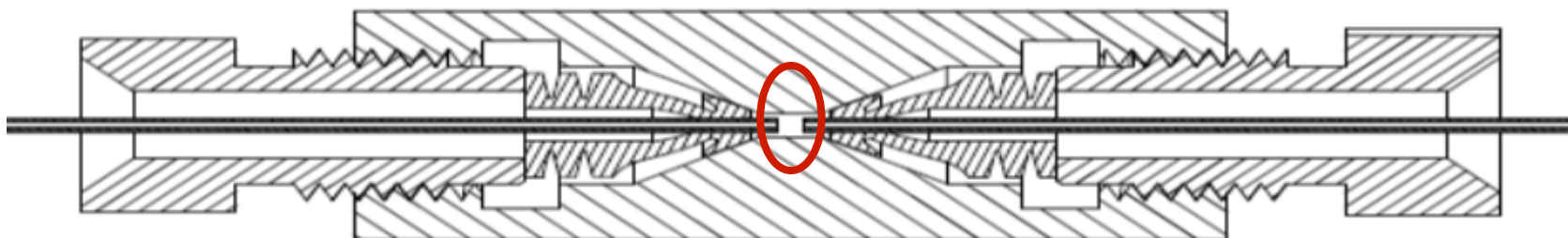
- Stainless Steel Micro Fluidic plates technology
- Deactivation essential to block active sites
- Column connection easy to assemble Release hole for stuck ferrules

Using Flexible Metal ferrules to overcome issues

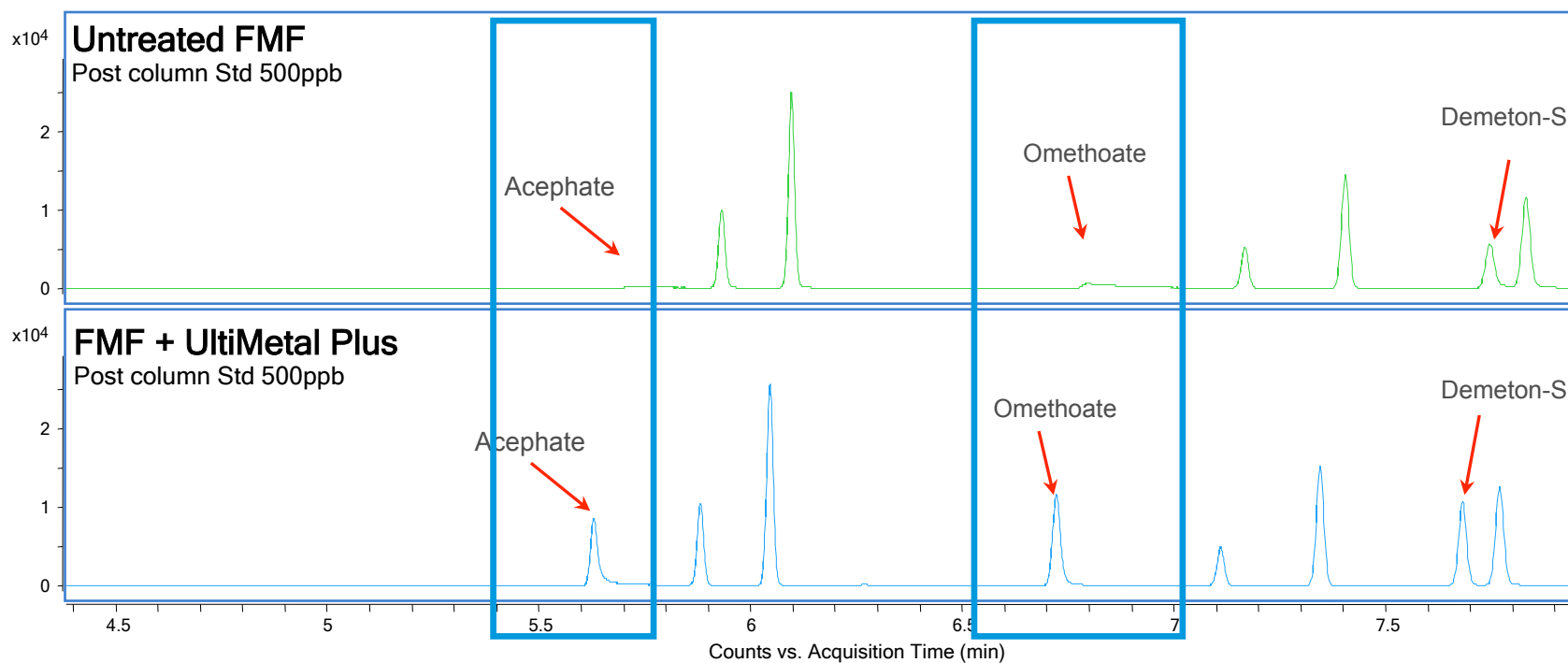
- UltiMetal Plus surface chemistry prevents activity
- Flexible design reduces risk of over tightening or column breaks
- Leak free seal remains after repeated temperature cycles



Impact of ferrule surface on inertness



Very small amount of ferrule surface exposed to active pesticides



Better connections: UltiMetal Plus Tubing and Fittings



- UltiMetal Plus Deactivated metal tubing and valves
- 0.53 and 0.25 mm ID guards and transfer lines
- Metal fittings (unions, tee's and nuts)
- Steel tubing (1/16", 1/8", 1/4")
- UltiMetal Plus fused silica guard columns



Ensure the entire chromatographic solution is inert and corrosion resistant to provide superior performance with improved peak shapes even for active compounds

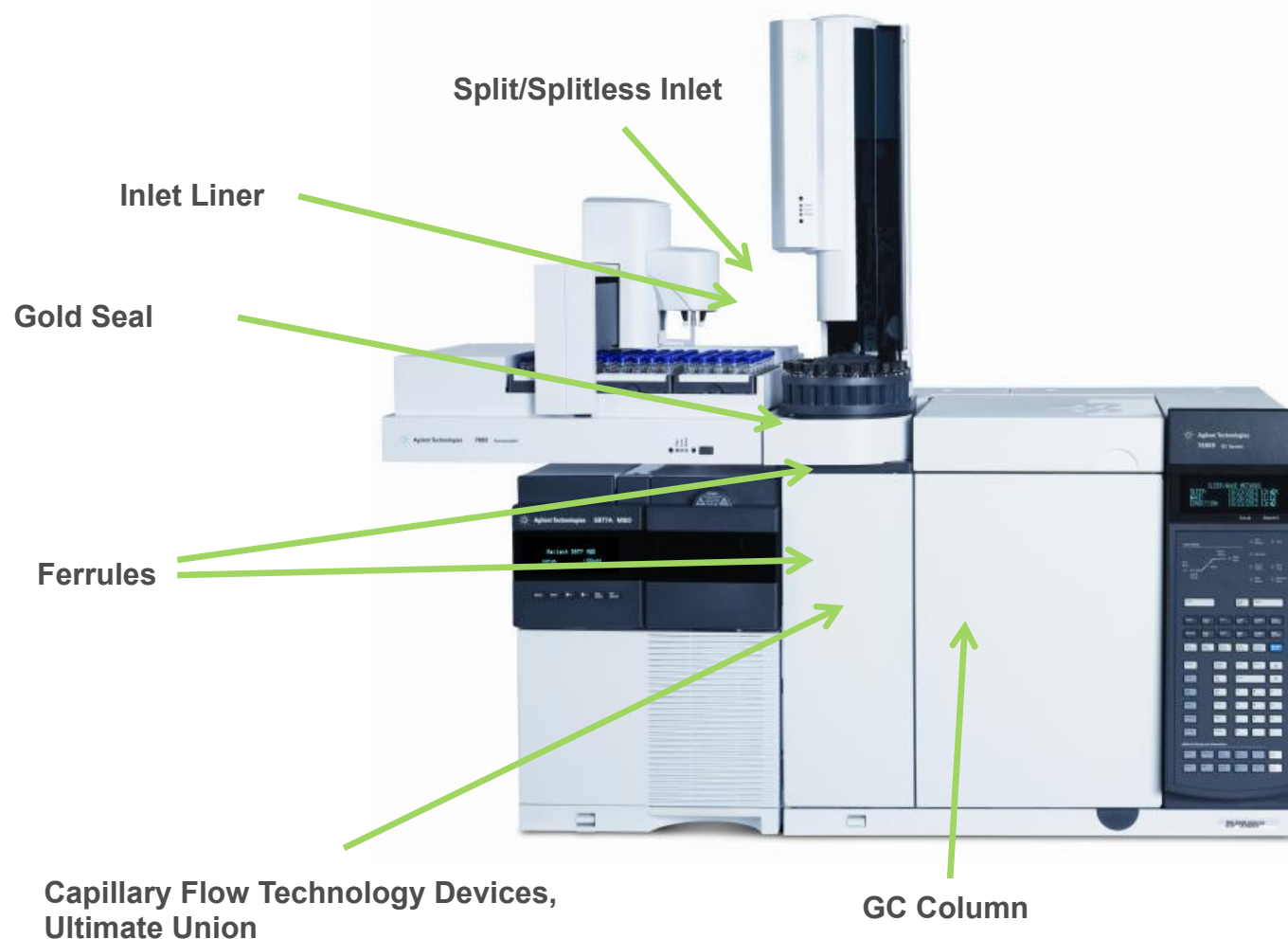


Making Better GC Connections

- Select supplies appropriate for your instrument and application
- Follow recommended assembly process
- Maximize productivity by using innovative tools and supplies
 - Column Installation Pre-swaging tools
 - Self Tightening column nuts
 - Ultra Inert Press Fit connectors
 - UltiMetal Plus Flexible metal ferrules
 - UltiMetal Plus tubing and fittings



Hot GC Surfaces in Contact with Samples the Need for Inertness



Agilent Inert Flow Path Solution

Ultimetal Plus Inlet Weldment, Shell and Transfer Lines



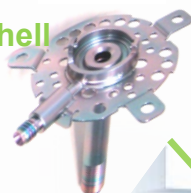
Ultra Inert Inlet Liner



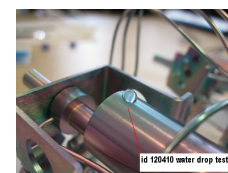
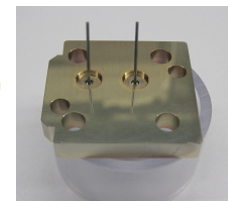
Ultimetal Plus Ferrules



Ultimetal Capillary Flow Technology Devices, Ultimate Union



Ultimetal Plus– TCD, FPD, NPD/FID Jets



Ultra Inert Gold Seal

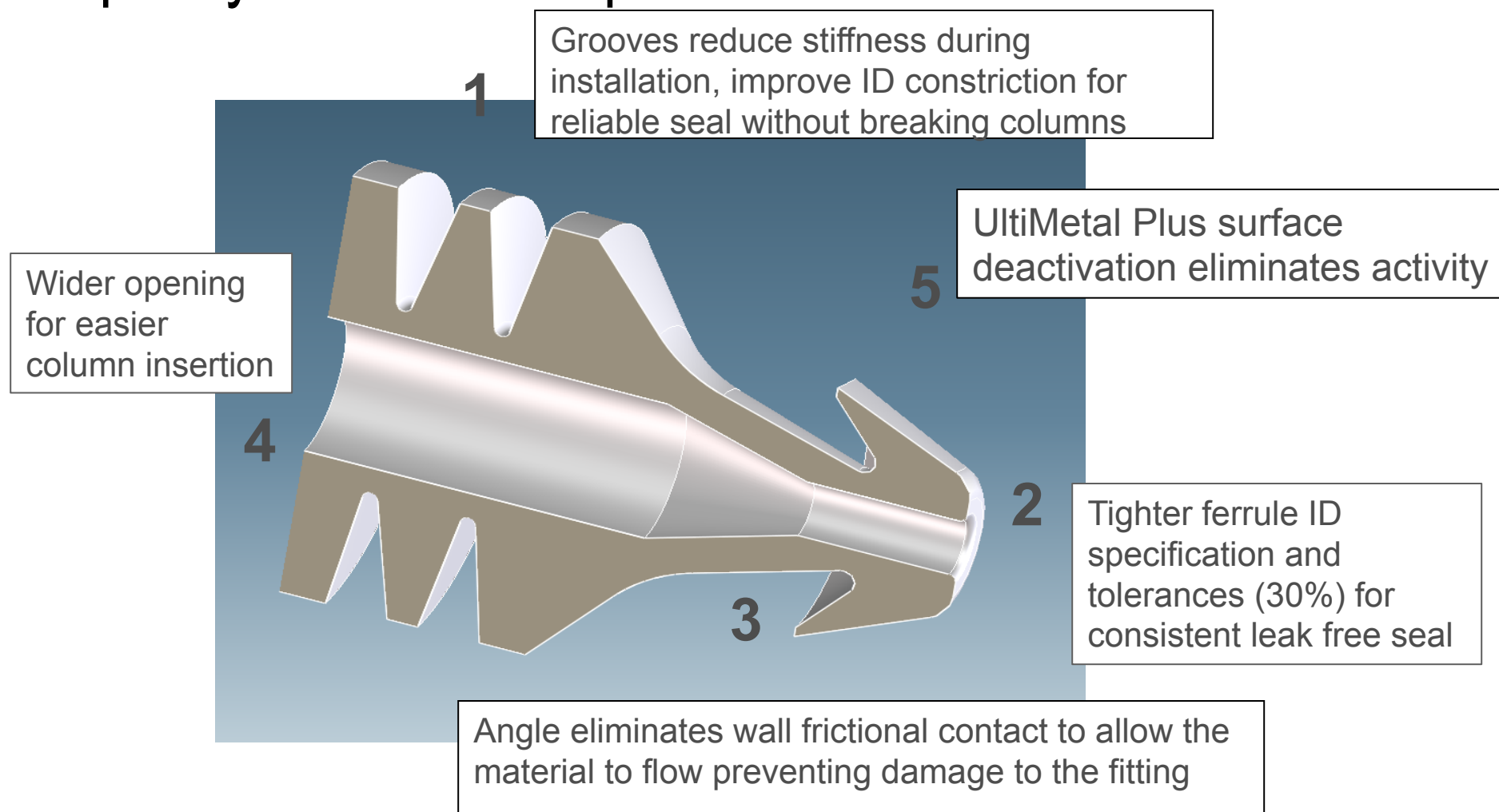


Ultra Inert GC Column



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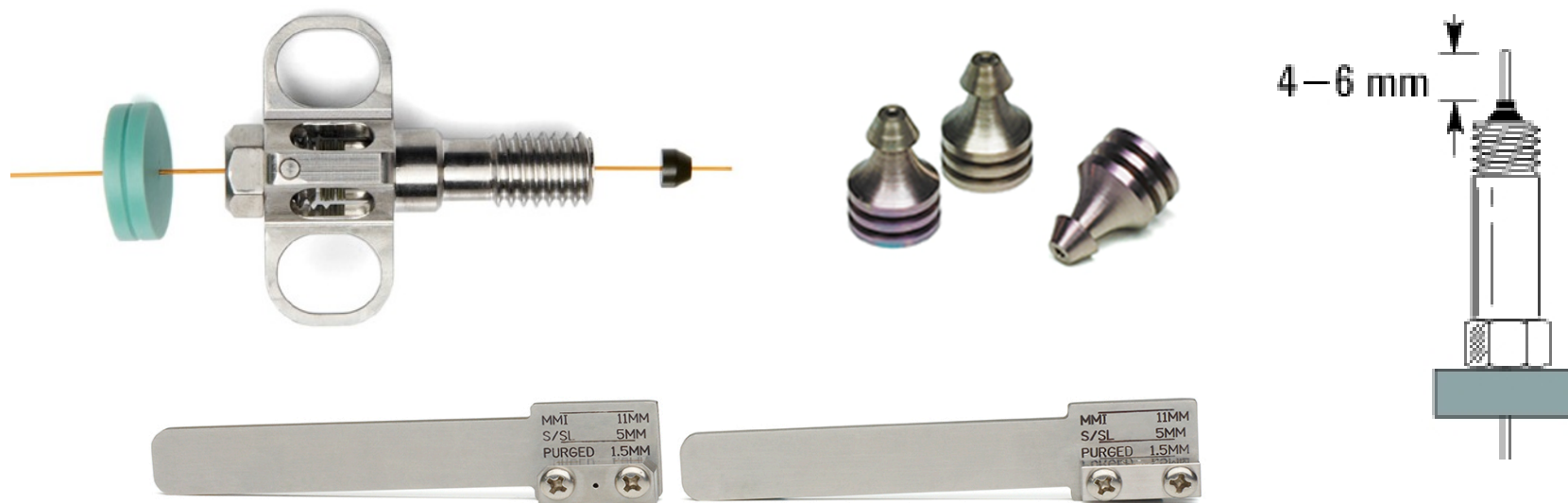
UltiMetal Plus Flexible Metal Ferrules improve Capillary Flow Technique column connections



Ferrule and Fittings

Maximizing Inertness

Inlet connections		
Recommended:		Inert Flow Path:
Nut		Nut
5190-6194	Column nut, Self Tightening Inlet/Detect	05921-21170 Column nut for long ferrule
Ferrule		Ferrule
5181-3323	Ferrule, 0.4mm VG 0.1-0.25 col 10/PK	G3188-27501 Ferrule, flexi inert 0.25mm col 10/PK
5062-3514	Ferrule, 0.5mm VG 0.32 col 10/PK	G3188-27502 Ferrule, flexi, inert 0.32mm col 10/PK
Tools:		Tools
G3440-80217	Column install pre-swaging tool, graphite ferrules	G3440-80218 Column install pre-swaging tool, metal ferrules



G3440-80217 / G3440-80218



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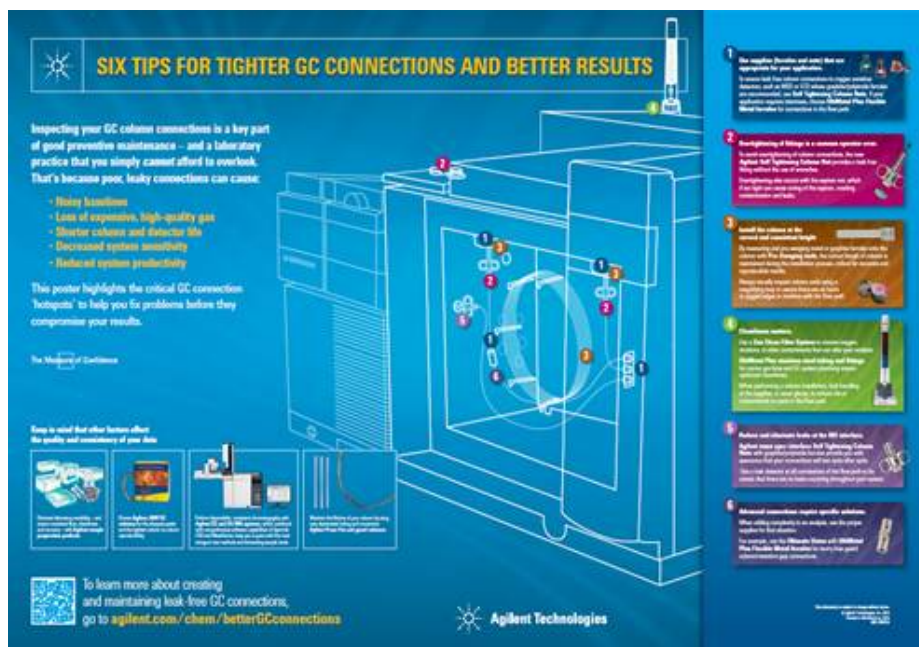
Inert Flow Path for the 7890B GC / 5977A MSD



NOW IT'S EASIER THAN EVER TO GET RELIABLE GC RESULTS THROUGH BETTER GC CONNECTIONS FROM AGILENT

www.agilent.com/chem/betterGCconnections

Order the poster...



View the video...



Acknowledgements

- Agilent colleagues
 - Lindy Miller
 - Yun Zou
 - Gary Lee
 - Paul Trip
 - Roger Firor
- You for your kind attention

Questions?



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