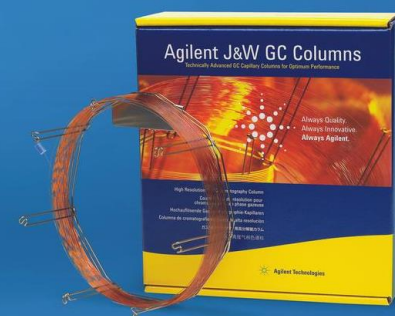
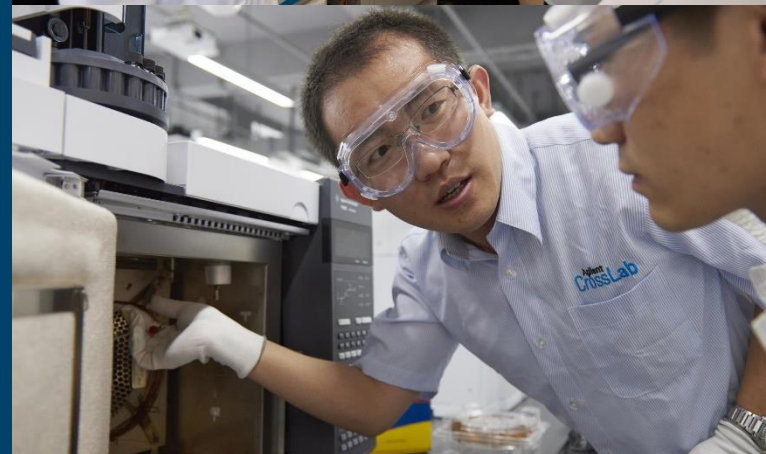


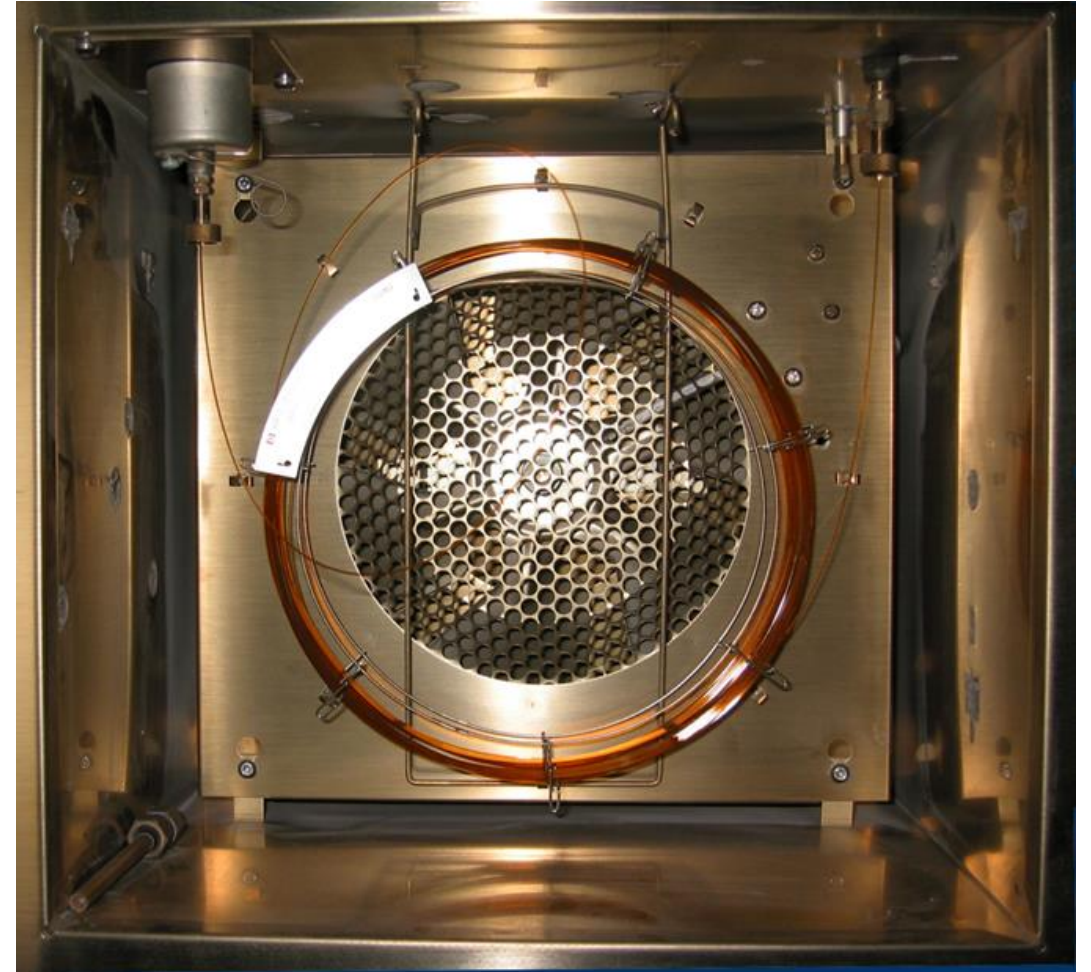
# Making Better Connections in GC and GC/MS Systems

Mark Sinnott  
GC Application Scientist  
16 February 2023



# GC Column Installation is a Challenge...

- A routine, basic procedure, but not necessarily easy to do:
  - Very small parts
  - Wrenches in cramped, dark space
- Two chances to get it wrong:
  - At the inlet and the detector
- What can go wrong?
  - Height into the fitting
  - Leaks
  - Activity
  - Broken Column



# Making Better GC Connections

- Select supplies appropriate to 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/Gold ferrules
- UltiMetal Plus tubing and fittings



# How Do You Make Better GC Column Connections?

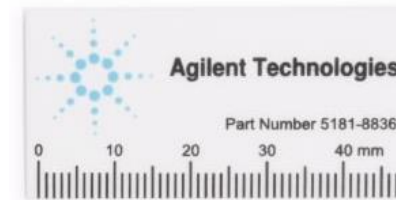
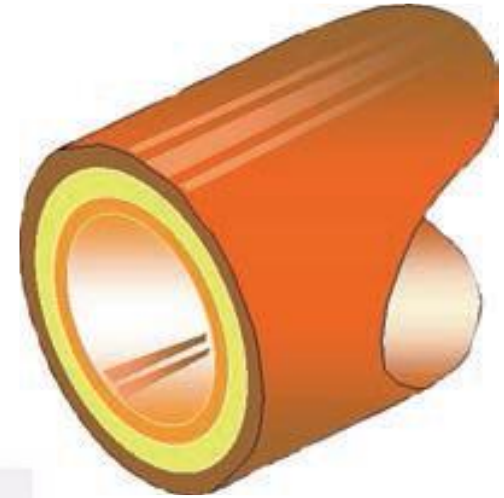
## 1. Start with the right tools and supplies

- 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, diamond tipped
- 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



5181-8836

# Supply Selection: Which Capillary Ferrules to Use?

	Composition	Re-use	Max T	Use	Limitation
	Graphite	Yes	450	FID, NPD, inlets	Contamination, permeable to air – not for oxygen-sensitive detectors
	Polyimide/graphite (85%/15%)	Limited	350	MS, ECD, FID, inlets	Still shrinks after thermal cycles, creating leaks; need to retighten regularly
	Flexible metal	No	450	Capillary Flow Technology - CFT (backflush, splitters)	May not seal well with damaged fittings or rough surfaces
	Gold flexible metal	No	450	Capillary Flow Technology - CFT (backflush, splitters)	Seals better, especially for scratched or damaged fittings (gold plating tends to fill microscopic scratches)
	Polyimide (Vespel)	Yes	280	Easy seal	Shrinks after heating, causing leaks after thermal cycle; isothermal only



Dial packaging

# Agilent Gold-Plated Flexible Metal Ferrules (FMFs)

Agilent is excited to announce the release of our “new” gold-plated flexible metal ferrules

- These newly launched ferrules improve on the existing flexible metal ferrule (FMF) design by applying a gold coating to ensure a leak-free connection **with capillary flow technology (CFT) devices** while providing enhanced ease-of-use.

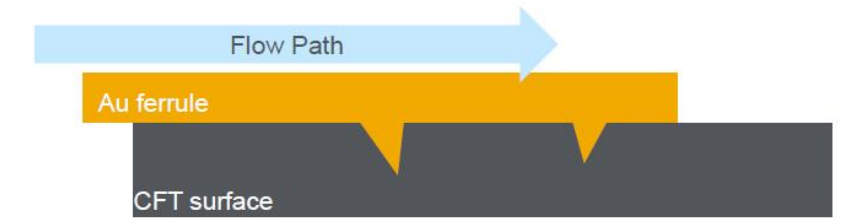
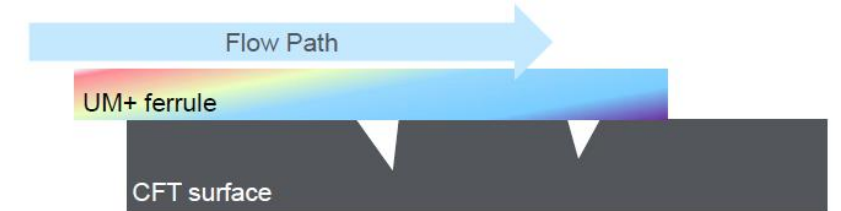
[Flexible Metal Ferrules Swaging Guide for UltiMetal Plus and Gold-Plated Ferrules \(agilent.com\)](https://www.agilent.com/chem/flexiferrule)



# The Highlights

## Review

- Flexible metal ferrules are technologically advanced offering ease-of-use and mechanically tight seals
- Gold-plating flows fill scratches and striations on the surface of the CFT device
- Create leak-free seals on first installation attempt
- Ideal for labs running backflush, Dean's switch, or GC x GC analyses



# Supply Selection: Graphite Ferrules

Often selected because of:

- Their high temperature range (450 °C)
- They are the least expensive capillary ferrules
- They are soft and easily conform to fitting shape and size
- Less prone to column crushing than graphite/vespel

## **But do not over tighten**

Over-tightening ferrules:

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

## **Remember**

Graphite is **not** recommended with MS, ECDs or with any capillary flow devices. Graphite is permeable to air, be very careful.

Active sites in the flow path



Extensive inlet maintenance needed





# Supply Selection: Graphite/Polyimide Blend Ferrules

Graphite/polyimide (Vespel) blend ferrules are very popular

## Recommendations

- Long style for mass spec
- Short for inlets, and other oxygen-sensitive detectors, like ECD



Soft enough to make a seal against rough surfaces

Fairly high temperature stability (350 °C)

Don't extrude beyond the fitting when tightened (like graphite)

Lower air permeability than pure graphite



# Capillary Column Nuts

Column nuts are determined by the instrument fitting  
Basic mechanical fittings with little enhancements  
Brass for the MS (soft to reduce xfer line damage)  
Some finger-tight designs for ease-of-use  
New design addresses ease-of-use and productivity issues of leaking and over-tightening



# Graphite/Polyimide Blend Capillary Ferrules

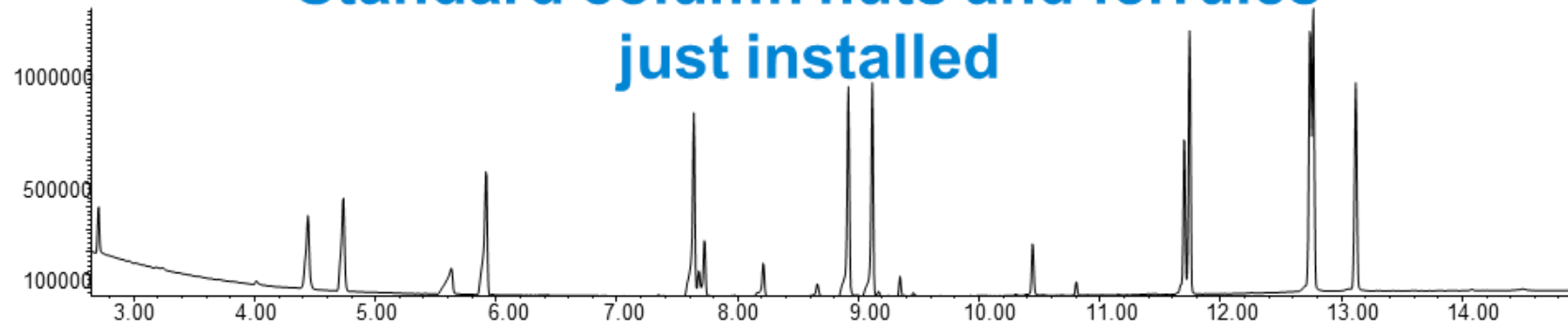
Unfortunately, there was a leak following normal temperature program runs

Studies show the leaking continues with use of the ferrules

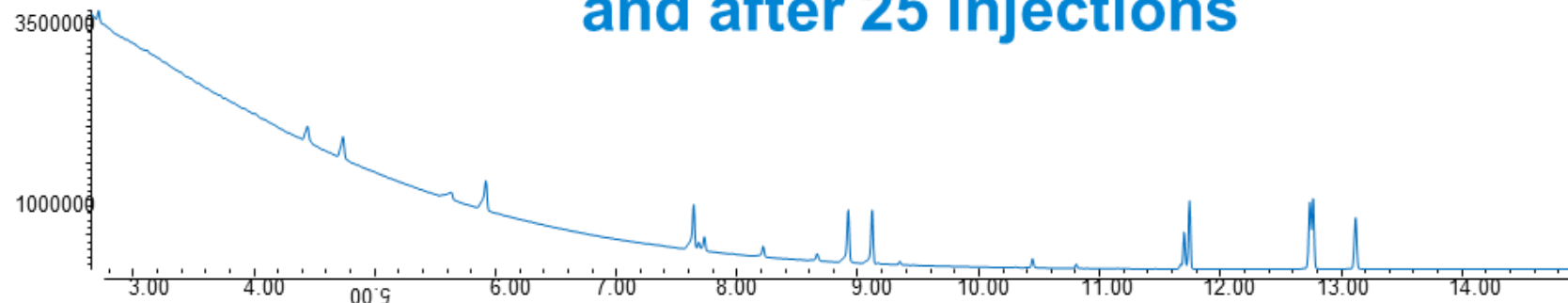
*Not* just after the first one or two runs

Frequent retightening of the fitting is needed to maintain a leak-free seal, and system performance and productivity

## Standard column nuts and ferrules just installed



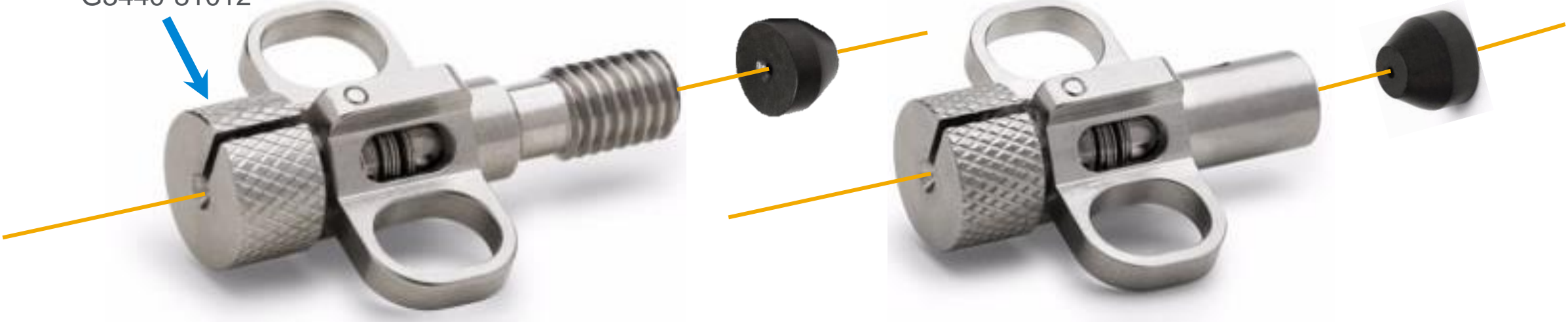
## and after 25 injections



# Better Connections: Collard 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 is needed for both ends of the column

Replacement Collar  
G3440-81012



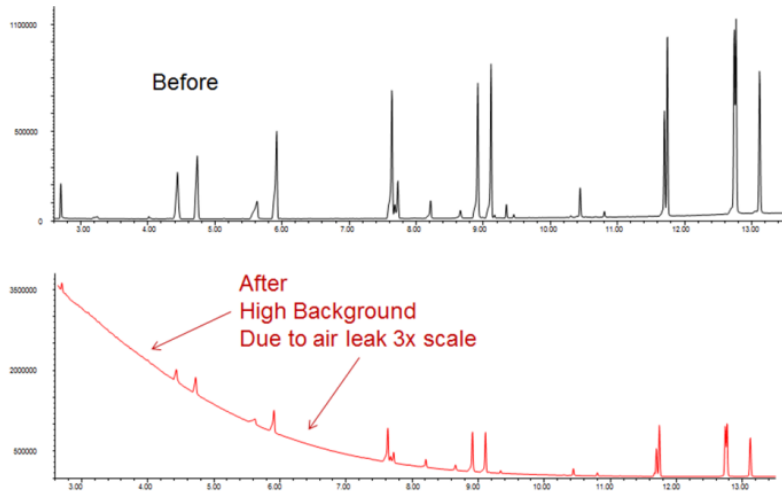
For inlet or detector  
p/n G3440-81011



For mass spectrometry transfer line  
p/n G3440-81013

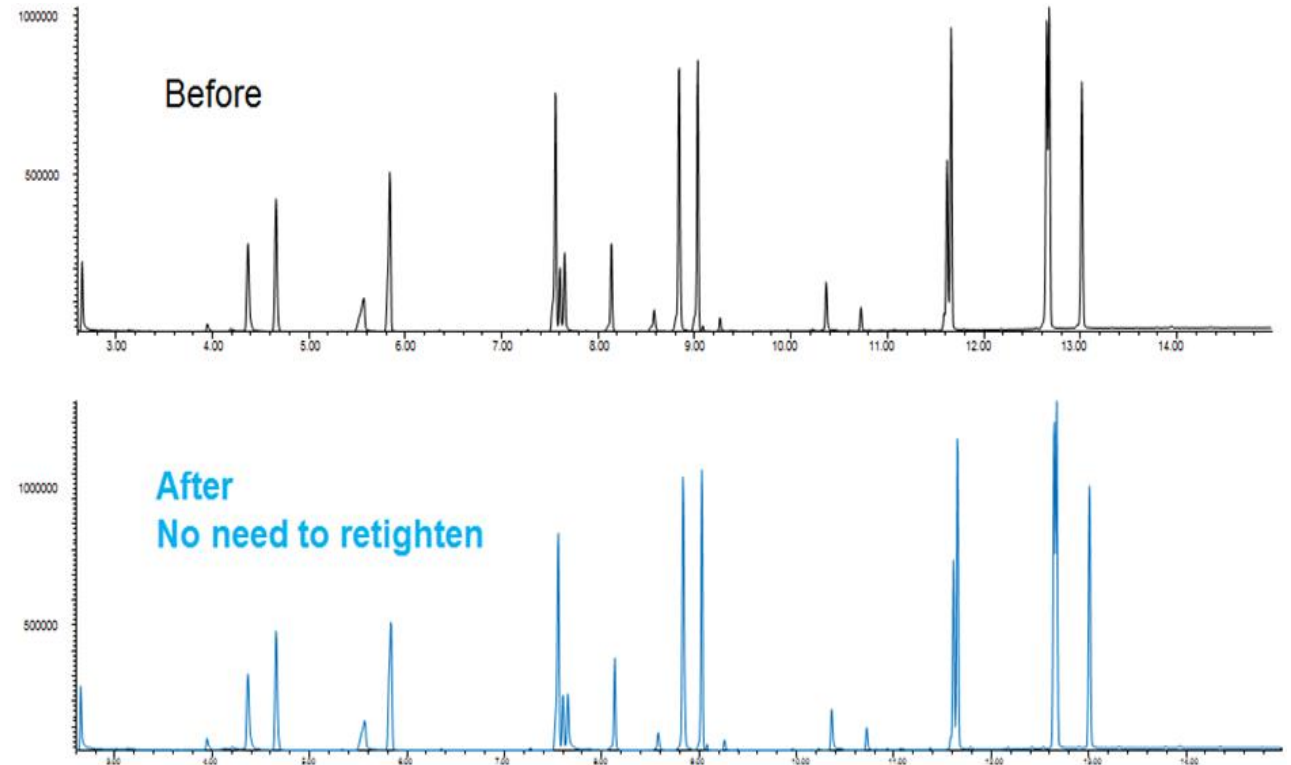


# Benefit of Self-Tightening Column Nuts



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 nut

Technical note: 5991-3612EN

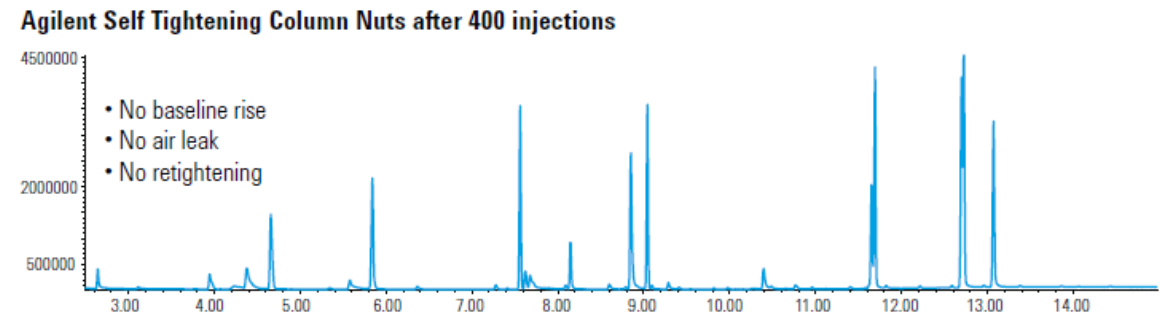
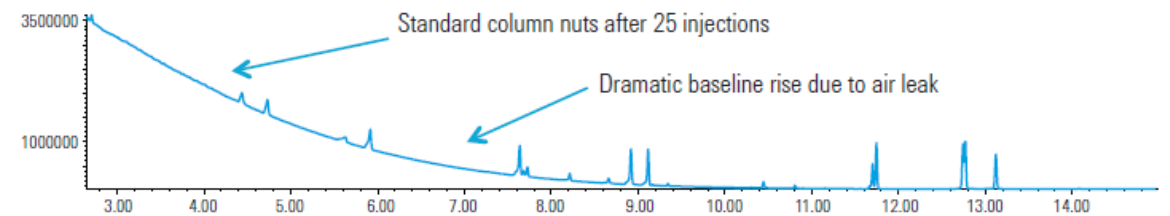
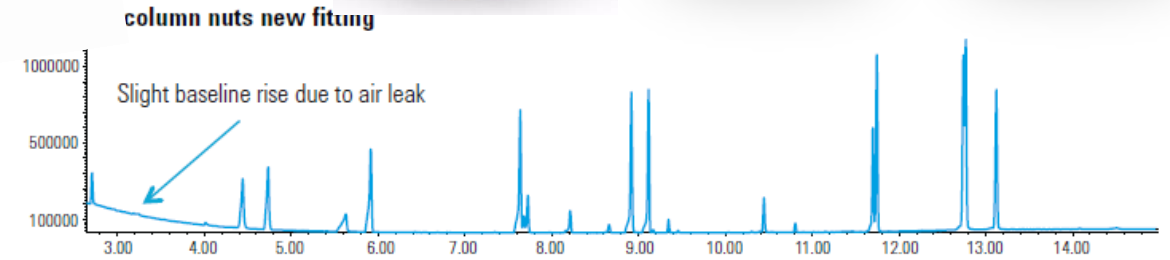
# Self Tightening Nuts: No Leaks, No Downtime, No Frustration

- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- No tools needed
- **Works only with short graphite/Vespel ferrules**



Part Number	Description
G3440-81013	Column Nut, Collared Self-Tightening MSD
G3440-81011	Column nut, Collared Self Tightening Inlet/Detect
G3440-81012	Collar for Self Tightening Nut

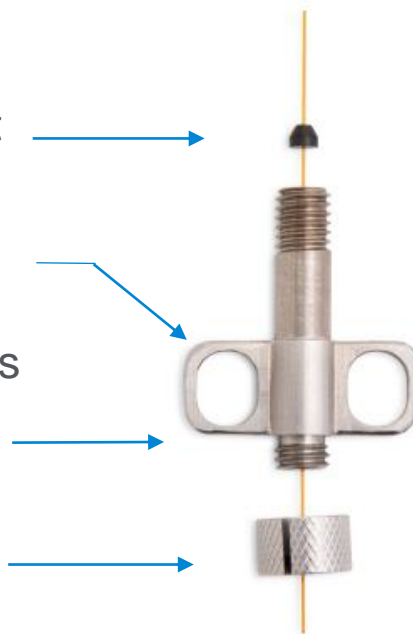
<https://www.agilent.com/en/video/gc-supplies-innovation>  
<https://www.agilent.com/en/video/stcn-inlet-detector>  
<https://www.agilent.com/en/video/stcn-mass-spec>



**400 injections**

# New Agilent Standard Winged Nut and Depth Guide

- Compatible with Agilent/HP style compact ferrules, including graphite ferrules
- Winged fastener design for easy engagement and tool-free install
- Hollow-body design with low thermal mass mitigates thermal lag during temperature cycling within the GC oven
- Removable locking-collar with soft-PTFE insert to secure column placement during install without damaging the analytical column



Don't confuse them with the Self-tightening nuts!



Inlet / Detector  
G3440-81018









MSD  
G3440-81019









- Easy to use template provides critical capillary column installation for the most popular Agilent GC configurations
  - SSL, MMI, purge-packed inlets
  - FID, TCD, NPD detectors
  - EI MSD source
- Compatible with the Agilent Self Tightening and winged column nuts



# Matching the Correct Nut with the Correct Ferrule

<u>Nuts/Ferrules for Inlets and Non-MS Detectors (Male)</u>			
Photo	Nut	Photo	Ferrule
	Traditional/ legacy nut		Short
	Wing nut		Short
	Self Tightening column nut		Short – G/V only

<u>Nuts/Ferrules MSD (Female – G/V Only)</u>			
Photo	Nut	Photo	Ferrule (G/V Only!)
	Traditional/ legacy nut		Long
	Wing nut		Long
	Self Tightening column nut		Short – G/V only



# Specialty Nuts for Non-Agilent Instruments

## Self Tightening column nuts

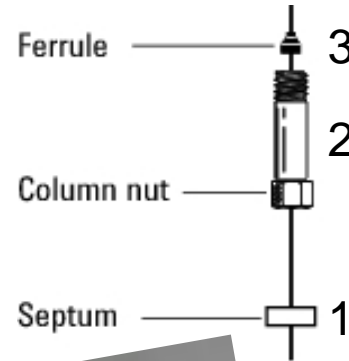
## Wing nuts

PART NUMBER	DESCRIPTION
8001-0015	Agilent Self Tightening column nut for Shimadzu GC/MS. Includes nut for SPL inlet, nut for MS transfer line connection, 0.4 mm graphite/polyimide ferrules (10/pk) for 250 µm columns
8001-0016	Agilent Self Tightening column nut kit for Shimadzu, includes 2 nuts, 10 ferrules for 250 µm columns
8001-0017	Agilent Self Tightening column nut kit for Shimadzu, includes 2 nuts, 10 ferrules for 320 µm columns
8001-0018	Agilent Self Tightening column nut kit for Shimadzu, includes 2 nuts, 10 ferrules for 530 µm columns
8001-0020	Agilent Self Tightening column nut, for Shimadzu QP-2010 MS with MS adaptor on the inlet. Includes 2 nuts for MS system connections, 0.4 mm graphite/polyimide ferrules (10/pk) for 250 µm columns.
8002-0010	Agilent Self Tightening column nuts for Thermo Trace and Focus with MS, for SPL inlet and 250 µm columns
8002-0013	Agilent Self Tightening column nuts for Thermo Trace 1300 with MS, for SPL inlet and 250 µm columns
8003-0010	Agilent Self Tightening column nuts for PerkinElmer, for SPL inlet and 250 µm columns
8004-0009	Agilent Self Tightening column nuts for Varian/Bruker, for SPL inlet and 250 µm columns
8004-0011	Agilent Self Tightening column nuts for Varian/Bruker, for SPL inlet and 530 µm columns

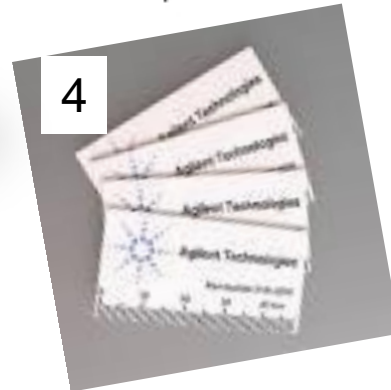
Part Number	Product Description	Ref Price
G3440-81018	Agilent Winged Column Nut, Collard, Inlet/Detector	\$57.00 USD
G3440-81019	Agilent Winged Column Nut, Collared, MSD	\$57.00 USD
G3440-88000	Agilent Capillary Column Depth Guide	\$62.00 USD
8001-0022	Shimadzu Winged Column Nut, Collared, Inlet/Detector	\$57.00 USD
8001-0022	Shimadzu Winged Column Nut, Collared, MSD	\$57.00 USD



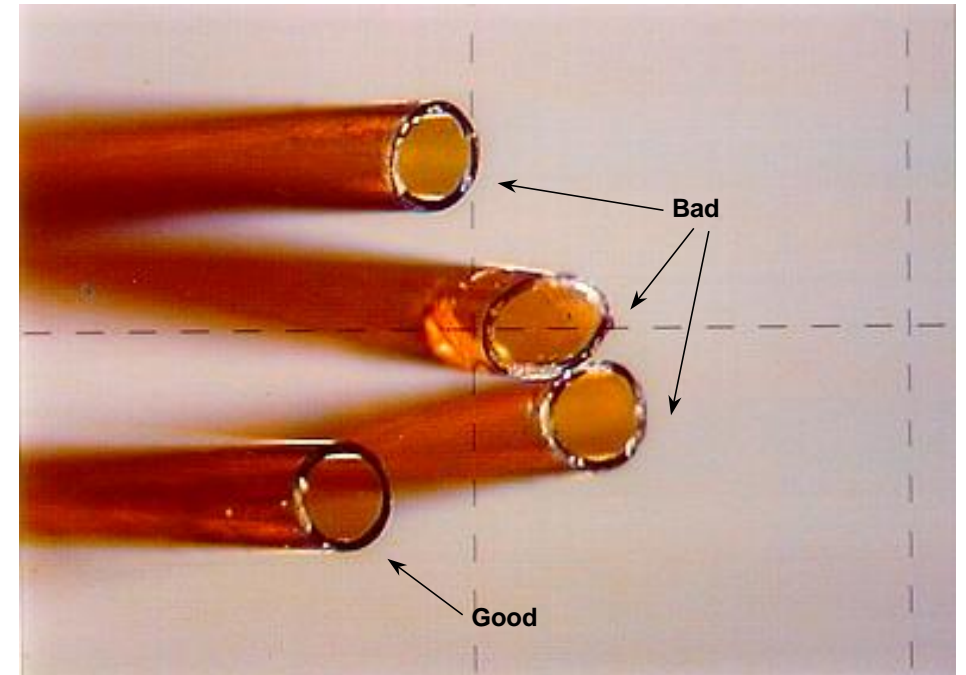
# Column Installation Assembly Process



- Thread through an inlet septum
- Pass column through the column nut
- Install ferrule onto the column tubing
- Then** make a fresh cut
- Inspect the cut; repeat the cut if there are any jagged or rough edges



430-1020

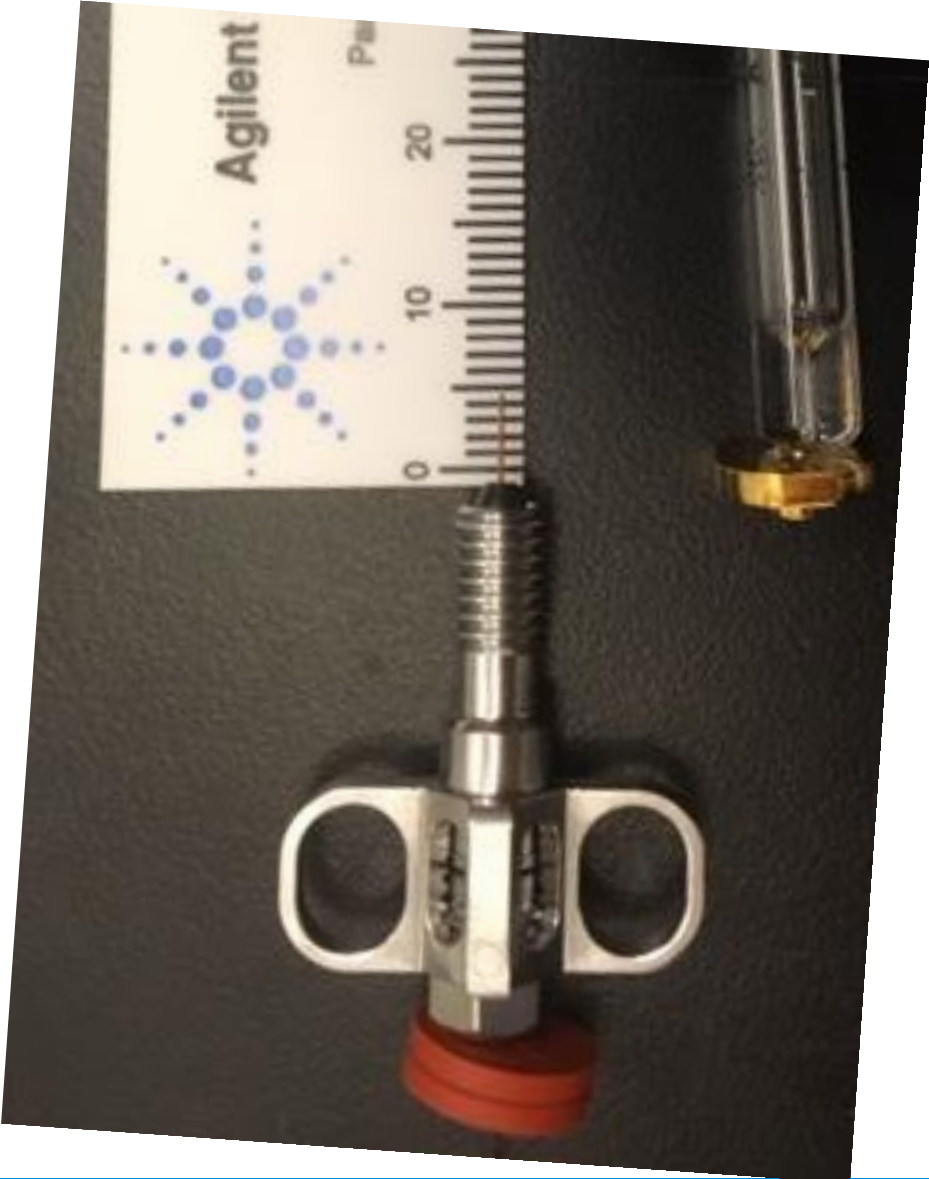


# Installation Distance Matters



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

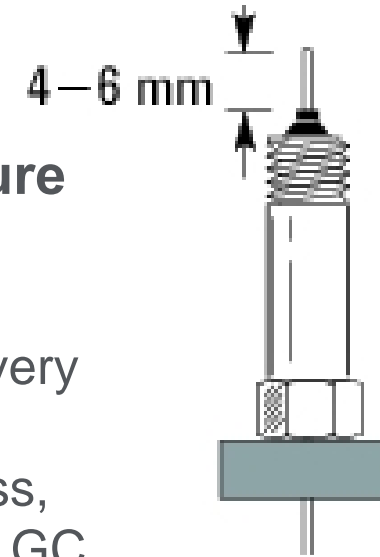
# Why Does Distance Matter?



# 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 preswaging tools for the split/splitless, multimode and purge packed inlets available for Agilent GC models 7890/7820 and 6890/6850, for graphite or metal ferrules



G3440-80217 Graphite ferrules



Metal ferrules G3440-80218



G1099-20030  
MS install tool



G3440-88000

# Pre-swaging Tool for CFT Devices



3-way splitter  
with make-up  
G3183-60501



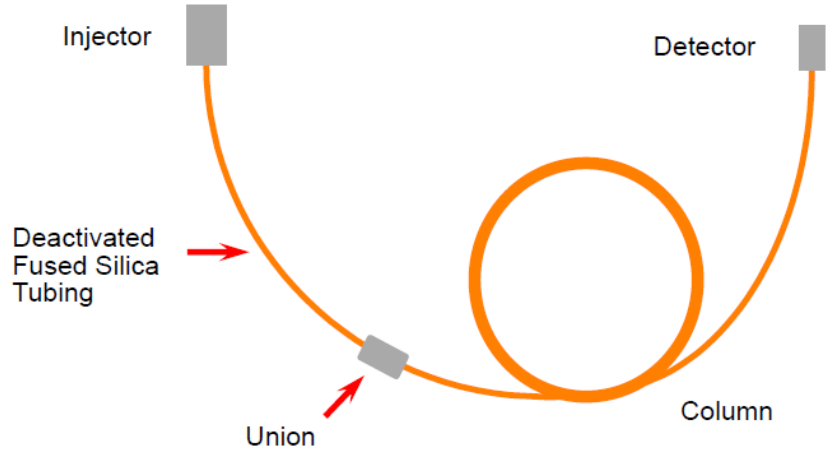
Ultimate Union kit  
G3182-61580



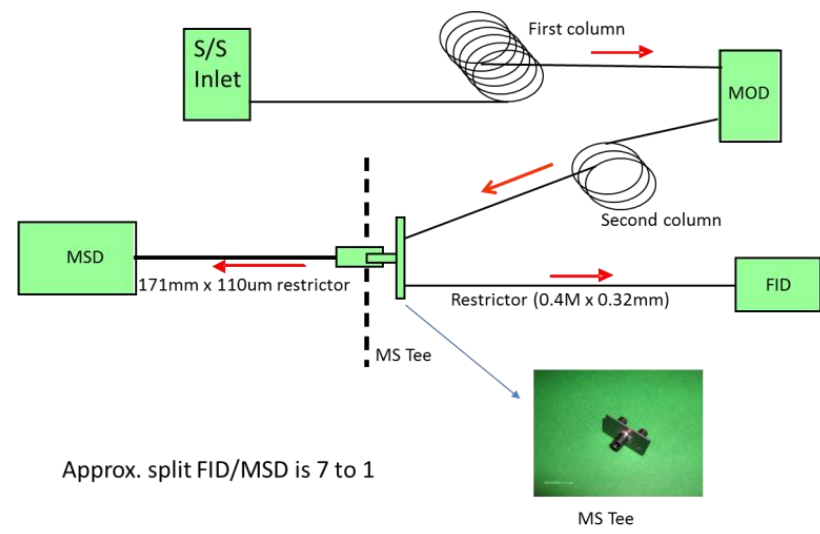
Inert tee  
G3184-60065

# Advanced Techniques Increase the Number of Connections

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



Powerful advanced GC x GC systems with many connections



# Better Connections: Beyond the Basics

## Sample splitting for productivity:

- Split effluent from a single column to two or more detectors on the same GC.
- Backflush – replacing a “bakeout” for high-boiling analytes that collect in the column then interfere with subsequent analyses

## GC x GC or two-dimensional GC:

- An approach used in some environmental, food and flavor, and hydrocarbon processing applications
- Separates complex mixtures using two independent columns with different stationary phase selectivity
- Columns are connected in a series and separated by a modulator

## 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
- Withstand the temperatures used in the GC analysis
- Not be technique-dependent – must be easy to do

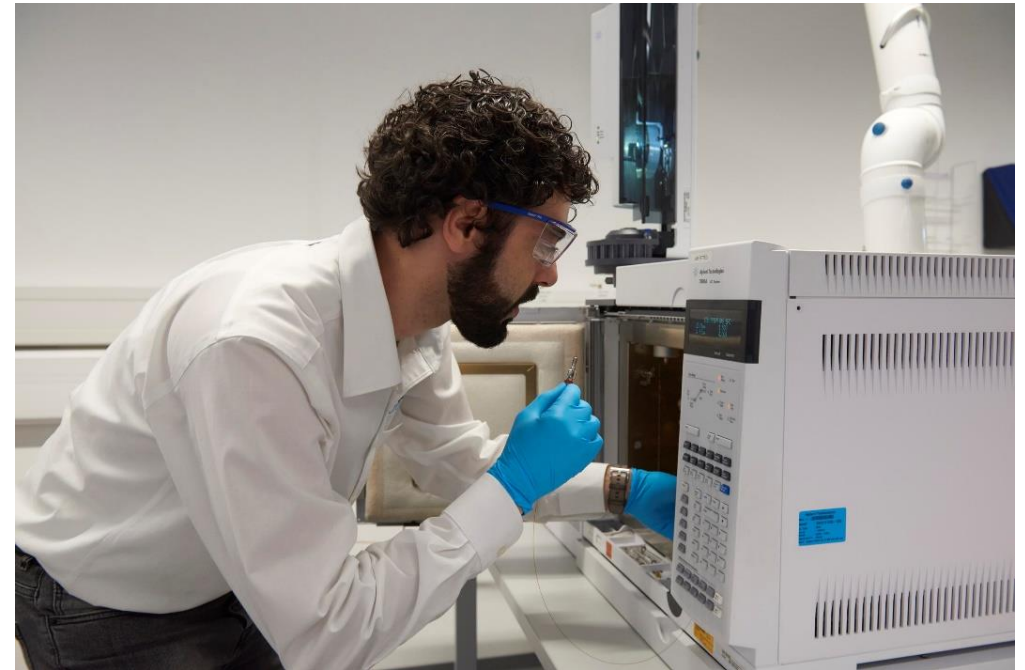


# The Problem with Splitters



Two-holed ferrules are inexpensive. Their challenges are:

1. Difficult installation
2. Poor inlet dynamics
3. Unpredictable splitting
4. Requires a larger-holed gold seal
5. Crowding in single taper inlet liners
6. Lack of guard column when used in the inlet



# The Problem with Splitters

The glass “Y” splitter overcomes many of the challenges of the two-holed ferrule and adds expense.

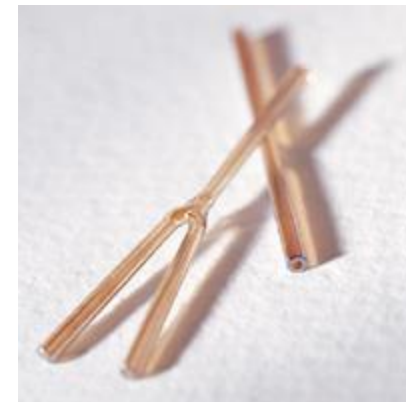
However, installing the connector is somewhat of an art, and even then, it is prone to leaking unpredictably.



## Why can't I get my Press-Union connector to seal?

[Press-Union connectors](#) are easy to use, but if they are not properly sealed, they can loosen due to thermal expansion during temperature-programmed runs. The keys to successful sealing are: 1) making a clean, square cut on the column and 2) moistening the end of the column with methanol or acetonitrile before seating it into the connector. A small amount of polyimide resin (Part # **500-1200**) also helps prevent the seal from separating during temperature cycling.

← Not so easy



# 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

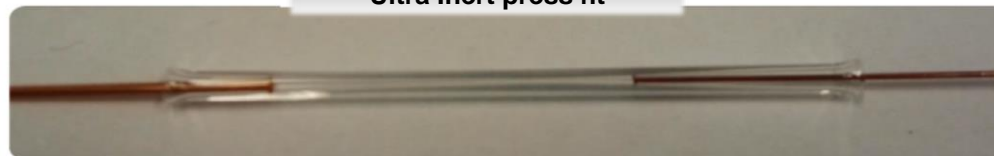
Easier to use – transparent deactivation gives visibility of the column connection



Traditional deactivated connector



Ultra Inert press fit



## Press Fit Part Numbers

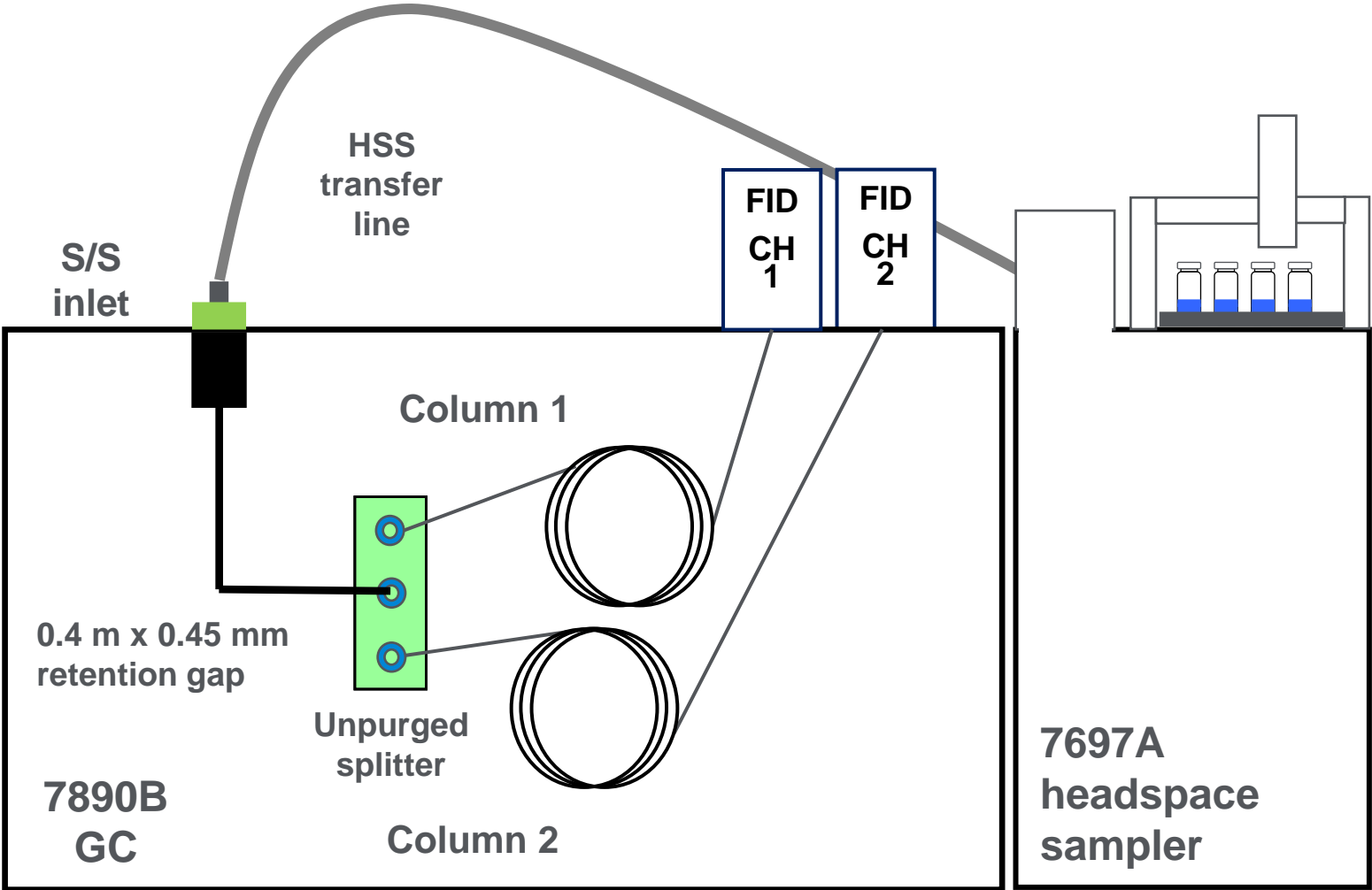
Universal press fits (Ultra Inert)	5190-6979
Universal “Y” splitter (Ultra Inert)	5190-6980
Sealing resin	500-1200



# 2 columns one injection: Agilent Solution



Inert tee  
G3184-60065



G3445B#683

# Better Connections: Capillary Flow Technology Devices

## UltiMetal Plus Ultimate Union/UltiMetal Plus tee

- Stainless steel microfluidic plates technology
- Deactivation essential to block active sites
- Column connection easy to assemble, release hole for stuck ferrules



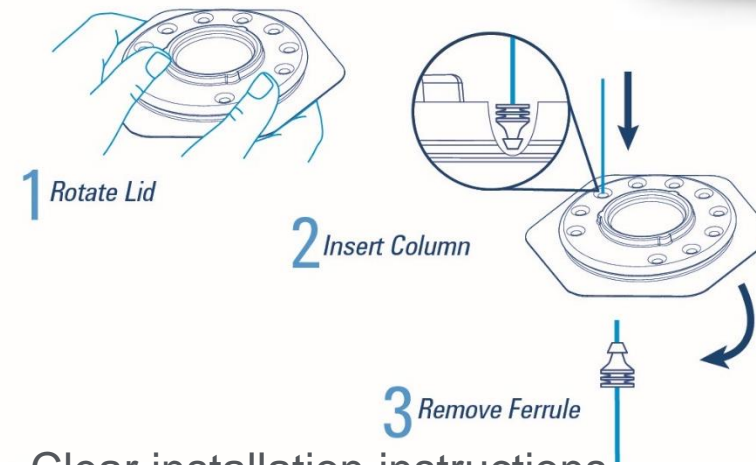
## Using flexible metal ferrules to overcome issues

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



# Flexi-Metal Ferrules: Touchless Dial Packaging

- Easy column insertion
- No lost ferrules
- No contamination risks
- Unique shape indicates column diameter
- Superior to SilTite™ ferrules



Clear installation instructions

[Flexible Metal Ferrules Swaging Guide for UltiMetal Plus and Gold-Plated Ferrules \(agilent.com\)](https://www.agilent.com/chem/flexiferrule)

# Added Value

**Table 1 Available UltiMetal Plus and Gold-plated Flexible Metal ferrule packages.**

Item	Compatible Column id	UM+ FMF part number	Gold-Plated FMF part number
1	0.1 to 0.25 mm	G3188-27501	G2855-28501
2	0.32 mm	G3188-27502	G2855-28502
3	0.45 to 0.53 mm	G3188-27503	G2855-28503
4	Plug	G3188-27504	N/A
5	0.25 to 0.32 mm UltiMetal	G3188-27505	G2855-28505
6	0.53 mm UltiMetal	G3188-27506	G2855-28506



Each id has its own unique design  
Ease of recognition, no mix-ups

# Agilent UI Gold Seal: Deactivated Gold Surface

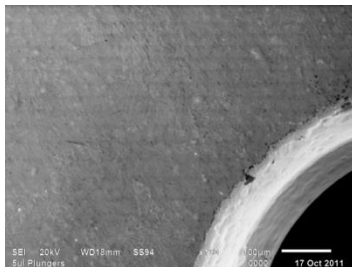
Soft gold plating is essential for proper sealing

Ultra Inert chemistry blocks active sites (gold is **not** inert)

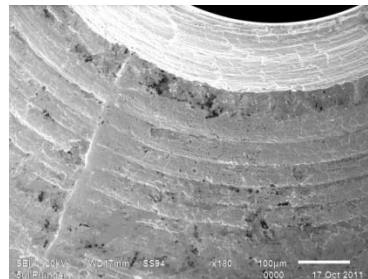
Smooth surface doesn't leak

Advantage Agilent

p/n 5190-6144 ea, 5190-6145, 10/pk (washer included)



Agilent MIM seal



Competitor's  
machined seal

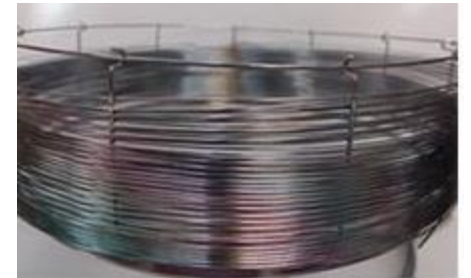
Reliable ppb and ppt  
measurements require  
attention to the little things



# 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, tees 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

# Agilent Inert Flow Solution

UltiMetal Plus inlet weldment, shell and transfer lines



Ultra Inert inlet liner



UltiMetal Plus ferrules  
Gold Ferrules



UltiMetal capillary flow technology devices, Ultimate Union



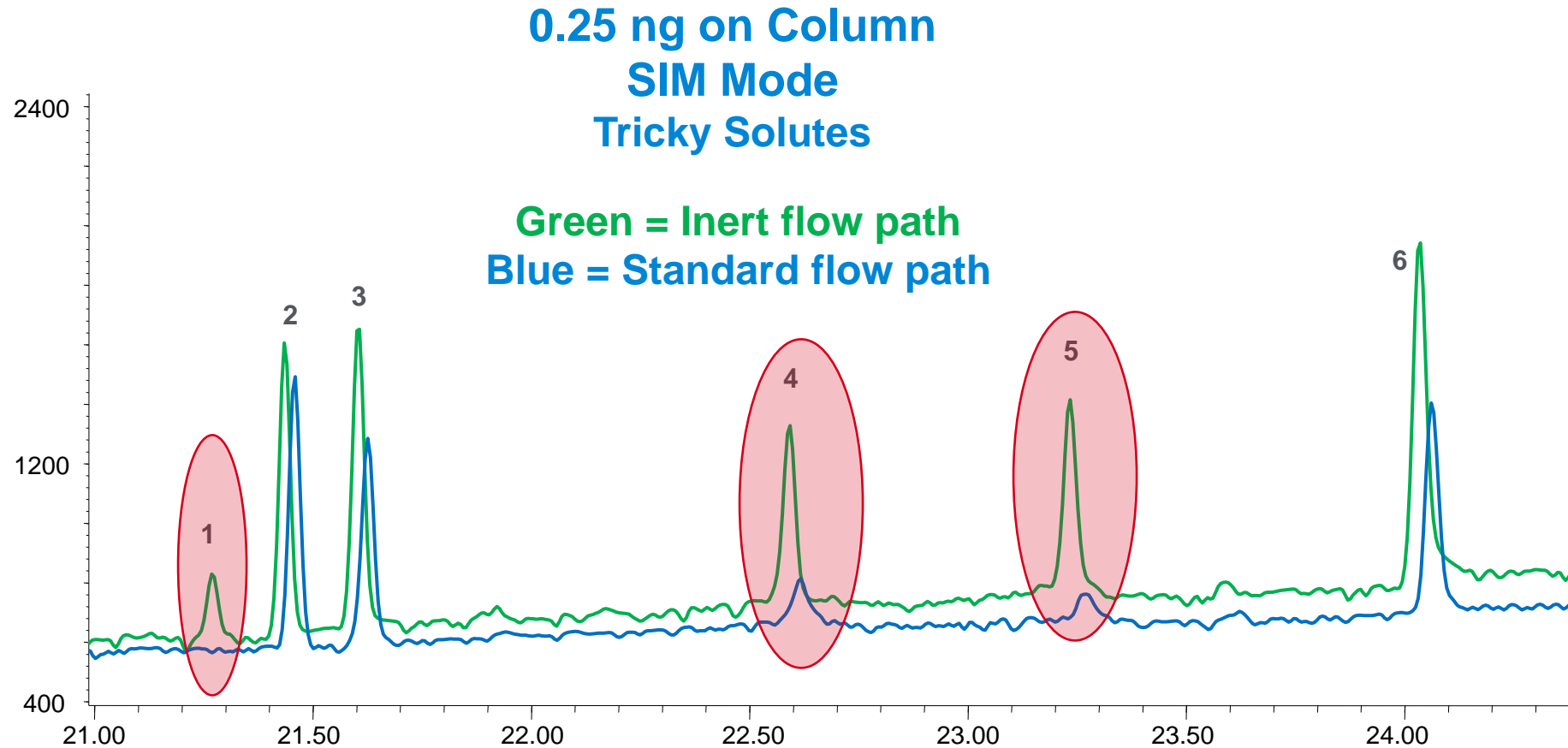
Ultra Inert gold seal

Ultra Inert GC column

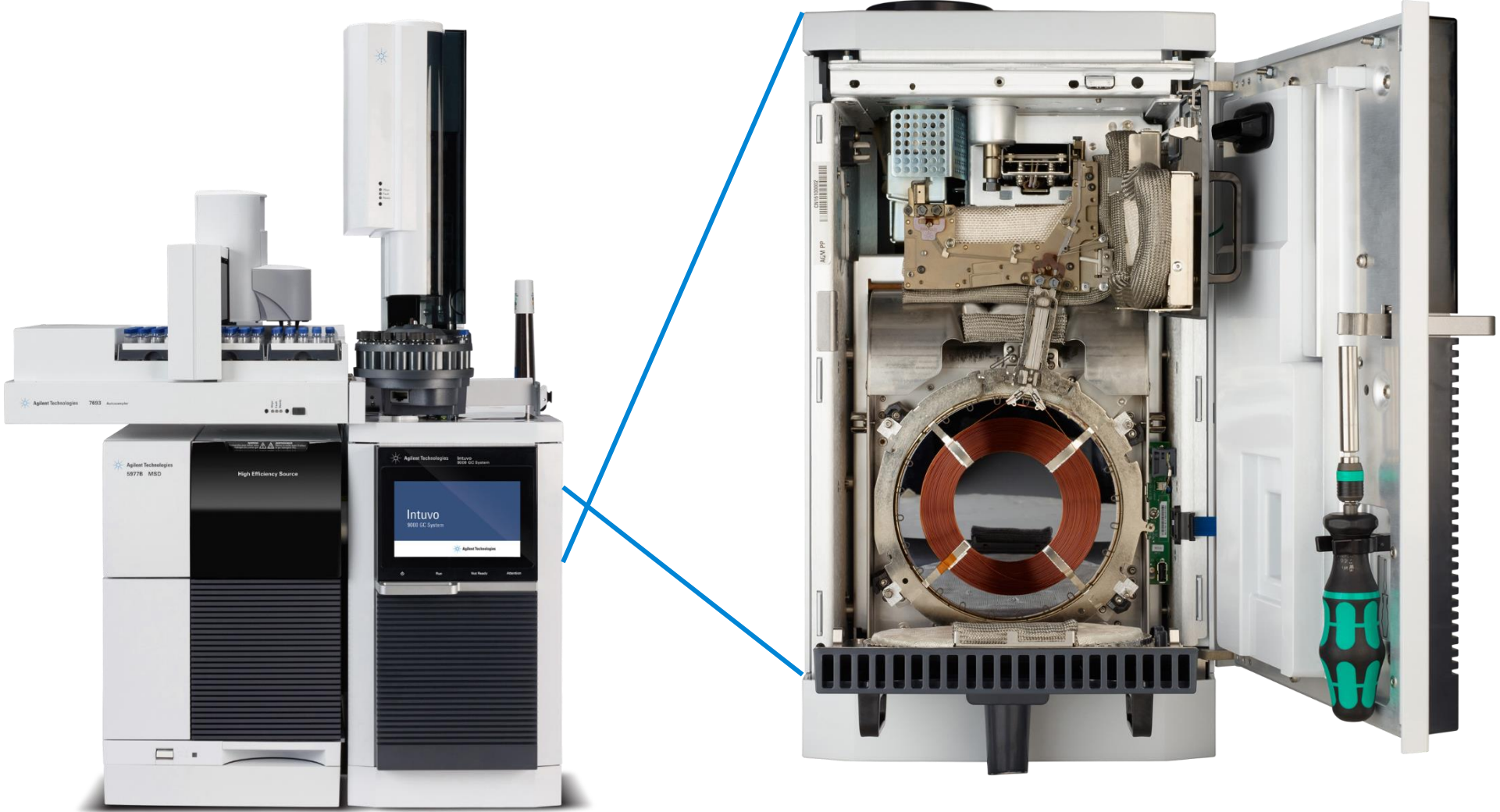


# Putting It All Together – Agilent Inert Flowpath

## Dramatic improvement at low levels

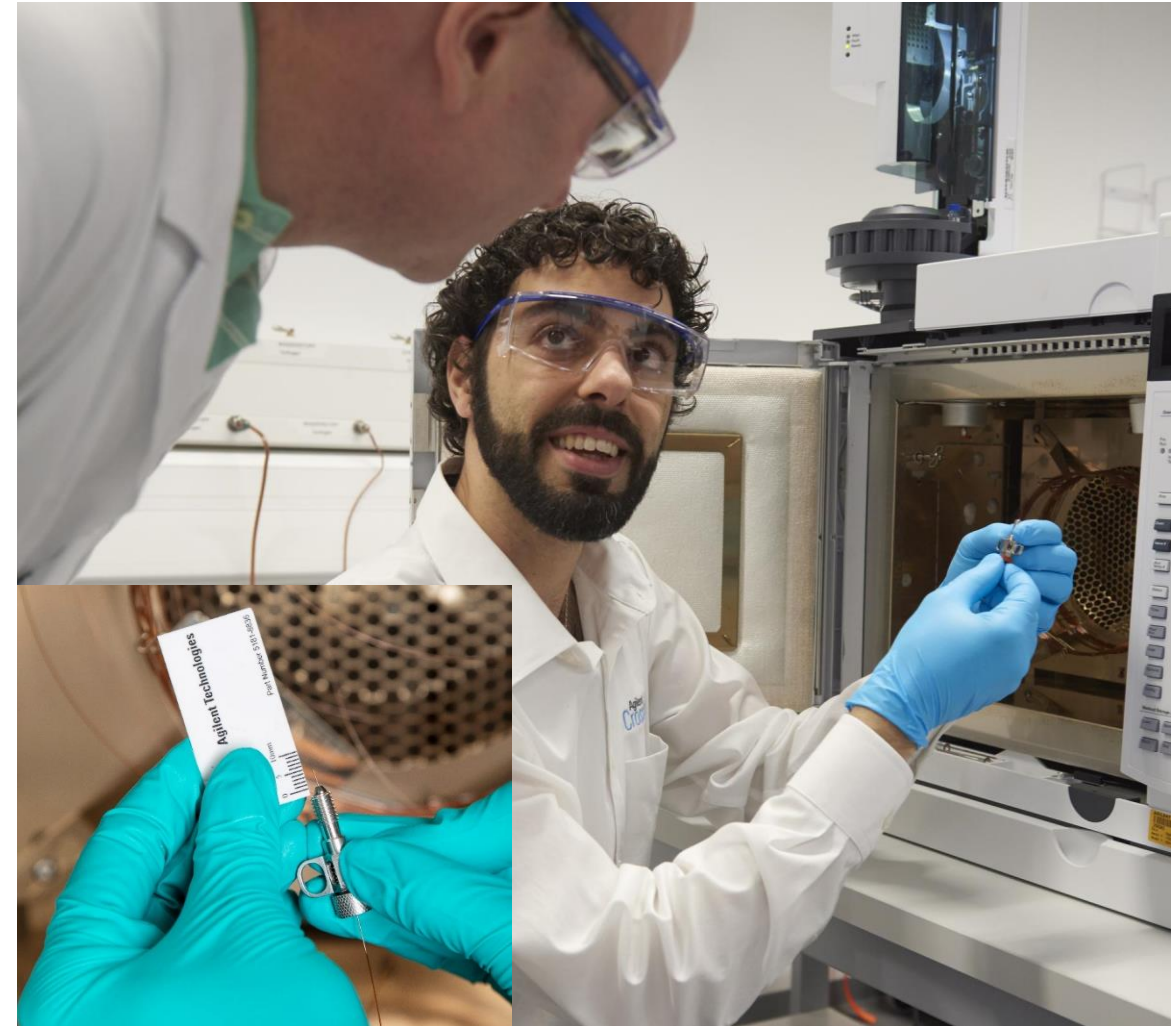


# The Agilent INTUVO 9000 GC System



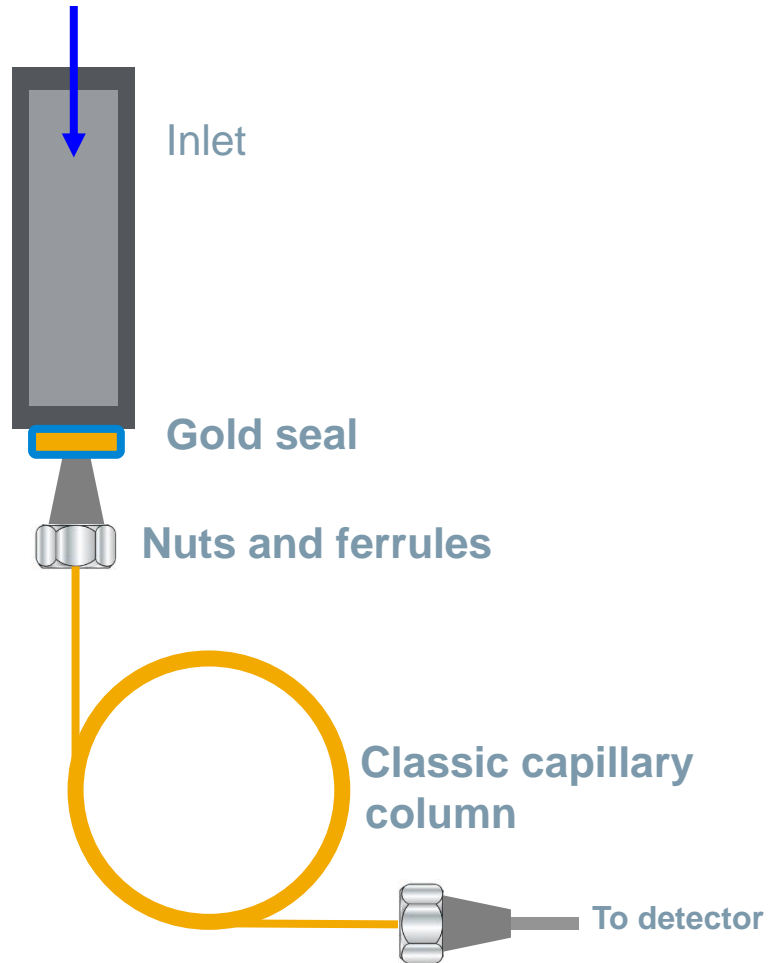
# Common Frustrations with GC

- Measuring column length correctly
- Cutting your column correctly
- How tight is too tight?
- Clipping columns to deal with active sites, then updating retention times

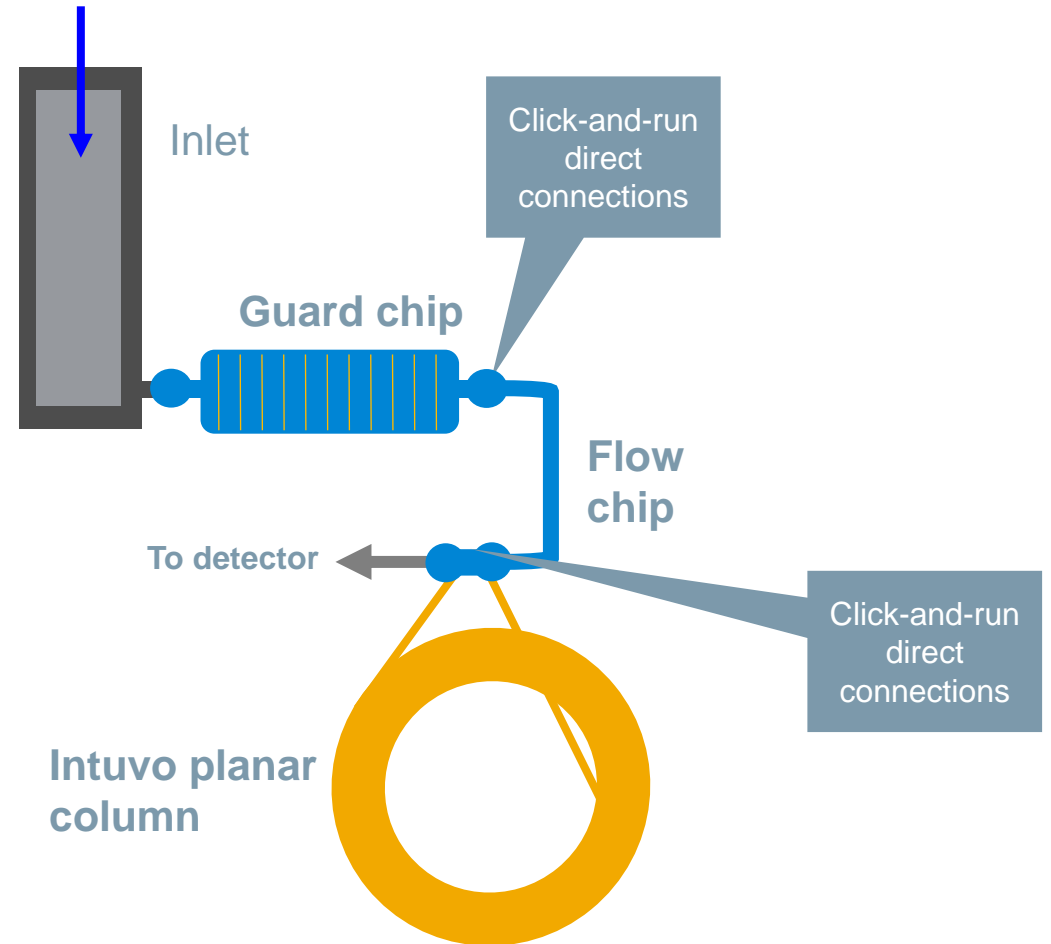


# Innovating the GC Flow Path

## Conventional flow path

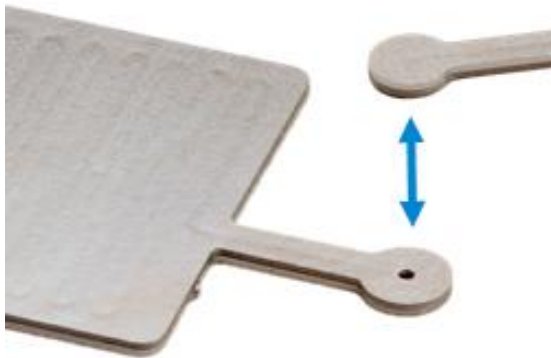


## Intuvo flow path

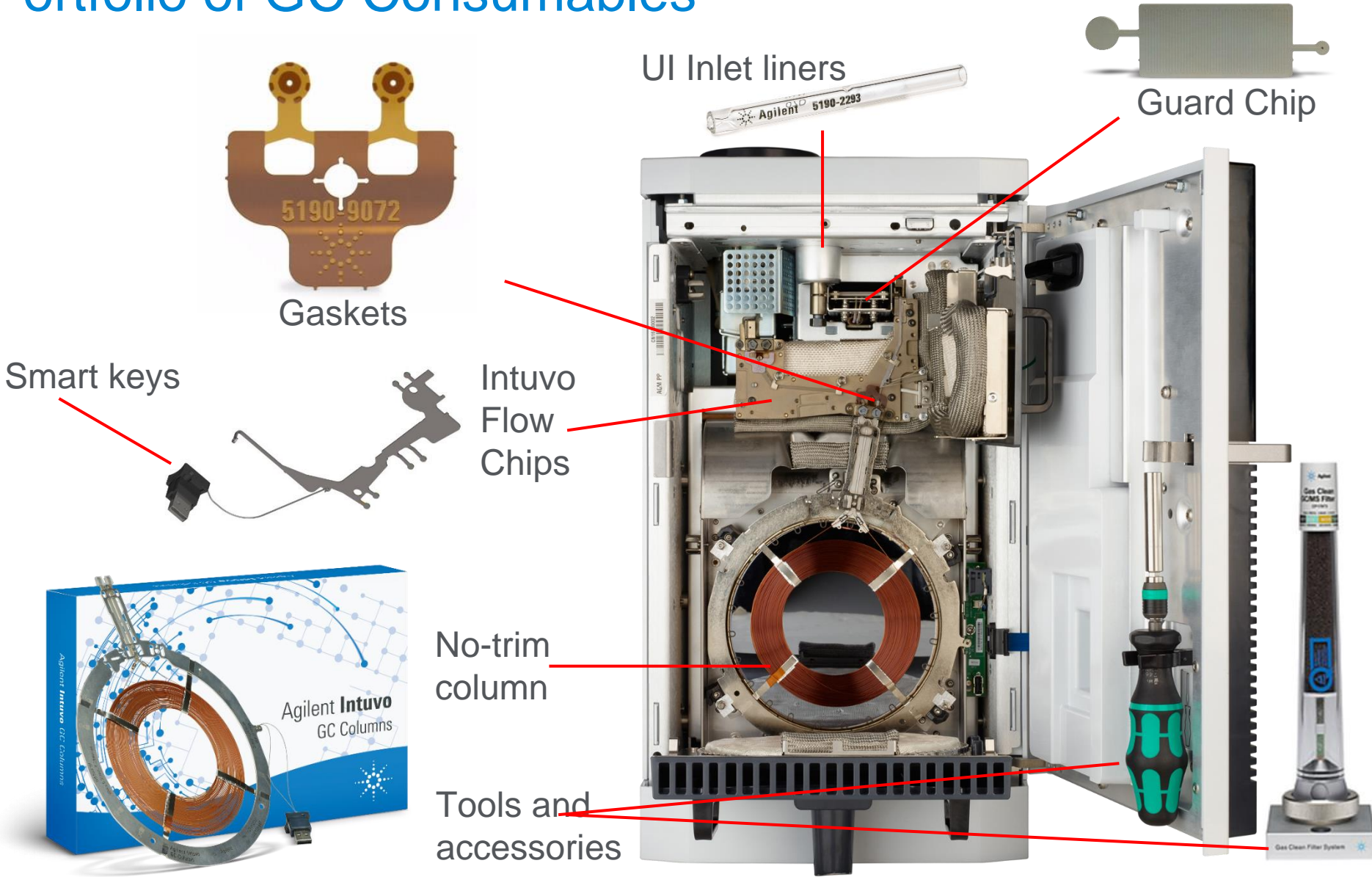


# Easier and Faster Maintenance with INTUVO

- No more ferrules
- Direct face seal connections
- Audible and tactile click lets you know connection is made
- Less unplanned downtime
- Fewer batch reruns and precious samples lost



# A New Portfolio of GC Consumables





# Guard/Jumper Chips



S/SL Jumper: G4587-60575 (ea)

Roughly 0.75 meters x 0.53 mm id →



S/SL: G4587-60565 (2/pk)



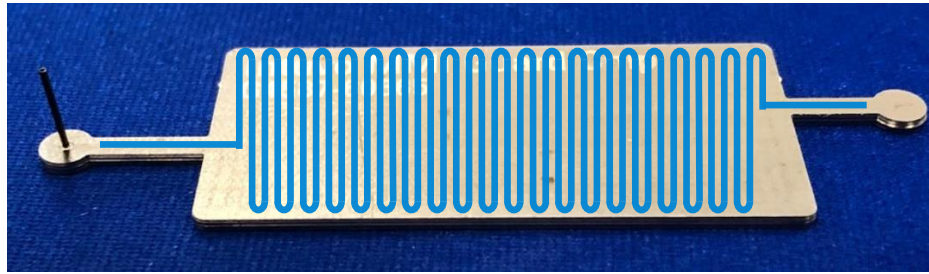
MMI: G4587-60665 (2/pk)

# Guard/Jumper Chips



S/SL Jumper: G4587-60575 (ea)

Roughly 0.75 meters x 0.53 mm id



MMI: G4587-60665 (2/pk)



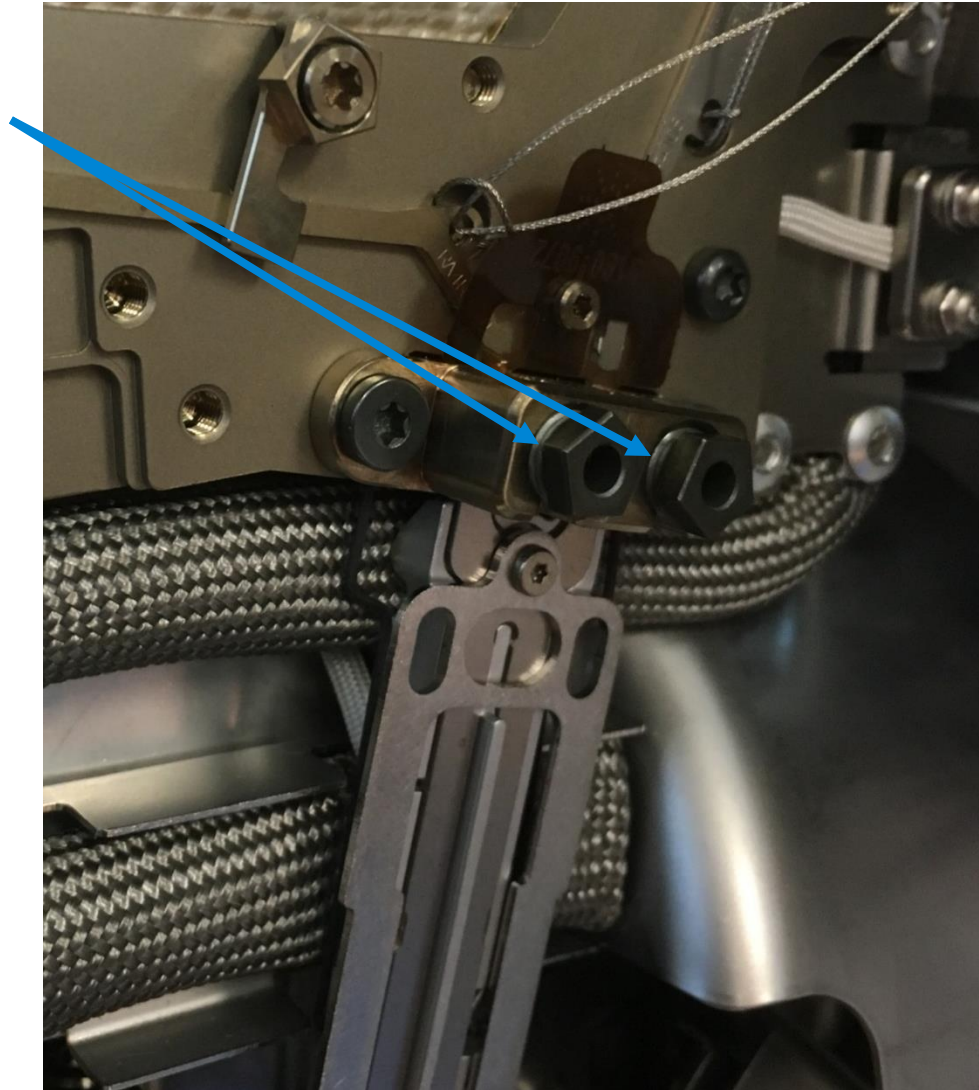
S/SL: G4587-60565 (2/pk)



# Tips to Assure a Good Column Installation

Finger tighten until only one thread on each of the two nuts is showing.

If more than one thread is showing, wiggle or reposition the column into place to further finger tighten the nuts to one thread.



# Tips to Assure a Good Column Installation

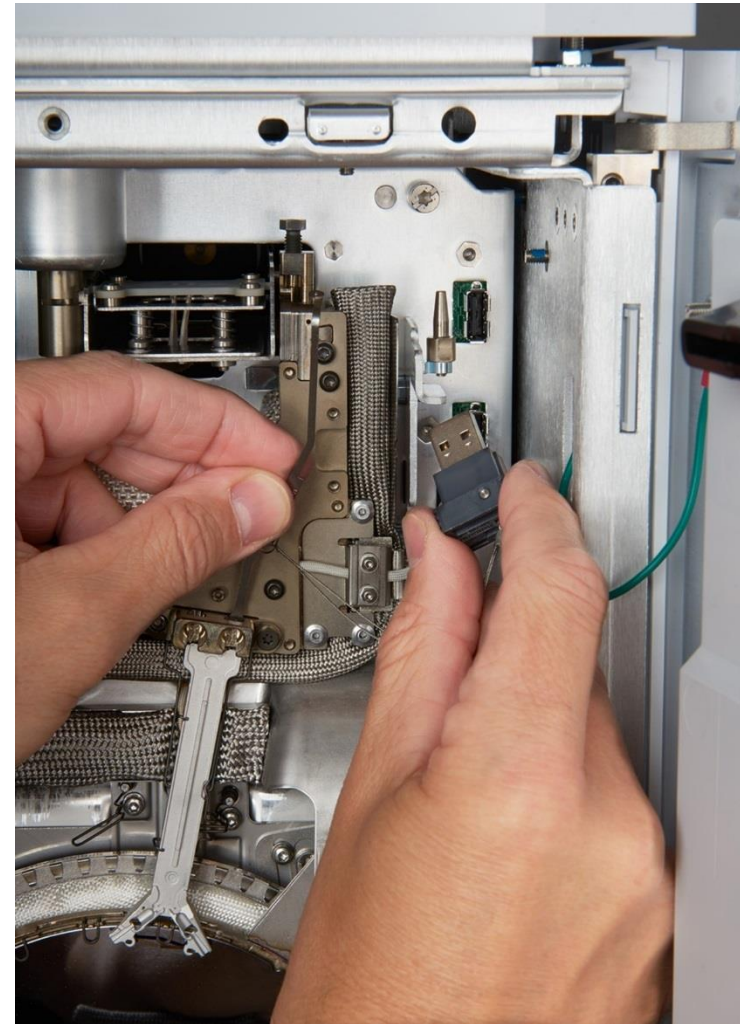
Check that the small integrated column nuts on the column are in form-fitted place on the heater in the instrument.

Click and run



# Smart Key Technology

- Smart chip tells your Intuvo what you have
- Sets temperature limits for you
- Keeps track of performance with read/writeable smart key



# Agilent Intuvo 9000 Videos:

## [The Agilent Intuvo 9000 GC System – Environmental Science Corporation \(ESC\)](#)

Discover higher GC productivity with the Agilent Intuvo 9000 GC system

Playing time: 4:00

## [The Agilent Intuvo 9000 GC System Story](#)

Learn more about the Agilent Intuvo 9000 GC system

Playing time: 2:21

## [The Agilent Intuvo 9000 GC System: Return on Investment. Return on Innovation.](#)

A testimonial regarding the return on investment on the Agilent Intuvo 9000 GC system

Playing time: 4:17

# Comparing the 7890 to the INTUVO (9000)

	7890				INTUVO	
<b>Footprint (H x W x D) inches</b>	19.2 x 22.9 x 20.2				20 x 10.7 x 27.2	
<b>Power Consumption</b>	2250 VA standard (2950 VA /220)				1296 VA standard (1548 VA/220)	
<b>Oven Ramp Rate</b>	<b>Temperature range</b>	<b>120 V oven</b>	<b>Fast oven</b>	<b>Fast oven with pillow</b>	Maximum achievable temperature ramp rate: 250 °C/min across all temperature ranges	
	50-70	75	120	120		
	70-115	45	95	120		
	115-175	40	65	110		
	175-300	30	45	80		
	300-450	20	35	65		
<b>Oven Cooling Rate</b>	Oven cool down (22 °C ambient) 450 to 50 °C in 4 minutes, (3.5 with oven insert accessory)				Oven cool down (22 °C ambient) 450 to 50 °C in less than 3 minutes	
<b>Heat Dissipation</b>	Standard oven ramp		7681 BTU/hour maximum		120 V	4424 BTU/hour maximum
	Fast oven ramp		10,071 BTU/hour maximum		200 – 240 V	5285 BTU/hour maximum

# Use Leak Detector or Electronics Duster to Find Your Leaks

## Why use a leak detector?

- High sensitivity
- Recommended for leak detection in gas plumbing and fittings



Flow meter/leak detector bundle - G6699A

## Typical Electronic Duster Components and Ions

<chem>CC(F)F</chem>	1,1-difluoroethane	<i>m/z</i> 51,65
<chem>CC(F)(F)F</chem>	1,1,1-trifluoroethane	<i>m/z</i> 69,83
<chem>CC(F)F(F)F</chem>	1,1,1,2-tetrafluoroethane	<i>m/z</i> 101

## Use electronics duster

- Hold can upright (don't spray liquid)
- Spray short bursts around possible leak points
- “Live” tune profiling for ions to pinpoint leak



# Ordering Guide

G6693A – CrossLab CS electronic leak detector

G6694A – Electronic leak detector cartridge

G6699A - CrossLab CS bundle: ADM flow meter and electronic leak detector

- The bundle will include one handheld, two cartridges, and a free carrying case.

G6694-60005 – Replacement probe filter

G6691-40500– Carrying case



Existing products:

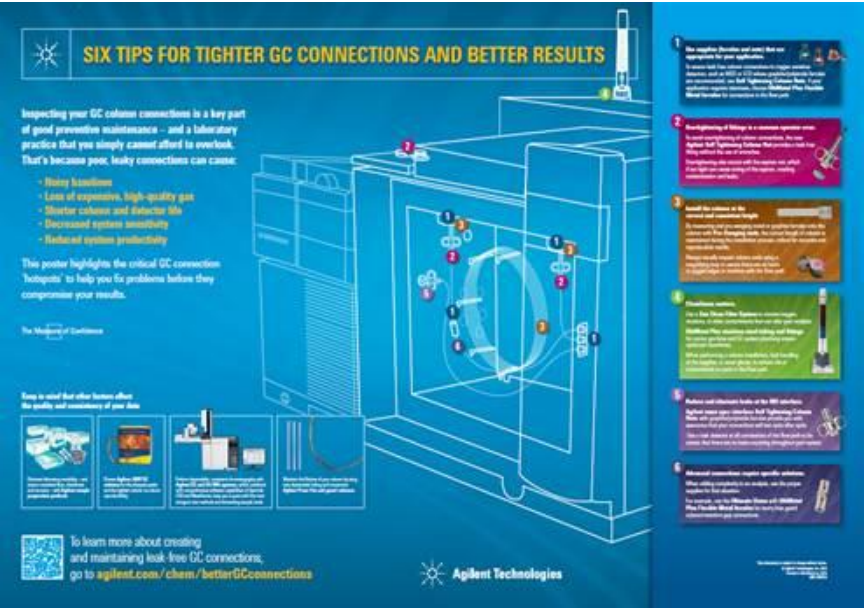
G6691A – CrossLab CS ADM flow meter

G6692A – ADM Flow meter cartridge\*

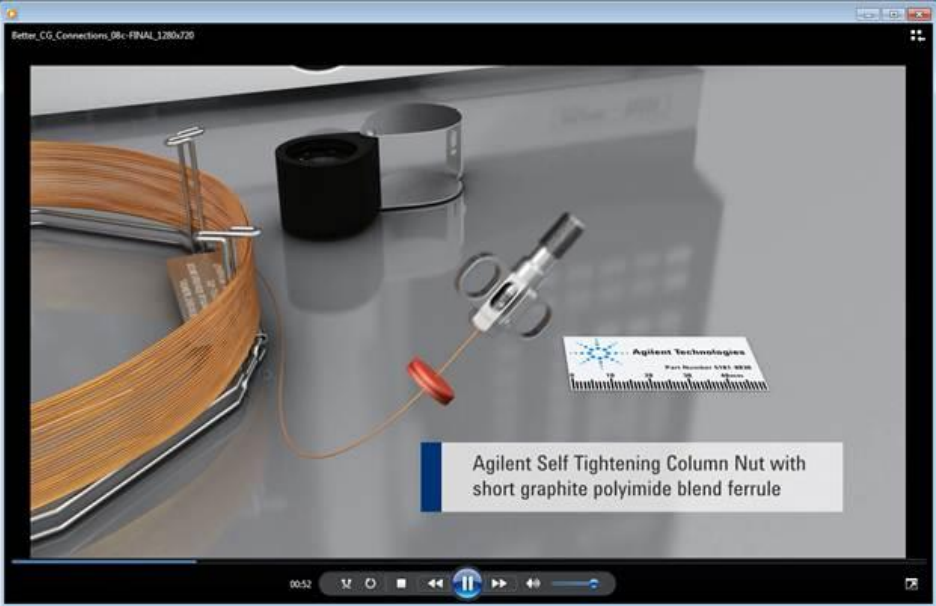
- Note that the ADM flow meter cartridge is ordered annually for calibration. The electronic leak detector does not need to be recalibrated.

Now it's easier than ever to get reliable GC results through better GC connections from Agilent, visit: [www.agilent.com/chem/betterGCconnections](http://www.agilent.com/chem/betterGCconnections)

Order the poster

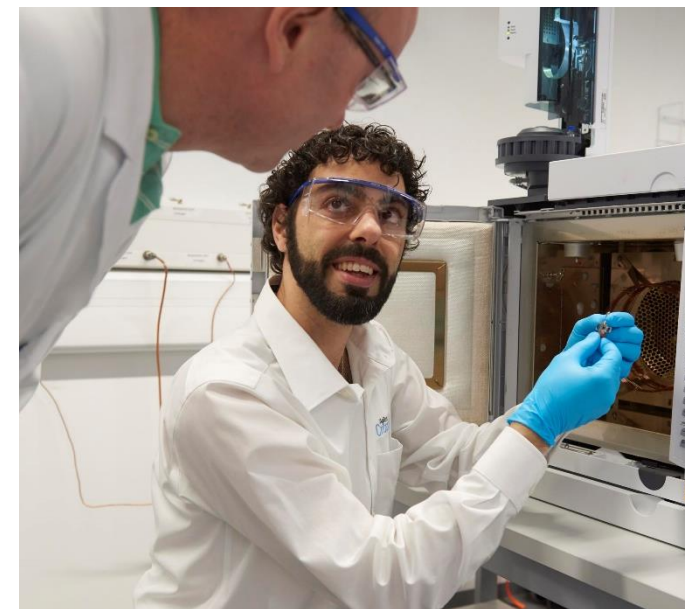


View the video

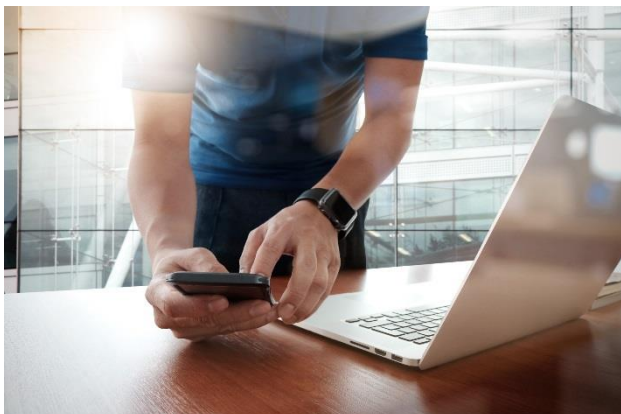


# Summary

- Make clean column cuts – use magnifier!
- Make sure to match nuts to the correct ferrule types, etc. (can be confusing!)
- Use proper swaging techniques/tools
- Ensure proper column distances for the given inlet/detector
- Pay attention to the entire flow path especially for tricky solutes (Ultra Inert)
- INTUVO GC makes connections simpler over-all (Click and go!)
- Use Techsupport when you have questions or issue!



# Contact Agilent Chemistries and Supplies Technical Support



1-800-227-9770 option 3, option 3:

Option 1 for GC and GC/MS columns and supplies

Option 2 for LC and LC/MS columns and supplies

Option 3 for sample preparation, filtration, and QuEChERS

Option 4 for spectroscopy supplies

Option 5 for chemical standards

Available in the U.S. and Canada 8–5, all time zones



[gc-column-support@agilent.com](mailto:gc-column-support@agilent.com)

[lc-column-support@agilent.com](mailto:lc-column-support@agilent.com)

[spp-support@agilent.com](mailto:spp-support@agilent.com)

[spectro-supplies-support@agilent.com](mailto:spectro-supplies-support@agilent.com)

[chem-standards-support@agilent.com](mailto:chem-standards-support@agilent.com)

# Questions

