A Beginner's Guide to Your GC Columns: Installation, Care, and Maintenance

Mark Sinnott and Ryan Birney Application Engineers May 26, 2021





Agenda

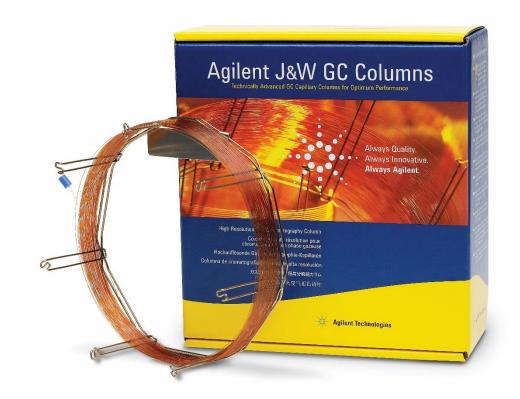
•Unboxing / "getting to know your column"

Install the column

•Preventive measures

•Corrective measures

•Latest instrument developments





Column Construction

Polyimide coating

Flexible polymeric coating; adds mechanical strength and temperature stability to fused silica

Fused silica

Amorphous glass-like tubing comprised of silicon dioxide; high temperature resistance, low reactivity. May contain rough edges creating active sites.

^r Deactivation

Chemical treatment layer; smooths fused silica surface to enhance inertness

Stationary phase

Polymeric coating atop deactivation layer; commonly comprised of polysiloxane- or polyethyleneglycol-based compounds

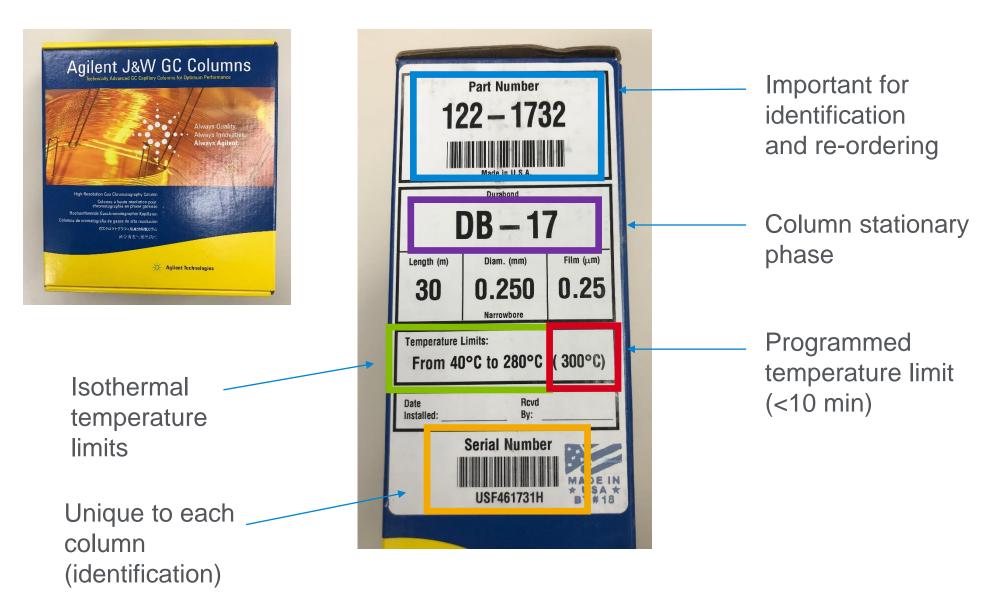


Agilent J&W Column Portfolio- DB, HP, CP, VF

Low Polarity			Mid Polarity			High Polarity			
CP-Sil 2	DB & HP-1ms UI	DB & HP-5ms UI	DB-XLB	DB-225ms	DB-ALC1	HP-88	DB-WAX	DB-WAX UI	
DB-MTBE	DB & HP-1ms	DB & HP-5ms	VF-Xms	DB-225	DB-Dioxin	CP-Sil 88	DB-WAXetr	DB-HeavyWAX	
CP-Select CB MTBE	VF-1ms	VF-5ms	DB-35ms UI	CP-Sil 43 CB	DB-200	DB-23	HP-INNOWax	DB-FATWAX UI	
	DB & HP-1	DB & HP-5	DB & VF-35ms	VF-1701ms	VF-200ms	VF-23ms	VF-WAXms		
	CP-Sil 5 CB	CP-Sil 8 CB	DB & HP-35	DB-1701	DB-210		CP-Wax 57 CB		
	Ultra 1	Ultra 2	DB & VF-17ms	CP-Sil 19 CB	DX-4		DB and HP-FFAP		
	DB-1ht	VF-DA	DB-17	HP-Blood Alcohol			DB-WAX FF		
	DB-2887	DB-5.625	HP-50+	DB-ALC2			CP-FFAP CB		
	DB-Petro/PONA	DB & VF-5ht	DB-17ht	DX-1			CP-WAX 58 FFAP CB		
	CP-Sil PONA CB	CP-Sil PAH CB	DB-608				CP-Wax 52 CB		
	DB-HT SimDist	Select Biodiesel	DB-TPH				CP-WAX 51		
	CP-SimDis	SE-54	DB-502.2				CP-Carbowax 400		
	CP-Volamine		HP-VOC				Carbowax 20M		
	Select Mineral Oil		DB-VRX				HP-20M		
	HP-101		DB-624				CAM		
	SE-30		VF-624ms				CP-TCEP		
			CP-Select 624 CB						
			DB-1301	Agilent J&W columns have over 50 different stationary phase offerings					
			VF-1301ms						
			CP-Sil 13 CB						



The "Unboxing" of the GC Column





What's Inside?





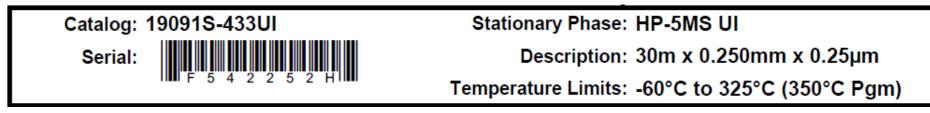
Column tag contains useful information



Column plug holds column ends together and protects against contamination. To put the column in storage, use this plug again or a piece of septa over the ends of the column.



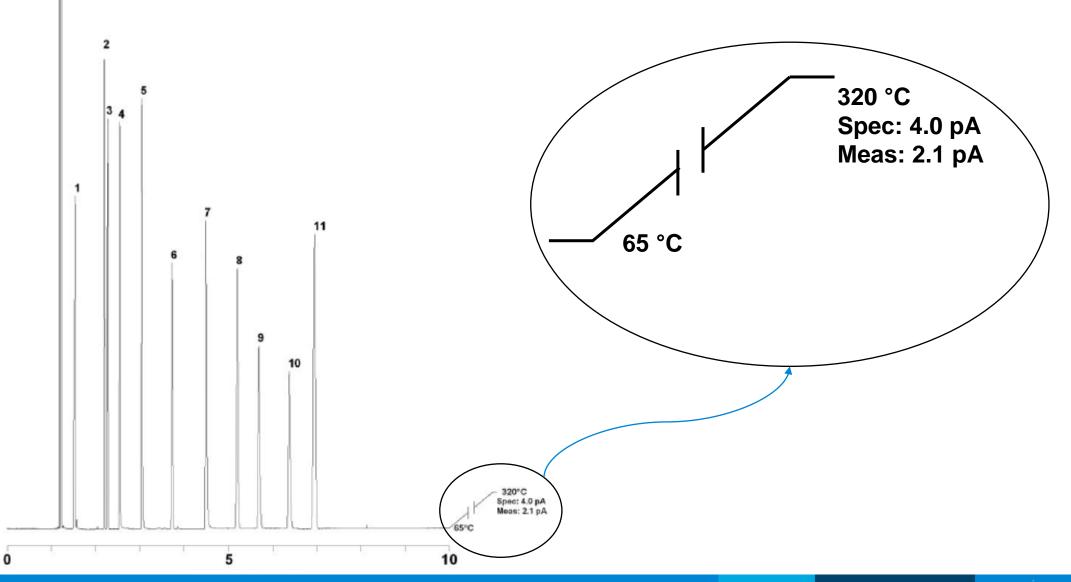
Column Performance Summary



Performance Res	Compound Identification	Retent. Time	Part. Ratio	1/2- Width	
Theoretical Plates/Meter:		1. PROPIONIC ACID 2. 1-OCTENE 3. n-OCTANE	1.543 2.203 2.282	0.30 0.86 0.92	0.027 0.015 0.016
DECANE	3208	4. 1,3-PROPANEDIOL 5. 4-METHYLPYRIDINE 6. n-NONANE 7. TRIMETHYLPHOSPHATE	2.552 3.051 3.738 4.482	1.15 1.57 2.15 2.78	0.020 0.021 0.027 0.033
Retention Index: n-PROPYLBENZENE 1-HEPTANOL	953.110 967.660	8. n-PROPYLBENZENE 9. 1-HEPTANOL 10. 3-OCTANONE 11. n-DECANE	4.462 5.193 5.682 6.368 6.940	3.38 3.79 4.37 4.85	0.033 0.038 0.041 0.047 0.053
Resolution: 1-OCTENE, n-OCTANE	2.97		ector: FID (3 Flow: 42.1 cm/sec Pentane (1		nl/min) nin)



Chromatographic Performance





Test Mixture Components

<u>Compounds</u> Hydrocarbons Purpose Efficiency Retention

FAMEs, PAHs Alcohols Acids Bases Retention Activity Acidic character Basic character



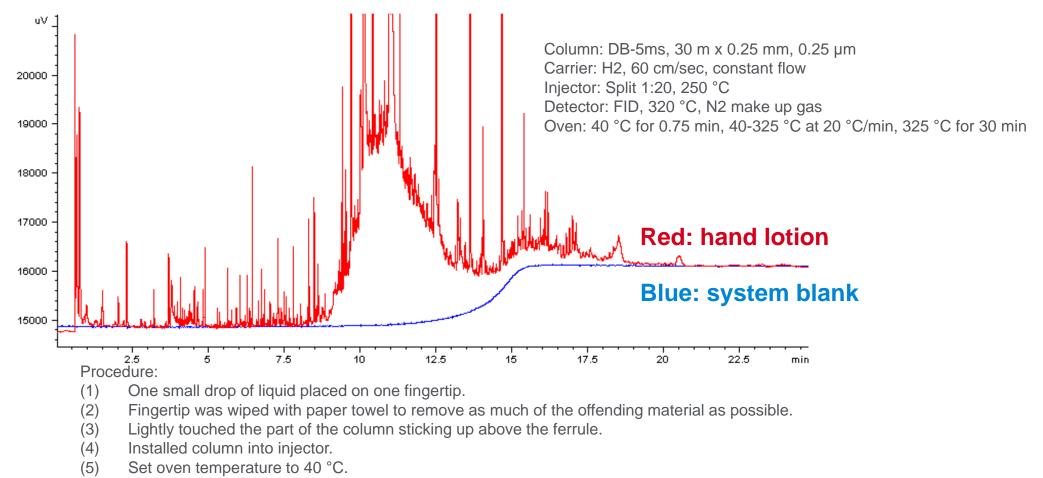
Column Installation Procedure

- Install the column
- Leak and installation check
- Column conditioning
- Bleed profile
- Test mix





Contamination from Hand Lotion



(6) Started oven temperature program as soon as oven reached 40 °C.



"Touchless" Packaging











Agilent

Column Installation What type of ferrule should I use?



Polyimide



Graphite





Polyimide/ graphite Flexible Metal

Composition	Re-use	Max Temperature (°C)	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, and so on)	May not seal well with damaged fittings or rough surfaces



"Short" ferrules for inlet and detector configurations on Agilent GCs



"Long" ferrules for MS transfer lines and MS interface nut



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 upon 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.



The Highlights

Review

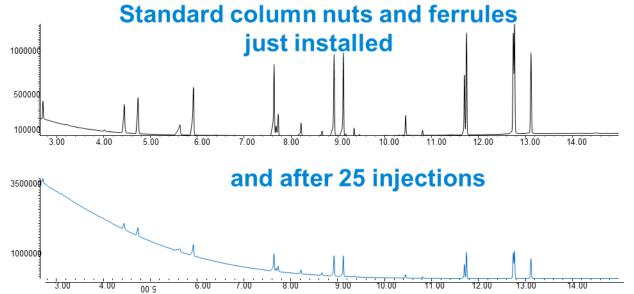
- Flexible Metal ferrules are technologically advanced to provide ease-of-use and mechanically tight seals
- Gold-plating flows to fill scratches and striations on the surface of the CFT device
- Creates leak-free seals on first installation attempt
- Ideal for labs running Backflush, Dean's switch,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



Graphite/Polyimide Blend Capillary Ferrules

Unfortunately, a leak occurred following normal temperature program runs

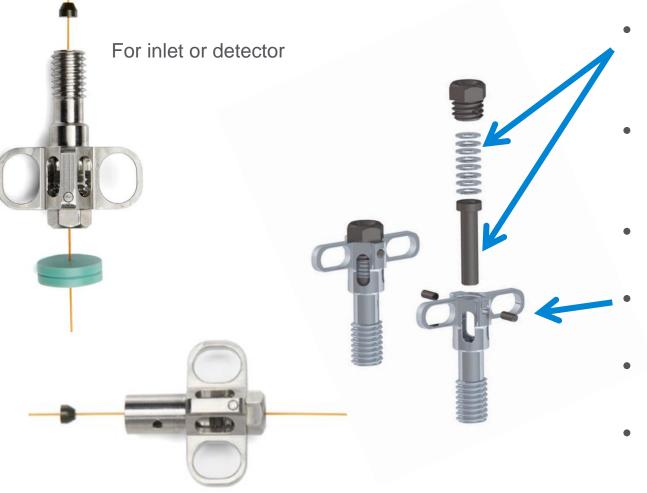
Studies show that the leaking continues with use of the ferrules (not just after the first one or two runs)



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



Column Installation: Self Tightening Column Nut

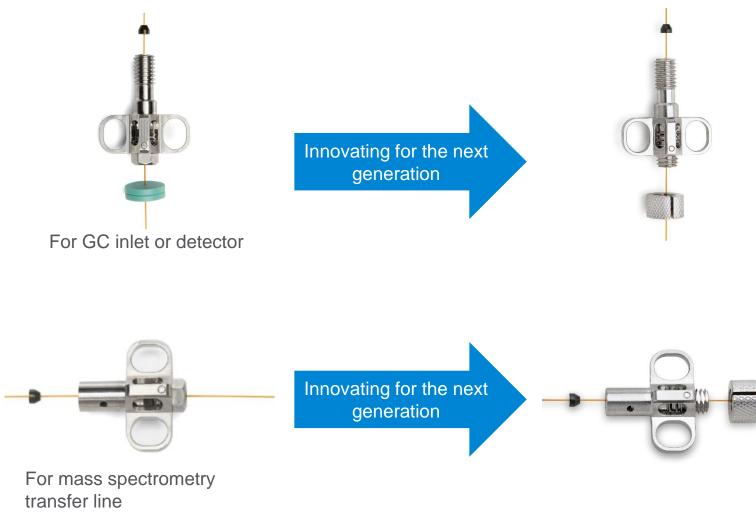


For mass spectrometry transfer line

- Spring driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- No leaks, no downtime, no frustration
- Wing design for finger tightening
- No tools needed
- No polymer materials for durability
- Compatible with **only** short graphite
- Vespel ferrules



Increasing Ease of Use Through Continued Innovation: Self Tightening Nuts



- Easier and faster to install
- Collar holds column in place
- Single-hand installation into inlet
- No tools needed





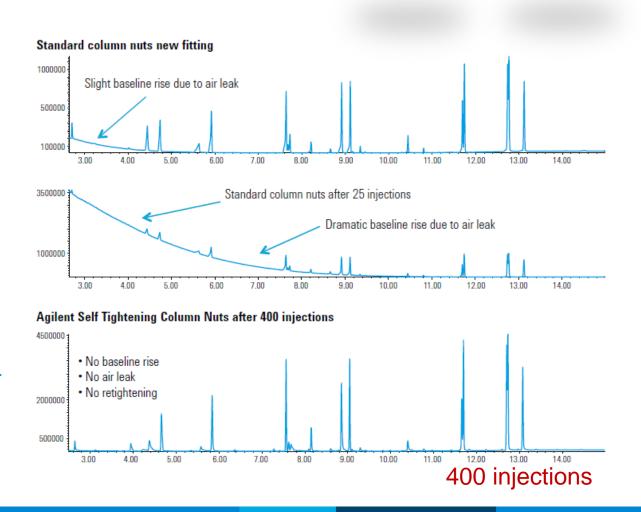
Self Tightening Nuts: No Leaks, No Downtime, No Frustration



- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- No tools needed

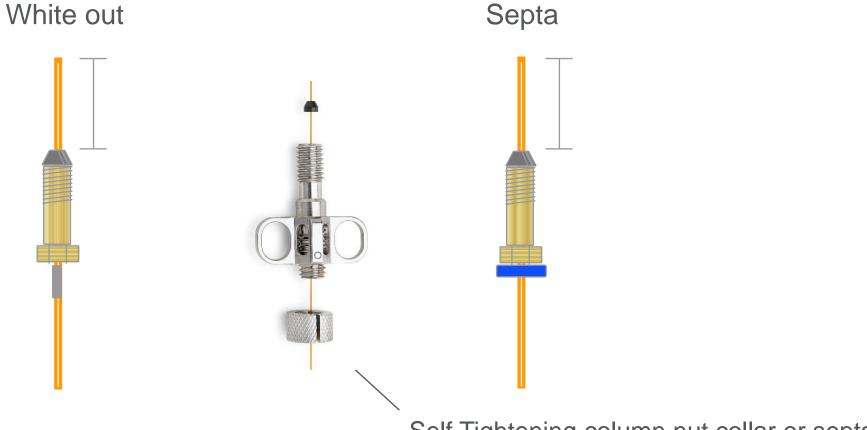
Part Number	Description
G3440-81013	Column Nut, Collared Self-Tightening MSD
G3440-81011	Column nut, Collared Self Tightening Inlet/Detect
G3440-81012	Collar for Self Tigthening Nut

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





Column Installation Measuring the right distance





Cutting the Column

Gently scribe through the polyimide coating

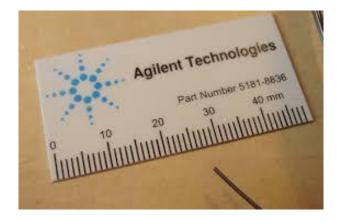
• Do not attempt to cut the glass

Recommended tools

- Diamond or carbide-tipped pencil, or sapphire cleaving tool
- Ceramic wafer
- Ocular

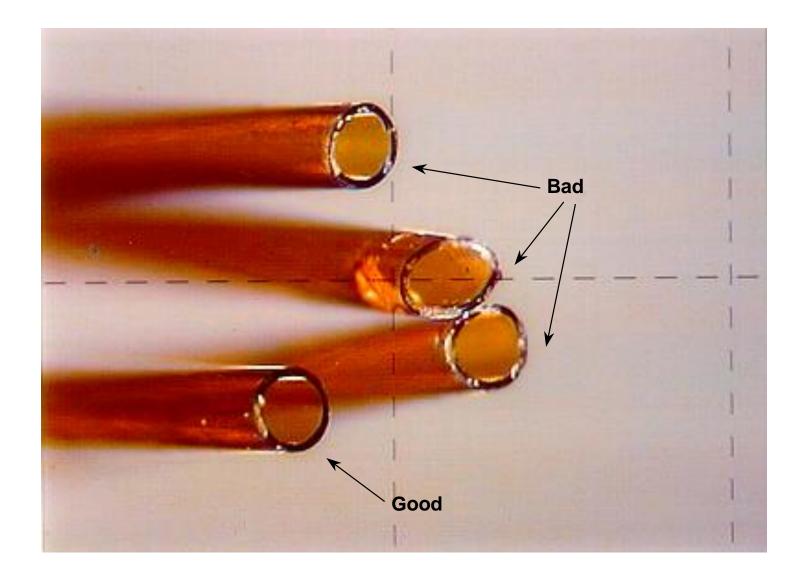
Do not use

• Scissors, file, and so on





Examples of Column Cuts

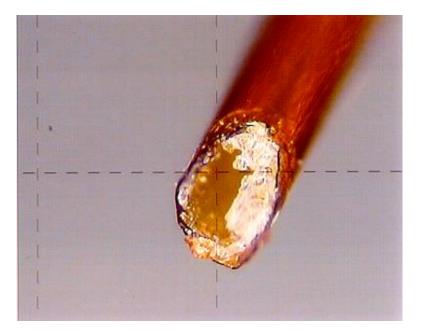




Column Installation

How tight is tight?





Overtightened ferrule





New Agilent Universal Fit GC Detector Jets

- Easier column installation and jet replacement reducing the risk of column damage
- Lubricant free threads reducing the risk of contamination
- Made of strong material reducing the risk of deforming
- Universally fits in both capillary column and packed column (adaptable) FID detectors



Previous Jets				New Universal Fit Jets			
Previous Jet PN	Jet Orifice ID (inch/mm)	Jet Length (inch/mm)	Fit of Detector Fitting Type	New Jet PN (use for re-order)	Jet Orifice ID (inch/ mm)	Jet Length (inch / mm)	Fit of Detector Fitting Type
19244-80560	0.011 / 0.29	2.4 / 62	FID, Adaptable	5200 0170	0.011 / 0.29	1.2 / 31	FID, Capillary & Adaptable
G1531-80560	0.011 / 0.29	1.7 / 43	FID, Capillary	5200-0176			
18710-20119	0.018 / 0.47	2.5 / 64	FID, Adaptable	5200-0177	0.018 / 0.47	1.2 / 31	FID, Capillary & Adaptable
19244-80620	0.018 / 0.47	2.4 / 62	FID, Adaptable				
G1531-80620	0.018 / 0.47	1.7 / 43	FID, Capillary				
18789-80070	0.030 / 0.76	2.5 / 64	FID, Adaptable	5200-0178	0.030 / 0.76	1.2 / 31	FID, Capillary & Adaptable
G1534-80580	0.011 / 0.29	2.0 / 52	NPD, Capillary	5200 0170	0.011 / 0.29	1.6.1.40	NPD, Capillary &
G1534-80590	0.011 / 0.29	2.8 / 71	NPD, Adaptable	5200-0179		1.6 / 40	Adaptable



Column Installation Leak check

Do not use snoop

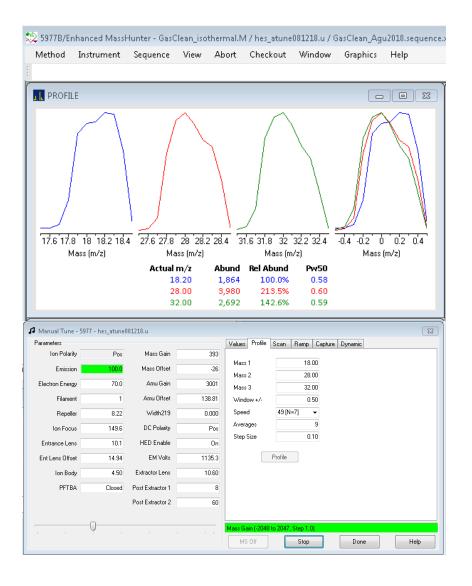
- Electronic leak detector
- IPA/water
- Inject a nonretained peak



Gas leak detector p/n G3388B



If My System is Leak-Free, What Should My Air Ion Abundances Be?



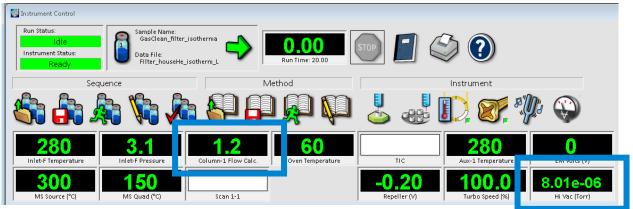
- These are just estimates!
 - $H_2O: \sim 2,000$ counts (less is ok)
 - N₂: ~10,000 counts(less is ok)*
 - O₂: ~3,000 counts (less is ok)

*Make sure to purge your Gas Clean filter

High vacuum gauge pressure (for SQ):

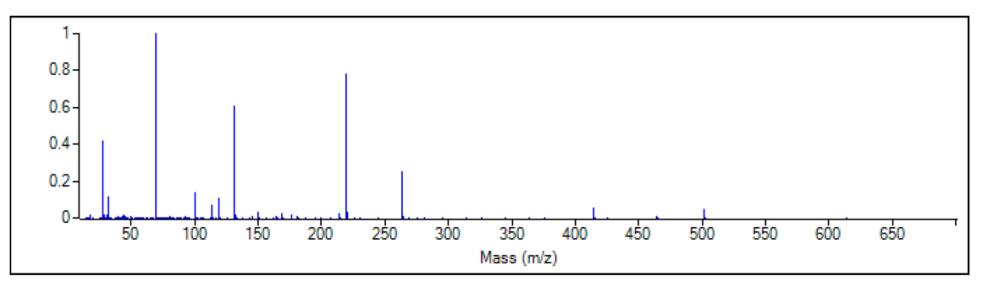
~1 x10⁻⁵ Torr[†]







Example Tune Report with Leak



Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	498,432	100.0%	70.00	6,216	1.2%
219.00	219.00	391,232	78.5%	220.00	18,216	4.7%
502.00	502.00	23,680	4.8%	503.00	2,467	10.4%

Air/Water Check: H20 ~1.8% N2 ~42.1% O2 ~11.4% CO2 ~1.3% N2/H20 ~2325.0%

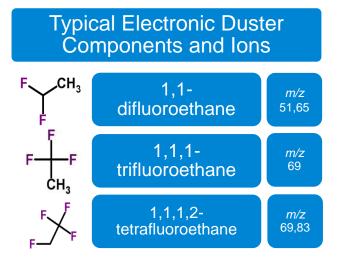
Column(1) Flow: 1.00 Column(2): 1.20 ml/min Interface Temp: 250

Use Leak Detector and/or Electronics Duster to Find Your Leaks

Why use a leak detector?

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





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



Leak and Installation Check

Inject a nonretained compound

Compound				
luted)				

The peak should be sharp and symmetrical



Nonretained Peak Shapes



Improper installation or injector leak

Check for:

- Too low of a split ratio
- Injector or septum leak
- Liner problem (broken, leaking, misplaced)
- Column position in injector and detector



Column Conditioning

System must be leak free before conditioning column

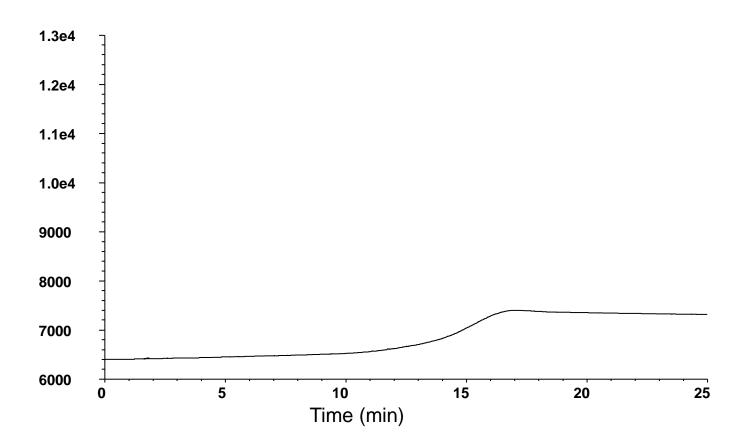
Heat the column to the <u>lower</u> of:

- Isothermal maximum temperature or 20 to 30 °C above highest operation temperature.
- Temperature programming is not necessary.

Stop conditioning when the stable baseline is obtained: 1 to 2 hours, usually



Generating a Bleed Profile



Temperature program the column without an injection*

*Agilent J&W DB-1 30 m x 0.32 mm id, 0.25 µm Temperature program // 40 °C, hold 1 min // 20 °C/min to 320 °C, hold 10 min



Own Test Mixture

- More specific to your application
- Selective detectors
- Concentrations specific to your application
- Use same instrument conditions
- Easiest to simply inject a calibration standard
- Store for future measure of column performance





ULTRA Scientific is Now Part of Agilent Technologies

Agilent ULTRA Chemical Standards have:

- Best in class online search, compare, and ordering capabilities
- Rapid shipping: 99.9% of orders dispatched within 24 to 48 hours (continental US only as of now)
- Custom standard solutions including our *new* online custom quoting tool, enabling customers to upload recipe formulations to and to modify the recipe before submitting it
 - Tool will allow customers to see the quote pricing instantly and allow them to check quote pricing based on quantity range
 - Check it out at <u>https://www.agilent.com/en/product/chemical-standards</u>
- Rigorously tested and manufactured under ISO 9001, ISO 17025, and ISO 17034 certifications
- Sample preparation materials, columns, supplies, instrumentation, and reference materials from a single source





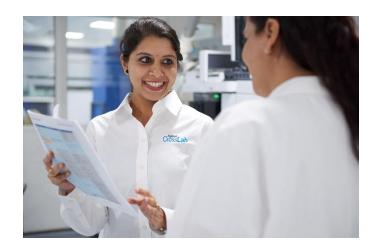
Proper Care of Your Column





Common Causes of Column Performance Degradation

- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O₂ damage)
- Chemical damage by samples
- Contamination

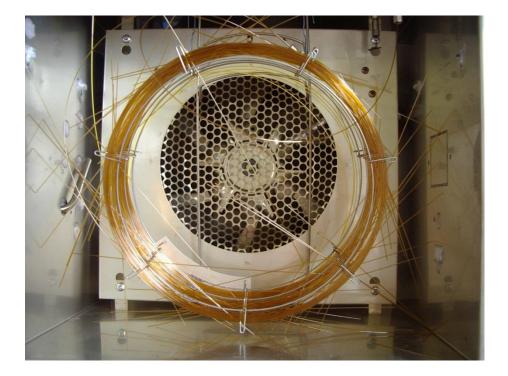






Physical Damage to the Polyimide Coating

- The smaller the tubing diameter, the more flexible it is
- Avoid scratches and abrasions
- Immediate breakage does not always occur upon physical damage



🔆 Agilent

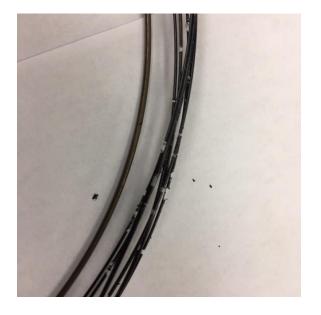
Thermal Damage

Degradation of the stationary phase is increased at higher temperatures

 Rapid degradation of the stationary phase (breakage along the polymer backbone) caused by excessively high temperatures

> Isothermal limit = indefinite time Programmed limit = 5-10 minutes

- Temporary "column failure" below lower temperature limit
- If this happens:
 - Disconnect column from detector
 - "Bake out" overnight at isothermal limit
 - Remove 10–15 cm from column end



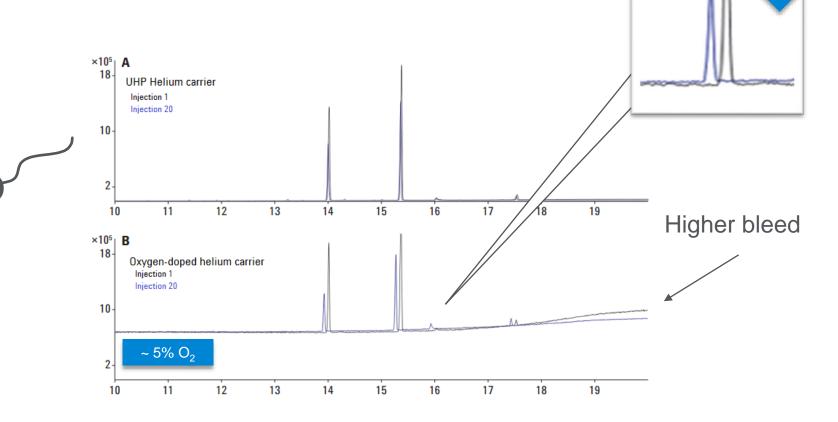
Column continuously exposed to temperatures above its temperature limit



Oxidation (O2 Damage)

Dimethylpolysiloxane

Oxygen in the carrier gas rapidly degrades the stationary phase. The damage is accelerated at higher temperatures. Damage along the polymer backbone is irreversible. (Premature filament failure/excessive source maintenance.)



IQ



Decreased retention

How to Prevent Column Damage by Oxygen

- High-quality carrier gas (four 9 s or greater)
- Leak free injector and carrier lines
 - Change septa
 - Maintain gas regulator fittings
- Appropriate impurity traps









Efficient, fast, easy



Chemical Damage

Bonded and crosslinked columns have excellent chemical resistance except for inorganic acids and bases.

HCI NH_3 KOH NaOH H_2SO_4 H_3PO_4 HF

Chemical damage will be evident by excessive bleed, lack of inertness or loss of resolution/retention.



Chemical Damage What to do if it happens

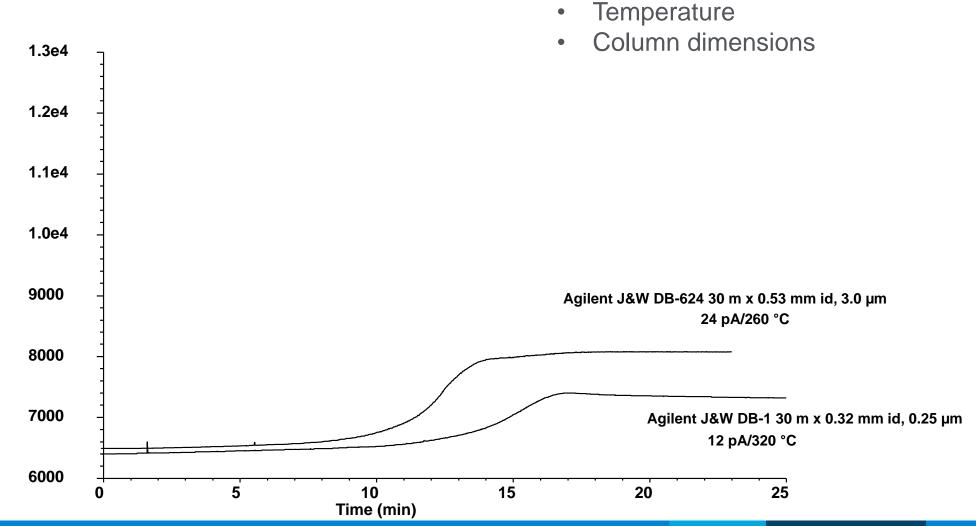
- Remove 0.5 to 1 m from the front of the columns
- Severe cases may require removal of up to 5 m





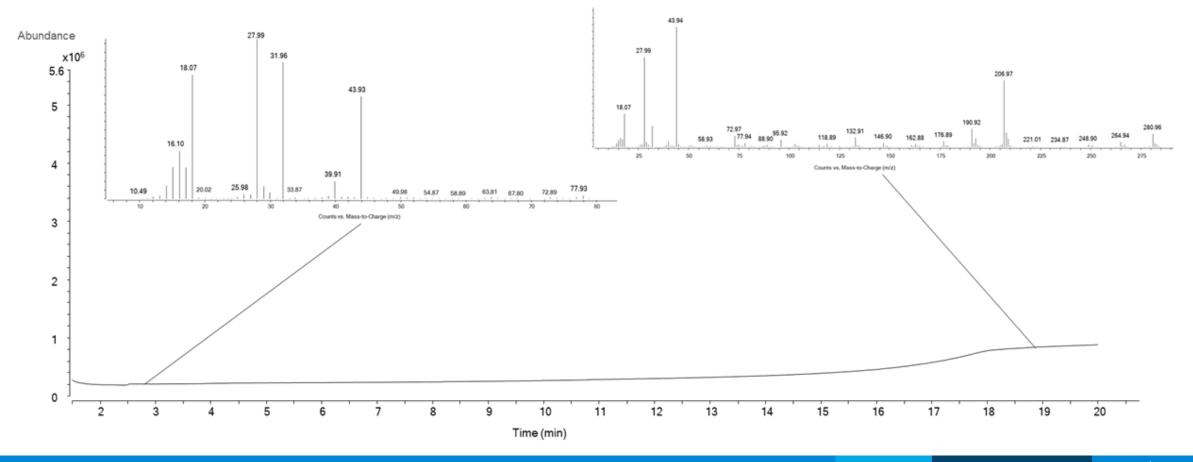
What is Normal Column Bleed?

Normal background signal generated by the elution of normal degradation products of the column stationary phase. Column bleed is influenced by: • Phase type





Mass Spectrum of Phenylmethylpolysiloxane Column Bleed Normal background (HP-5ms UI)





What is a Bleed Problem?

An abnormal elevated baseline at high temperature

It is not

- A high baseline at low temperature
- Wandering or drifting baseline at any temperature

Discrete peaks



Column Contamination and Symptoms

- Fouling of GC and column by contaminants
- Mimics nearly every chromatographic problem

- Poor peak shape
- Loss of separation (resolution)
- Changes in retention
- Reduced peak size
- Baseline disturbances (semivolatiles only)



Typical Samples That Contain a Large Amount of Residues

Biological (blood, urine, tissue, plants)

Soils Foods

Wastewater Sludges

All samples contain residues (even standards)

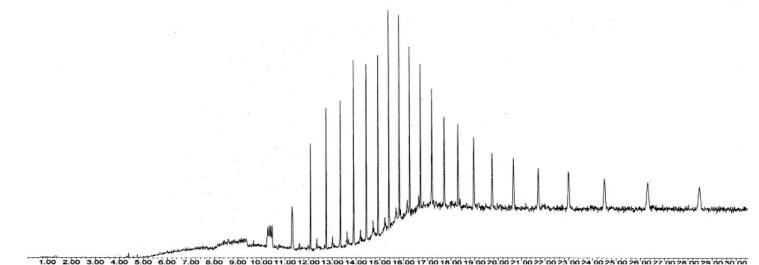


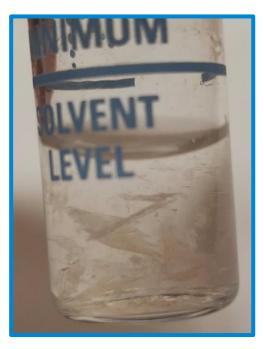


Other Sources of Contamination

- Septum and ferrule particles
- Gas and trap impurities
- Unknown sources (such as vials and syringes)

Sample vial septum bleed profile:





Contaminated wash solvent



Types of Residues

Nonvolatile residues

• Any portion of the sample that does not elute from the column or remains in the injector.

Semivolatile residues

• Any portion of the sample that elutes from the column after the current chromatographic run.



Methods to Minimize Nonvolatile Residue Problems

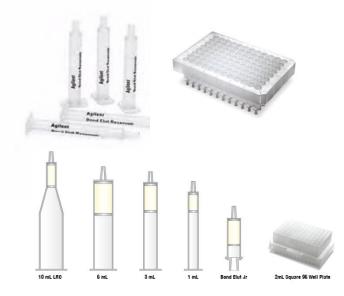
- Sample cleanup
- Packed injection port liners
- Guard columns







Offline Options for Sample Matrix Removal



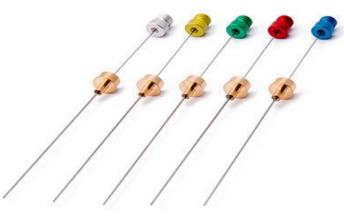
Bond Elut solid phase extraction cartridges and plates



Filter vials



QuEChERS



SPME



Captiva EMR-Lipid filtration cartridges and plates



Chem Elut S



Captiva syringe filters



Filtration – Captiva Filter Vials



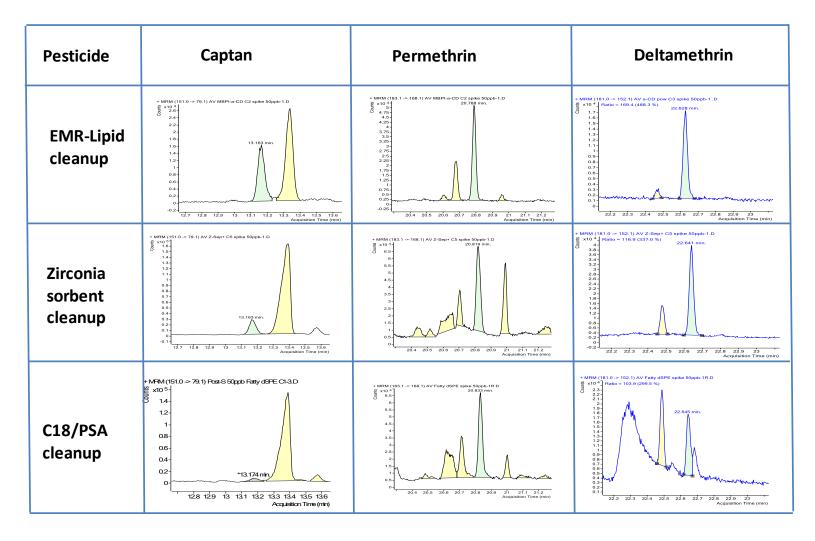
Part Number	Description	
5191-5933	PTFE filter vial, 0.45 µm, 100/pk	
5191-5934	PTFE filter vial, 0.20 µm, 100/pk	
5191-5935	Nylon filter vial, 0.45 µm, 100/pk	
5191-5936	Nylon filter vial, 0.20 µm, 100/pk	
5191-5939	RC filter vial, 0.45 µm, 100/pk	
5191-5940	RC filter vial, 0.20 µm, 100/pk	
5191-5941	PES filter vial, 0.45 µm, 100/pk	
5191-5942	PES filter vial, 0.20 µm, 100/pk	
5191-5943	Vial closure tool	

Agilent.com/chem/filtervials Filter vials user guide: 5994-0814EN

See appendix for solvent compatibility poster request

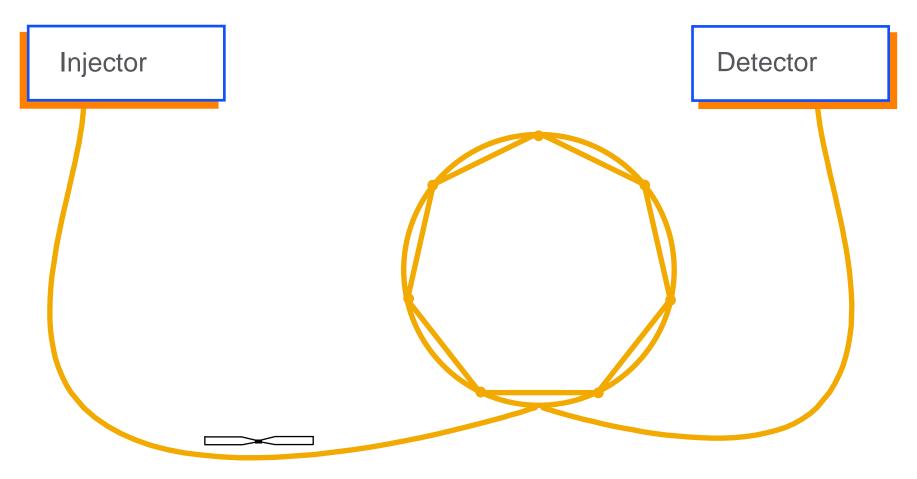


Captiva EMR–Lipid Cleanup Improves Analytes S/N Ratio and Integration Accuracy on GC/MS(/MS) of Pesticides in Olive Oil





Guard Column or Retention Gap



The guard column is 3–5 m of deactivated fused silica tubing with the same diameter as the analytical column. It is connected with a zero dead volume union.



Nonvolatile Contamination What to do if it happens

- Do not "bake out" the column
- Front end maintenance
 - Clean or change the injector liner
 - Clean the injector
 - Cut off 0.5-1 m of the front of the column
- Turn the column around
- Cut the column in half



Semivolatile Contamination What to do if it happens

- "Bake out" the column
 - Limit to 1-2 hours
 - Longer times may polymerize some contamination and reduce column life
- Solvent rinse the column



Instrumentation: Leveraging Intelligence Innovations





Introducing the Agilent 8890 GC System Flexible and expandable to meet your needs today and tomorrow



Future-proof: Ready for anything

- Powerful next generation electronic architecture
- Expanded smart-connected functionality
- Full suite of inlets, detectors, and accessories, CFT, Deans switch, backflush, GC x GC, dual simultaneous injection
- Six valves, eight heated zones, plus LVO
- Generation 6 precision EPC
- Smart keys
- 7-inch color touch display





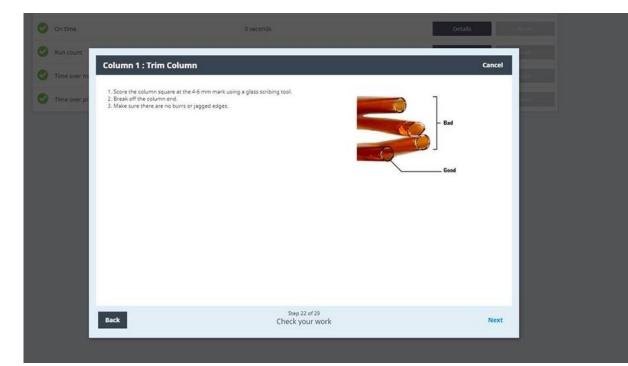
Agilent 8890 GC System Smart-connected GC

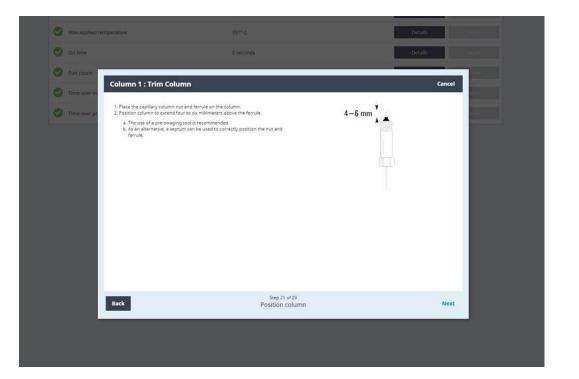
Modern intuitive interface

- 7-inch color touch screen
 - Configuration
 - Status
 - Methods
 - Sequence info
 - Troubleshooting, diagnostics, and help
- Real-time chromatographic evaluation
 - Blank evaluation
 - Detector evaluation



Some Examples of Guided Troubleshooting/Step-by-Step Guides on the Agilent 8890 GC System







GC Columns with Smart Key (for the Agilent 8890 GC only)

For immediate identification and use monitoring of your GC column

- Available with the Agilent 8890 GC model only
- Can track use of a GC column
- Smart key contains GC column information, including:
 - Part and serial numbers
 - Number of injections/runs
 - Time at/above temperature limits
 - Date installed
 - Temperature limits GC columns
 - If more than one column is installed, temperature is determined by lowest column smart key installed (DB-WAX vs DB-5)
 - Column length/trimming done edited in "column maintenance mode" in software and rewritten to smart key
 - S/N of last instrument installed in if it was in an Agilent 8890
 GC







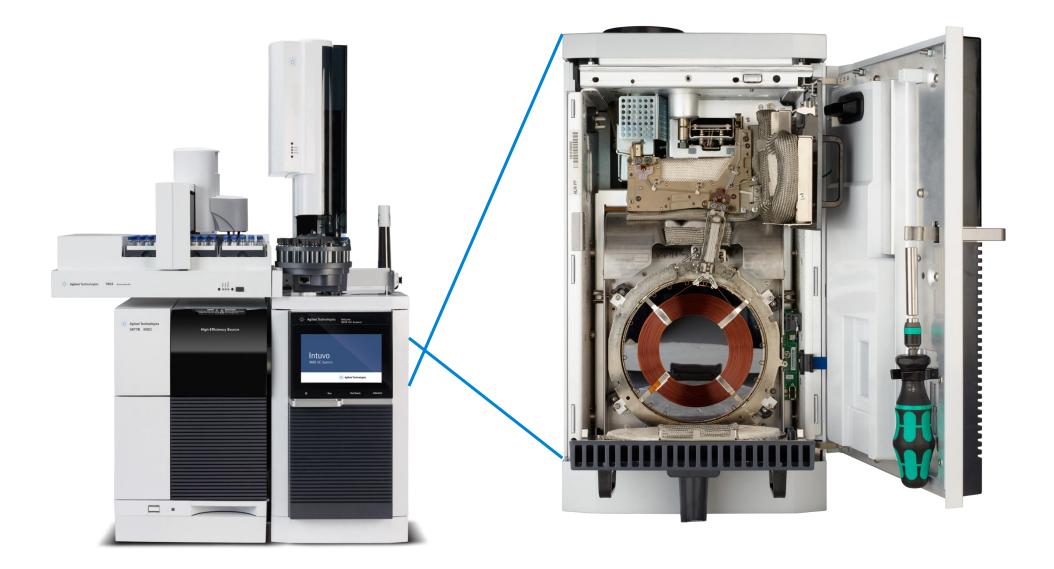
Other Resources

	Resources	Weblinks
1	Agilent 8890 GC brochure	URL: <u>www.agilent.com/cs/library/brochures/brochure-</u> <u>gc-8890-5994-0476en-agilent.pdf</u>
2	Smart key product page (not for ordering smart keys)	URL: www.agilent.com/chem/smartkey8890
3	Instruction sheet	URL: <u>www.agilent.com/cs/library/instructionsheet/publi</u> <u>c/insert-smart%20key-8890-5994-0700en-</u> <u>agilent.pdf</u>





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



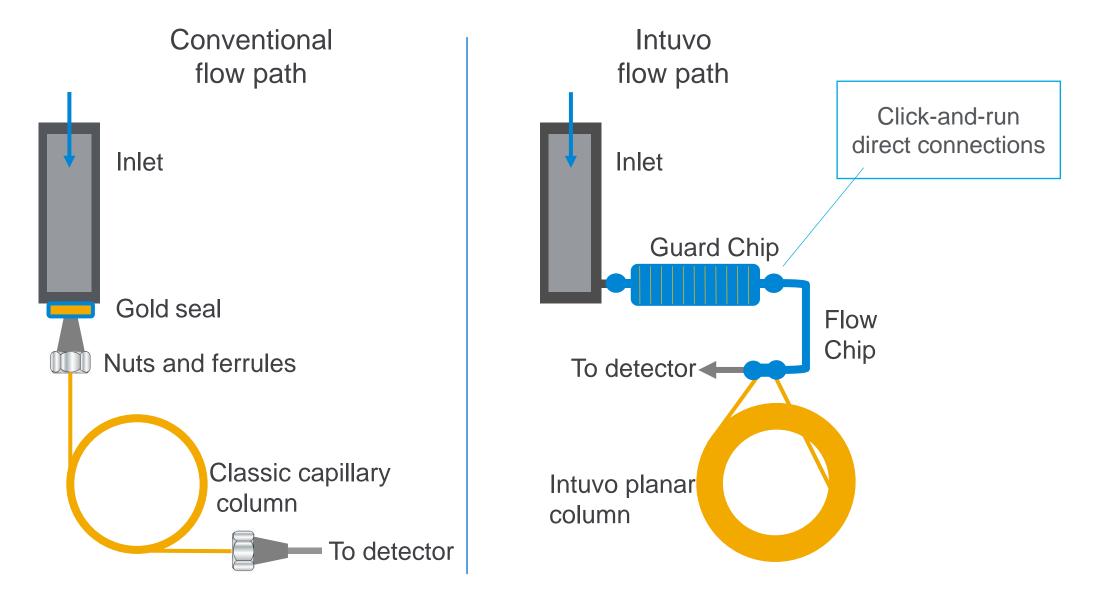
Common Care and Maintenance Scheme for GC Columns

- 1. Cut off 6 inches to 1 foot from the inlet end of the column
- 2. Bake out the column for no more than 2 hours
- 3. Cut off more of the column (repeat as necessary)





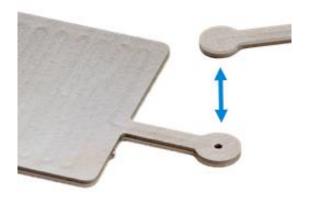
Innovating the GC Flow Path





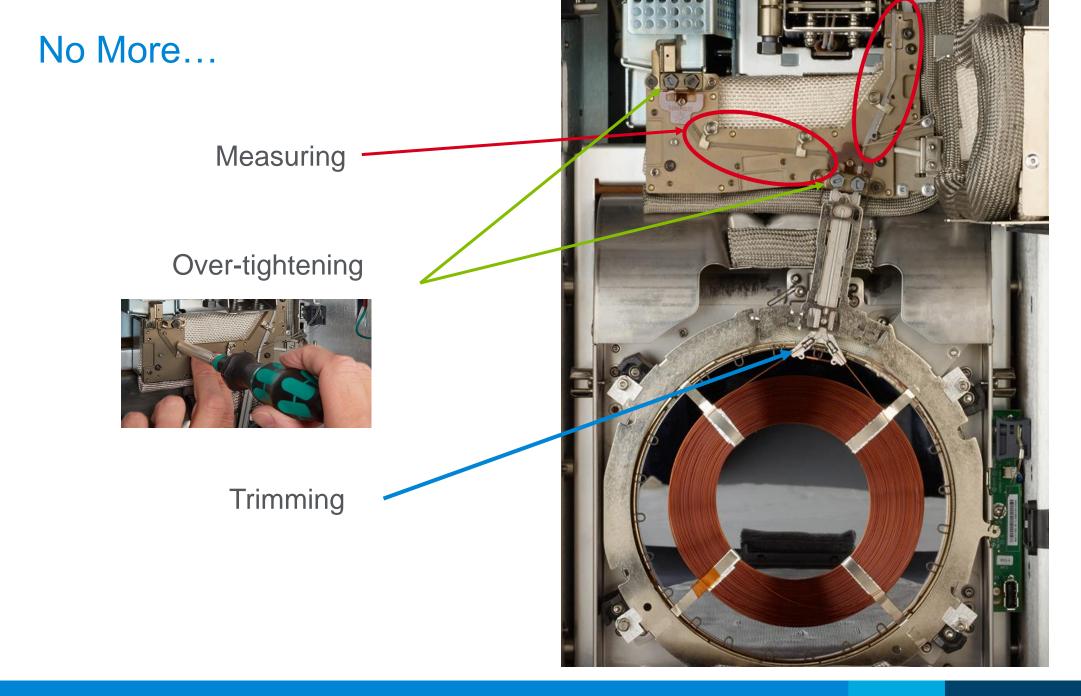
Easier and Faster Maintenance with Intuvo

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



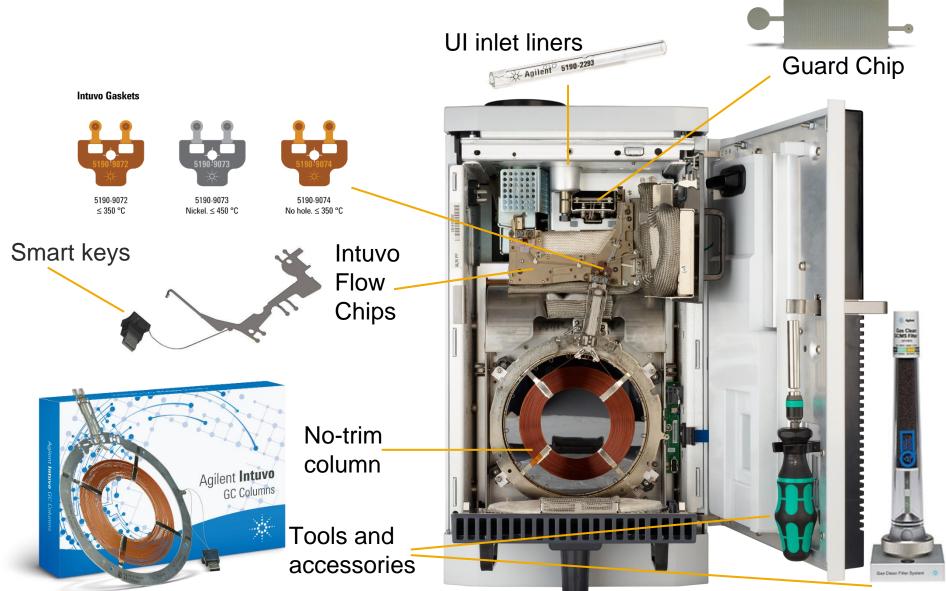








A New Portfolio of GC Consumables



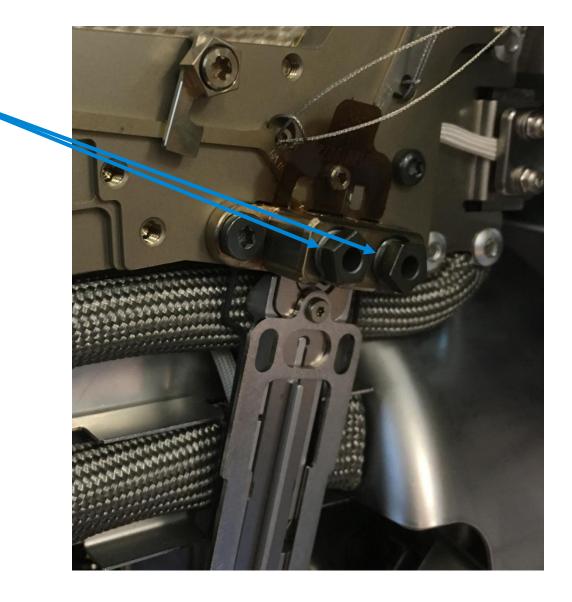
69



Tips to Ensure 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 Ensure 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.

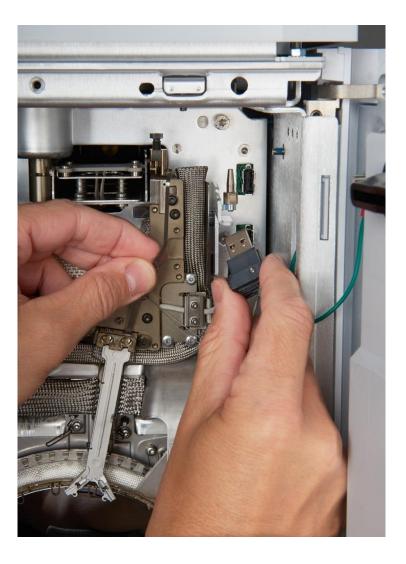




Smart Key Technology

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





Agilent Intuvo 9000 Videos

<u>The Agilent Intuvo 9000 GC System – Environmental Science Corporation (ESC)</u> 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 about the return on investment on the Agilent Intuvo 9000 GC System Playing time: 4:17



Always Remember

- Start with a good installation
- Maintain an oxygen-free system
- Avoid physical, thermal, and chemical damage
- Take steps to prevent contamination





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 USA and Canada 8–5, all time zones



gc-column-support@agilent.com Ic-column-support@agilent.com spp-support@agilent.com spectro-supplies-support@agilent.com chem-standards-support@agilent.com

