

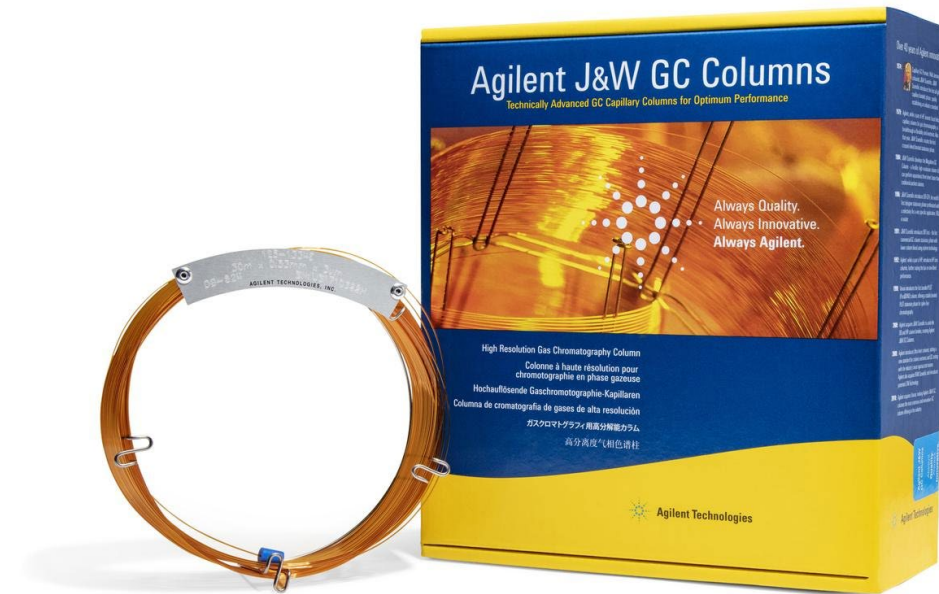
Back to Basics: Installation, Care, and Maintenance of GC Columns

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Application Engineers
July 21, 2022

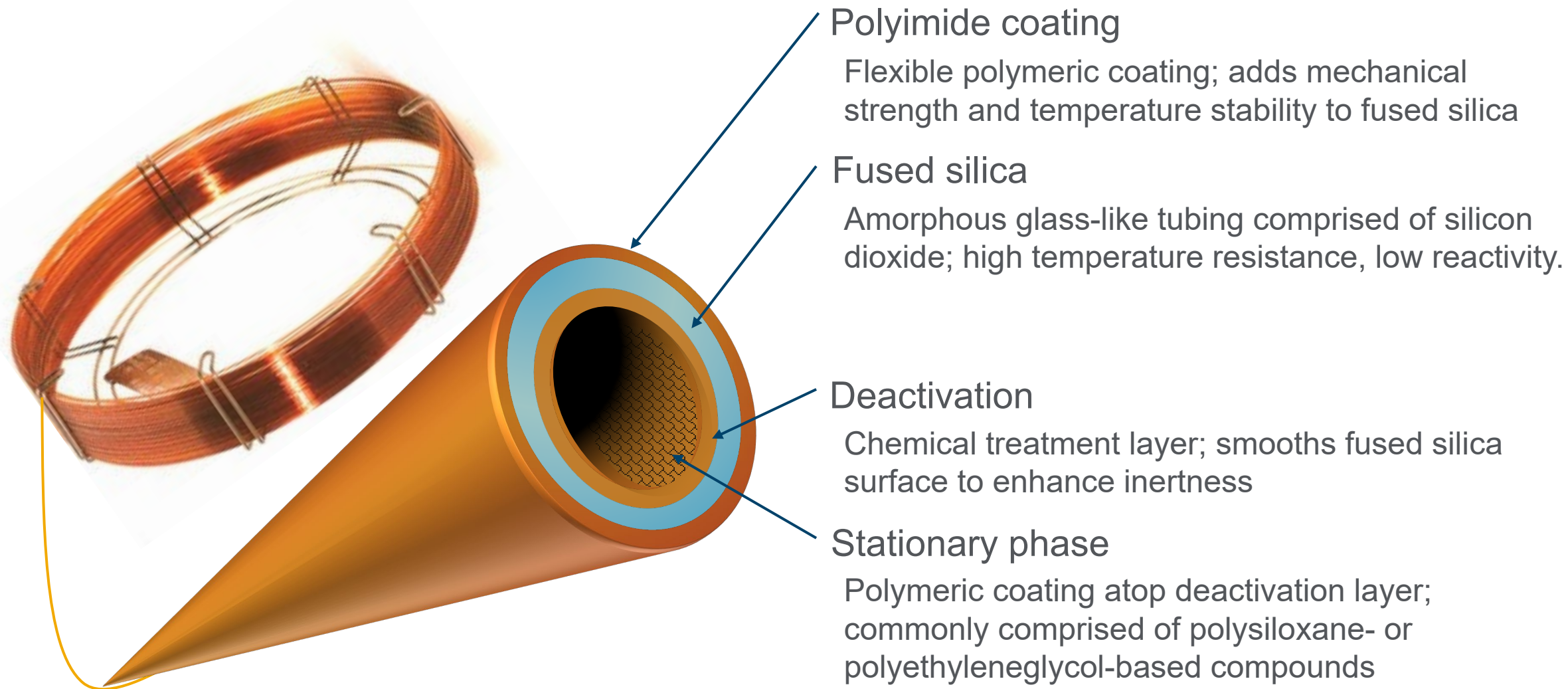


Agenda

- Unboxing / “getting to know your column”
- Install the column
- Preventive measures
- Corrective measures
- Latest instrument developments
 - 8890 / 8860 / INTUVO



Column Construction

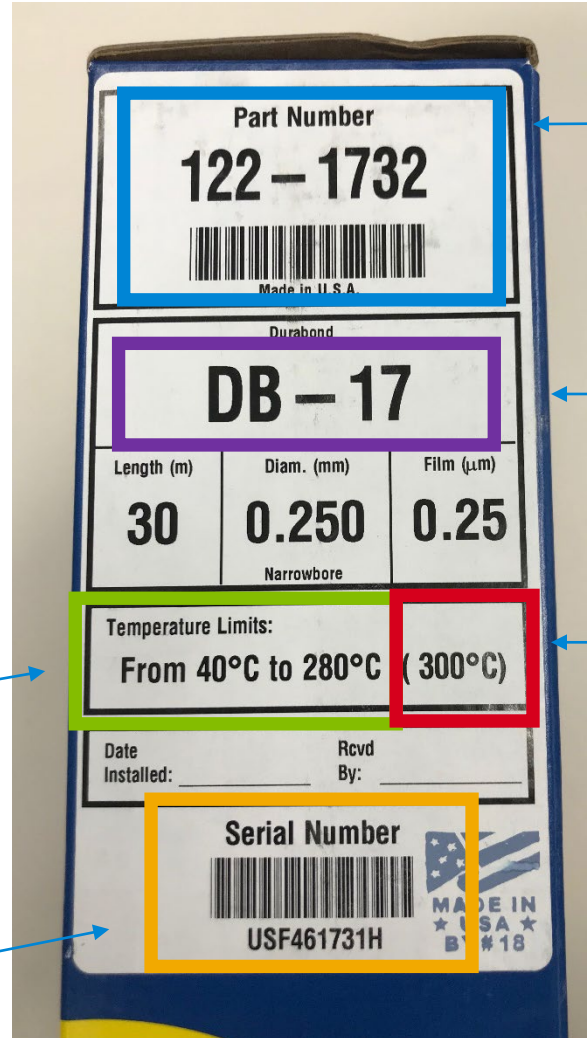
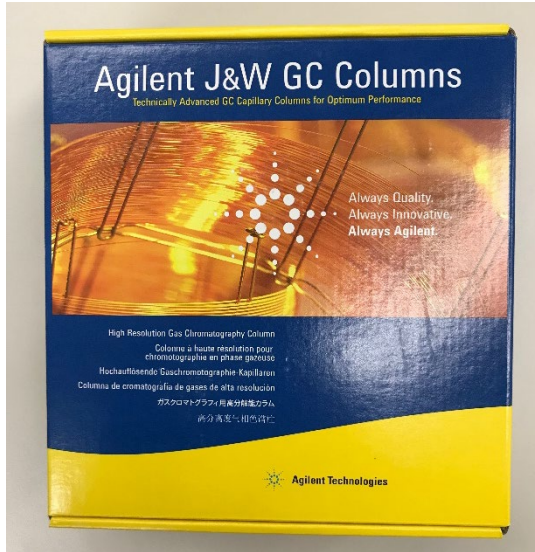


Agilent J&W Column Portfolio- DB, HP, CP, VF

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					
			VF-1301ms					
			CP-Sil 13 CB					

Agilent J&W columns have over 50 different stationary phase offerings

The “Unboxing” of the GC Column



Important for identification and re-ordering

Column stationary phase

Programmed temperature limit (<10 min)

Isothermal temperature limits

Unique to each column (identification)

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

Catalog: 19091S-433UI

Serial:



Stationary Phase: HP-5MS UI

Description: 30m x 0.250mm x 0.25µm

Temperature Limits: -60°C to 325°C (350°C Pgm)

Performance Results

Theoretical Plates/Meter:

n-DECANE 3208

Retention Index:

n-PROPYLBENZENE 953.110

1-HEPTANOL 967.660

Resolution:

1-OCTENE, n-OCTANE 2.97

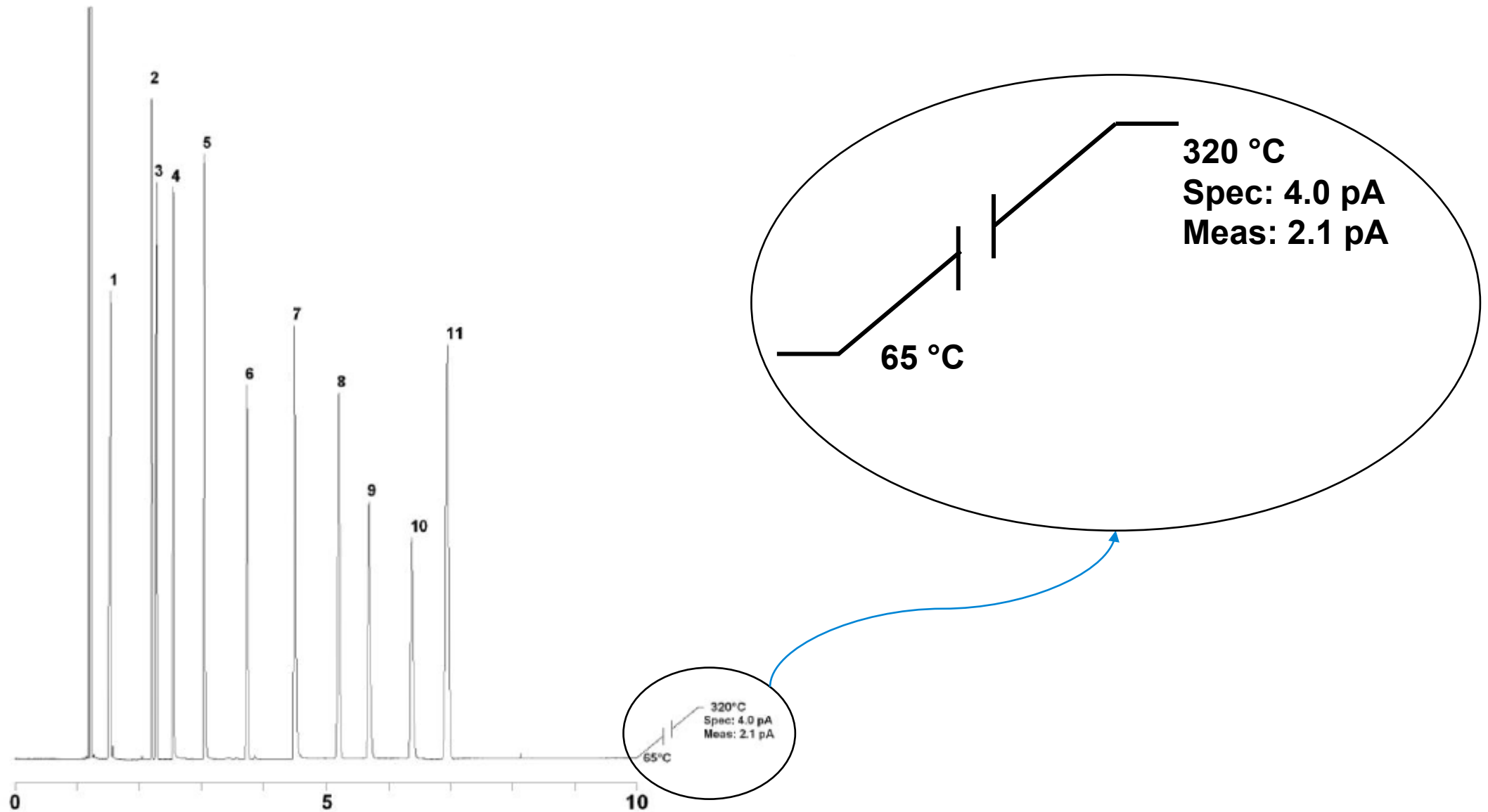
Compound Identification

Compound Identification	Retent. Time	Part. Ratio	1/2-Width
1. PROPIONIC ACID	1.543	0.30	0.027
2. 1-OCTENE	2.203	0.86	0.015
3. n-OCTANE	2.282	0.92	0.016
4. 1,3-PROPANEDIOL	2.552	1.15	0.020
5. 4-METHYLPYRIDINE	3.051	1.57	0.021
6. n-NONANE	3.738	2.15	0.027
7. TRIMETHYLPHOSPHATE	4.482	2.78	0.033
8. n-PROPYLBENZENE	5.193	3.38	0.038
9. 1-HEPTANOL	5.682	3.79	0.041
10. 3-OCTANONE	6.368	4.37	0.047
11. n-DECANE	6.940	4.85	0.053

Test Conditions

Inlet: Split (250°C) Detector: FID (325°C)
 Carrier Gas: Hydrogen Flow: 42.1 cm/sec (1.2 ml/min)
 Holdup Compound: Pentane (1.187-min)
 Temperature Program: Isothermal at 65°C

Chromatographic Performance



Test Mixture Components

Compounds
Hydrocarbons

Purpose
Efficiency
Retention

FAMEs, PAHs
Alcohols
Acids
Bases

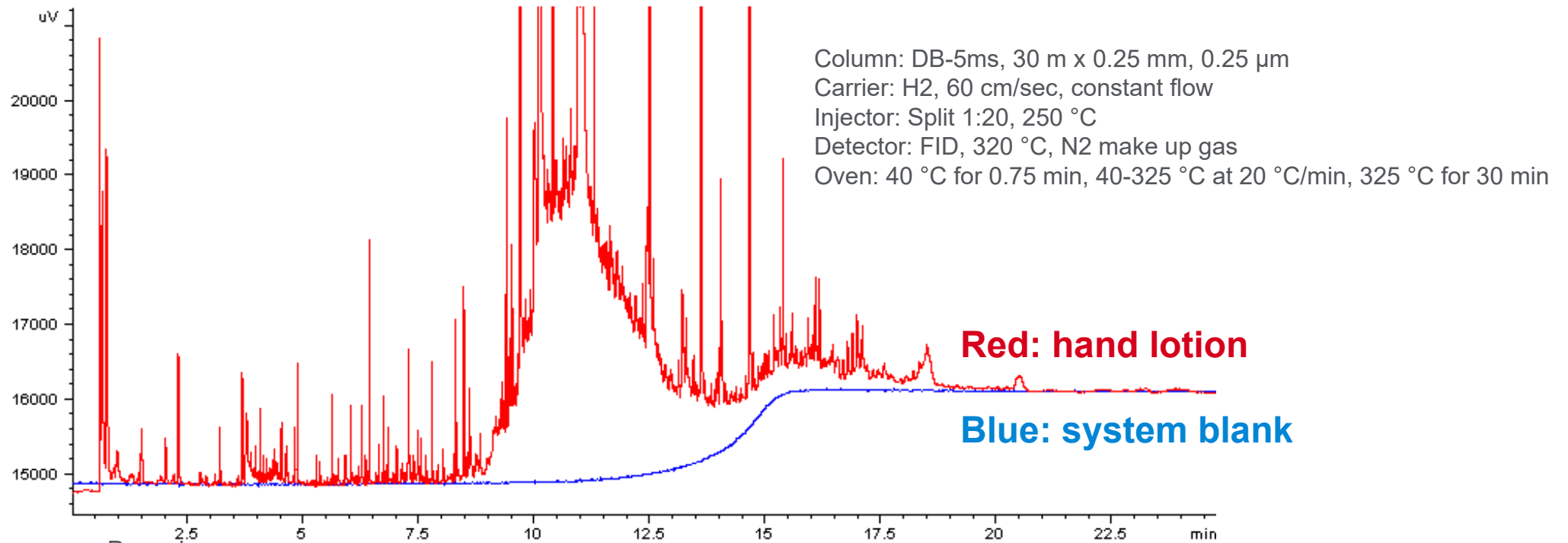
Retention
Activity
Acidic character
Basic character

Column Installation Procedure

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



Contamination from Hand Lotion



Procedure:

- (1) One small drop of 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 40 °C.
- (6) Started oven temperature program as soon as oven reached 40 °C.

“Touchless” Packaging



Column Installation

What type of ferrule should I use?



Polyimide



Graphite



Polyimide/
graphite



Flexible
Metal

Composition	Re-use	Max Temperature (°C)	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, inlets	Still shrink after thermal cycles creating leaks; need to retighten regularly
Polyimide (Vespel)	Yes	280	Easy seal	Shrink after heating causing leaks after thermal cycle; isothermal only
Flexible Metal	No	450	Capillary flow technology (backflush, splitters, and so on)	May not seal well with damaged fittings or rough surfaces



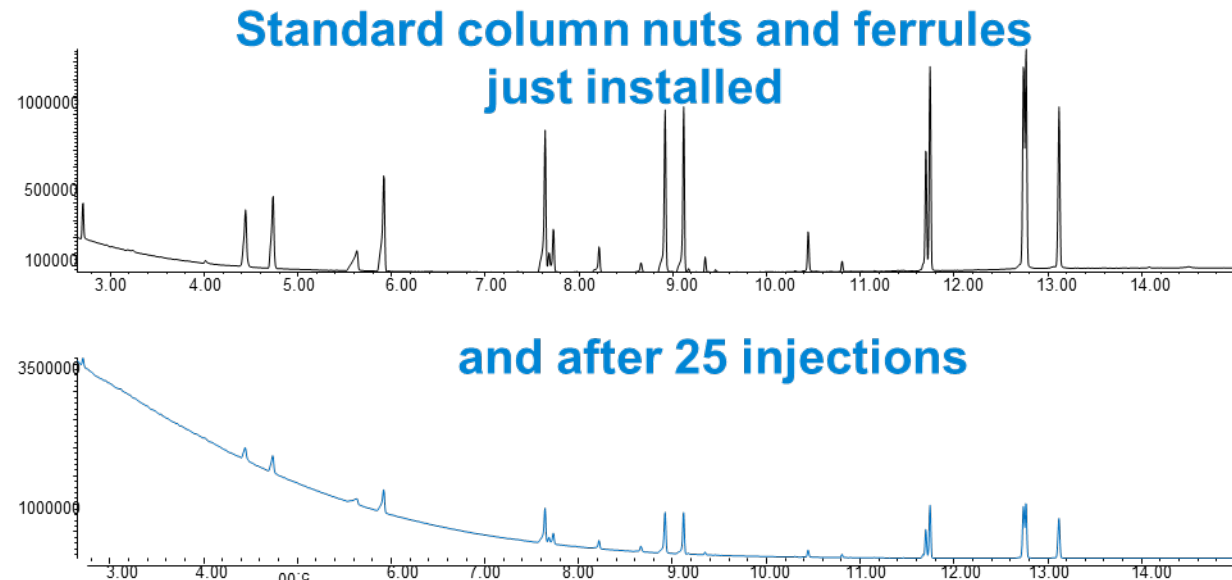
“Short” ferrules for inlet and detector configurations on Agilent GCs



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

Graphite/Polyimide Blend Capillary Ferrules

Unfortunately, a leak occurred following normal temperature program runs (**especially true with MSDs**)
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

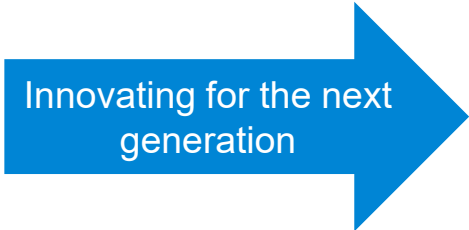


- Compatible **only** with short graphite/vespel!!
- Spring driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- No leaks, no downtime, no frustration
- Wing design for finger tightening
- No tools needed
- No polymer materials for durability
- Vespel ferrules

Increasing Ease of Use Through Continued Innovation: Self Tightening Nuts



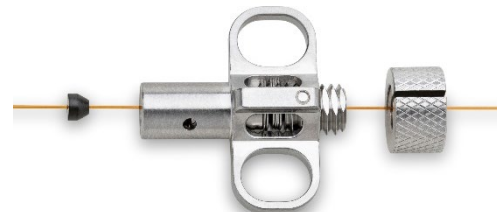
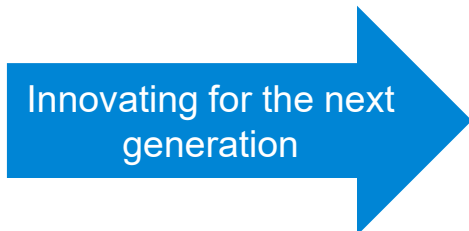
For GC inlet or detector



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



For mass spectrometry transfer line



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



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.

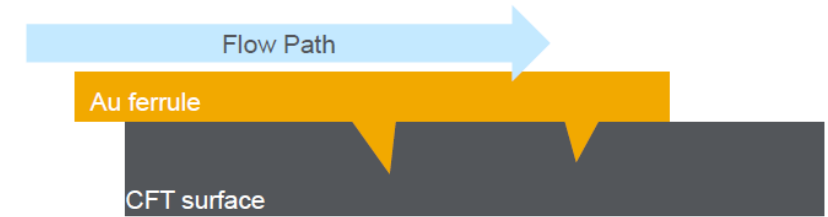
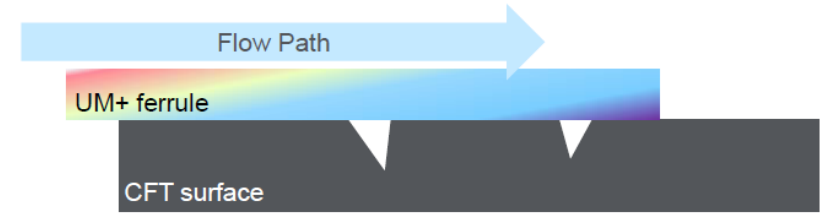
[Flexible Metal Ferrules Swaging Guide for UltiMetal Plus and Gold-Plated Ferrules \(agilent.com\)](https://www.agilent.com/chem/flexferrule)



The Highlights

Review

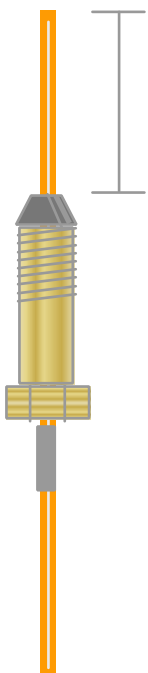
- Flexible Metal ferrules are technologically advanced to provide ease-of-use and mechanically tight seals
- Gold-plating flows to fill scratches and striations on the surface of the CFT device
- Creates leak-free seals on first installation attempt
- Ideal for labs running Backflush, Dean's switch,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



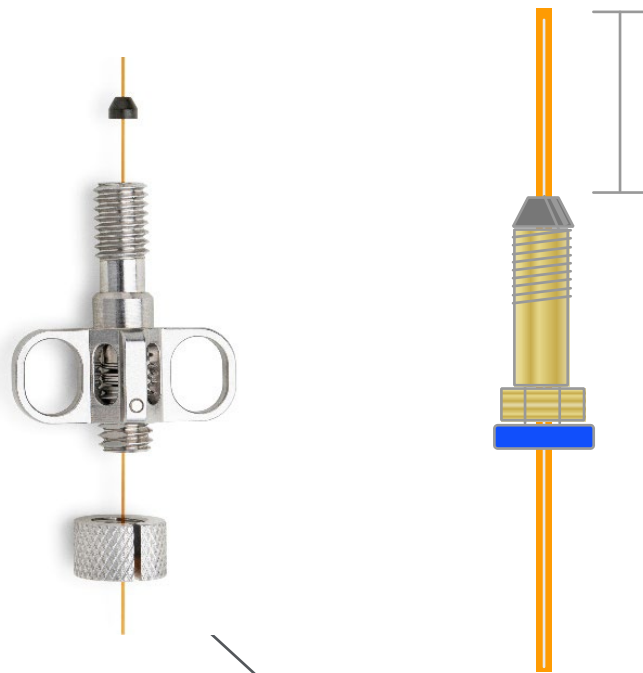
Column Installation

Measuring the right distance

White out



Septa



Split/Splitless
MMI

4-6 mm*
12-15 mm*

*Try to keep this distance the same with every install/method; small differences here can make large differences in performance

Self Tightening column nut collar or septa

Cutting the Column

Gently scribe through the polyimide coating

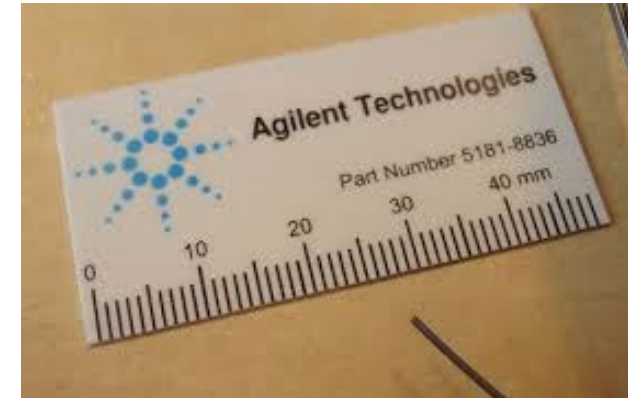
- Do not attempt to cut the glass

Recommended tools

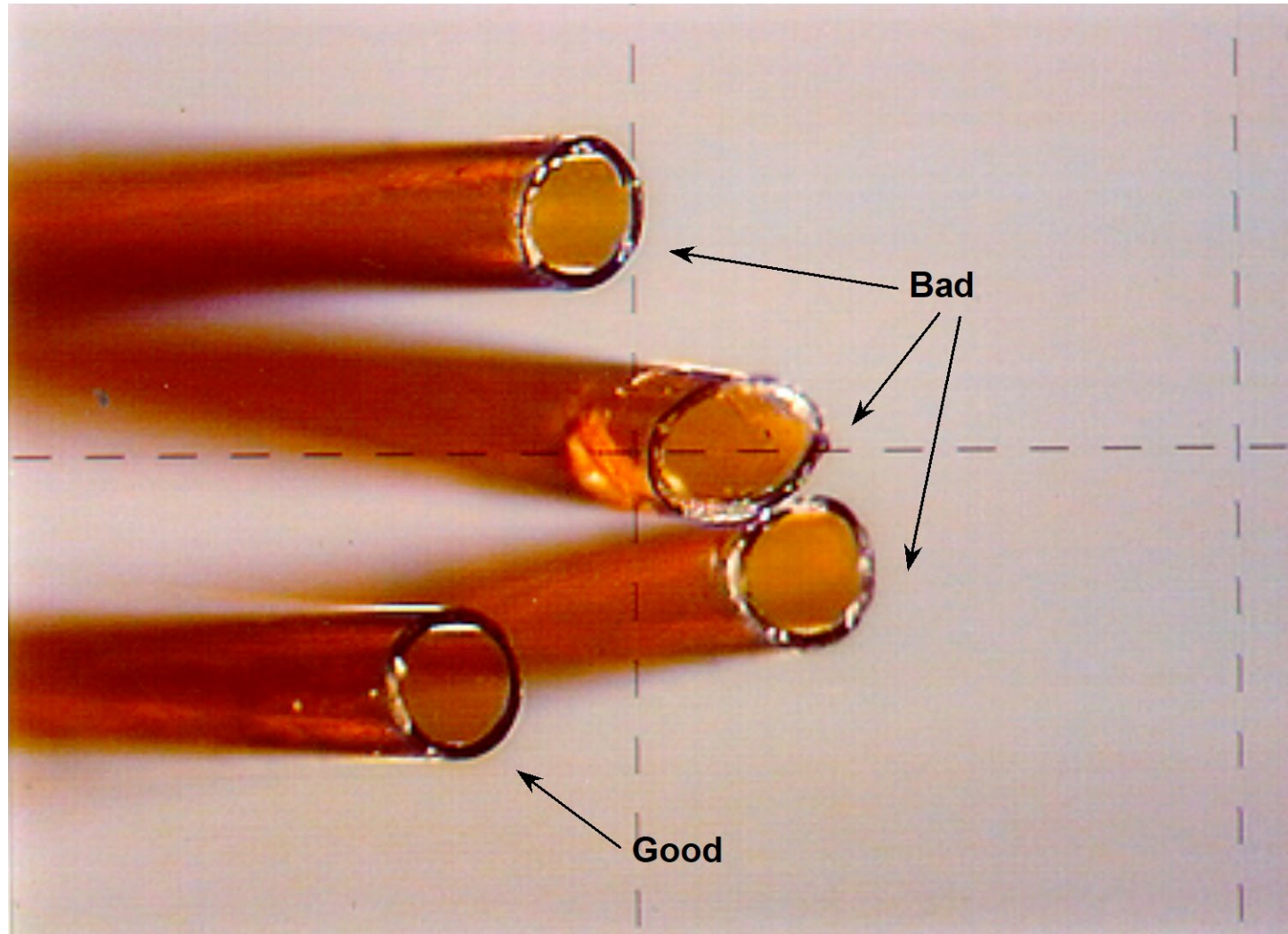
- Diamond or carbide-tipped pencil, or sapphire cleaving tool
- Ceramic wafer (Use “smooth” edge)
- Ocular

Do not use

- Scissors, file, and so on

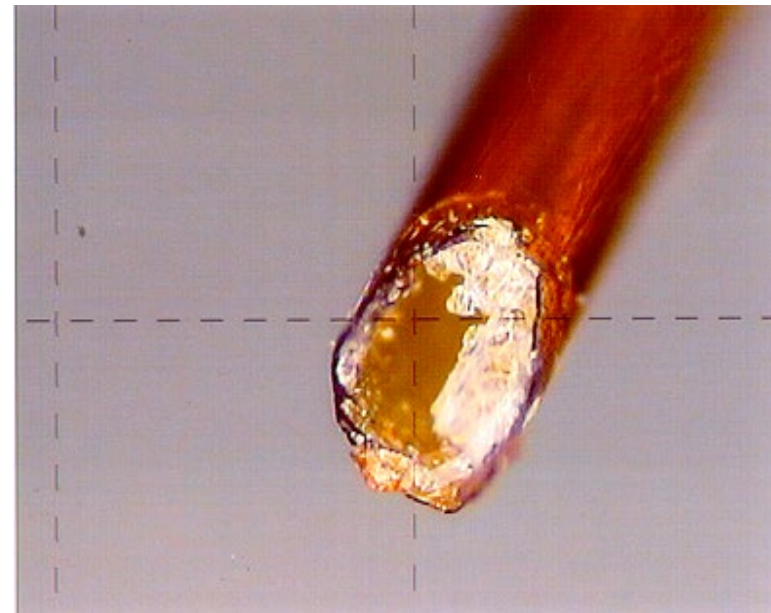


Examples of Column Cuts



Column Installation

How tight is tight?
Finger tight + $\frac{1}{4}$ turn



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	5200-0176	0.011 / 0.29	1.2 / 31	FID, Capillary & Adaptable
G1531-80560	0.011 / 0.29	1.7 / 43	FID, Capillary				
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	5200-0179	0.011 / 0.29	1.6 / 40	NPD, Capillary & Adaptable
G1534-80590	0.011 / 0.29	2.8 / 71	NPD, Adaptable				

Column Installation

Leak check

Do not use snoop

- Electronic leak detector
- IPA/water
- Inject a non-retained peak



The Cost of Leaks

Cost of gases

Reduced consumable lifetime

(O₂ + high temp will quickly destroy the column)

Reduced productivity from downtime

Detector noise and elevated baselines

Time in troubleshooting



It is critical that every customer checks for leaks. They should have the best tool for the job! Check valves, fittings, and traps for leaks after every maintenance, and after thermal cycling as these can loosen some types of fittings.

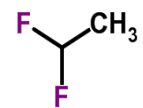
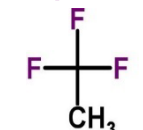
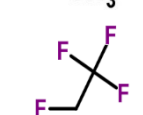
Use Leak Detector and/or Electronics Duster to Find Your Leaks

Why use a leak detector?

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



Typical Electronic Duster Components and Ions

	1,1- difluoroethane	<i>m/z</i> 51,65
	1,1,1- trifluoroethane	<i>m/z</i> 69
	1,1,1,2- tetrafluoroethane	<i>m/z</i> 69,83

For MSD use electronics duster / canned “air”

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

Agilent CrossLab CS (Cartridge System)

Features:

- Exchangeable cartridge with ADM Flow Meter
- Audible alarm
- Automatic Notification of Probe Filter Replacement
- Ergonomic and robust design
- Universal 3AA or USB power
- USB connects to web interface for added functionality and firmware updates
- Easy to view OLED Screen
- Kickstand

Leak detector
cartridge

Handheld



ADM Flow Meter
cartridge

Ordering Guide

G6693A – CrossLab CS Electronic Leak Detector

G6694A – Electronic Leak Detector Cartridge G6699A -
CrossLab CS Bundle: ADM Flow Meter and Electronic Leak

Detector G6691A – ADM Flow meter

- The bundle will include 1 handheld, 2 cartridges, and a 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!

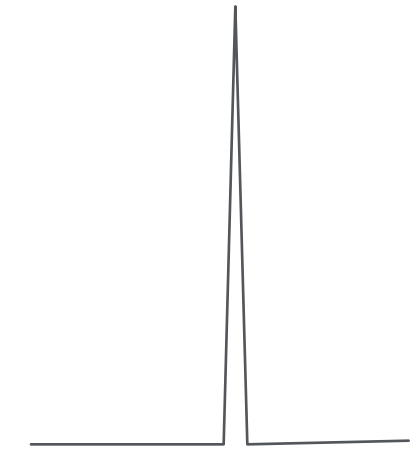
Leak and Installation Check

Inject a non-retained compound

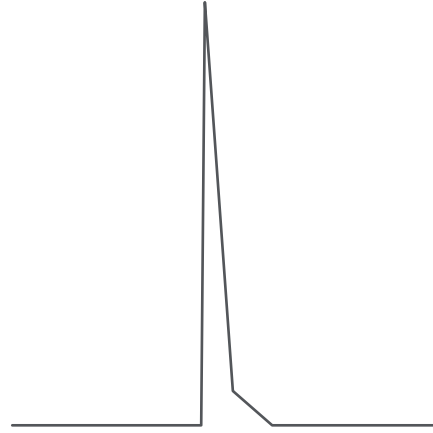
Detector	Compound
FID	Methane or butane
ECD	MeCl ₂ (headspace or diluted)
NPD	CH ₃ CN-acetonitrile (headspace or diluted)
TCD	Air
MS	Air or butane

The peak should be sharp and symmetrical

Non-retained Peak Shapes



Good installation



Improper installation or injector leak

$$\bar{F} = \frac{\pi r^2 L}{t_o}$$

\bar{F} = Flow rate (mL/min)
 r = Column radius (cm)
 L = Column length (cm)
 t_o = Retention time (sec)

\bar{F} is dependent on column temperature

$$\bar{\mu} = \frac{L}{t_o}$$

$\bar{\mu}$ = Average linear velocity (cm/sec)
 L = Column length (cm)
 t_o = Retention time (sec)

He 35-40 cm/sec
 H₂ 45-60 cm/sec

$\bar{\mu}$ is dependent on column temperature

Check for:

- Too low of a split ratio
- Injector or septum leak
- Liner problem (broken, leaking, misplaced)
- Column position in injector and detector
- Can use RT to determine actual column flow / velocity

Column Conditioning

System must be leak free before conditioning column

Heat the column to the lower of:

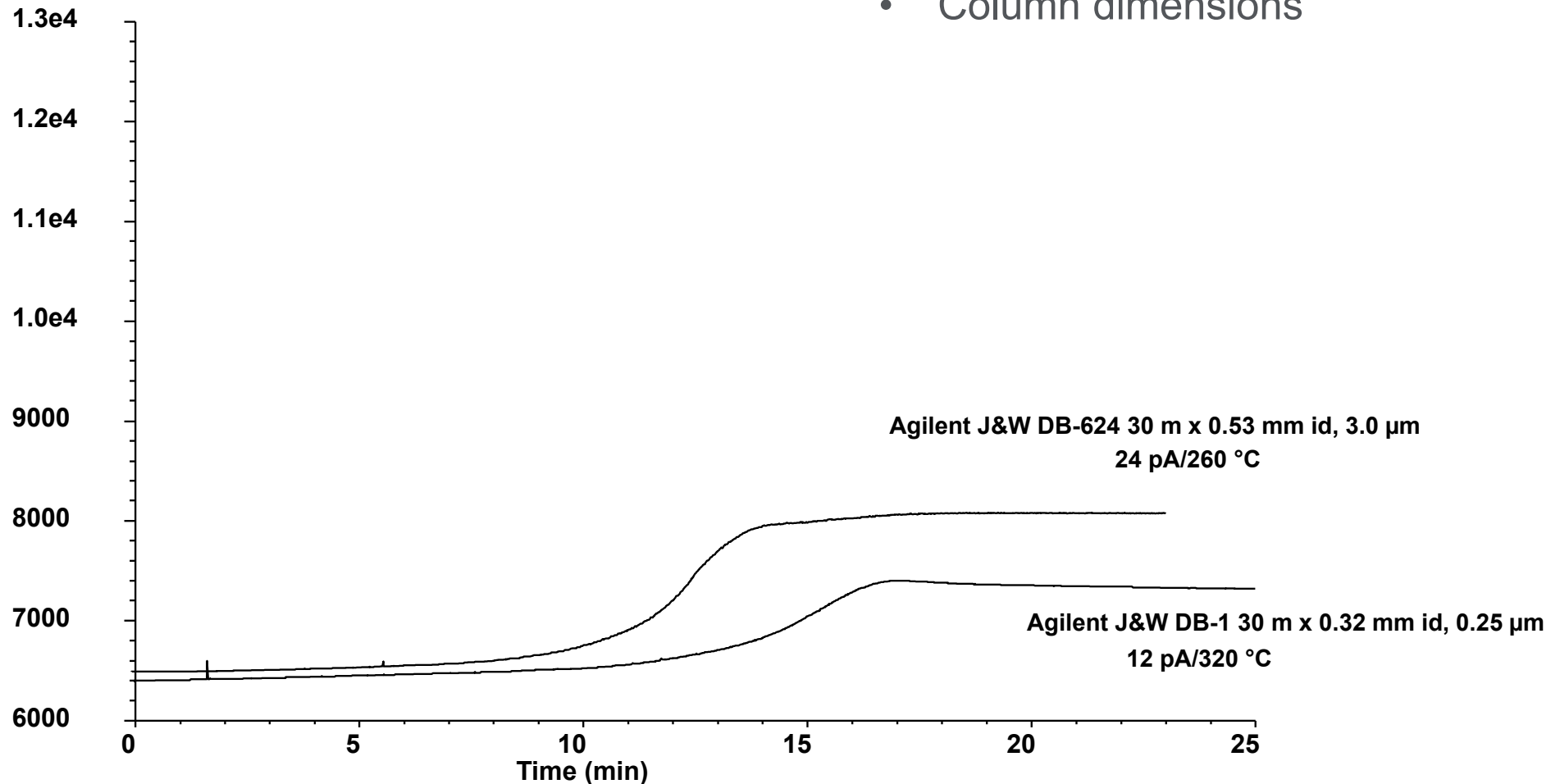
- Isothermal maximum temperature **or** 20 to 30 °C above highest operation temperature in your GC method.
- Temperature programming is not necessary.
- Be certain to observe that the baseline begins to stabilize/drop at the final conditioning temperature
 - if the base line continues to rise after final temperature is reached, this indicates a leak. **Stop immediately to investigate!**

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

What is Normal Column Bleed?

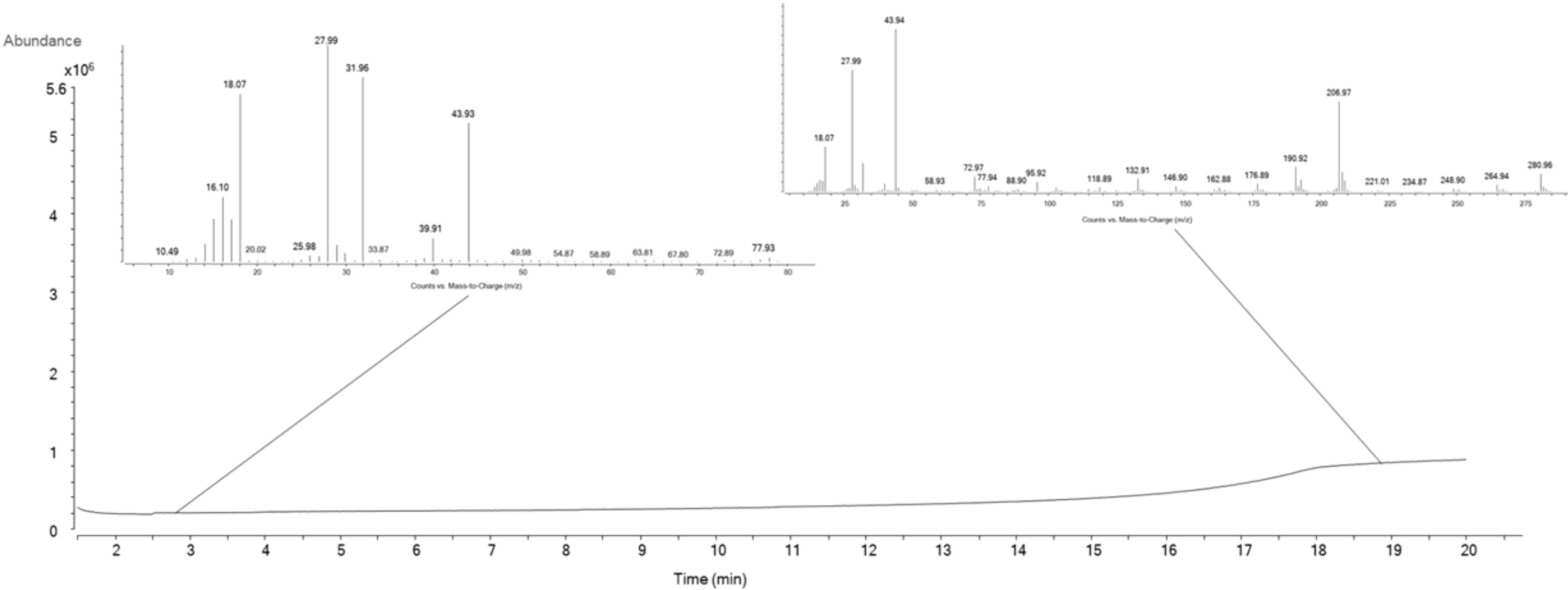
Normal background signal generated by the elution of normal degradation products of the column stationary phase. Column bleed is influenced by:

- Phase type
- Temperature
- Column dimensions



Mass Spectrum of Phenylmethylpolysiloxane Column Bleed

Normal background (HP-5ms UI)



What is a Bleed Problem?

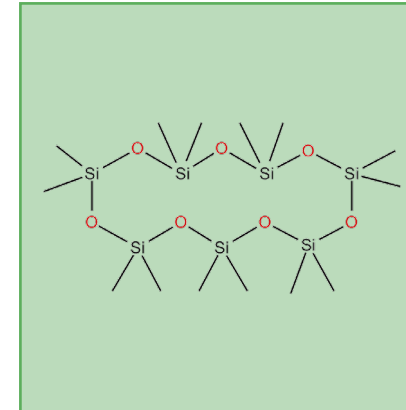
An abnormal elevated baseline at high temperature

It is not:

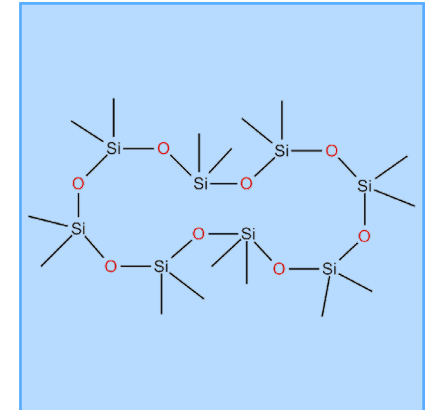
A high baseline at low temperature

Wandering or drifting baseline at any temperature

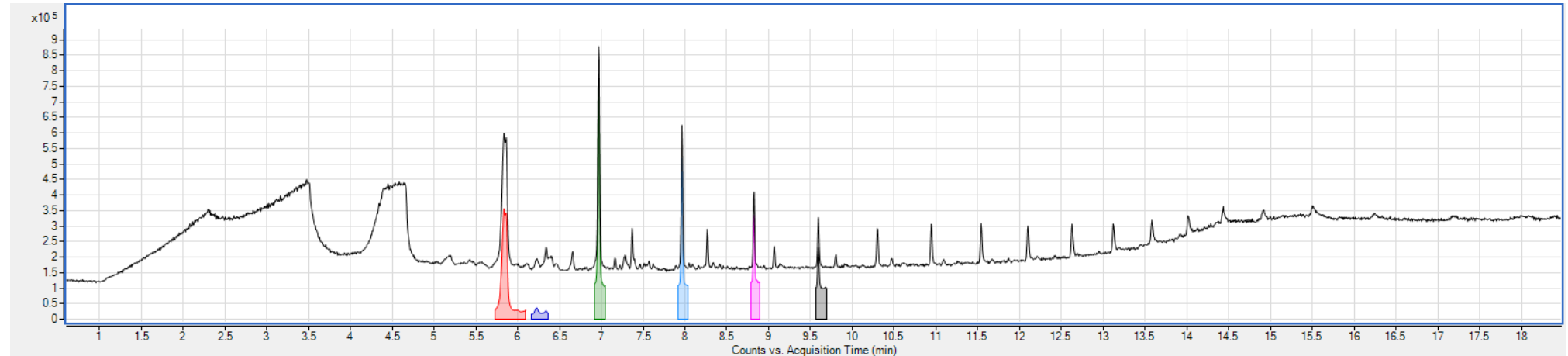
Discrete peaks



Tetradecamethyl
cycloheptasiloxane

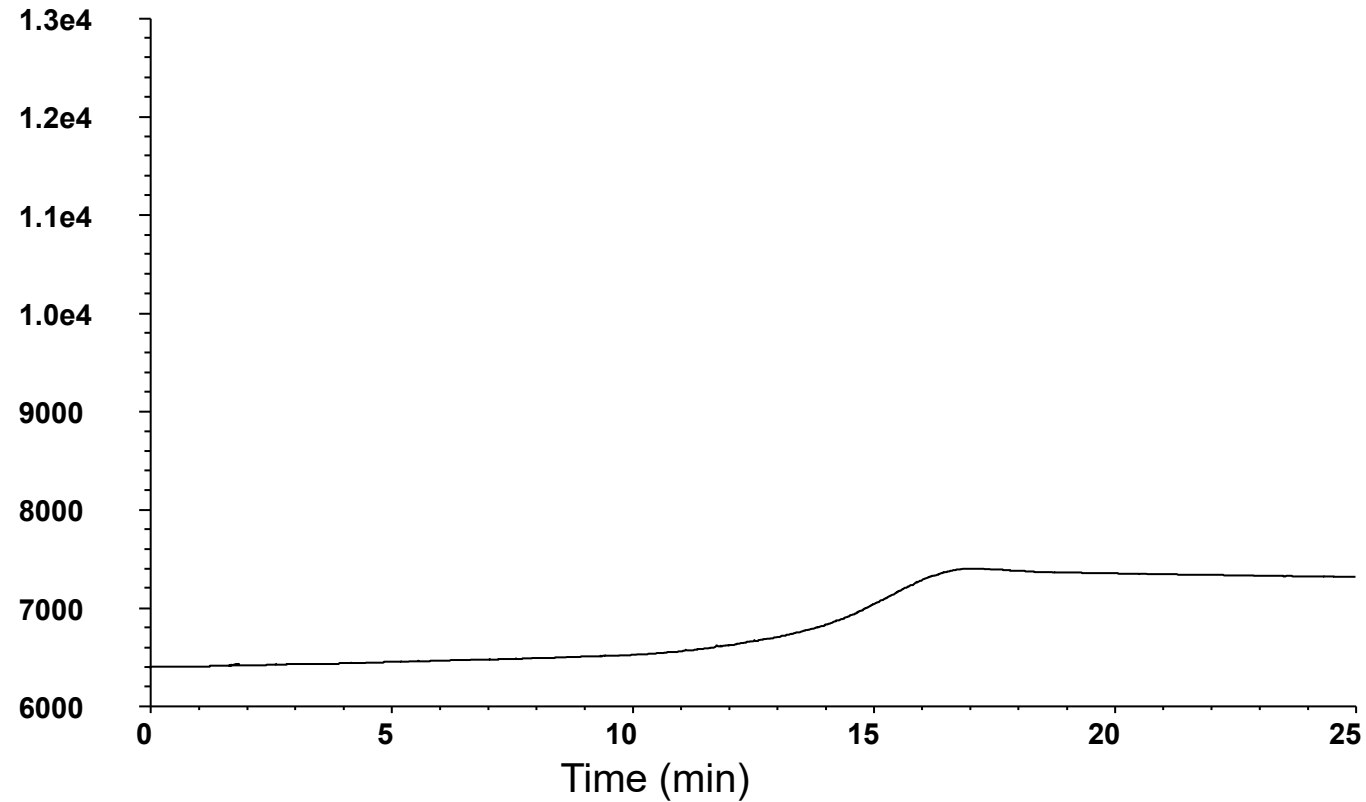


Hexadecamethyl
cyclooctasiloxane



Generating a Bleed Profile

Temperature program the column without an injection*



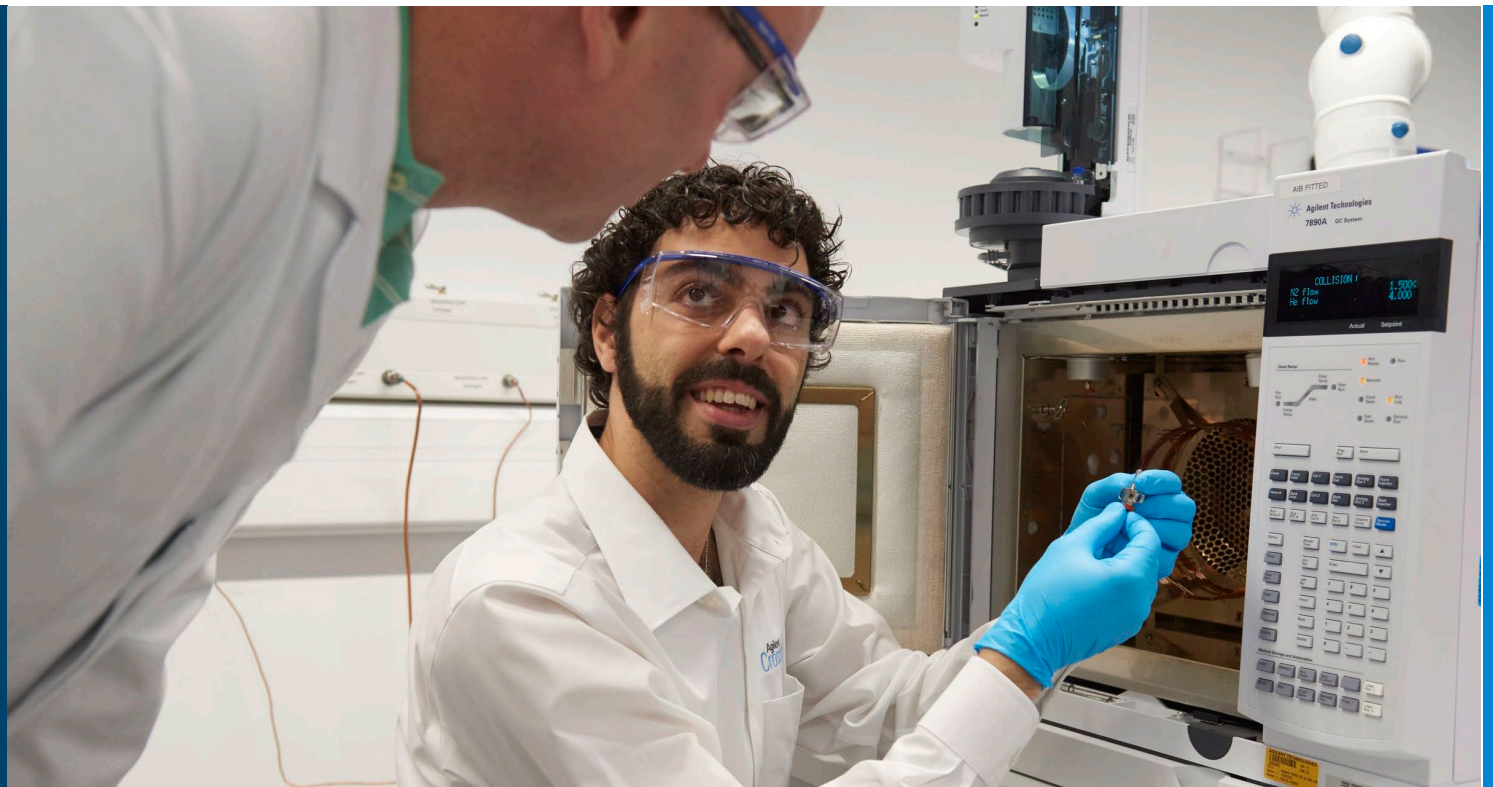
*Agilent J&W DB-1 30 m x 0.32 mm id, 0.25 μ m
Temperature program // 40 °C, hold 1 min // 20 °C/min to 320 °C, hold 10 min

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
- Agilent now has standards (formerly ULTRA Scientific)
 - <https://www.agilent.com/en/product/chemical-standards>

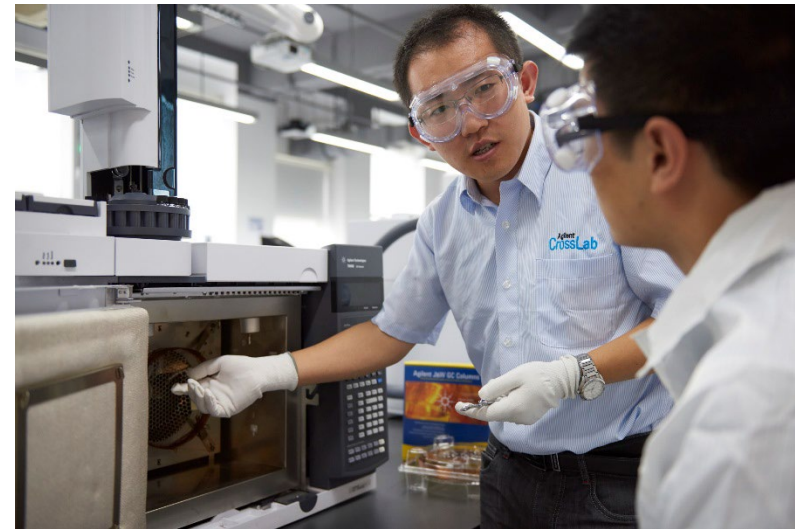
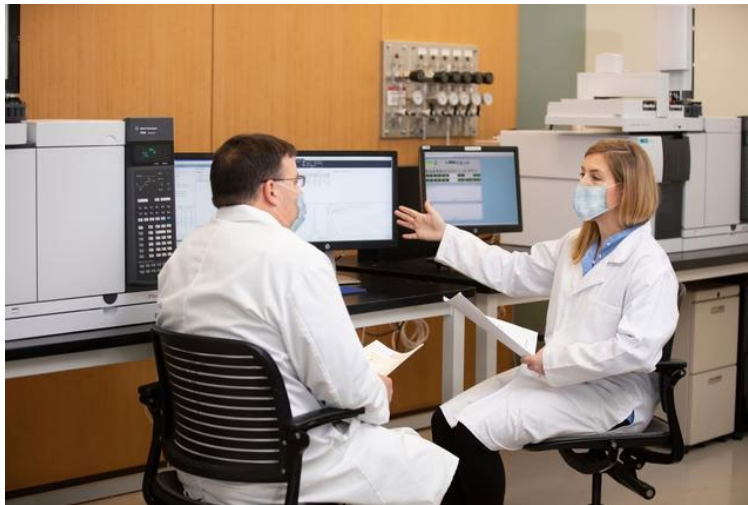


Proper Care of Your Column



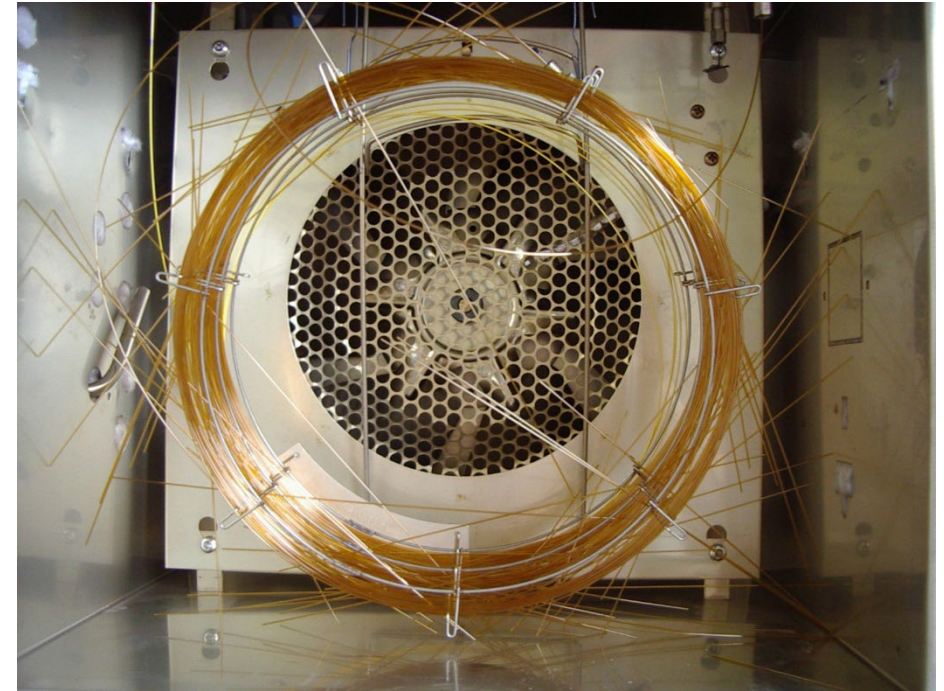
Common Causes of Column Performance Degradation

- 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



Thermal Damage

Degradation of the stationary phase is increased at higher temperatures

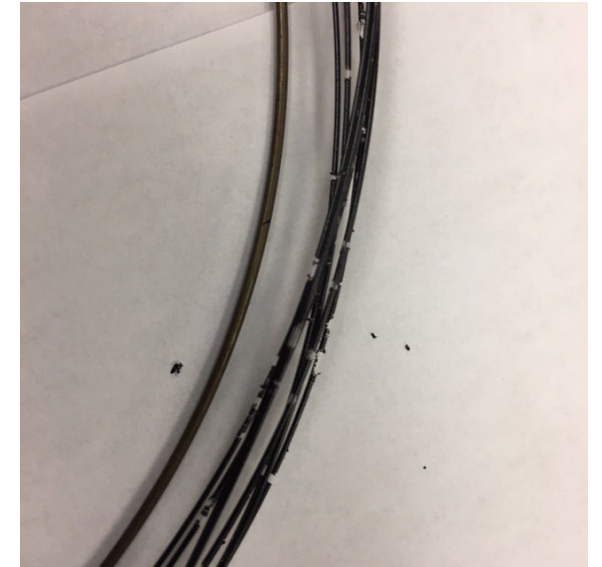
- Rapid degradation of the stationary phase (breakage along the polymer backbone) caused by excessively high temperatures

Isothermal limit = indefinite time

Programmed limit = 5–10 minutes

- If this happens:
 - Disconnect column from detector
 - “Bake out” overnight at isothermal limit
 - Remove 10–15 cm from column end

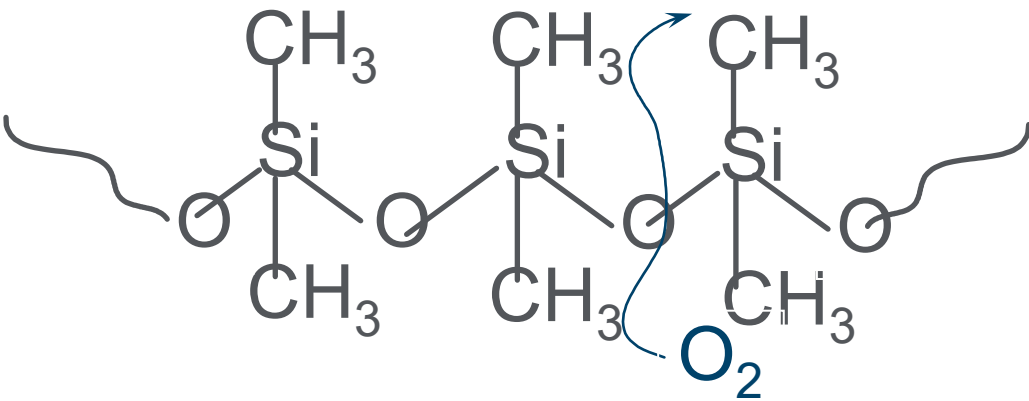
-Below lower temperature limit essentially “freezes” the phase rendering it useless, but only while below this temperature (i.e. its reversible)



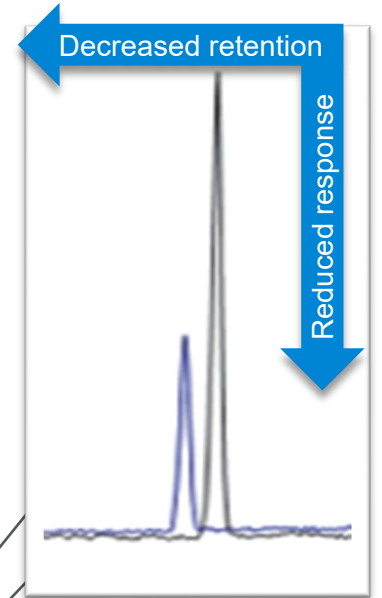
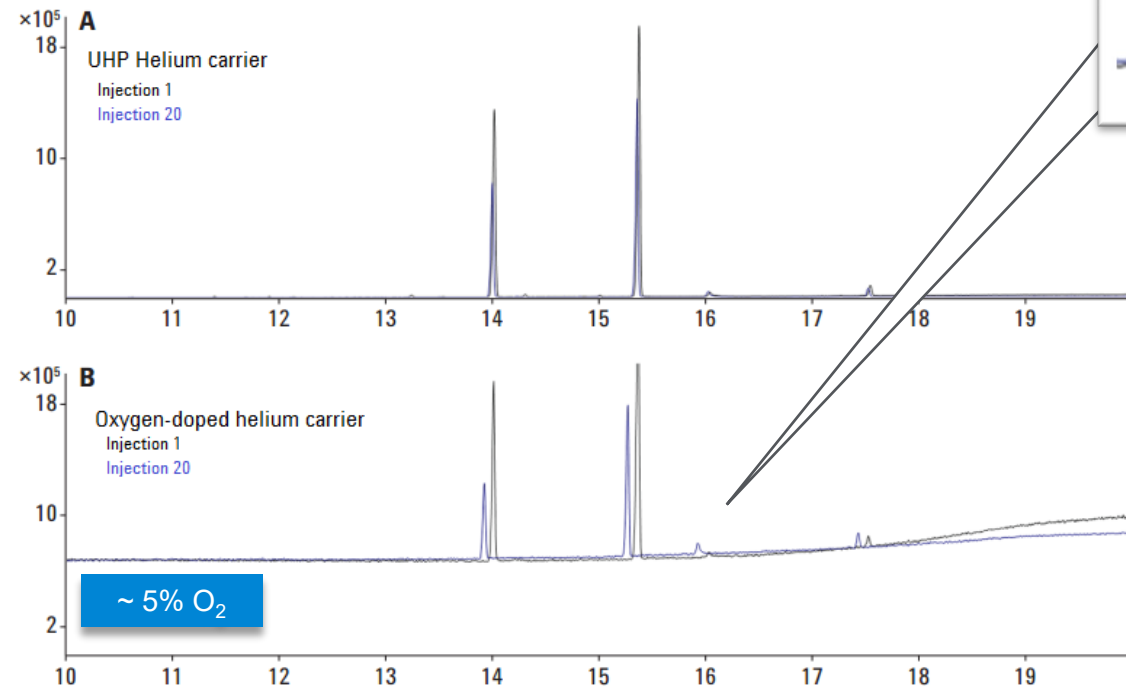
Column continuously exposed to temperatures above its temperature limit

Oxidation (O2 Damage)

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.)

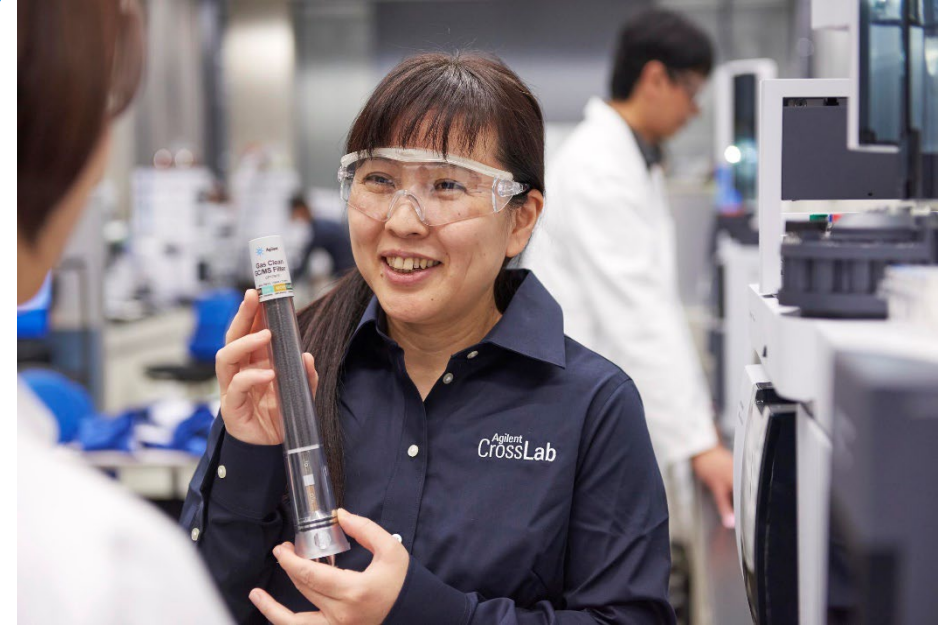


Dimethylpolysiloxane



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.

HCl NH₃ KOH NaOH

H₂SO₄ H₃PO₄ 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



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)

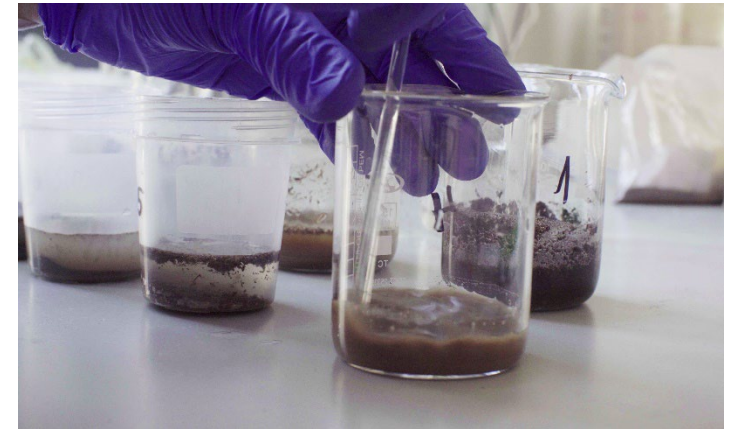
Typical Samples That Contain a Large Amount of Residues

Biological (blood, urine, tissue, plants)

Soils Foods

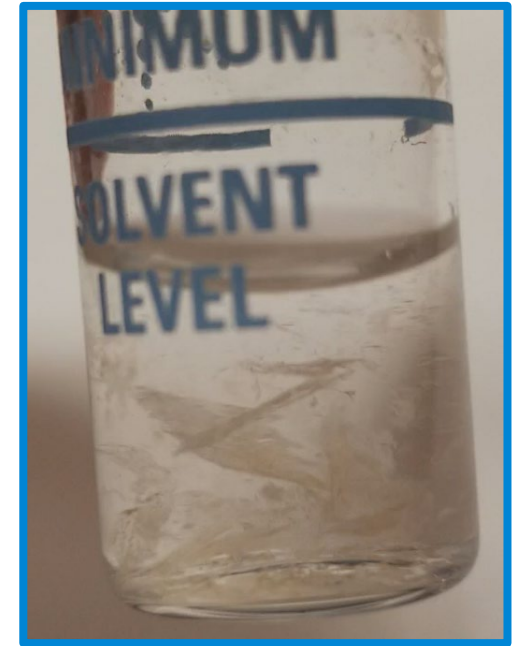
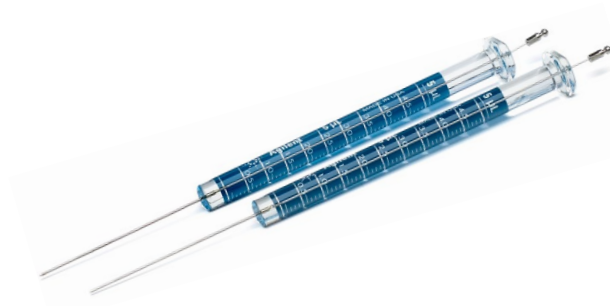
Wastewater Sludges

All samples contain residues (even standards)

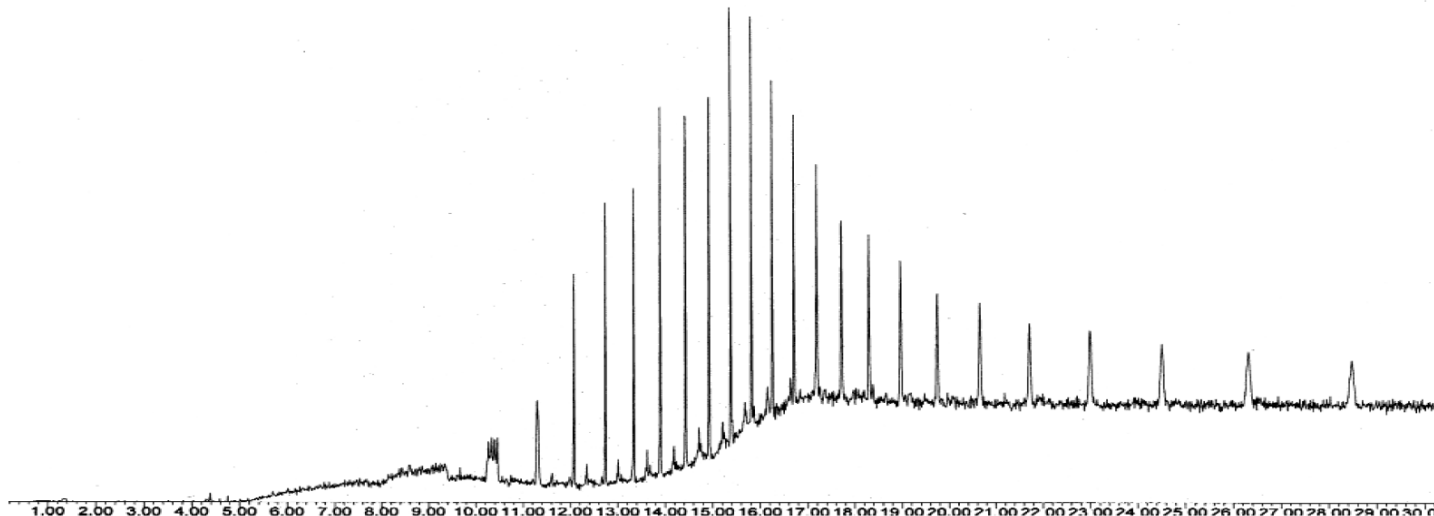


Other Sources of Contamination

- Septum and ferrule particles
- Gas 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

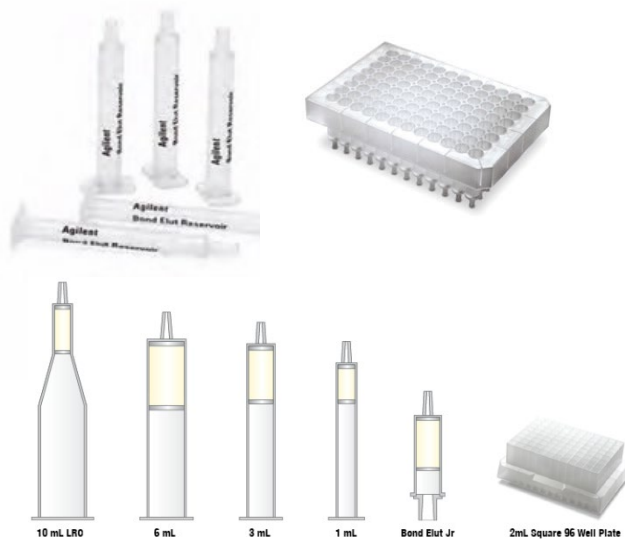
- Any portion of the sample that elutes from the column after the current chromatographic run.

Methods to Minimize Nonvolatile Residue Problems

- Sample cleanup
- Packed injection port liners
- Guard columns



Offline Options for Sample Matrix Removal



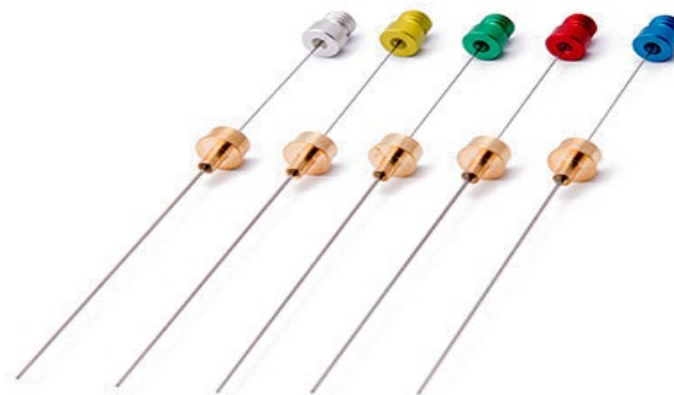
Bond Elut solid phase extraction cartridges and plates



Filter vials



QuEChERS



SPME



Captiva EMR-Lipid filtration cartridges and plates



Chem Elut S



Captiva syringe filters

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 connected with a zero dead volume union.

Dura-Guard columns have integrated guards (no union)

Nonvolatile Contamination

What to do if it happens

- Do not “bake out” the column
- Front end maintenance
 - Change the injector liner + gold seal
 - Clean the injector
 - Cut off 0.5–1 m of the front of the column
- Turn the column around
- Cut the column in half



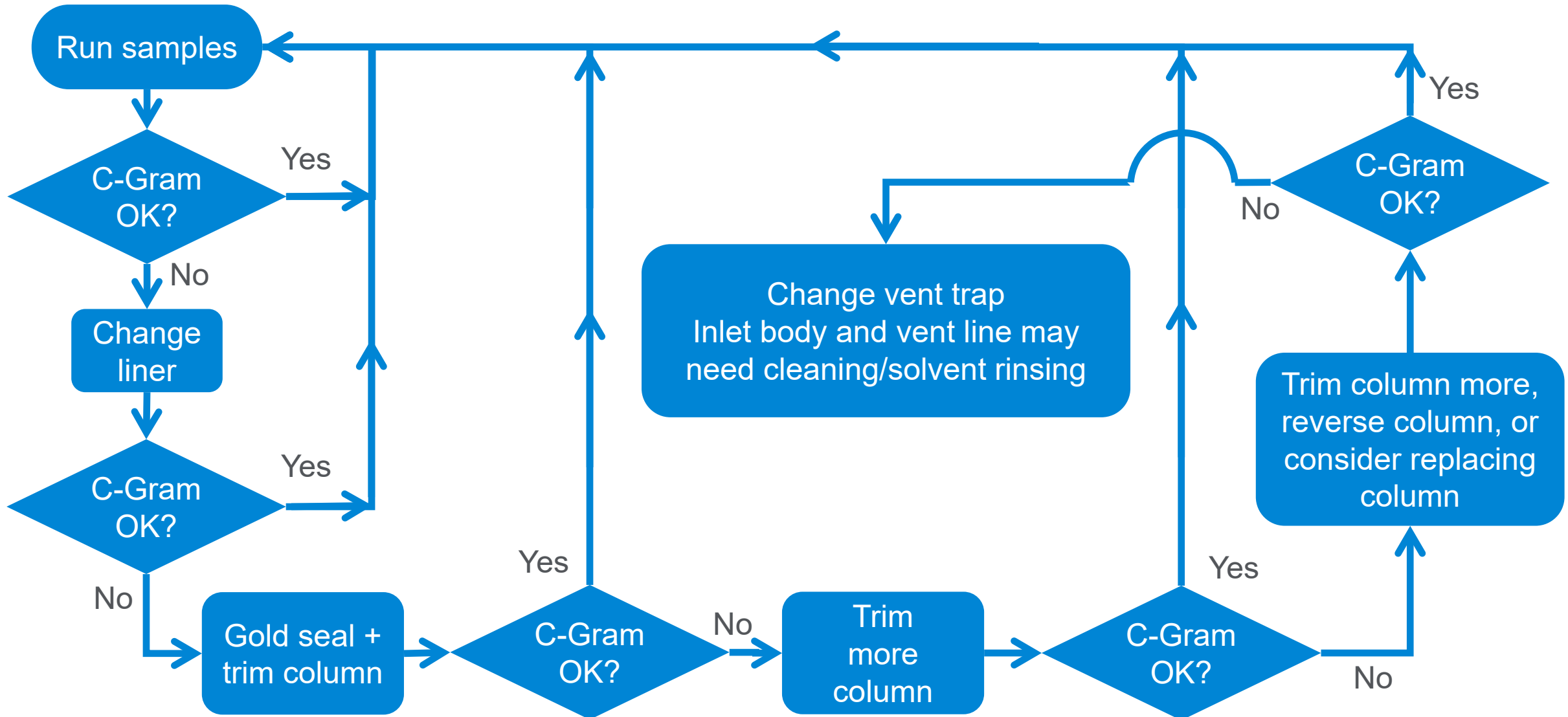
Semivolatile Contamination

What to do if it happens

- Front end maintenance
 - Change the injector liner + gold seal
 - Clean the injector
 - Cut off 0.5–1 m of the front of the column
- “Bake out” the column
 - Limit to 1–2 hours
 - Longer times may polymerize some contamination and reduce column life



Inlet Maintenance Flow Chart



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



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

For immediate identification and use monitoring of your GC column

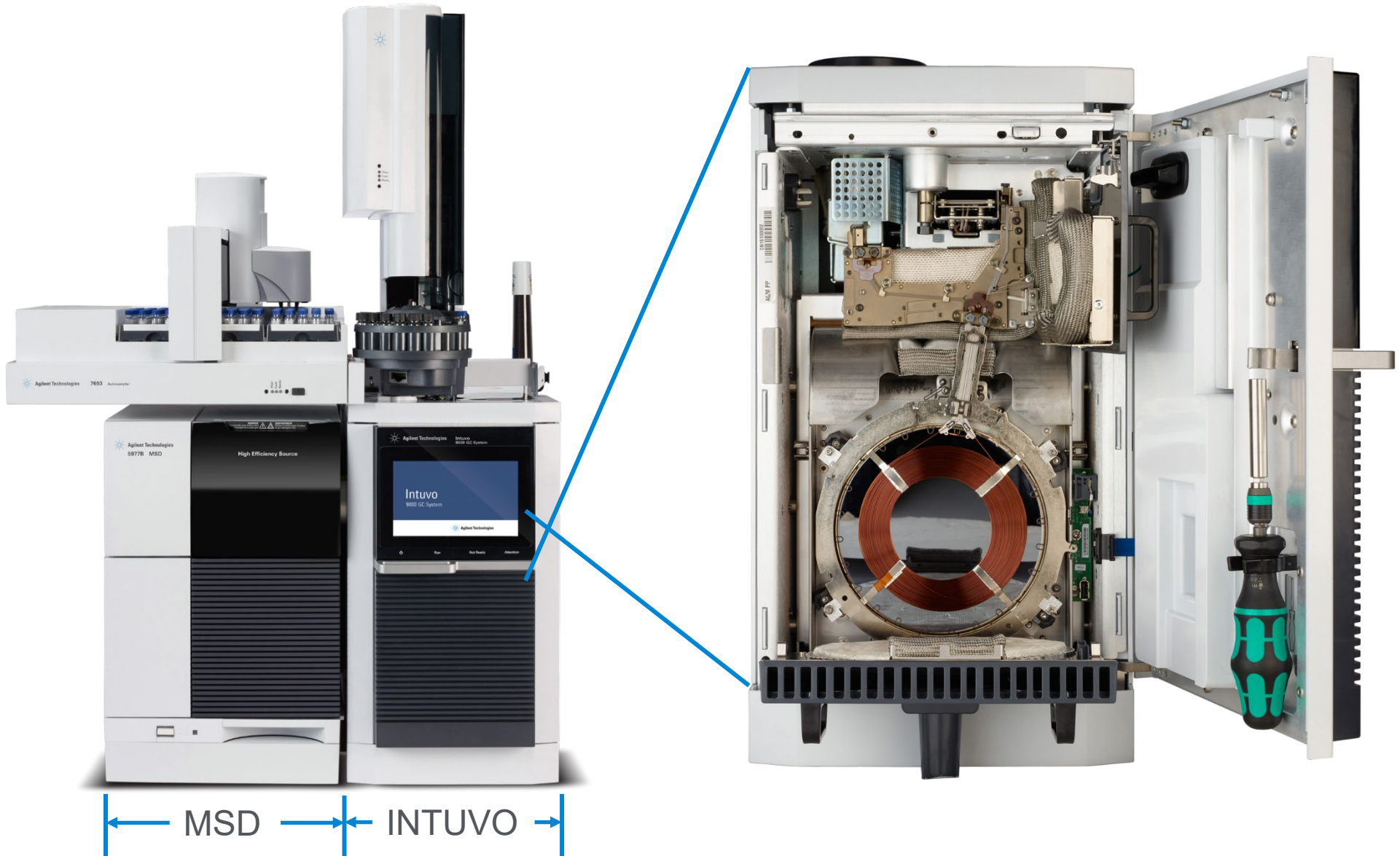
- Available with the Agilent 8890 GC model only
- Can track use of a GC column
- Smart key contains GC column information, including:
 - Part and serial numbers
 - Number of injections/runs
 - Time at/above temperature limits
 - Date installed
 - Temperature limits – GC columns
 - If more than one column 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



Common Frustrations with GC

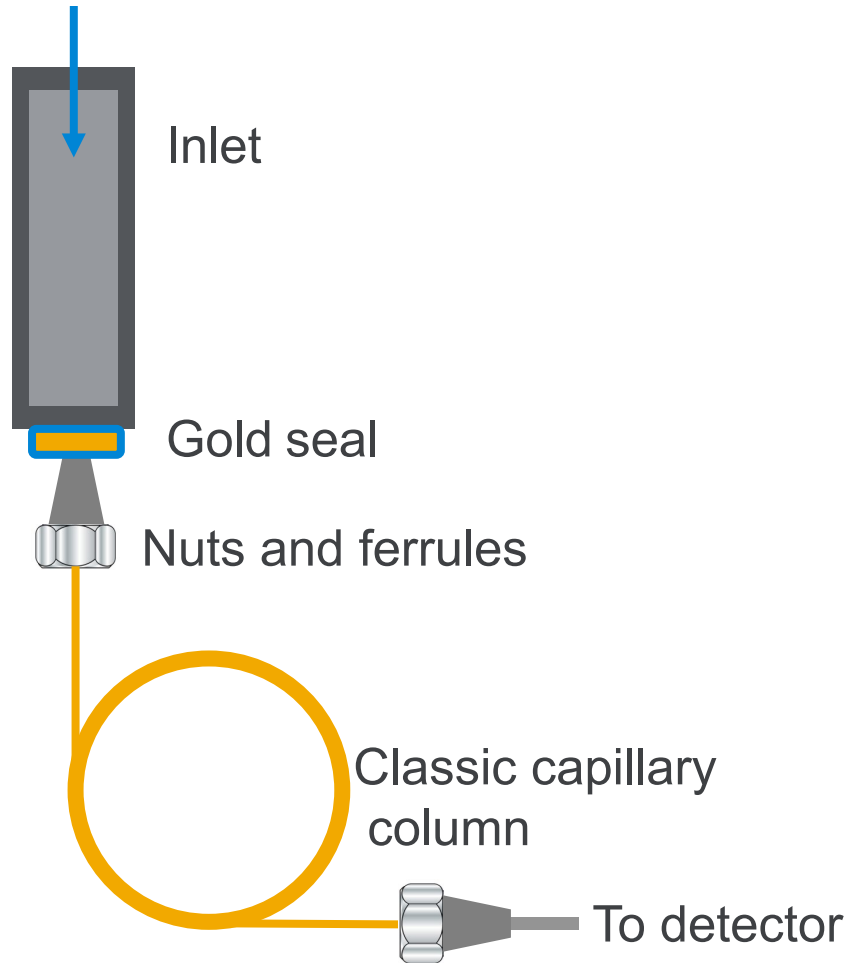
- Measuring column length correctly
- Cutting your column correctly
- How tight is too tight?
- Clipping columns to deal with active sites, then updating retention times

Agilent Intuvo 9000 GC System

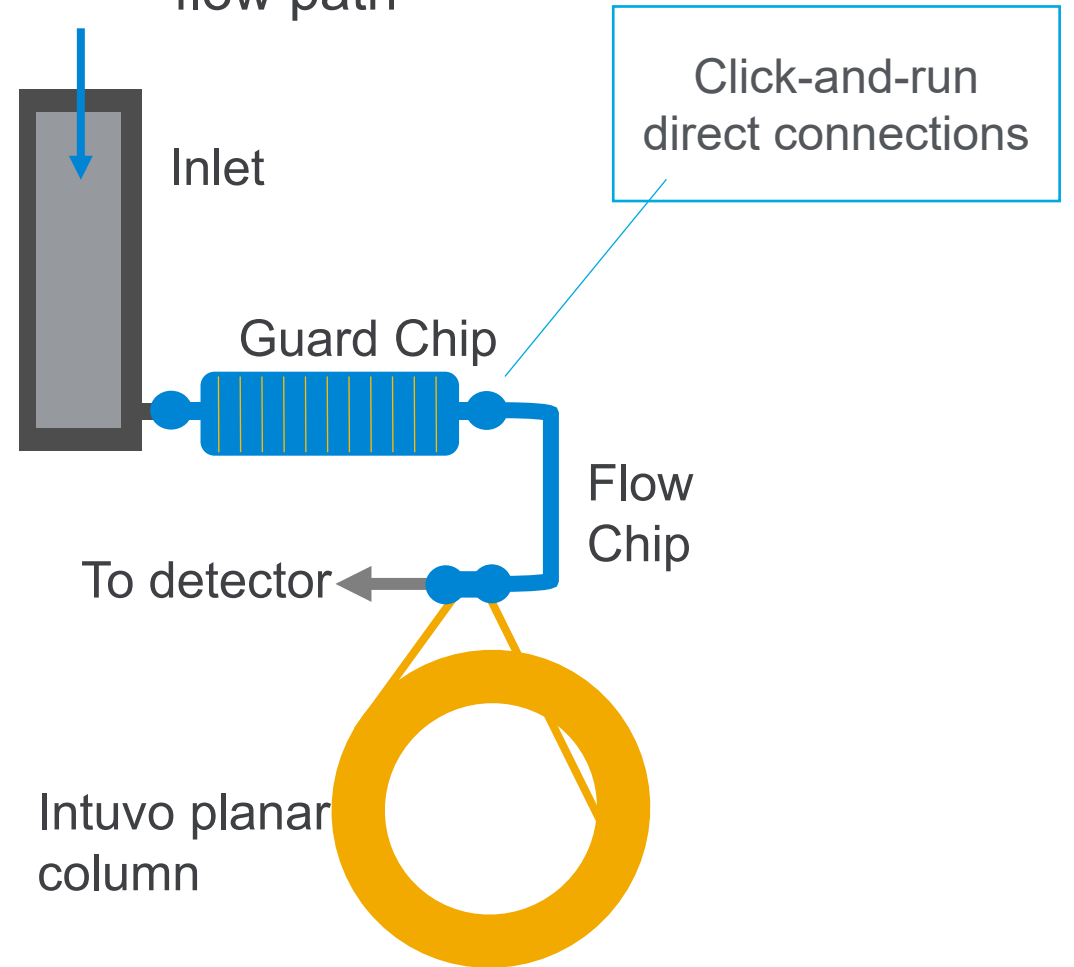


Innovating the GC Flow Path

Conventional flow path

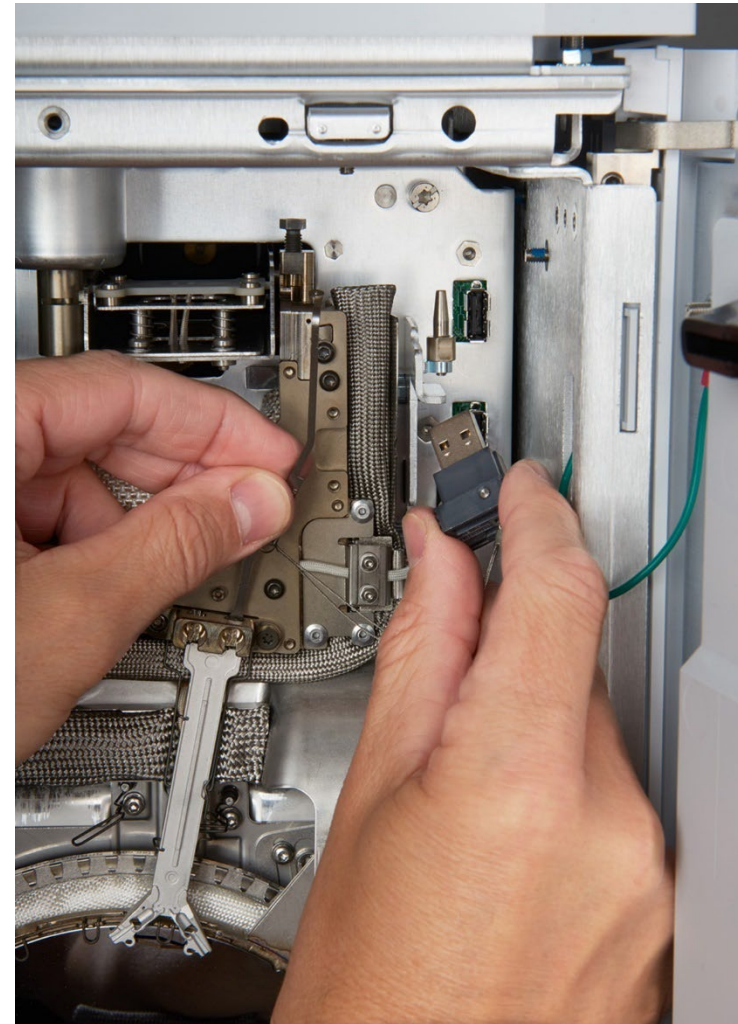


Intuvo flow path



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



Always Remember

- Start with a good installation
- Maintain an oxygen-free system
- Avoid physical, thermal, and chemical damage
- Take steps to prevent contamination
- Perform front-end maintenance regularly to stay ahead of problem



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

lc-column-support@agilent.com

spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com

Other Resources

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



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 about the return on investment on the Agilent Intuvo 9000 GC System

Playing time: 4:17

<https://www.agilent.com/cs/library/usermanuals/public/G4580-90008.pdf>