Keeping your GC Column Happy and Healthy: Installation, Care, and Maintenance

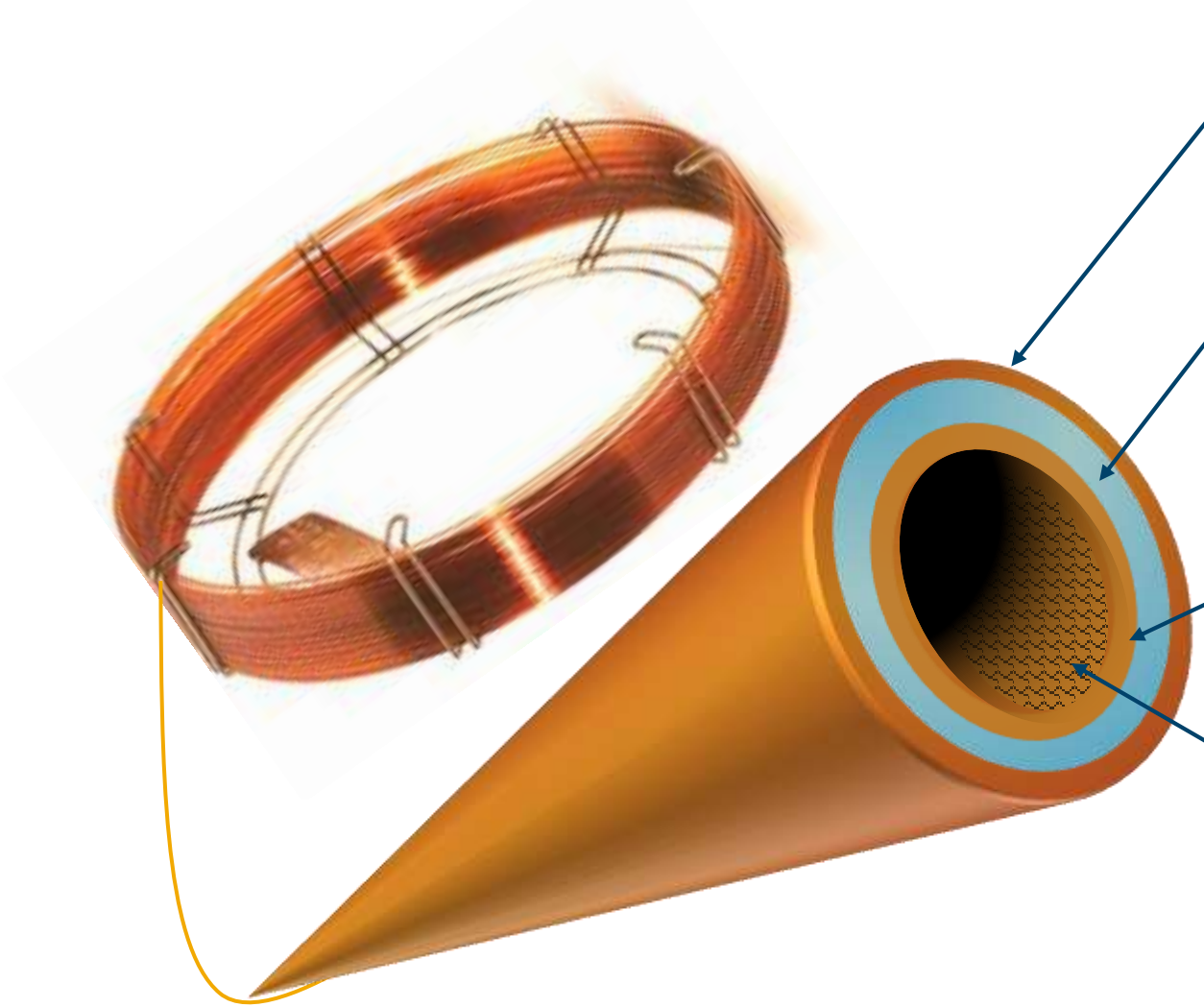
Alexander Ucci and Mark Sinnott
Application Engineers
May 30, 2019

Agilent
Trusted Answers

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.

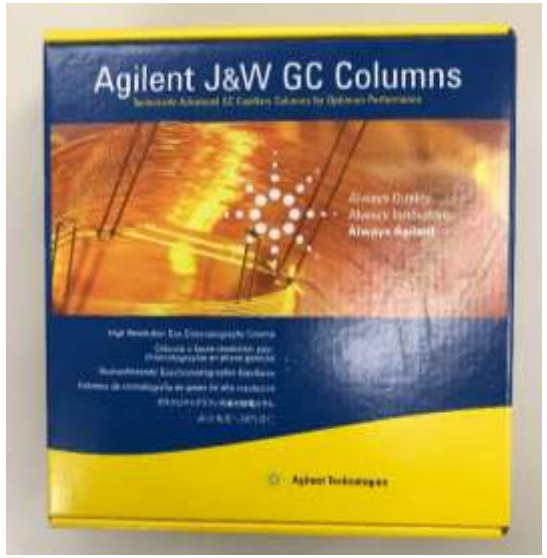
Deactivation

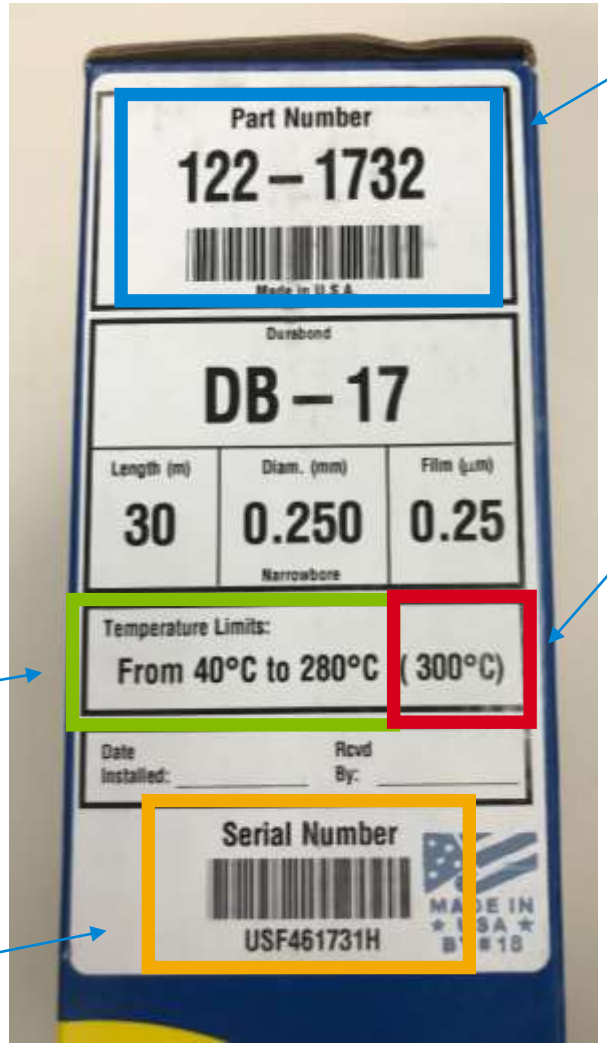
Chemical treatment layer; smooths fused silica surface to enhance inertness

Stationary phase

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

The “Unboxing” of the GC Column





Important for identification and re-ordering

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: F 5 4 2 2 5 2 H

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:

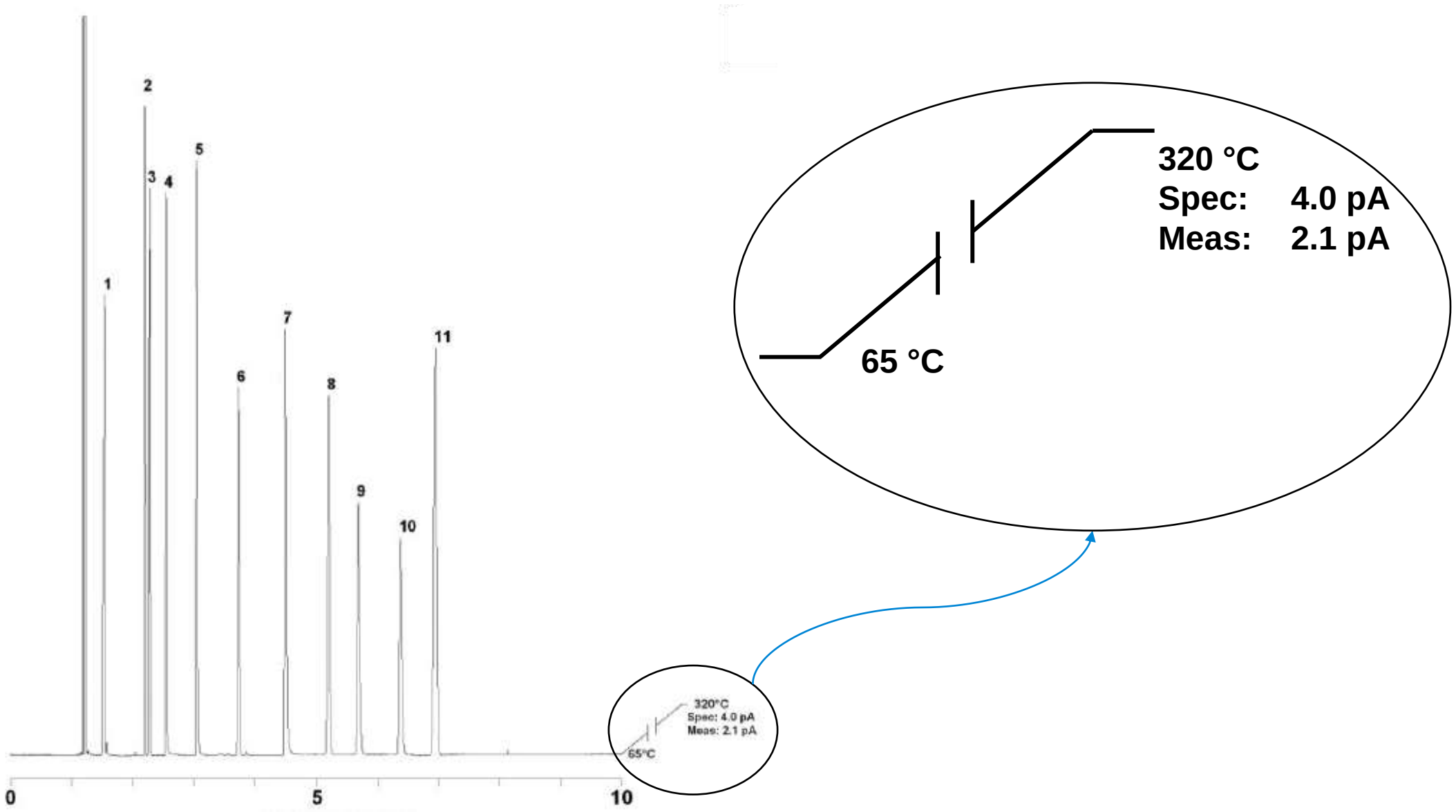
1-OCTENE, n-OCTANE	2.97

Compound Identification	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



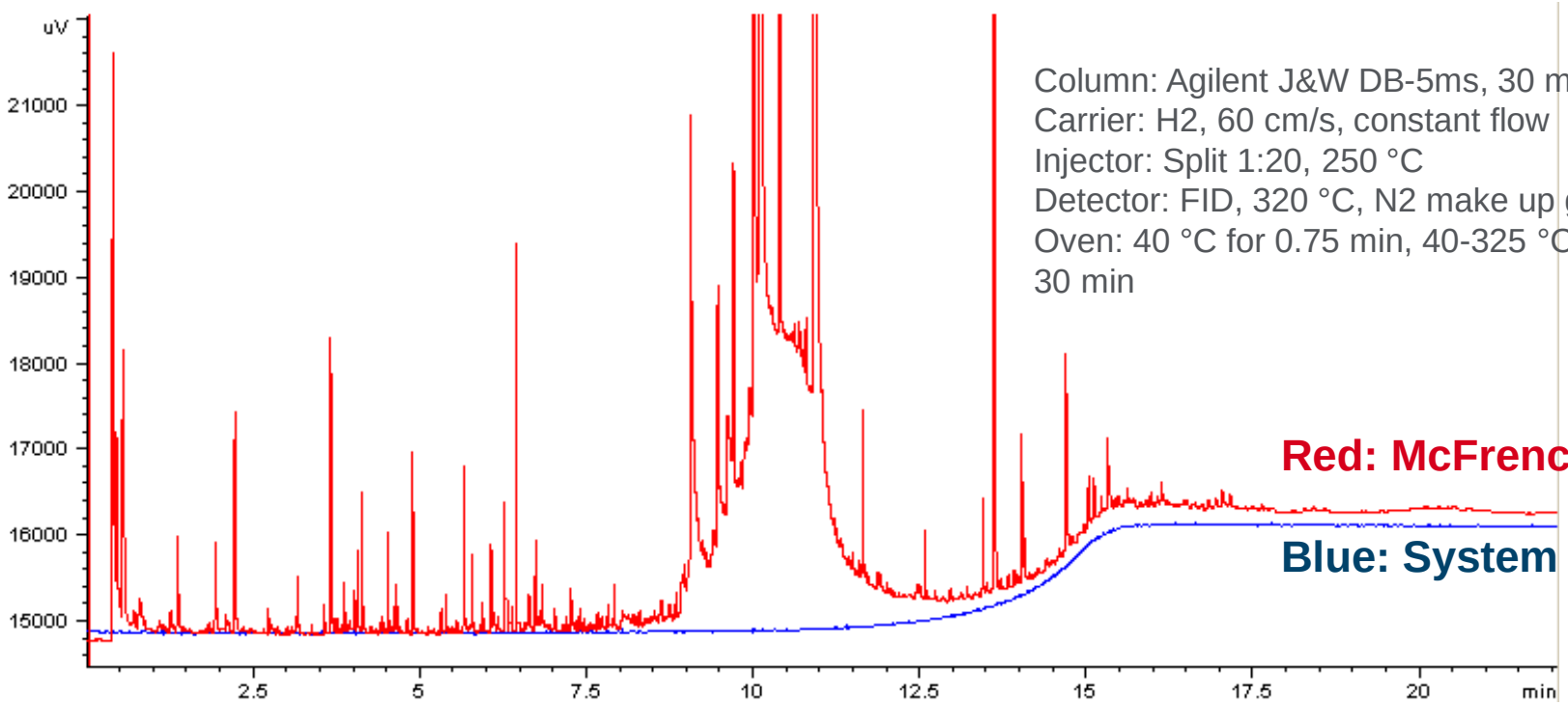
Test Mixture Components

Compounds	Purpose
Hydrocarbons	Efficiency Retention
FAMEs, PAHs	Retention
Alcohols	Activity
Acids	Acidic character
Bases	Basic character

Column Installation Procedure

- Install the column
- Leak and installation check
- Column conditioning
- Setting linear velocity or flow rate
- Bleed profile
- Test mix

Contamination from French Fry Grease



Column: Agilent J&W DB-5ms, 30 m x 0.25 mm, 0.25 µm
Carrier: H2, 60 cm/s, constant flow
Injector: Split 1:20, 250 °C
Detector: FID, 320 °C, N2 make up gas
Oven: 40 °C for 0.75 min, 40-325 °C at 20 °C/min, 325 °C for 30 min

Red: McFrench fry

Blue: System blank

Procedure:

(1) Held French fry for 5 s.
(2) Fingertip was wiped with paper towel to remove as much of the contamination 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 when oven reached 40 °C.

“Touchless” Packaging

Column Installation

What type of ferrule should I use?

Polyimide

Graphite

Polyimide/ graphite

Flexible Metal

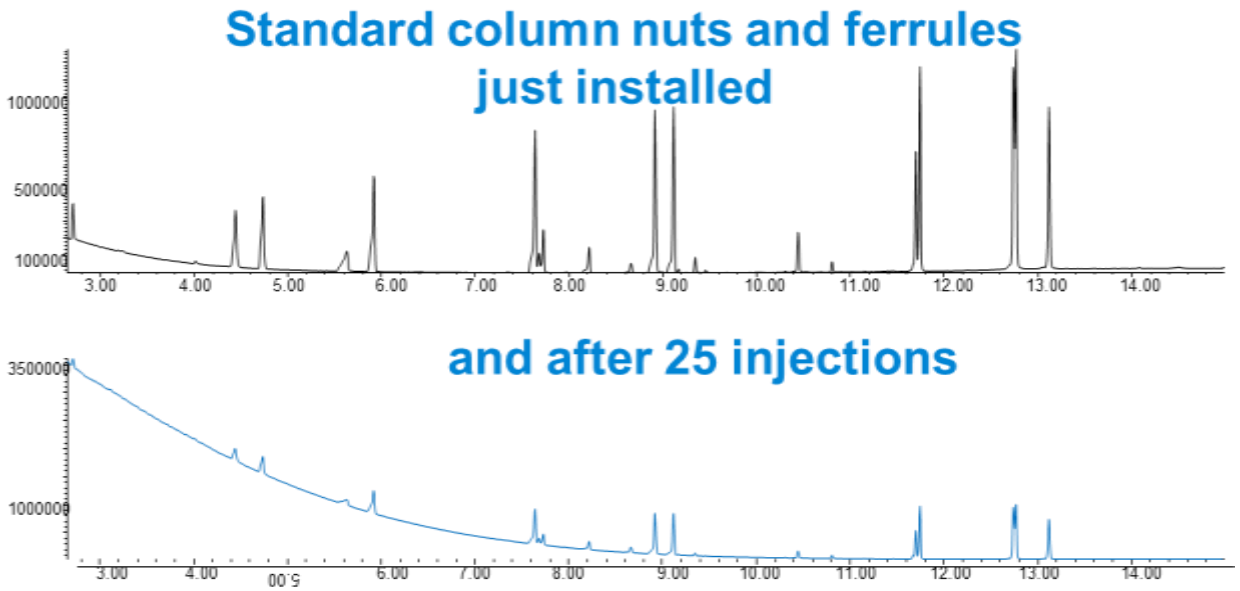
Composition	Re-use	Max Temperature (°C)	Use	Limitation
Polyimide (Vespel)	Yes	280	Easy seal	Shrink after heating causing leaks after thermal cycle; isothermal only
Graphite	Yes	450	FID, NPD, inlets	Contamination, permeable to air – not for oxygen sensitive detectors
Polyimide/graphite (85%/15%)	Limited	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

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.

Better Connections: Self Tightening Column Nuts

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

For inlet or detector
p/n 5190-5233

For mass spectrometer transfer line
p/n 5190-6194

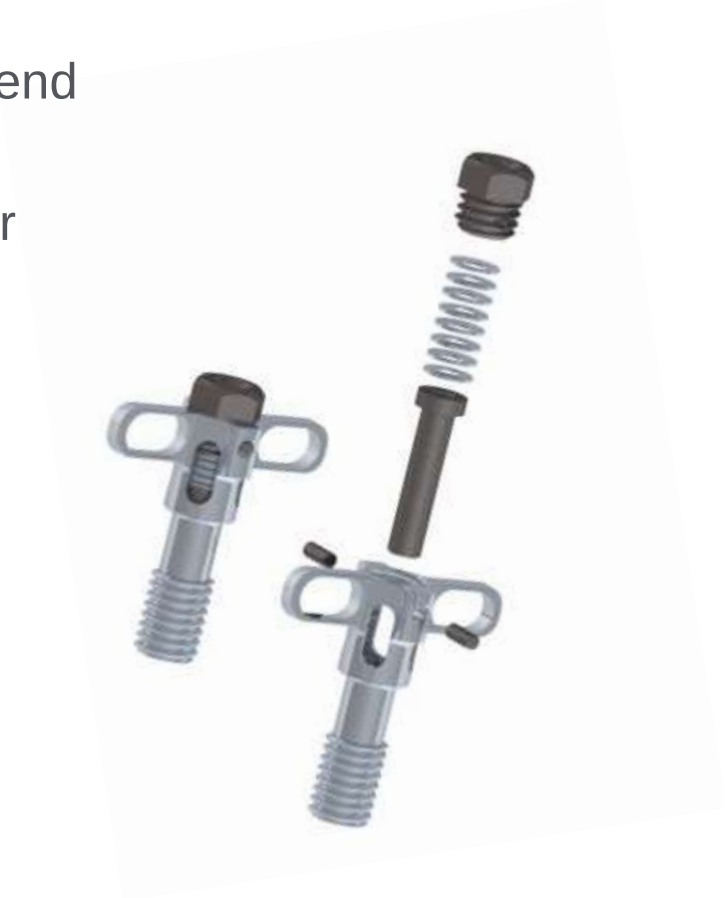
A short ferrule exposes more thread of the fitting for better sealing.

How Do Self Tightening Column Nuts Work?

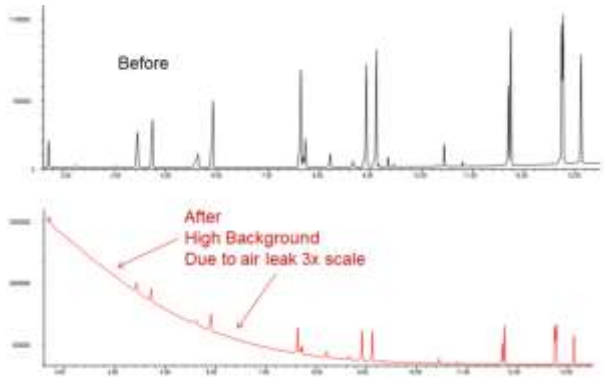
- Ease-of-use – install in dark, small space in GC oven without wrenches
- Wing design for finger-tight installation with graphite/polyimide blend ferrules
- No tools, dramatically reduces force preventing over-tightening or damage
- Robust stainless steel construction

Plus

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

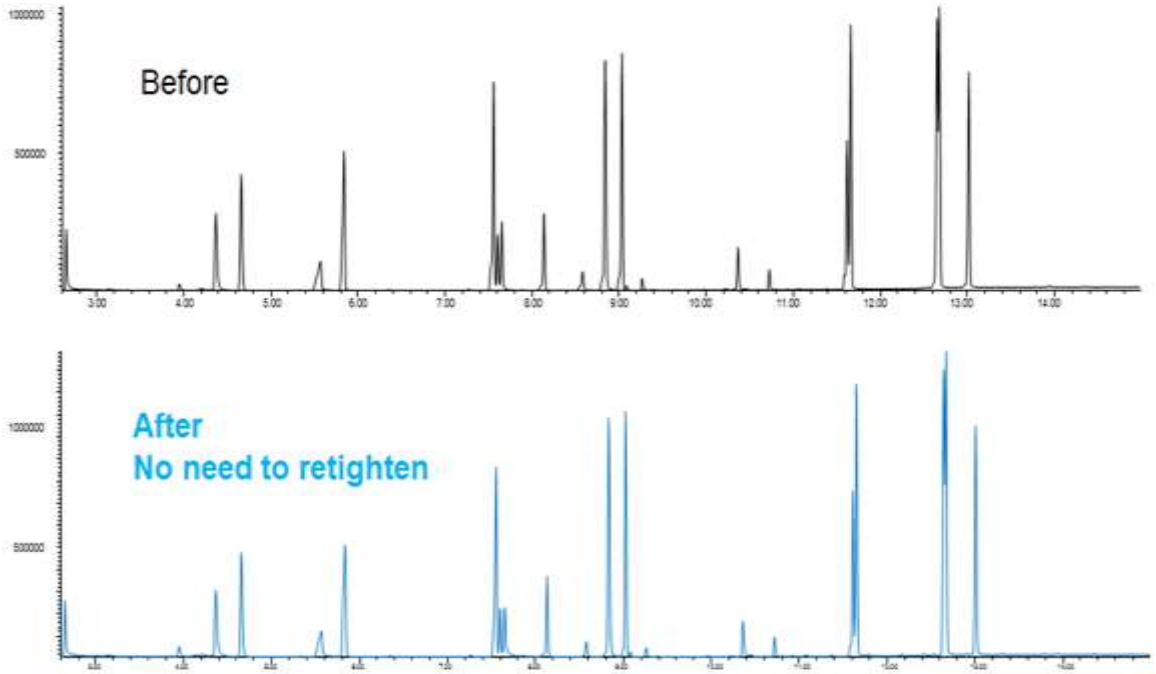


Benefit of Self Tightening Column Nuts



Takes you from this…

…to this



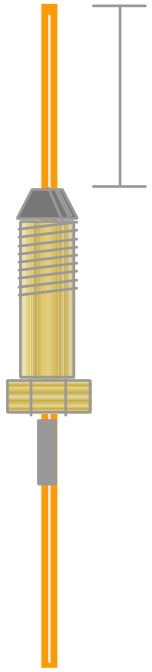
Without retightening, the baseline remains flat after 400 runs with no indication of leaks when using the Self Tightening column nut.

Ref. Technical note: 5991-3612EN

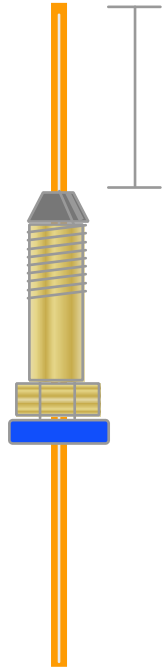
Column Installation

Measuring the right distance

White out



Septa

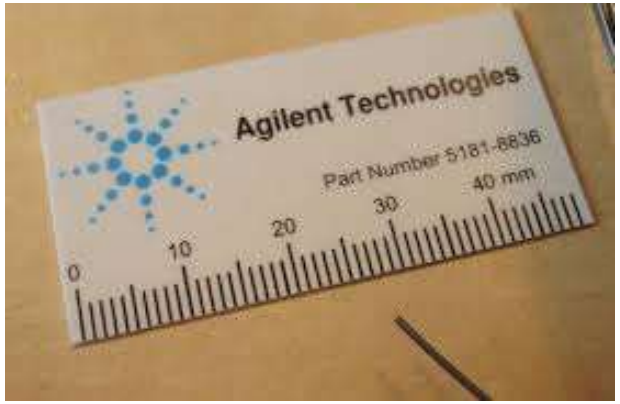


Extra septa

Cutting the Column

Gently scribe through the polyimide coating

- Do not attempt to cut the glass



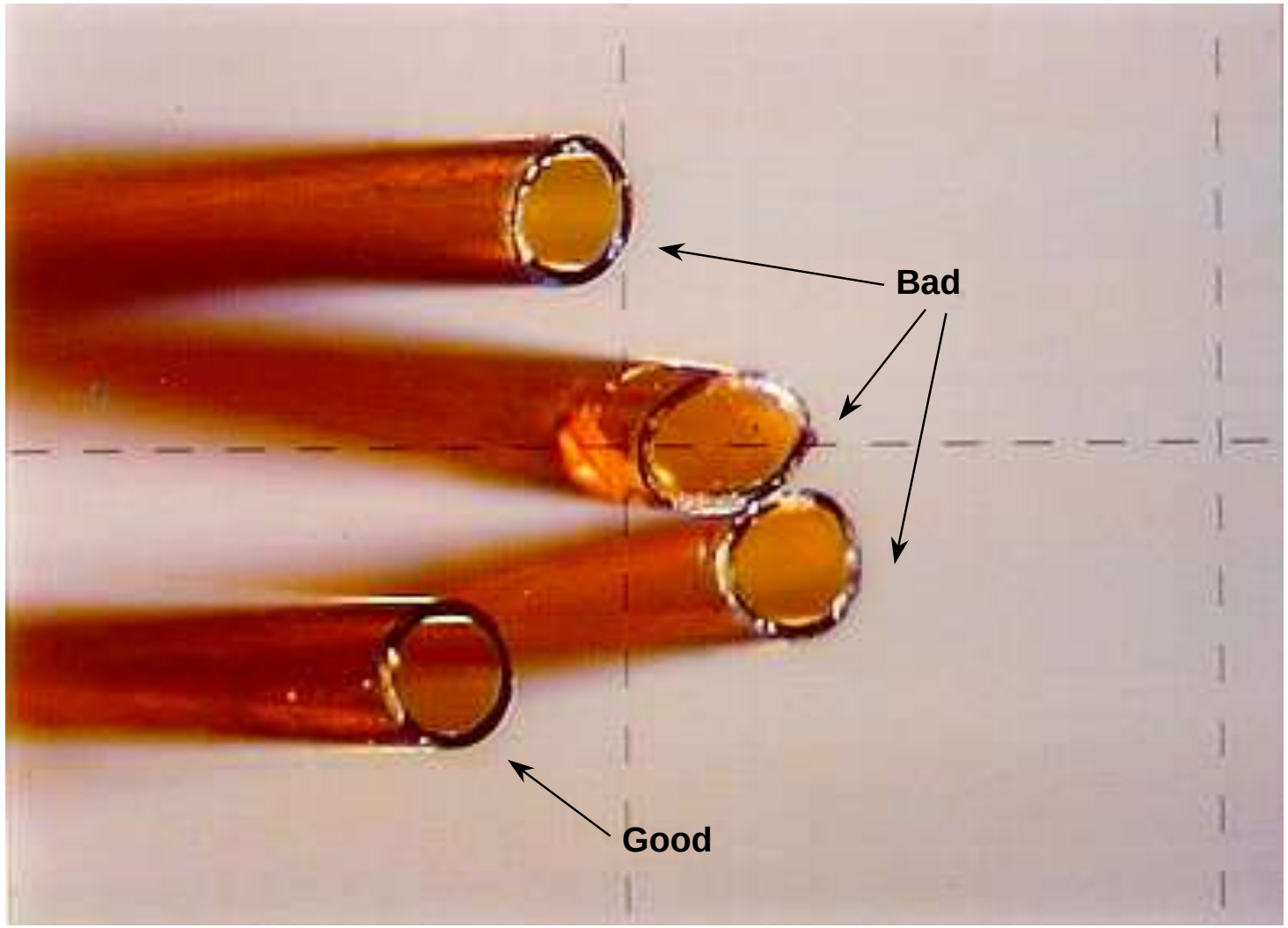
Recommended tools

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

Do not use

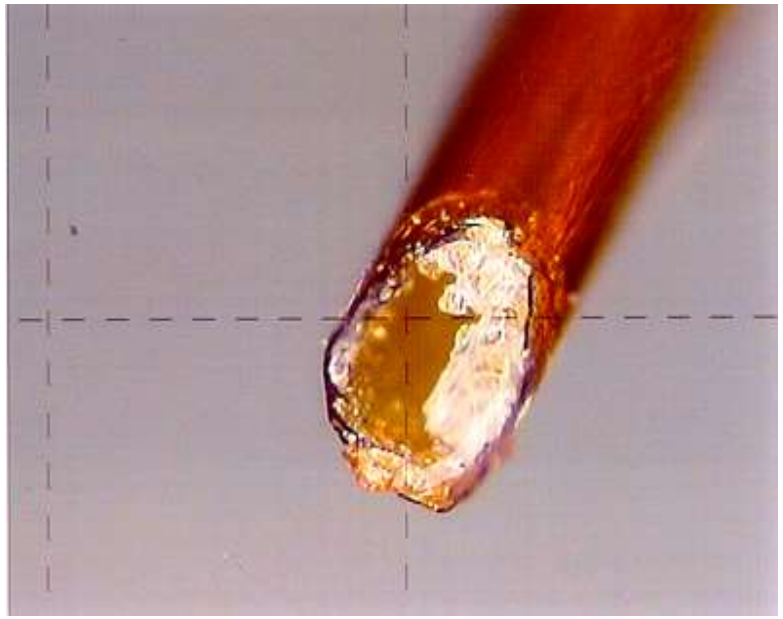
- Scissors, file, and so on

Examples of Column Cuts



Column Installation

How tight is tight?



Over-tightened ferrule

Column Installation
Leak check

Do not use snoop

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

Gas leak detector
p/n G3388B

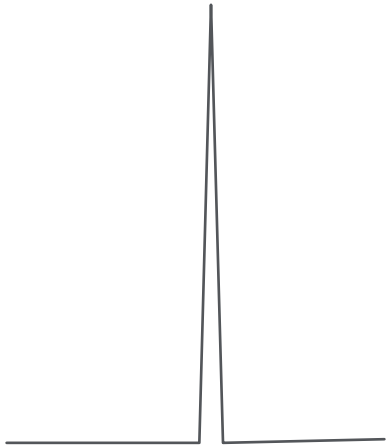
Leak and Installation Check

Inject a nonretained compound

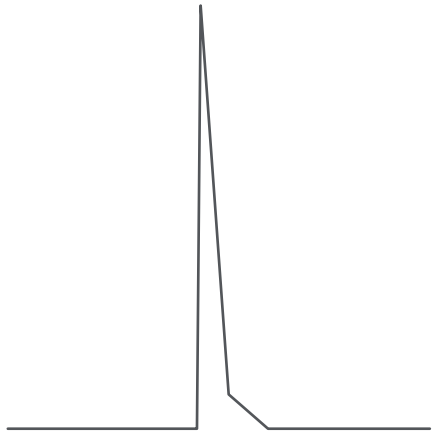
Detector	Compound
FID	Methane or butane
ECD	$MeCl_2$ (headspace or diluted)
NPD	CH_3CN-acetonitrile (headspace or diluted)
TCD	Air
MS	Air or butane

The peak should be sharp and symmetrical

Nonretained Peak Shapes



Good installation



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

Calculating Linear Velocity

Inject a nonretained compound and obtain the retention time:

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

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

He 20-40 cm/s
H_2 35-55 cm/s

μ is *dependent* on column temperature, but is *independent* of column dimensions

Calculating Flow Rate

Inject a nonretained compound and obtain the retention time:

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

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

$\bar{F}$ is dependent on column temperature
Measuring flow with a flow meter is often inaccurate

Column Conditioning

System must be leak free before conditioning column

Heat the column to the lower of:

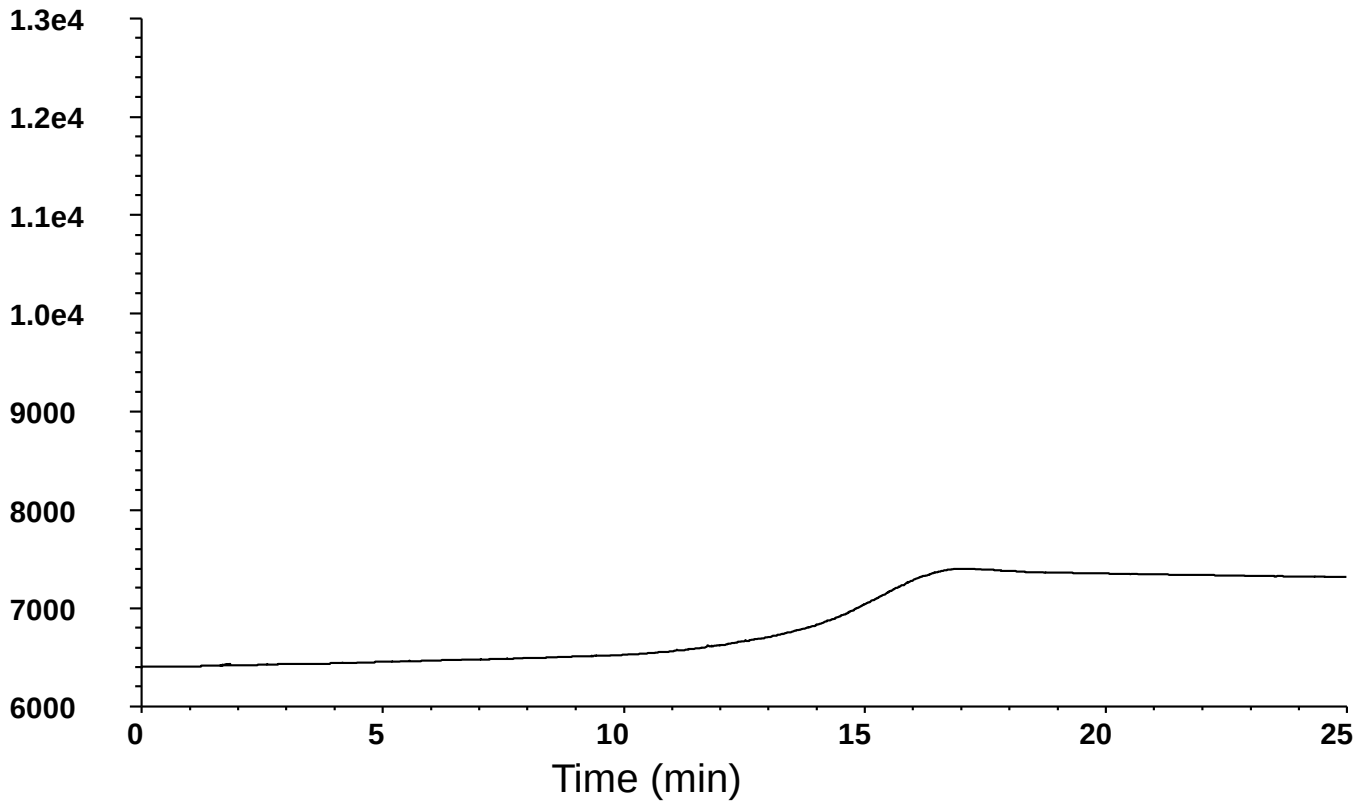
- Isothermal maximum temperature **or** 20 to 30 °C above highest operation temperature.
- Temperature programming is not necessary.

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
- Custom solutions
- Sample preparation materials, columns, supplies, instrumentation, and reference materials from a single source
- Rigorously tested and manufactured under ISO 9001, ISO 17025, and Guide 34 certifications

So you can calibrate with confidence and maximize accuracy

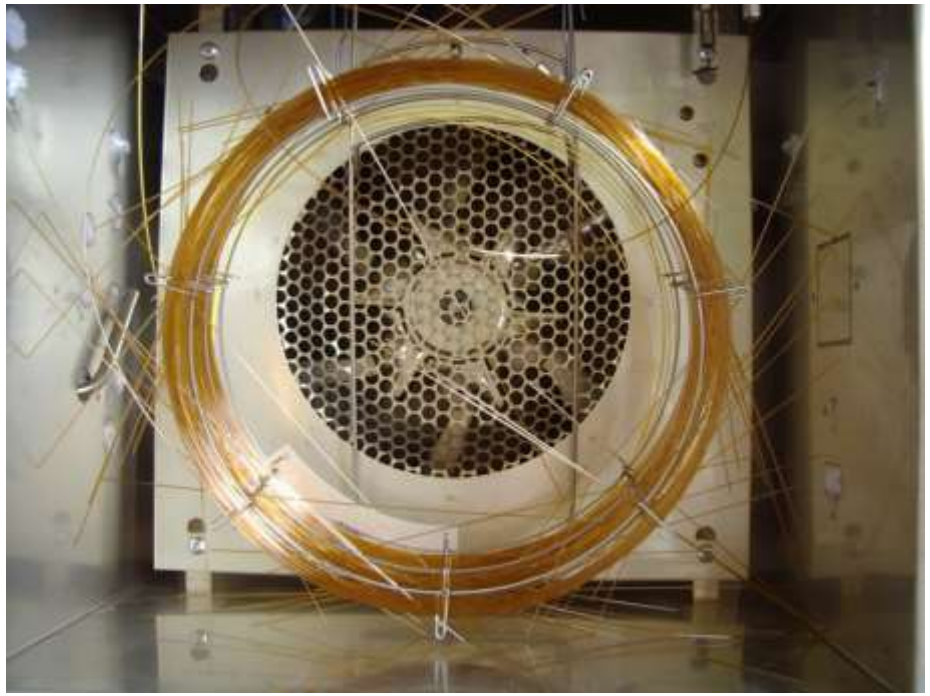
Proper Care of your Column

Common Causes of Column Performance Degradation

- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O_2 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

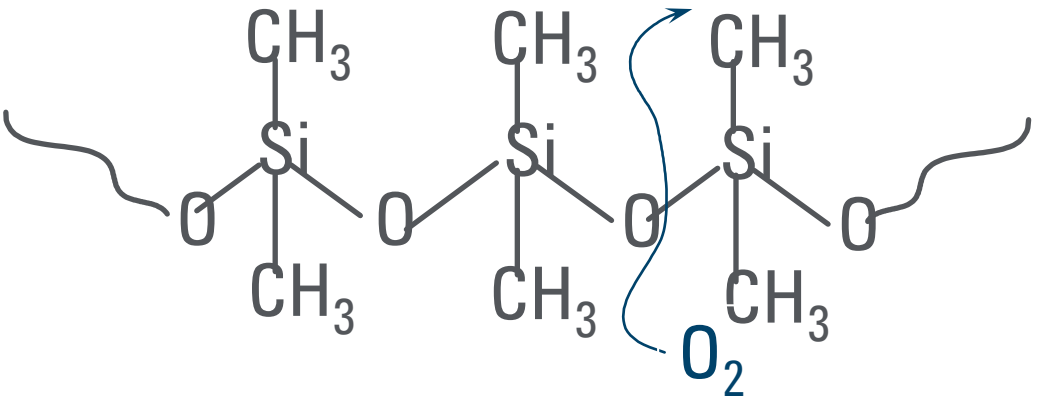
  Isothermal limit = indefinite time
  Programmed limit = 5-10 minutes

- 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

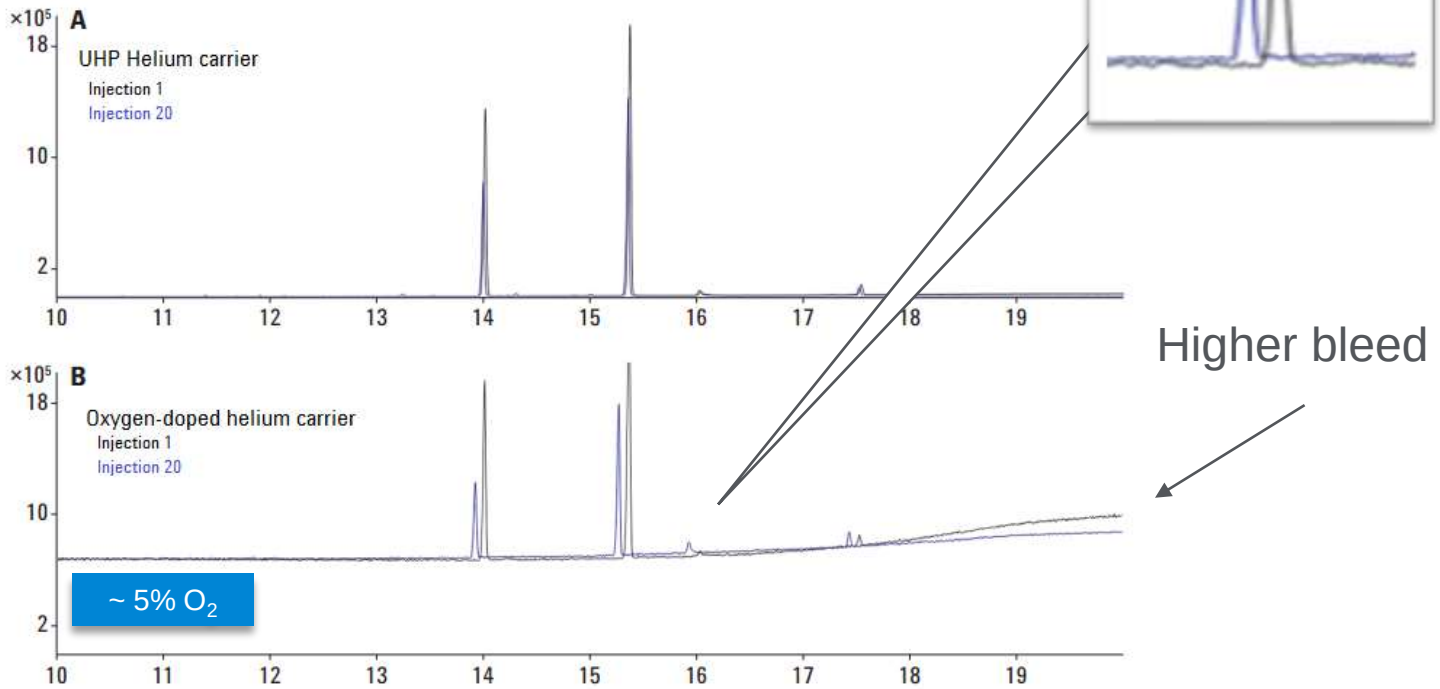
Column continuously exposed to temperatures above its temperature limit

Oxidation (O_2 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 9s or greater)
- Leak free injector and carrier lines
  - Change septa
  - Maintain gas regulator fittings
- Appropriate impurity traps

Efficient, fast, easy

Chemical Damage

Bonded and cross-linked columns have excellent chemical resistance except for inorganic acids and bases.

HCl 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

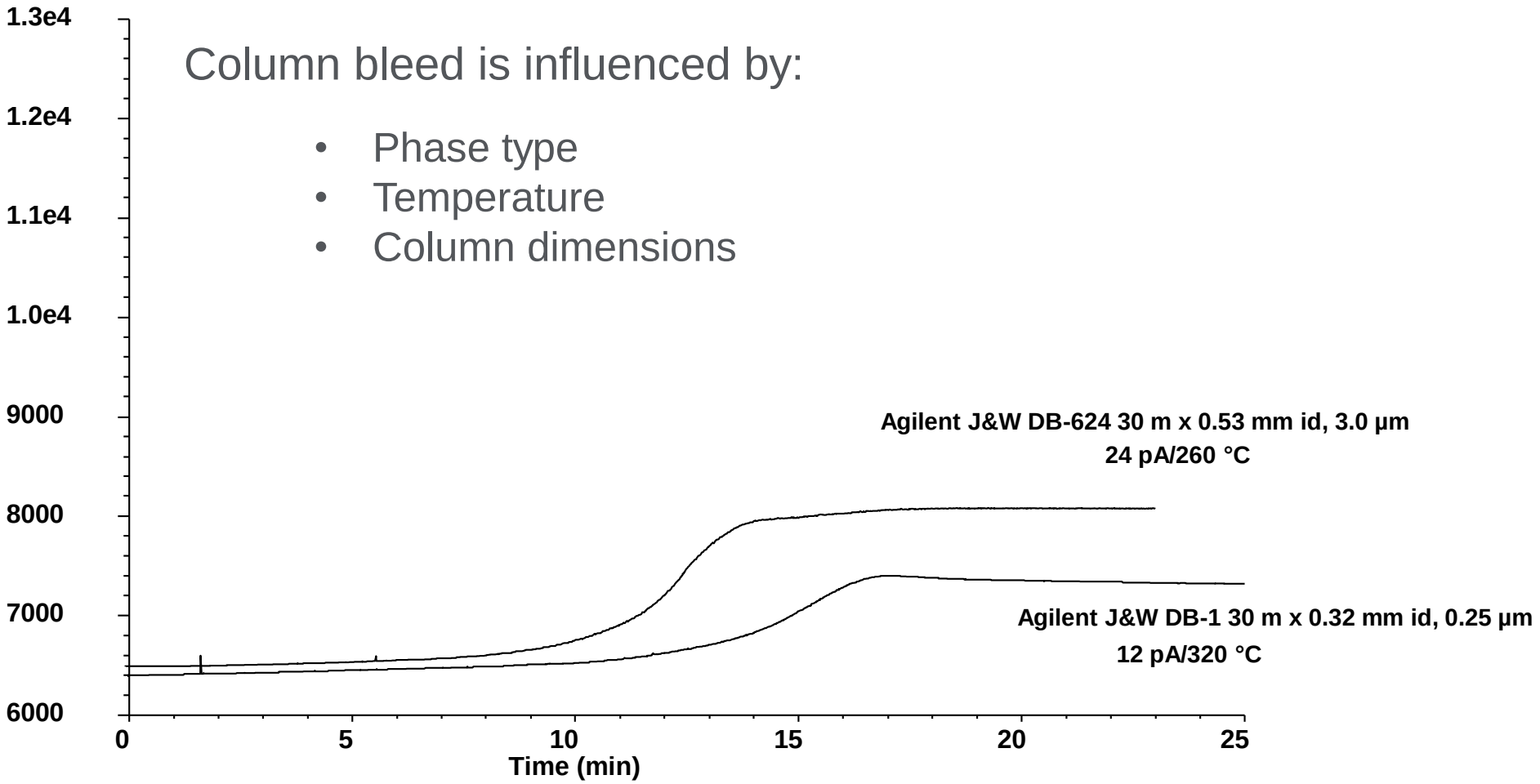
- Remove 0.5 to 1 m from the front of the columns
- Severe cases may require removal of up to 5 m

What is Normal Column Bleed?

Normal background signal generated by the elution of normal degradation products of the column stationary phase

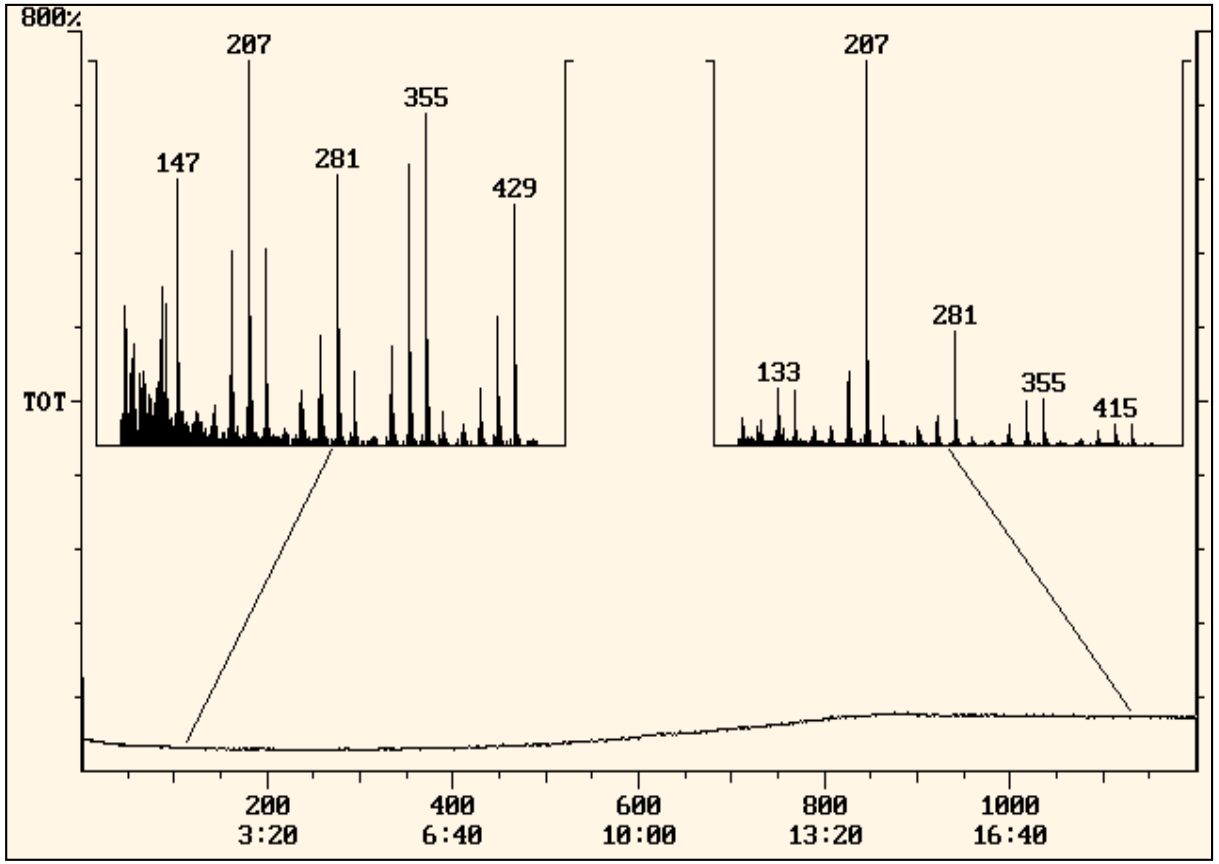
Column bleed is influenced by:

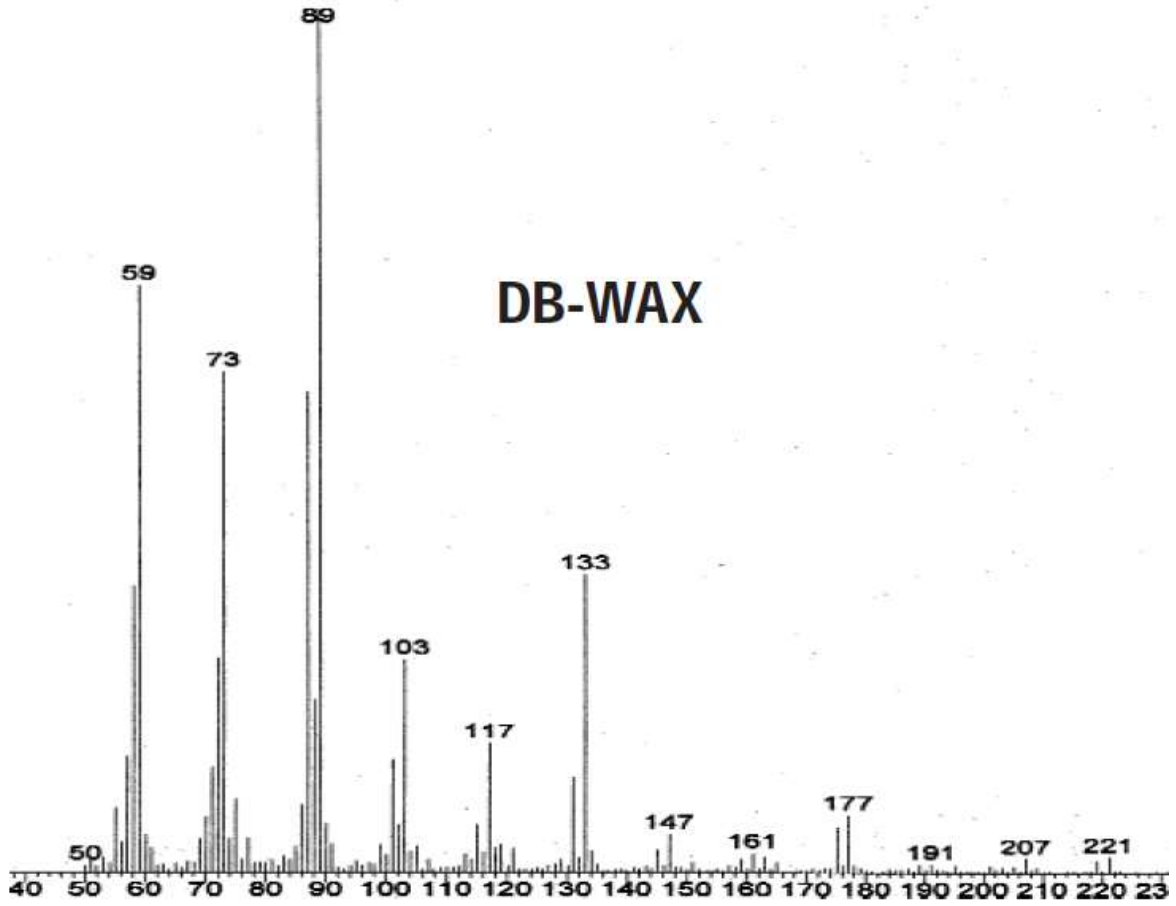
- Phase type
- Temperature
- Column dimensions



Mass Spectrum of Phenylmethylpolysiloxane Column Bleed

Normal background





Mass spectral library search is not always accurate

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 problems

- 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

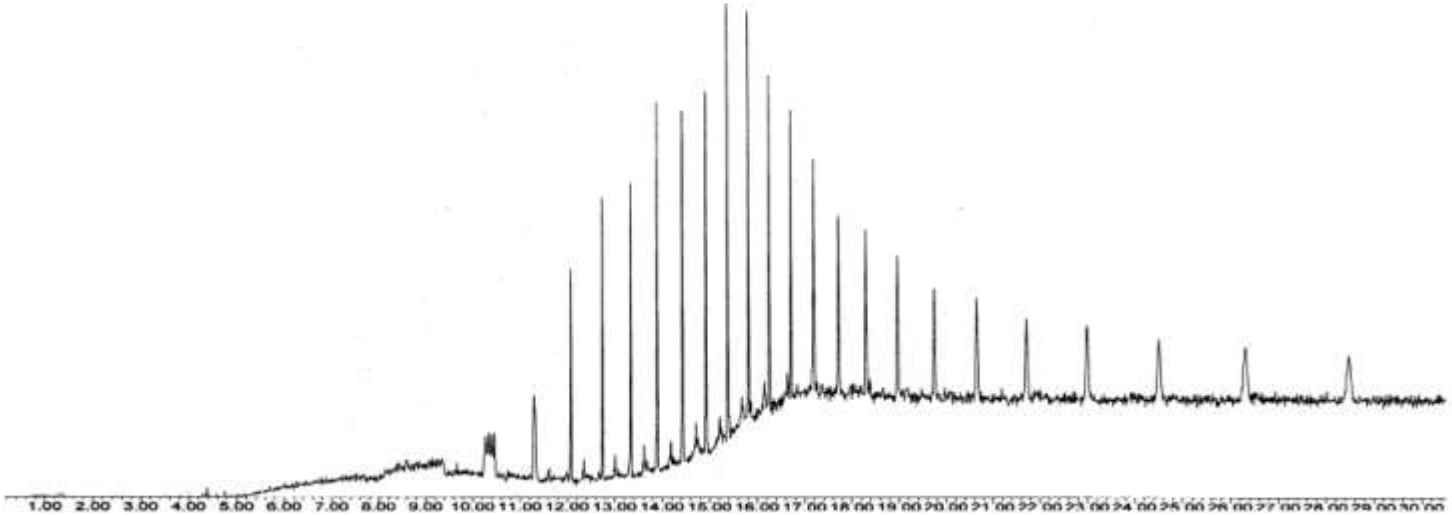
All samples contain residues (even standards)

Other Sources of Contamination

- Septum and ferrule particles
- Gas and trap impurities
- Unknown sources (vials, syringes, and so on)

Contaminated wash solvent

Sample Vial Septum Bleed Profile:



Types of Residues

Nonvolatile residues

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

Semivolatile residues

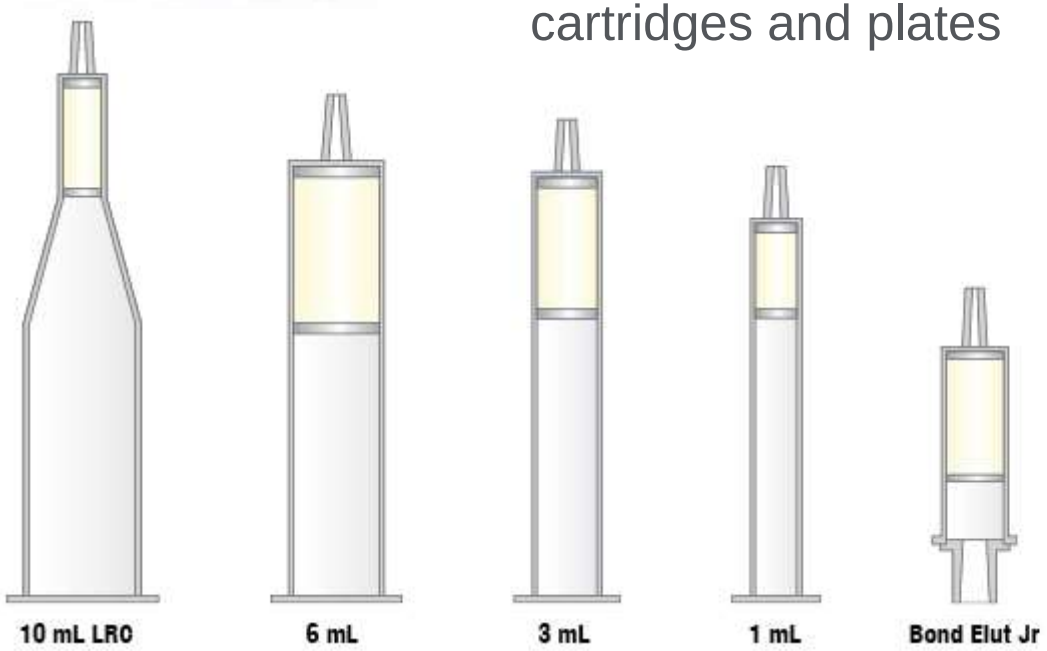
- Any portion of the sample that elutes from the column after the current chromatographic run.

Methods to Minimize Nonvolatile Residue Problems

- Sample cleanup
- Packed injection port liners
- Guard columns

Agilent Bond Elut Sample Cleanup Products

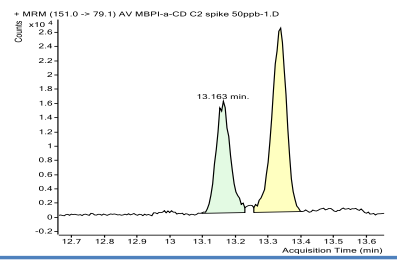
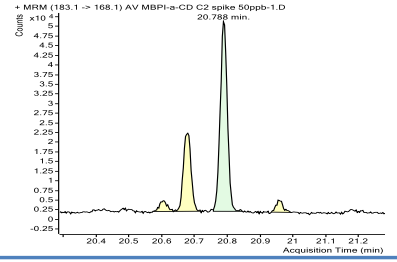
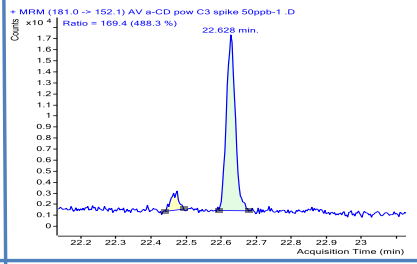
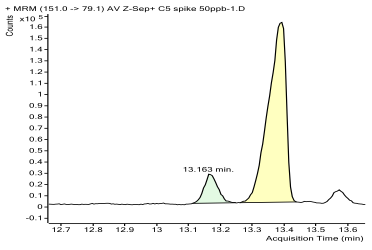
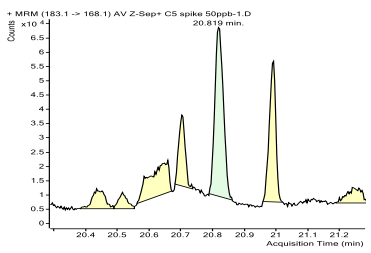
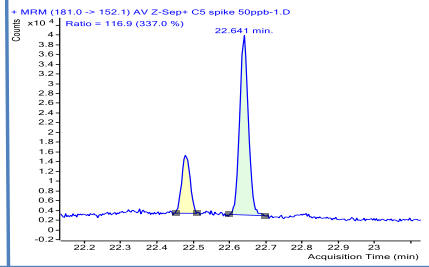
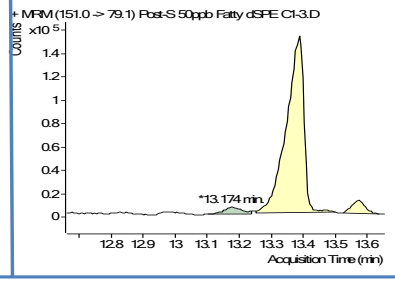
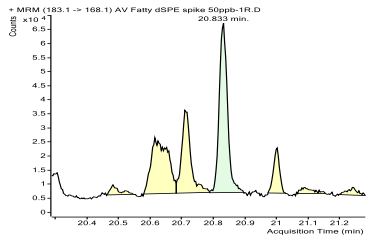
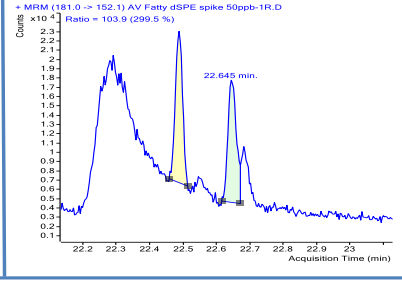
Solid Phase Extraction cartridges and plates



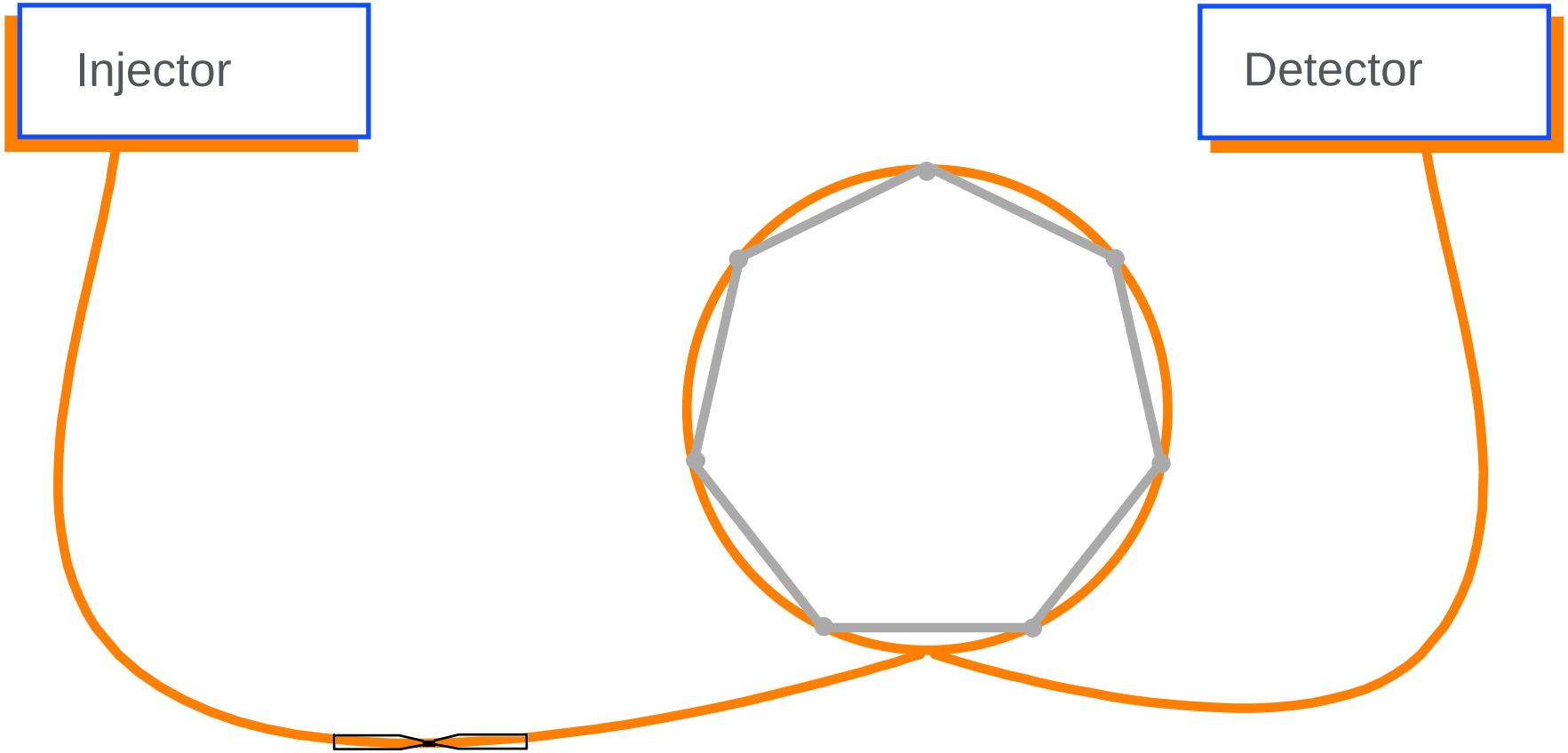
Filtration cartridges and plates

Captiva EMR Lipid

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

Pesticide	Captan	Permethrin	Deltamethrin
EMR-Lipid cleanup			
Zirconia sorbent cleanup			
C18/PSA cleanup			

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
- Solvent rinse the column (see appendix)
- 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

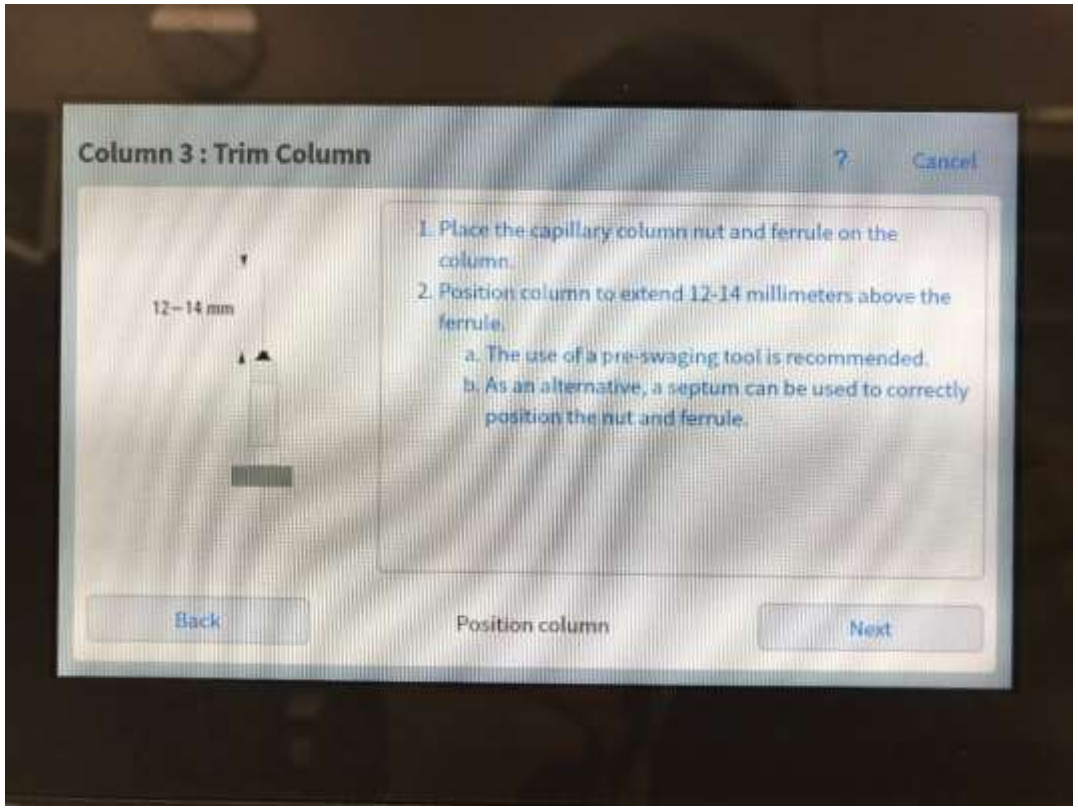
Smart-connected GC

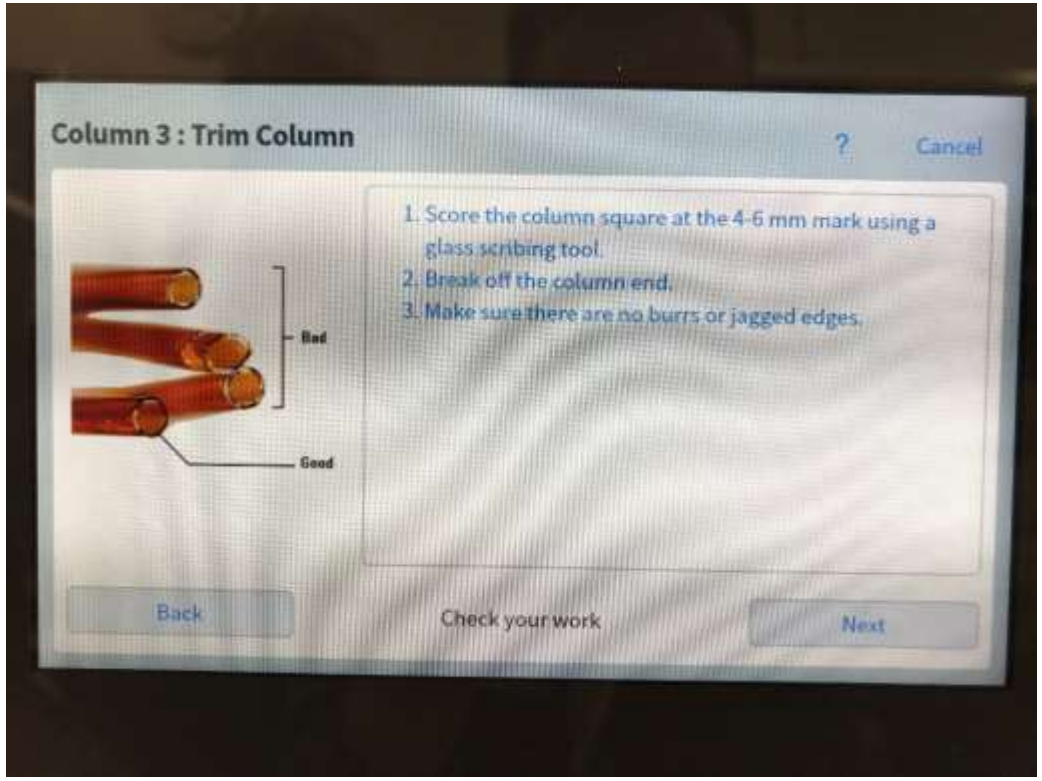
Modern intuitive interface

- 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)

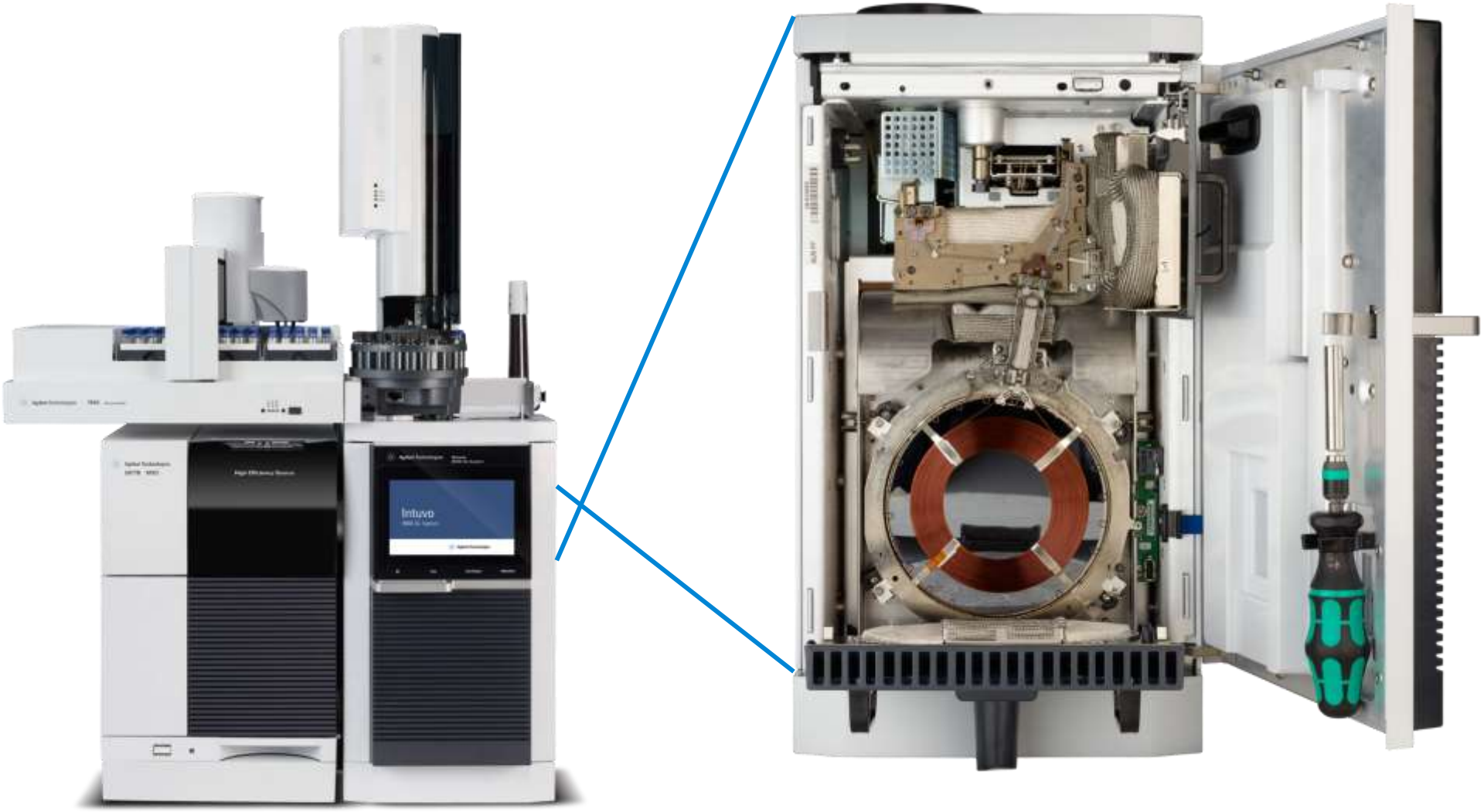
For immediate identification and use monitoring of your GC column

- 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:https://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:https://www.agilent.com/cs/library/instructionsheet/public/insert-smart%20key-8890-5994-0700en-agilent.pdf

Agilent Intuvo 9000 GC System



Common Frustrations with GC

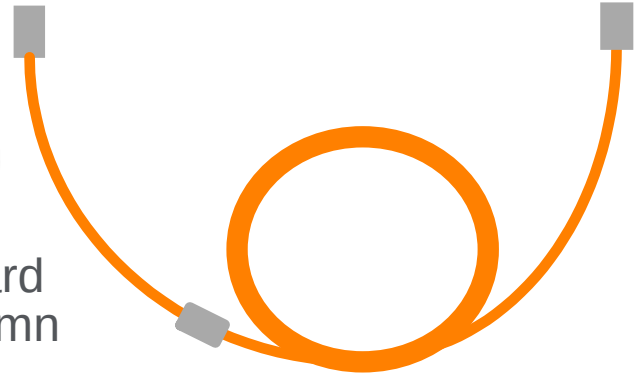
- Measuring column length correctly
- Cutting your column correctly
- How tight is too tight?
- Clipping columns to deal with active sites, then updating retention times

Common Care and Maintenance Scheme for GC Columns

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

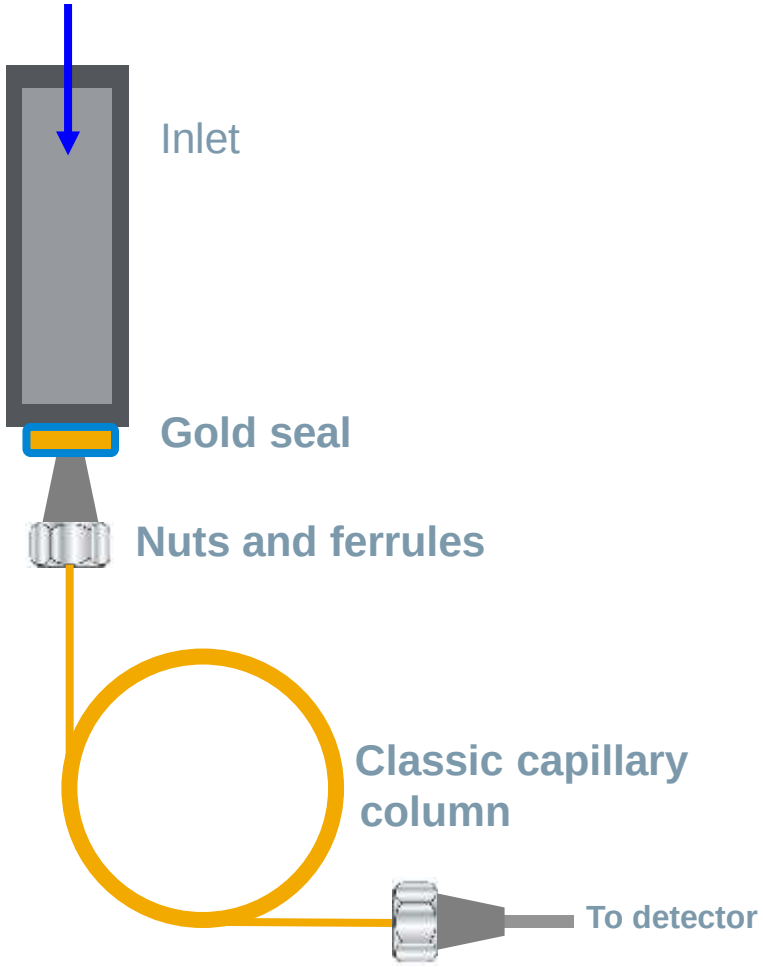
Intuvo…

Change the Guard Chip

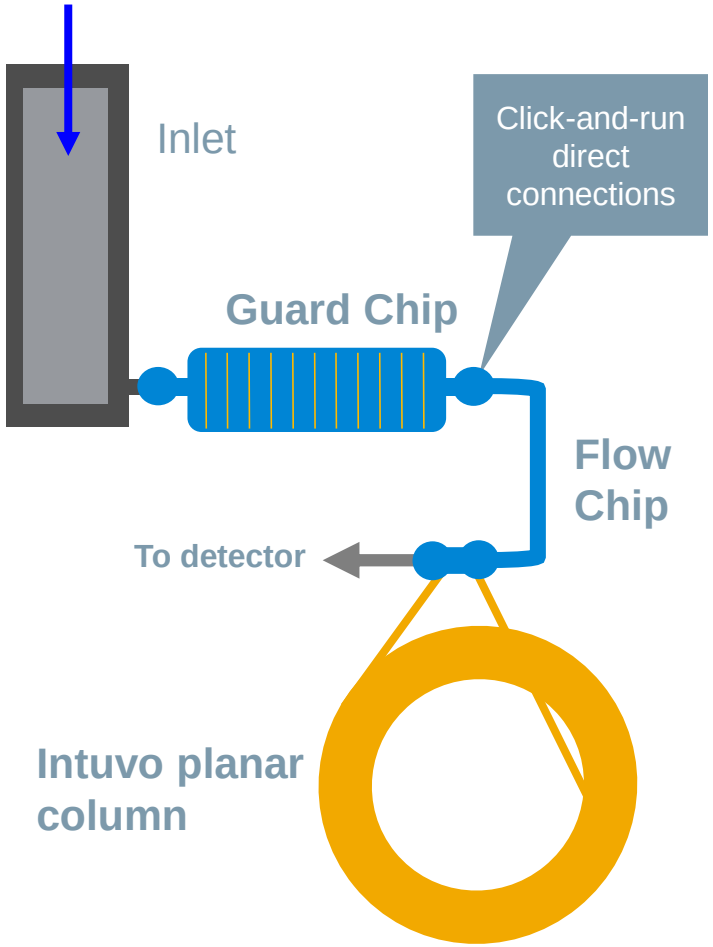


Innovating the GC Flow Path

Conventional flow path

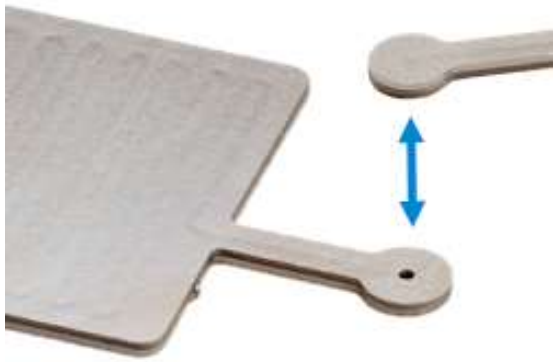


Intuvo flow path



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, fewer samples lost

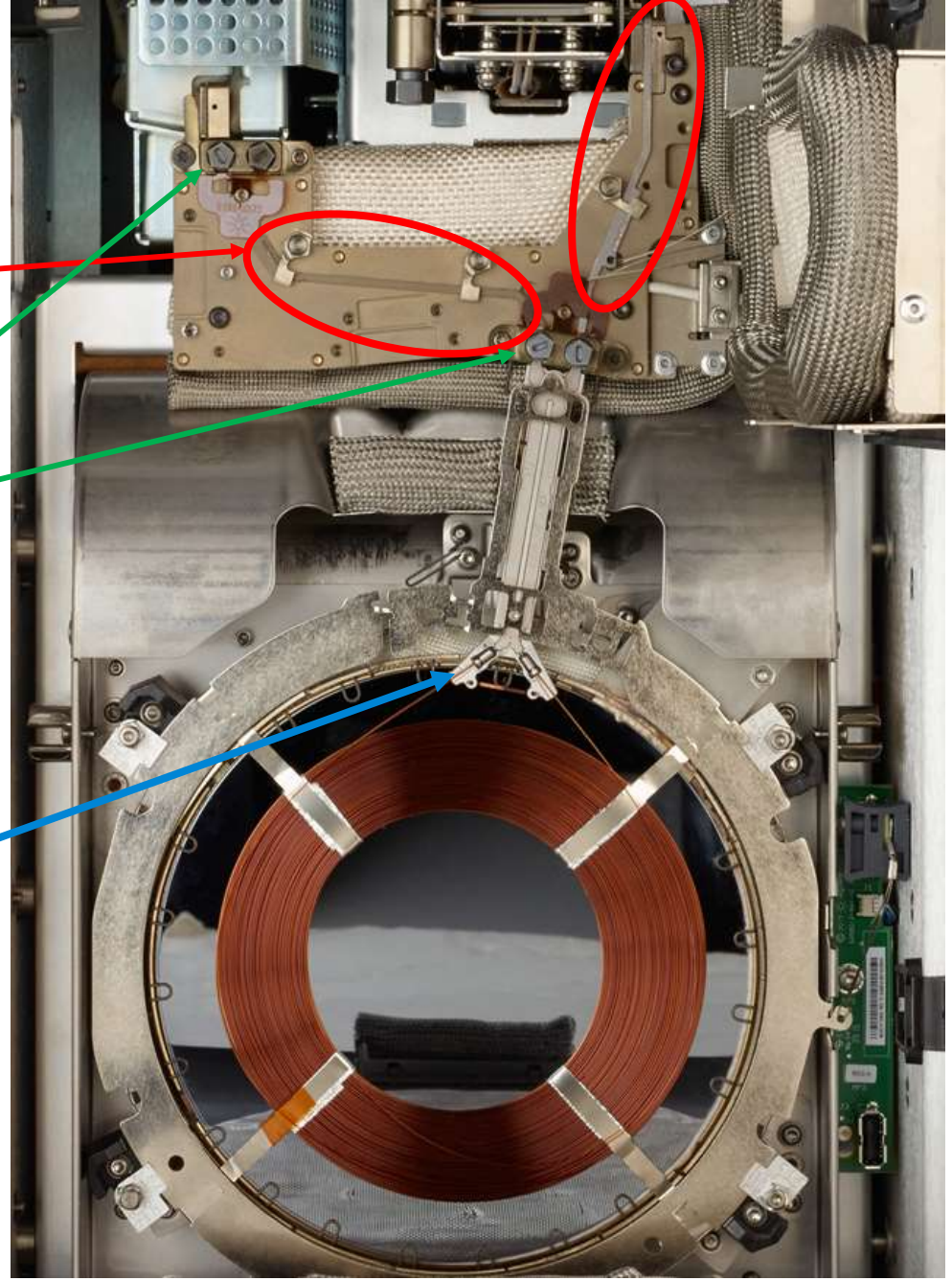


No More

Measuring

Over-tightening

Trimming



A New Portfolio of GC Consumables

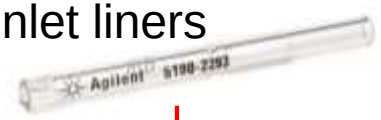
Smart Keys

Intuvo Flow Chips

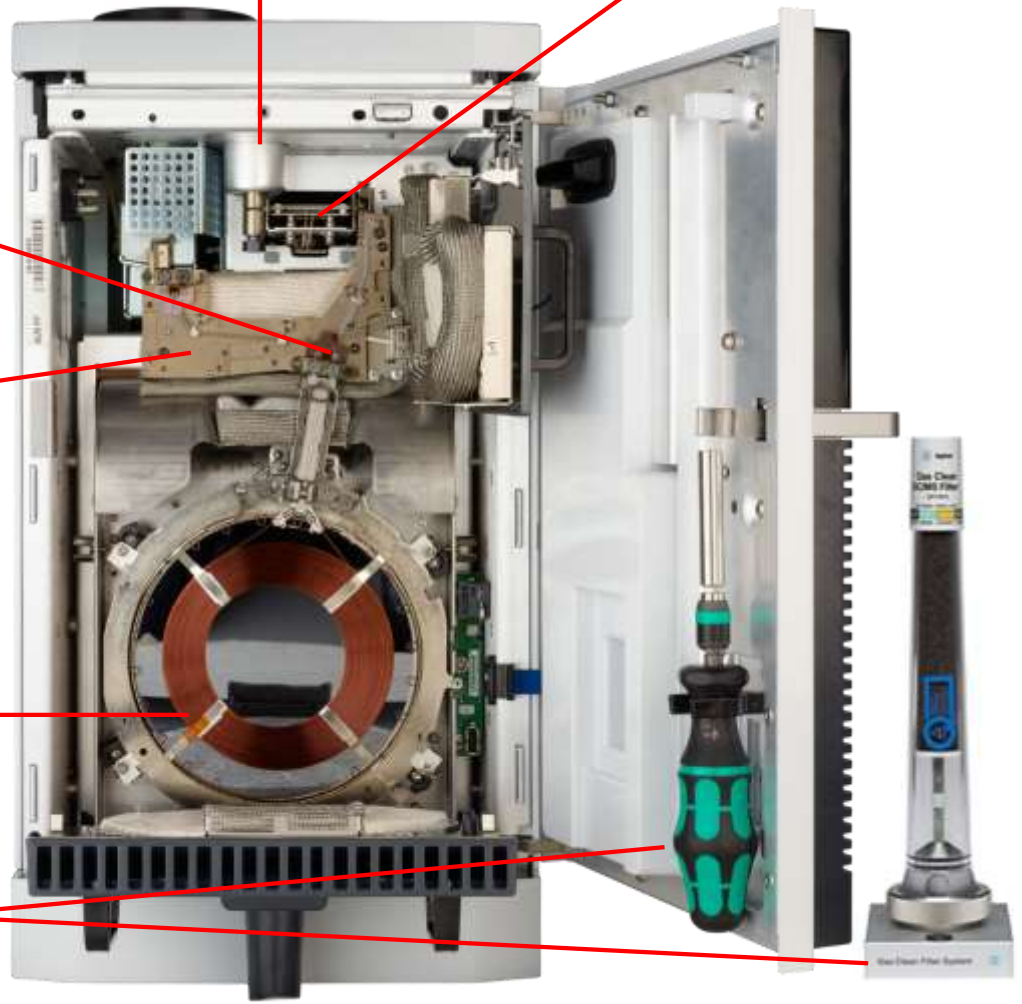
No-trim column

Tools and accessories

UI inlet liners



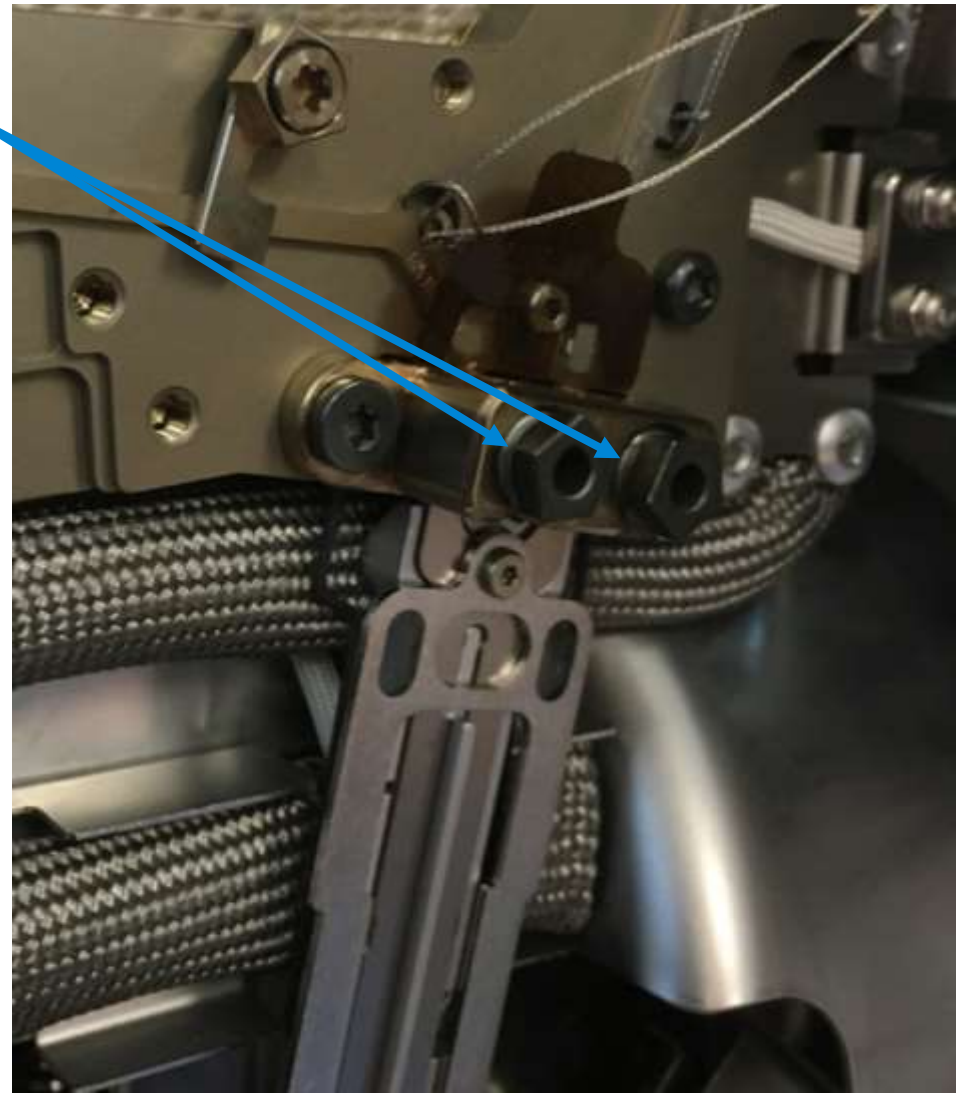
Guard Chip



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.



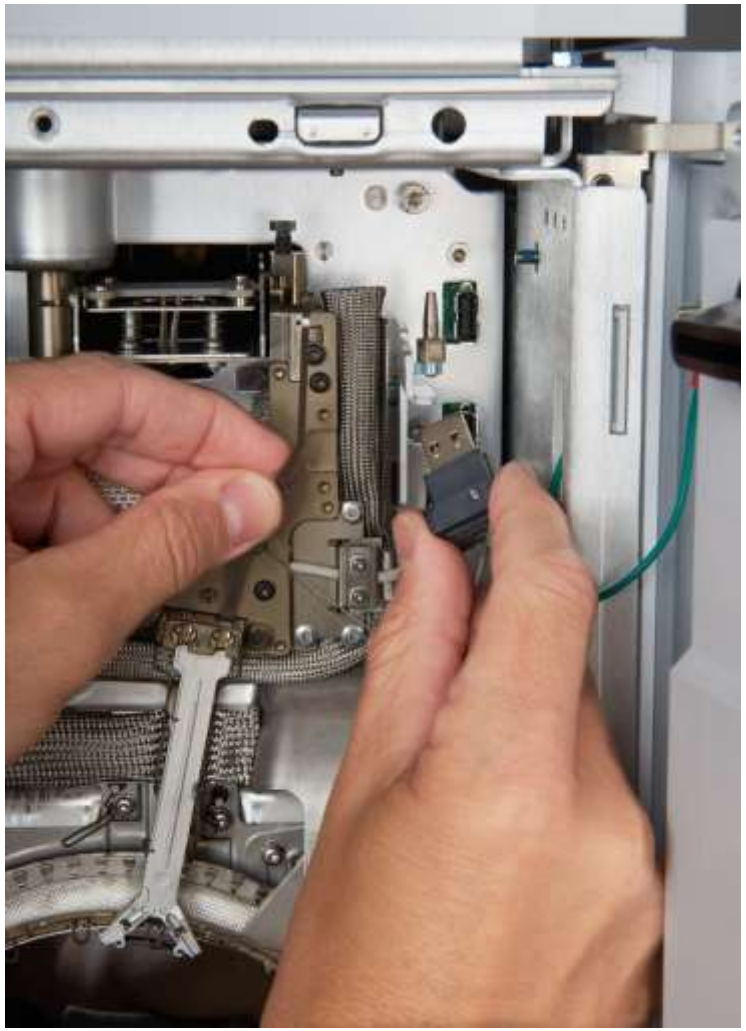
Tips to Assure a Good Column Installation

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
- Keep 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 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

lc-column-support@agilent.com

spp-support@agilent.com

spectro-supplies-support@agilent.com

chem-standards-support@agilent.com