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

Alexander Ucci and Mark Sinnott
Application Engineers
May 30, 2019
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.
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Column Performance Summary

Catalog: 19091S-433UI
Serial: [Barcode]
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

<table>
<thead>
<tr>
<th>Theoretical Plates/Meter:</th>
<th>n-DECANE</th>
<th>3208</th>
</tr>
</thead>
<tbody>
<tr>
<td>Retention Index:</td>
<td>n-PROPYLENEDIAMINE</td>
<td>953.110</td>
</tr>
<tr>
<td></td>
<td>1-HEPTANOL</td>
<td>967.660</td>
</tr>
<tr>
<td>Resolution:</td>
<td>1-OCTENE, n-OCTANE</td>
<td>2.97</td>
</tr>
</tbody>
</table>

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

Test Conditions

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

May 31, 2019
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Chromatographic Performance

Spec: 4.0 pA
Meas: 2.1 pA

320 °C
65 °C
## Test Mixture Components

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrocarbons</td>
<td>Efficiency</td>
</tr>
<tr>
<td></td>
<td>Retention</td>
</tr>
<tr>
<td>FAMEs, PAHs</td>
<td>Retention</td>
</tr>
<tr>
<td>Alcohols</td>
<td>Activity</td>
</tr>
<tr>
<td>Acids</td>
<td>Acidic character</td>
</tr>
<tr>
<td>Bases</td>
<td>Basic character</td>
</tr>
</tbody>
</table>
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

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?

<table>
<thead>
<tr>
<th>Composition</th>
<th>Re-use</th>
<th>Max Temperature (°C)</th>
<th>Use</th>
<th>Limitation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyimide (Vespel)</td>
<td>Yes</td>
<td>280</td>
<td>Easy seal</td>
<td>Shrink after heating causing leaks after thermal cycle; isothermal only</td>
</tr>
<tr>
<td>Graphite</td>
<td>Yes</td>
<td>450</td>
<td>FID, NPD, inlets</td>
<td>Contamination, permeable to air – not for oxygen sensitive detectors</td>
</tr>
<tr>
<td>Polyimide/graphite</td>
<td>Limited</td>
<td>350</td>
<td>MS, ECD, inlets</td>
<td>Still shrink after thermal cycles creating leaks; need to retighten regularly</td>
</tr>
<tr>
<td>Flexible Metal</td>
<td>No</td>
<td>450</td>
<td>Capillary flow technology (backflush, splitters, and so on)</td>
<td>May not seal well with damaged fittings or rough surfaces</td>
</tr>
</tbody>
</table>

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

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

Composition | Re-use | Max Temperature (°C) | Use                      | Limitation                                                                 |
<table>
<thead>
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<th></th>
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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.

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

For inlet or detector p/n 5190-5233

For mass spectrometer transfer line p/n 5190-6194
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

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
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*

<table>
<thead>
<tr>
<th>Detector</th>
<th>Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>FID</td>
<td>Methane or butane</td>
</tr>
<tr>
<td>ECD</td>
<td>MeCl$_2$ (headspace or diluted)</td>
</tr>
<tr>
<td>NPD</td>
<td>CH$_3$CN-acetonitrile (headspace or diluted)</td>
</tr>
<tr>
<td>TCD</td>
<td>Air</td>
</tr>
<tr>
<td>MS</td>
<td>Air or butane</td>
</tr>
</tbody>
</table>

The peak should be sharp and symmetrical
Nonretained Peak Shapes

Check for:

- Too low of a split ratio
- Injector or septum leak
- Liner problem (broken, leaking, misplaced)
- Column position in injector and detector
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\textsubscript{2}  35-55 cm/s

\( \mu \) is \textit{dependent} on column temperature, but is \textit{independent} of column dimensions.
Calculating Flow Rate

Inject a nonretained compound and obtain the retention time:

\[ F = \frac{\pi r^2 L}{t_o} \]

- \( F \) = 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
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 (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

Decreased retention

Reduced response

Higher bleed

~ 5% O₂
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
Chemical Damage

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

HCl  NH$_3$  KOH  NaOH
H$_2$SO$_4$  H$_3$PO$_4$  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
- Wastewater
- Sludges
- Foods

*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)

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
Agilent Bond Elut Sample Cleanup Products

Solid Phase Extraction cartridges and plates

Filtration cartridges and plates

Captiva EMR Lipid

10 mL LRO
6 mL
3 mL
1 mL
Bond Elut Jr
Captiva EMR–Lipid Cleanup Improves Analytes S/N Ratio and Integration Accuracy on GC/MS(/MS) of Pesticides in Olive Oil

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Captan</th>
<th>Permethrin</th>
<th>Deltamethrin</th>
</tr>
</thead>
<tbody>
<tr>
<td>EMR-Lipid cleanup</td>
<td><img src="image1.png" alt="Graph" /></td>
<td><img src="image2.png" alt="Graph" /></td>
<td><img src="image3.png" alt="Graph" /></td>
</tr>
<tr>
<td>Zirconia sorbent cleanup</td>
<td><img src="image4.png" alt="Graph" /></td>
<td><img src="image5.png" alt="Graph" /></td>
<td><img src="image6.png" alt="Graph" /></td>
</tr>
<tr>
<td>C18/PSA cleanup</td>
<td><img src="image7.png" alt="Graph" /></td>
<td><img src="image8.png" alt="Graph" /></td>
<td><img src="image9.png" alt="Graph" /></td>
</tr>
</tbody>
</table>

Captan Permethrin
EMR-Lipid cleanup
Zirconia sorbent cleanup
C18/PSA cleanup
Deltamethrin
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
<table>
<thead>
<tr>
<th>Resources</th>
<th>Weblinks</th>
</tr>
</thead>
<tbody>
<tr>
<td>2    Smart Key product page (not for ordering Smart Keys)</td>
<td>URL: <a href="http://www.Agilent.com/chem/smartkey8890">www.Agilent.com/chem/smartkey8890</a></td>
</tr>
</tbody>
</table>
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

- Inlet
- Gold seal
- Nuts and ferrules
- Classic capillary column
- To detector

Intuvo flow path

- Inlet
- Guard Chip
- Click-and-run direct connections
- Flow Chip
- To detector
- Intuvo planar column
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

- UI inlet liners
- Guard Chip
- Smart Keys
- Intuvo Flow Chips
- No-trim column
- Tools and accessories
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