Contamination, Peak Shape, and Retention Time Problems
Plan Ahead for a Smooth Trip

“What Phileas Fogg had won his wager, and made his journey around the world in eighty days. To do this he had employed every means of conveyance – steamers, railways, carriages, yachts, trading-vessels, sledges, elephants.”

Jules Verne, Around the World in Eighty Days

• What can you do to reduce or anticipate potential chromatography problems?
  - Instrument
  - Sample prep
  - Column
Hidden Hazards of GC

-Bad Gas
-Inlet Contamination
-Leaks
-Dirty Samples
-Wrong Column Choice
Bad Gas Stinks!

- Use ultra-high purity carrier gas (99.9995% or greater)
- Use the appropriate gas traps
- Oxygen in carrier gas is detrimental to GC, resulting in:
  - Reduced response
  - Elevated background
  - Irreversible column damage
  - Premature filament failure
  - Excessive source maintenance
- Agilent has a wide range of gas filters
  - GasClean oxygen and moisture filters have indicators
    - Replace when needed
    - Correct any leaks present

- Decreased retention
- Reduced response

---

**Agilent GC/MS filter**

Agilent P/N CP17973

~ 5% $O_2$

*Decreased retention*

*Reduced response*
Other Gas Traps Available

Renewable Gas Purification System

Please contact us at GC-column-support@agilent.com for assistance with setting up gas filters!

In-Line Large Traps

Refillable Moisture Traps
Choosing the Right Syringe

**Fixed Needle Syringes (shown)**
- Typically abbreviated FN
- Needle “cemented” to barrel using epoxy
- Typically used in autosamplers
- Preferred for applications requiring trace level samples
- Recommended for use where probability of needle bending is minimal
- Can be heated up to 70°C

**Removable Needle Syringes**
- Typically abbreviated RN
- Allows use of various needle point styles
- Threaded connection with PTFE sealing ferrule that can be tightened to compensate for wear
- Can be heated up to 120°C
- Can be prone to leakage
- Recommended for chlorinated solvents

**Standard plungers**
- Fit tightly within syringe barrel
- Limit loss of volatile sample
- Individually fitted to the syringe
- Not replaceable/Not interchangeable
- Recommended for analysis of liquid samples

**PTFE-tipped (shown)**
- Limit sample deposit adsorption
- **Forms gas-tight seal**
- Replaceable
- Requires maintenance to maintain PTFE seal
- Recommended for:
  - “Dirty” samples
  - Highly volatile samples
  - Gas injections
  - Chlorinated solvents

**Cone Tip/PS AS (shown)**
Used in Agilent autosamplers for optimum performance and reliability by reducing septum coring.

**Bevel Tip/PS 2**
General purpose, excellent choice for transferring liquids from ampoules or vials. For manual GC injections, a bevel tip is preferred for optimum septum penetration with minimal coring.

**Side Hole Tip/PS 5**
Recommended for thin gauged septa and large volume- or gas injections.
Vials

• Choose high quality vials and caps
• Poorly constructed vial septa → siloxanes → bleed peaks
• Low quality vial → leach contaminants into sample
• Choose the right cap/septa for your solvent

<table>
<thead>
<tr>
<th>High performance septa</th>
<th>Thin PTFE</th>
<th>PTFE/Silicone*</th>
<th>PTFE/Silicone/PTFE*</th>
<th>PTFE/Red rubber</th>
<th>Fluoroelastomer</th>
<th>Butyl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range</td>
<td>40 °C to 300 °C</td>
<td>Up to 260 °C</td>
<td>-40 °C to 200 °C</td>
<td>-40 °C to 200 °C</td>
<td>-40 °C to 90 °C</td>
<td>-40 °C to 260 °C</td>
</tr>
<tr>
<td>Use for multiple injections</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>Price</td>
<td>More expensive</td>
<td>Very economical</td>
<td>Economical</td>
<td>Most expensive</td>
<td>Very economical</td>
<td>Economical</td>
</tr>
<tr>
<td>Resistance to coring</td>
<td>Excellent</td>
<td>None</td>
<td>Excellent</td>
<td>Yes</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>Recommended for storage</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>Best for</td>
<td>High temperature headspace applications</td>
<td>Superior chemical inertness, short cycle times, and single injections</td>
<td>Most common HPLC and GC analyses, not as resistant to coring as P/S/P</td>
<td>Superior performance for ultra trace analysis, repeat injections, and internal standards</td>
<td>Chlorosilanes, more economical option for single injections</td>
<td>Chlorinated solvents, higher temperatures</td>
</tr>
</tbody>
</table>

* Agilent silicone is platinum cured (versus peroxide cured), making it more inert and less likely to interact with samples.
** For up to 1 hour.
Sample Clean-Up
Filtration, Solid Phase Extraction, QUECHERS, and more!
Why perform Sample Clean-Up?

- To acquire desired sensitivity/selectivity
- To reduce contamination/carryover issues
- Use of sensitive and expensive instruments: *Protect your investment!!!*

Pesticides in Avocado *without* SP

Pesticides in Avocado *with* SP

Curtain plate after injection of 25 samples with extractions from raisins without cleanup
Challenge: Instrument Contamination

GC System Component Contamination

GC Inlet Liner

GC Inlet Seal

“I’m emailing in regards to the QuEChERS kits my lab has recently begun using. We thought we’d send along a couple pictures of the difference they have made in our biological sample clean-up process. This is a comparison of the GC inlet liner after a run of approximately 50 samples with and without using the kits. Enjoy! Those samples were extracted from adipose tissue, for reference.”
Sample Clean-up Tools to Help you on your Journey

Solid Phase Extraction Cartridges and Plates

QUECHERS

Filtration Cartridges and Plates

Syringe Filters

Captiva EMR Lipid
Captiva EMR-Lipid

- One of Agilent’s newest products with a 2 in 1 benefit of removing proteins and lipids
- Simple pass-through format
- Solvent-retention frit in 1 mL cartridge/96-well plate format for in well protein precipitation (in situ)
  - Unique cartridge/well construction minimizes clogging – and **ensures protein and lipid removal** (no cloudy samples)
- 3 and 6 mL cartridge format for larger samples
  - Do not contain solvent retain frit which allow for gravity flow
  - Protein precipitation performed offline (QUECHERS, etc.)
- Unique cartridge/well construction minimizes clogging – and **ensures protein and lipid removal** (no cloudy samples)
- High analyte recoveries
- Effective use will reduce ion suppression, increase analyte sensitivity, and detection, and extend the lifetime of your analytical column
Enhanced Matrix Removal: **EMR-Lipid**

**EMR-Lipid sorbent technology effectively traps lipids through two mechanisms:**

- **Size exclusion** – Unbranched hydrocarbon chains (lipids) enter the sorbent; bulky analytes do not

- **Sorbent chemistry** – Lipid chains that enter the sorbent are trapped by hydrophobic interactions

**EMR-Lipid = Finger Trap**

Finger = carbon chain of lipids
Effective phospholipid removal

$m/z$ 184 precursor ion scan profile

- Protein precipitation only
- In-well protein precipitation with Captiva EMR—Lipid
Protein Precipitation vs. Captiva EMR-Lipid RSD and Peak Area

Lipids cause reproducibility problems resulting in high RSD values
Using Captiva EMR-Lipid $\rightarrow$ low RSD values and higher peak areas
Higher peak area due to less ion suppression $\rightarrow$ can lead to lower detection limits
Screening of pesticide residues in fruit and vegetables

- Developed to make sample cleanup of food faster, simpler, less expensive, and greener

Now used with other matrices and compound classes as well

Commercially available kits allow for ease of use and convenience leading to increased throughput

Consists of two steps, and thus 2 kits:

Step 1: Liquid Extraction

Step 2: Dispersive SPE / Interference Removal
What are the Benefits of QuEChERS?

- QuEChERS Approach: **Extract +250 compounds at one time**
- QuEChERS methodology is non-selective technique, **does not remove all the matrix**
- Final extract amenable to GC/MS or LC/MS
- **Reduced solvent and labor, increased lab productivity**

**QuEChERS Approach Advantages**

- ~30 minutes to extract multiple samples at once
- Minimal solvent usage per sample: 10-15 mL
- Chlorinated Solvents: None

*If you can weigh, pipette, shake and your lab has a centrifuge, you can perform QuEChERS*
Productivity Benefits with Sample Preparation

More Matrix Removal = Less Matrix Entering System = Time and Cost Savings!

✓ Less matrix build-up
  – Less interferences
  – Improved S/N
  – Better reproducibility

✓ Better chromatography
  – Less time spent on data analysis/manual integration
  – Less time spent on re-runs/recalibrations

✓ Less maintenance
  – Less instrument down-time
  – Saves $$ on consumables/services

✓ Less troubleshooting
  – “Is it my column or my MS”? 
  – Less instrument down-time
Inlet and Supplies
Inlet

• Injection Efficiency:
  • Main function of the inlet is to produce a narrow sample band at the head of the column
  • One of the most important aspects to any high resolution GC method

• Must be reproducible

• The liner volume must be large enough to accommodate the solvent’s phase transformation into a vapor (Back-Flash)

• The vast majority of chromatography problems are “front-end” related

• Many consumables to replace: septa, liner, gold seal

• Inlet body must be cleaned/solvent rinsed periodically (**No steel brushes please!**)
Septa

• Typical cost of 1 Premium Septum (list), $1.25
• Typical cost of 1 GC Column, 30 m x 0.25 mm ID, $450.
• “Don’t step over a dollar to pick up a dime!”
• Proactively change inlet septa.
• Agilent’s packing eliminates contamination of septa
• “centerguide septa” puts less train on syringe compared to solid septa
• Do not overtighten septum nut- septum can begin to “bulge” out
• Should tighten nut until c-clamp on top stops turning, then ½ to ¾ turn more

Removed during PM
What is glass wool used for?

**Filtration**
- Prevents nonvolatile matrix from entering column

**Vaporization**
- Provides volatilization surface for liquid injections, promotes mixing with carrier gas

**Needle wiping**
- Increases reproducibility by wiping needle after injection

Does liner diameter have an effect?

**Inner diameter**
- Small inner diameter for gas analysis
- Larger inner diameter for liquid analysis

**Outer diameter**
- Large od ideal for splitless injections
- Slower transfer, snug fit directs flow within liner

Could you clarify the effect of top taper on sample backflash?
Split liners:

**Split/splitless liner with glass wool, low pressure drop**

Split injections have higher carrier gas flow through liner to help split sample
- Faster transfer onto column
- Split liners have a smaller outer diameter than splitless liners to help flow circulate

If potential exists for sample discrimination between low and high boiling components
- Use a liner with wool

**Ultra Inert** liners enable excellent peak shapes for tricky analytes
- 5190-2295 is recommended liner- Single taper, low pressure drop

**Glass wool** plug in upper position wipes needle, avoids sample discrimination improves P&A, and collects septum particulate

**Low pressure drop** bead promotes better carrier gas flow for split injections

Touchless packaging ensures contamination-free installation

Preinstalled o-ring
Splitless liners

Single Taper with or without wool

Splitless has lower flows through liner
- Splitless liners are typically wider for a more snug fit  
  - Ensures all available flow funnels through the liner, not around
- Do NOT do split injections on a splitless liner  
  - Poor reproducibility, not enough room for flow

**Ultra Inert** liners enable excellent peak shapes for tricky analytes
- 5190-2293 is recommended splitless liner- Single taper, with wool

In low carrier gas flow splitless analysis, a **bottom taper** helps focus analytes onto head of column

Small plug of **glass wool** near bottom of liner filters matrix
Ultra Inert Inlet Liners:

1. Ultra Inert deactivated inlet liners provide higher response for sensitive compounds
2. Ultra Inert **Glass wool liners** deliver benefits of glass wool w/o loss of active compounds
3. QC tested & certified for consistent performance

**Productivity:**
Touchless packaging with pre-installed o-ring: quick & easy hassle free installation
Liner Selection

• Liner is where sample is volatilized so selection is important

• Liner Variables
  • Liner volume
  • Liner treatments or deactivation
  • Special characteristics (glass wool, taper, etc.)

• When choosing a liner for your application, consider all three aspects to give you the best chromatography
  • what type of inlet is your GC?
  • What is the application?
  • Injection technique (split, splitless, etc.)?

• You may need to experiment with several liner types to find the best one for your method.
Agilent UI Gold Seal: Deactivated gold surface

- Soft gold plating is essential for proper sealing
- Ultra Inert chemistry blocks active sites (gold is NOT inert)
- Smooth surface doesn't leak (Injected molded)
- Advantage Agilent

Reliable ppb and ppt measurements require attention to the little things!
Split/Splitless Inlet: The Split Vent Trap

What is it???

Where is it???
On a 7890 GC
Anatomy of a Capillary GC Column

Polyimide

Fused Silica

Stationary Phase polymer
Column Installation & Tools

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, etc.

5181-8836 (4/pk)
Column Installation: Good Cuts and Clean Hands

Examples of Column Cuts-The GOOD the BAD and the UGLY!

Don’t overtighten!!!
Contamination from Liquid Soap

Column: DB-5ms, 30m x 0.25mm, 0.25um
Carrier: H2, 60 cm/sec, constant flow
Injector: split 1:20, 250C
Detector: FID, 320C, N2 makeup gas
Oven: 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

Red: liquid soap
Blue: system blank

Procedure:
(1) One very 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 40C.
(6) Started oven temperature program as soon as oven reached 40C.
## Ferrules

<table>
<thead>
<tr>
<th>Composition</th>
<th>Max T</th>
<th>Use</th>
<th>Limitation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vespel</td>
<td>280</td>
<td>Easy seal</td>
<td>Leaks after T cycle, iso only</td>
</tr>
<tr>
<td>Vespel/graphite</td>
<td>350</td>
<td>MS</td>
<td>Retighten after T cycle</td>
</tr>
<tr>
<td>Graphite</td>
<td>450</td>
<td>Not MS</td>
<td>Contamination, leakages</td>
</tr>
<tr>
<td>Ultimetal Plus Flexible Metal Ferrules</td>
<td>450</td>
<td>MS, CFT</td>
<td>Overtightening can damage fitting</td>
</tr>
</tbody>
</table>

“Short” ferrules for detector and inlet configurations on Agilent GC’s, provide a robust seal.

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

**Dial packaging**
Graphite / Polyimide blend capillary ferrules

Unfortunately … leak following normal temperature program runs

Studies show the leaking continues with use of the ferrules - *Not* just after the first one or two runs

Frequent re-tightening of the fitting is needed to maintain a leak-free seal – and 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 only one type of ferrule needed for both ends of the column!

Short ferrule exposes more thread of the fitting for better sealing

For inlet or detector
P/N 5190-5233

For mass spec 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. Tech note: 5991-3612EN

Take you from this…. .... to this!
## When do I change what?

<table>
<thead>
<tr>
<th>Item</th>
<th>Typical Schedule</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Septum Nut</td>
<td>3-6 months</td>
<td>Septum nut can get warm and shed metal particle into the liner. Replace to minimize activity in the inlet/liner.</td>
</tr>
<tr>
<td>Syringe</td>
<td>Every 3 months</td>
<td>Check movement of plunger and replace if it does not move freely and cannot be cleaned.</td>
</tr>
<tr>
<td>Gold Seal</td>
<td>Monthly</td>
<td>At a minimum replace when trimming the front end of the column.</td>
</tr>
<tr>
<td>Split Vent Trap</td>
<td>6 months-1 year</td>
<td>Often forgotten. Can also cause retention instability.</td>
</tr>
<tr>
<td>Liner</td>
<td>Weekly</td>
<td>The liner takes the brunt of the sample load/residues. Replace often to help prevent unwanted down time.</td>
</tr>
<tr>
<td>Trim/Replace column</td>
<td>Weekly-Monthly</td>
<td>When experiencing chromatographic problems trim ½ to 1 meter of the front end of the column. Replace liner, septum and gold seal.</td>
</tr>
<tr>
<td>Inlet Setpa</td>
<td>100-200 injections</td>
<td>Depends a bit on septum type and manual/auto injections.</td>
</tr>
</tbody>
</table>

Schedule is an approximation of average usage requirements. Actual frequency is application and sample specific. Use your chromatography as a guide to developing a normal maintenance schedule.
Column Installation
Leak Check

DO NOT USE SNOOP

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

G3388B Leak Detector
# Leak and Installation Check

Inject a non-retained compound vs DB-1

<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>MeCl₂ (headspace or diluted)</td>
</tr>
<tr>
<td>NPD</td>
<td>CH₃CN-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
Non-Retained 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
ADM Flow Meter

- Replaceable Calibration Cartridge
- Automatic Notification of Cartridge Replacement
- Ergonomic and robust design
- Universal 3AA or USB power
- USB connects to web interface for added functionality
- Easy to view OLED Screen
- Kickstand
Modes of Flow Measurement

Press “Power/Mode” button

Press “Power/Mode” button

Press “Power/Mode” button

Press “Power/Mode” button
New Record Feature!

Press “Select”

Unique to NEW ADM Flow Meter!

Customer Benefit:
Easier to complete IQ/OQ or troubleshoot detectors with multiple flows
Column Conditioning

System must be leak free before conditioning column

Condition with the column connected to the detector so response can be monitored

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 hour in most cases
Column Installation to MSD

➢ Best to condition the column attached to the MSD
  ➢ By conditioning into the MSD you can observe for leaks and correct them prior to ever elevating oven temperature (high temp + O2 will quickly kill a column).
  ➢ MSD is a perfect leak detector, why not take advantage of this capability?
  ➢ Normal bleed products will not harm or dirty source
  ➢ If you condition not connected to the MSD and oxidize the phase from a leak, the column will bleed; customers then blame column.
  ➢ Oxygen WILL back diffuse into the column which will oxidize the last 6 – 12 inches

➢ Open source door and set column distance according to the service manual (1 – 4 mm)
➢ Tighten self-tightening column nut
➢ Close source door and pump down
➢ Press firmly against source door until vacuum takes over; do not tighten the source door nut / shipping clamp bolt
# JW Column Portfolio- DB, HP, CP, VF

<table>
<thead>
<tr>
<th>Low Polarity</th>
<th>Mid Polarity</th>
<th>High Polarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-Sil 2</td>
<td>DB-XLB</td>
<td>HP-88</td>
</tr>
<tr>
<td>DB-MTBE</td>
<td>DB-225MS</td>
<td>DB-WAX</td>
</tr>
<tr>
<td>CP-Select CB MTBE</td>
<td>DB-ALC1</td>
<td>CP-TCEP</td>
</tr>
<tr>
<td>DB-1HT</td>
<td>DB-17</td>
<td>DB-WAX FF</td>
</tr>
<tr>
<td>CP-Sil 5 CB</td>
<td>HP-Blood Alcohol</td>
<td>DB-WAX FF</td>
</tr>
<tr>
<td>Ultra 1</td>
<td>DB-17</td>
<td>DB-WAX FF</td>
</tr>
<tr>
<td>DB-2887</td>
<td>HP-50+</td>
<td>DB-WAX FF</td>
</tr>
<tr>
<td>DB-Petro/PONA</td>
<td>DB-17HT</td>
<td>CP-WAX 58 FFAP CB</td>
</tr>
<tr>
<td>CP-Sil PONA CB</td>
<td>DB-608</td>
<td>CP-WAX 52 CB</td>
</tr>
<tr>
<td>DB-HT SimDis</td>
<td>DB-TPH</td>
<td>CP-WAX 51</td>
</tr>
<tr>
<td>CP-SimDis</td>
<td>DB-502.2</td>
<td>CP-Carbowax 400</td>
</tr>
<tr>
<td>CP-Volamine</td>
<td>HP-VOC</td>
<td>Carbowax 20M</td>
</tr>
<tr>
<td>Select Mineral Oil</td>
<td>DB-VRX</td>
<td>HP-20M</td>
</tr>
<tr>
<td>HP-101</td>
<td>DB-624</td>
<td>CAM</td>
</tr>
<tr>
<td>SE-30</td>
<td>VF-624MS</td>
<td></td>
</tr>
<tr>
<td>CP-Select 624 CB</td>
<td>DB-1301</td>
<td></td>
</tr>
<tr>
<td></td>
<td>VF-1301MS</td>
<td></td>
</tr>
<tr>
<td></td>
<td>CP-Sil 13 CB</td>
<td></td>
</tr>
</tbody>
</table>

Agilent J&W has over 50 different stationary phase offerings
The Power of Selectivity: Start with the right phase

**DB-1**
15m x 0.32mm, 0.25µm
Oven:
40°C for 2 min
40-120°C at 5°C/min

**DB-Wax**
15m, 0.32mm, 0.25µm
Oven:
80-190°C at 20°C/min
Which column types/dimensions produce higher bleed?

➢ Polarity: More polar = higher bleed
➢ Low polarity = More thermally stable
  ➢ look at temperature limits as a general indicator of thermal stability
➢ The more total mass of polymer in the column the higher the bleed (within a given phase)
  ➢ Larger diameters
  ➢ Longer columns
  ➢ Thicker films
Column Bleed is Influenced by:

- Phase type
- Temperature
- Column dimensions

Graph showing time (min.) on the x-axis and a logarithmic scale on the y-axis ranging from 6000 to 1.3e4. Two lines represent different conditions:

- DB-624 30M x .53mm I.D., 3.0µm
  - 24 pA / 260°C

- DB-1 30m x .32mm I.D., .25µm
  - 12 pA / 320°C
Column Inertness: What does it mean?

➢ Easier to describe “lack of inertness”
   ➢ Peak Tailing (reversible interaction)
   ➢ Loss of compound all together (irreversible interaction)

➢ A high level of flow path inertness will produce peaks from active compounds that are not degraded and will look “normal”/symmetrical

➢ The negative effects the column has towards challenging compounds
   ➢ Acids
   ➢ Bases
   ➢ Hydrogen Bonding
   ➢ i.e. 2,4-DNP, Endrin, DOA, Etc.
### Ultra Inert Test Mix – DB-5MS Ultra Inert v. Competitors

1. **1-Propionic acid**
2. **1-Octene**
3. **n-Octane**
4. **4-Picoline**
5. **n-Nonane**
6. **Trimethyl phosphate**
7. **1,2-Pentanediol**
8. **n-Propylbenzene**
9. **1-Heptanol**
10. **3-Octanone**
11. **n-Decane**

---

**Agilent J&W DB-5ms Ultra Inert**

30m x 0.25mm x 0.25um

(P/N 122-5532UI)
# Ultra Inert Phases

<table>
<thead>
<tr>
<th>Column</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>DB-1ms UI</td>
<td>DB-Select 624 UI 467</td>
</tr>
<tr>
<td>HP-1ms UI</td>
<td>DB-Wax UI</td>
</tr>
<tr>
<td>DB-5ms UI</td>
<td>DB-35ms UI</td>
</tr>
<tr>
<td>HP-5ms UI</td>
<td>DB- BAC1 UI</td>
</tr>
<tr>
<td>DB-624 UI</td>
<td>DB-BAC2 UI</td>
</tr>
</tbody>
</table>

UI columns use engineered proprietary deactivation and are QC tested with VERY demanding probes that will exploit weaknesses in inertness.

**Same Selectivity, more Inertness!**
The Best Type of Column for GC/MS

➢ Low bleed
  ➢ “ms” phases are best but not required
  ➢ ms and msUI phases have same bleed

➢ Low flow
  ➢ ≤2 ml/min for HES and Diffusion pumps
    ➢ This includes during pressure pulse
  ➢ ≤4 mL/min for Turbo
    ➢ ≤ 2 is still best for optimum performance
  ➢ Maximum diameter 0.32mm,
    (however 0.25mm ID or smaller is best)
  ➢ 30m x 0.25mm by far most common
The guard column is 3 - 5 meters of deactivated fused silica tubing with the same diameter as the analytical column. It is connected with a zero dead volume union.
Integrated Guards - DuraGuard

- No union
- Possible for any DB column 0.18mm and larger
- Limited offering “off-the-shelf”

<table>
<thead>
<tr>
<th>DuraGuard</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase</td>
<td>ID (mm)</td>
<td>Length (m)</td>
<td>Film (µm)</td>
<td>Guard Length (m)</td>
<td>Part No.</td>
</tr>
<tr>
<td>DB-1</td>
<td>0.25</td>
<td>30</td>
<td>0.25</td>
<td>10</td>
<td>122-1032G</td>
</tr>
<tr>
<td>DB-XLB</td>
<td>0.25</td>
<td>30</td>
<td>0.25</td>
<td>10</td>
<td>122-1232G</td>
</tr>
<tr>
<td>DB-5ms</td>
<td>0.25</td>
<td>30</td>
<td>0.25</td>
<td>10</td>
<td>122-5532G</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.50</td>
<td>10</td>
<td>122-5536G</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.00</td>
<td>10</td>
<td>122-5533G</td>
</tr>
<tr>
<td></td>
<td></td>
<td>60</td>
<td>0.25</td>
<td>10</td>
<td>122-5562G</td>
</tr>
<tr>
<td></td>
<td>0.32</td>
<td>30</td>
<td>1.00</td>
<td>10</td>
<td>123-5533G</td>
</tr>
<tr>
<td></td>
<td>0.53</td>
<td>30</td>
<td>0.50</td>
<td>10</td>
<td>125-5537G</td>
</tr>
<tr>
<td>DB-5.625</td>
<td>0.18</td>
<td>20</td>
<td>0.36</td>
<td>5</td>
<td>121-5622G5</td>
</tr>
<tr>
<td></td>
<td>0.25</td>
<td>30</td>
<td>0.25</td>
<td>5</td>
<td>122-5631G5</td>
</tr>
<tr>
<td>DB-1701</td>
<td>0.53</td>
<td>30</td>
<td>1.00</td>
<td>10</td>
<td>125-0732G</td>
</tr>
<tr>
<td>DB-624</td>
<td>0.53</td>
<td>30</td>
<td>3.00</td>
<td>5</td>
<td>125-1334G5</td>
</tr>
</tbody>
</table>
Misc Tools - ferrule removal, pre-swagers

RFT-2500
RFT-5300
RMP-5005
9301-0985

Metal Ferrules, G3440-80218
Graphite Ferrules, G3440-80217

430-1020
20 x magnifier

UltiMetal Plus Flexible Metal ferrules

Pre-swaging tool, G2855-60200
Detectors

- FID – Most common; will detect anything that will “burn” in the flame to produce ions…anything organic
- TCD – Universal detector, non-destructive; choice of carrier gas determines sensitivity
- MSD – Qual and quant
- ECD – Halogens; extremely sensitive
FID

Adaptable fitting

Capillary optimized fitting

Cleaning kit
Other consumables like ignitor

Ignitor glow plug assembly,
19231-60680
TCD – 2 types of connections for column

- Standard design

---

<table>
<thead>
<tr>
<th>TCD Ferrules</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column ID (mm)</td>
</tr>
<tr>
<td>----------------</td>
</tr>
<tr>
<td>0.53</td>
</tr>
<tr>
<td>0.32</td>
</tr>
<tr>
<td>0.25/0.2/0.1</td>
</tr>
<tr>
<td>No hole</td>
</tr>
<tr>
<td>TCD back ferrule for 1/8 in. detector fitting, 10/pk</td>
</tr>
</tbody>
</table>
### SST / Inert Ion Source Assembly

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
<th>Part number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Gold plated set screw</td>
<td>G1999-20022</td>
</tr>
<tr>
<td>2</td>
<td>Gold Plated Screw</td>
<td>G3870-20021</td>
</tr>
<tr>
<td>3</td>
<td>Interface socket</td>
<td>G1099-20136</td>
</tr>
<tr>
<td>4</td>
<td>Ion Source body for SST</td>
<td>G1099-20130</td>
</tr>
<tr>
<td></td>
<td>Ion Source body for Inert</td>
<td>G2589-20043</td>
</tr>
<tr>
<td>5</td>
<td>Draw out Cylinder</td>
<td>G1072-20008</td>
</tr>
<tr>
<td>6</td>
<td>Draw out plate for SST - 3mm</td>
<td>05971-20134</td>
</tr>
<tr>
<td></td>
<td>Draw out plate for SST - 6mm</td>
<td>G3163-20530</td>
</tr>
<tr>
<td></td>
<td>Draw out plate for Inert - 3mm</td>
<td>G2589-20100</td>
</tr>
<tr>
<td></td>
<td>Draw out plate for Inert - 6mm</td>
<td>G2589-20045</td>
</tr>
<tr>
<td></td>
<td>Draw out plate for Inert - 9mm</td>
<td>G3440-20022</td>
</tr>
<tr>
<td>7</td>
<td>4-turn filament</td>
<td>G7005-60061</td>
</tr>
<tr>
<td>8</td>
<td>Spring Washer</td>
<td>3050-1374</td>
</tr>
<tr>
<td>9</td>
<td>Lens insulator for SST EI / Inert EI</td>
<td>G3170-20530</td>
</tr>
<tr>
<td>10</td>
<td>Entrance Lens</td>
<td>G3170-20126</td>
</tr>
<tr>
<td>11</td>
<td>Ion focus Lens</td>
<td>05971-20143</td>
</tr>
<tr>
<td>12</td>
<td>350 Repeller Assembly SST EI</td>
<td>G3870-67172</td>
</tr>
<tr>
<td></td>
<td>350 Repeller Assembly Inert EI</td>
<td>G3870-67173</td>
</tr>
</tbody>
</table>
You can reduce or prevent problems by thinking ahead
Some instrument parts should be replaced on a regular basis, before there is a problem
Develop a maintenance routine that works for you
Sample clean-up is a powerful tool in addressing common chromatography and mass spectrometry challenges
Choose the best column for your sample and conditions
Contact Agilent Chemistries and Supplies Technical Support

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

**Option 1 for GC/GCMS Columns and Supplies**

Option 2 for LC/LCMS Columns and Supplies

Option 3 for Sample Prep Products, Filtration and QuEChERS

Option 4 for Spectroscopy Supplies

Available in the USA 8-5 all time zones

**gc-column-support@agilent.com**

**lc-column-support@agilent.com**

**spp-support@agilent.com**

**spectro-supplies-support@agilent.com**

GC columns and supplies