New Agilent J&W Ultra Inert Capillary GC Columns

Raising the Bar for CONSISTENT Column Inertness Performance

Trace Level Analysis Made Routine
Outline

• What is inertness?
• Evolution of capillary column QC Testing
• Value of Consistent Column Inertness and Exceptionally Low Bleed
• Application examples
• Take away messages
GC System Inertness
What do we mean?
Problems with poor inertness usually limited to “active” solutes.

For example:
Alcohols & Diols (-OH), Phenols (O-H), Amines (-NH3), Acids (COOH), ThioIls & Sulfur in general like to tail.

Thermally labile and structurally “strained” solutes will breakdown or rearrange, e.g., DDT, Endrin, Carbamates, Nitroglycerines.
What Does GC System Inertness Look Like?

Easier question: What does poor inertness look like?

Symptoms of poor GC system inertness:

* Tailing peaks
* Reduced peak response
* No peak response
* Extra peaks!
* Poor linearity of a peak – usually at low concentrations
* Unstable detector baseline
GC System Inertness
What do we mean?

Problems with poor inertness usually limited to “active” solutes.

Tailing or breakdown of “benign” solutes is symptomatic of a more generalized system problem, usually related to gross contamination.
Possible Inertness Problem Areas

Inlet
- liner, liner packing, gold seal, stainless steel

Consumables
- septa, syringe, vial, caps, inserts, solvents

Column

GC Detector
- source geometry, material, column interface, acquisition rates

Temperatures
- inlet, transfer line, source, quads, oven

Other method factors i.e. samples and standards preparation
How important is **Column** inertness to overall **Flowpath Inertness**?

**GC Flowpath Surface Areas**

<table>
<thead>
<tr>
<th>Surface Area</th>
<th>l (cm)</th>
<th>d (cm)</th>
<th>pi</th>
<th>(cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liner</td>
<td>7</td>
<td>0.2</td>
<td>3.142</td>
<td>4.4</td>
</tr>
<tr>
<td>Seal</td>
<td>0.4</td>
<td>0.8</td>
<td>3.142</td>
<td>1.0</td>
</tr>
<tr>
<td>Column</td>
<td>3000</td>
<td>0.025</td>
<td>3.142</td>
<td>235.6</td>
</tr>
</tbody>
</table>
Where does column activity come from?

Isolated Geminal    Vicinal Siloxane

Sterically difficult to “cap” all of them—estimates 40-65% capped with traditional deactivation.

Non-traditional sources such as trace impurities in starting materials and manufacturing lines.
Traditional Deactivations

Dichlorodimethylsilane, various silizanes, etc… “endcaps”

Traditional deactivation has gaps in surface coverage due to bulky TMS type moieties, and tight fused silica lattice, and is somewhat inert and chemically resistant.
DB-5ms and HP-5ms Engineered Deactivations

Polymeric Deactivation Technology

“Binds” at multiple points with many silanols

“Blankets” sterically hindered active silanols, fewer silanols
What does Column Activity look like?

Tailing, and loss of response.

<table>
<thead>
<tr>
<th>Probe</th>
<th>0.5 µl inj, 1:50 split, (ng)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methane</td>
</tr>
<tr>
<td>2</td>
<td>Propionic Acid</td>
</tr>
<tr>
<td>3</td>
<td>Octane</td>
</tr>
<tr>
<td>4</td>
<td>Nitrobutane</td>
</tr>
<tr>
<td>5</td>
<td>4-Picoline</td>
</tr>
<tr>
<td>6</td>
<td>Trimethyl Phosphate</td>
</tr>
<tr>
<td>7</td>
<td>1,2-Pentanediol</td>
</tr>
<tr>
<td>8</td>
<td>Propylbenzene</td>
</tr>
<tr>
<td>9</td>
<td>1-Hetpanol</td>
</tr>
<tr>
<td>10</td>
<td>3-Octanone</td>
</tr>
<tr>
<td>11</td>
<td>Decane</td>
</tr>
</tbody>
</table>

Loss of peak height, but same area counts

Loss of peak height and area counts

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What are the specific benefits of High Inertness?

Greater sensitivity for traditional trace active analytes
meet RRF requirements with greater ease
more runs before maintenance

Greater reliability for ultra-trace non-traditionally active analytes (<100 ppb PAHs, Chlorinated dioxins, etc...)
Who benefits from High Inertness Columns?

Anyone doing trace analysis of active analytes
Environmental semivolatile analysts
Pesticide residue analysts
Forensic/Drug analysts

Anyone in Industry, Government, or Academia interested in ultra-trace amounts of even modestly active analytes
**Test Probes and Column Activity QC Testing**

- Test probes are vital to ensure the quality and reproducibility of GC columns
  - Properly deactivated
  - Contain the correct amount of stationary phase
  - Consistent batch-to-batch relative retention time

- Test probes can either highlight or mask the deficiencies of a column, normally include:
  - An organic acid (peak tailing or lost response of acid indicates the column is basic)
  - A base (peak tailing or lost response of base indicates the column is acidic)
  - An alcohol (gives indication of any oxygen damage or exposed silanols)
  - Non-active probes (e.g. alkanes)

- Good test probes allows the probative portion of the test module to penetrate and fully interact with the columns stationary phase and surface.
  - Low molecular weight
  - Low boiling points
  - No steric shielding of active group
Weak Probes vs. Strong Probes

Weak Probes

Acidic and basic portion of the molecules are shielded by the methyl groups of the 2,6-dimethyl substituted phenyl ring

Stronger Probes

Active end of each compound is available to interact with any active sites on the columns
How is High GC column Inertness Assured?

Not like this!

Alcohol

“Acid”

“Base”

not an Agilent test!
Grob-Type Mix - QC Testing of the 80s

1. 1-Octanol
2. n-Undecane
3. 2,6-Dimethylphenol
4. 2,6-Dimethylaniline
5. n-Dodecane
6. Naphthalene
7. 1-Decanol
8. n-Tridecane
9. Methyl decanoate

Agilent J&W DB-5ms Ultra Inert
30m x 0.25mm x 0.25um (P/N 122-5532UI)

- Less demanding test mix
- Less probative probes for column activity
- Elevated oven temperature at 120°C allows the molecules to sweep past active sites and mask solute/column interactions.

Restek Rxi-5SIL MS
30m x 0.25mm x 0.25um

Sampler: Agilent 7683B, 5 µL syringe (Agilent part # 5181-1273), 1.5 µL split injection, 4 ng each component
Carrier: Hydrogen constant pressure 37 cm/s
Inlet: Split/splitless; 250 °C, 1.4 ml/min. column flow, split flow 100 ml/min.
Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)
Oven: 120 °C isothermal
Detection: FID at 325 °C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

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DB-5ms Test Mix – QC Testing of the 90s

Agilent J&W DB-5ms
Ultra Inert
30m x 0.25mm x 0.25um
(P/N 122-5532UI)

Restek Rxi-5SIL MS
30m x 0.25mm x 0.25um

1. 2-Ethylhexanic acid
2. 1,6-Hexanediol
3. 4-Chlorophenol
4. Tridecane
5. 1-Methylnaphthalene
6. 1-Undecanol
7. Tetradecane
8. Dichlorohexylamine

Sampler: Agilent 7683B, 5 µL syringe (Agilent part # 5181-1273), 1.5 µL split injection, 4 ng each component
Carrier: Hydrogen constant pressure 38 cm/s
Inlet: Split/splitless; 250 °C, 1.4 ml/min. column flow, split flow 75 ml/min.
Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)
Oven: 125 °C isothermal
Detection: FID at 320 °C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

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How Agilent Assures High Inertness on the HP-5ms columns, with Every Test

### Performance Results

<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>UNDECANE</td>
<td>2.058</td>
<td>0.70</td>
<td>0.015</td>
</tr>
<tr>
<td>4-CHLOROPHENOL</td>
<td>3.222</td>
<td>1.18</td>
<td>0.023</td>
</tr>
<tr>
<td>1-DECYLAMINE</td>
<td>3.813</td>
<td>1.58</td>
<td>0.024</td>
</tr>
<tr>
<td>TETRDECANE</td>
<td>4.528</td>
<td>2.07</td>
<td>0.028</td>
</tr>
<tr>
<td>METHYL CAPRATE</td>
<td>4.946</td>
<td>2.35</td>
<td>0.031</td>
</tr>
<tr>
<td>TETRADECANE</td>
<td>6.677</td>
<td>3.52</td>
<td>0.043</td>
</tr>
<tr>
<td>ACENAPHTHYLENE</td>
<td>8.700</td>
<td>4.89</td>
<td>0.061</td>
</tr>
<tr>
<td>1-DODECANOL</td>
<td>9.155</td>
<td>5.20</td>
<td>0.062</td>
</tr>
<tr>
<td>PENTADECANOE</td>
<td>10.293</td>
<td>5.97</td>
<td>0.070</td>
</tr>
</tbody>
</table>

### Theoretical Plates/Meter

PENTADECANOE

3545

### Retention Index

- METHYL CAPRATE: 1124.1
- ACENAPHTHYLENE: 148.2
- 1-DODECANOL: 1473.8

### Peak Height Ratio

- 1-DODECANOL, TETRDECANE: 0.54
- 1-DECYLAMINE, TETRDECANE: 1.43
- 4-CHLOROPHENOL, TETRDECANE: 1.12

### Test Conditions

- **Inlet:** Split (275°C)
- **Detector:** FID (325°C)
- **Carrier Gas:** Hydrogen
- **Flow:** 33.9 cm/sec (1.0 ml/min)
- **Holdup Compound:** Pentane (1.477 min)
- **Temperature Program:** Isothermal at 135°C

### Acid

![Acid Structure](image)

### Base

![Base Structure](image)

### Alcohol

![Alcohol Structure](image)

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How Agilent Assures High Inertness on the DB-5ms columns, with Every Test

Performance Results

<table>
<thead>
<tr>
<th>Compound Identification</th>
<th>Retent.</th>
<th>Part.</th>
<th>1/2-Wide</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 2-ETHYLHEXANOIC ACID</td>
<td>2.990</td>
<td>1.31</td>
<td>0.022</td>
</tr>
<tr>
<td>2. 1,6-HEXANEDIOL</td>
<td>2.990</td>
<td>1.31</td>
<td>0.022</td>
</tr>
<tr>
<td>3. 4-CHLOROPHENOL</td>
<td>3.635</td>
<td>1.80</td>
<td>0.027</td>
</tr>
<tr>
<td>4. TRIDECANE</td>
<td>6.283</td>
<td>3.84</td>
<td>0.043</td>
</tr>
<tr>
<td>5. 1-METHYLNAPHTHALENE</td>
<td>7.820</td>
<td>5.03</td>
<td>0.051</td>
</tr>
<tr>
<td>6. 1-UNDECANOL</td>
<td>8.963</td>
<td>5.91</td>
<td>0.060</td>
</tr>
<tr>
<td>7. TETRADECANE</td>
<td>9.877</td>
<td>6.62</td>
<td>0.064</td>
</tr>
<tr>
<td>8. DICYCLOHEXYLAMINE</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Test Conditions

- Inlet: Split (250°C)
- Detector: FID (300°C)
- Carrier Gas: Hydrogen
- Flow: 38.6 cm/sec (1.2 ml/min)
- Holdup Compound: Methane (1.297 ml/min)
- Temperature Program: Isothermal at 125°C

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Ultra Inert Test Mix – QC Testing for Today’s Demanding Applications

- Carefully selected very demanding test probes for in-depth evaluation of column inertness
- Test temperature 65°C (isothermal), well below that normally used in conventional tests

<table>
<thead>
<tr>
<th>Probe</th>
<th>(ng on column)</th>
<th>Column functional test</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 1-Propionic acid</td>
<td>1.0</td>
<td>Basicity</td>
</tr>
<tr>
<td>2. 1-Octene</td>
<td>0.5</td>
<td>Polarity</td>
</tr>
<tr>
<td>3. n-Octane</td>
<td>0.5</td>
<td>Hydrocarbon marker</td>
</tr>
<tr>
<td>4. 4-Picoline</td>
<td>1.0</td>
<td>Acidity</td>
</tr>
<tr>
<td>5. n-Nonane</td>
<td>1.0</td>
<td>Hydrocarbon marker</td>
</tr>
<tr>
<td>6. Trimethyl phosphate</td>
<td>1.0</td>
<td>Acidity</td>
</tr>
<tr>
<td>7. 1,2-Pentanediol</td>
<td>1.0</td>
<td>Silanol</td>
</tr>
<tr>
<td>8. n-Propylbenzene</td>
<td>1.0</td>
<td>Hydrocarbon marker</td>
</tr>
<tr>
<td>9. 1-Heptanol</td>
<td>1.0</td>
<td>Silanol</td>
</tr>
<tr>
<td>10. 3-Octanone</td>
<td>1.0</td>
<td>Polarity</td>
</tr>
<tr>
<td>11. n-Decane</td>
<td>1.0</td>
<td>Hydrocarbon marker</td>
</tr>
</tbody>
</table>

Sampler: Agilent 7683B, 0.5 µL syringe (Agilent part # 5188-5246), 0.02 µL split injection
Carrier: Hydrogen constant pressure, 38 cm/s
Inlet: Split/splitless; 250 °C, 1.4 ml/min. column flow, split flow 900 ml/min., gas saver flow 75 ml/min. on at 2.0 min.
Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)
Oven: 65 °C isothermal
Detection: FID at 325 °C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min., nitrogen makeup
Ultra Inert Test Mix on Restek Rxi-5Sil MS

All highlighted peaks have poor peak shape – poor column deactivation

- The Restek column showed very poor performance when tested against the Über One test mix.
- Less demanding test probes masked the column activity for this Restek column:
  - The same column performed well with Grob-type test mix and DB-5ms test mix
Ultra Inert Test Mix on a Phenomenex Column

Phenomenex ZB-5ms
30m x 0.25mm x 0.25um

All highlighted peaks have poor peak shape with obvious loss of responses.

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

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Ultra Inert Test Mix on Agilent J&W DB-5ms Ultra Inert

- Nice Peak Shapes for challenging active compounds
- Increased peak heights for accurate integration and detection of trace samples
- Routine analysis of demanding analytes now feasible

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Ultra Inert Test Mix on Agilent J&W DB-5ms Ultra Inert

- Nice Peak Shapes for challenging active compounds
- Increased peak heights for accurate integration and detection of trace samples
- Routine analysis of demanding analytes now feasible
Test Mix Summary

- Grob-type mix not probative for inertness
- DB-5ms text mix is a good test for the 90s
- Über One mix probes inertness and differentiates an excellent column from a mediocre one
- Well designed test mix uncovers potential adsorption of acid and base analytes and raises the bar in inertness QC
Challenges and Needs of Today’s Laboratories

• Challenges
  - Qualification/quantification of trace samples
  - Keep instrument up and running

• Needs
  - Lower detection limits
  - Improved stability in GC or GC/MS system

<table>
<thead>
<tr>
<th>Lower Detection Limit</th>
<th>Increase signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduce noise</td>
<td></td>
</tr>
<tr>
<td>Injection system (septa, liners, connections)</td>
<td>Sample concentration</td>
</tr>
<tr>
<td>Carrier gas and detector gases</td>
<td>Sample size</td>
</tr>
<tr>
<td>Leaks</td>
<td>Inert injection and detection port sleeves/liner</td>
</tr>
<tr>
<td>Temperature setting</td>
<td>Gas velocity or temp program rate</td>
</tr>
<tr>
<td>Stationary phase and column bleed</td>
<td>Column inertness</td>
</tr>
</tbody>
</table>

• Only when a column exhibits both low bleed and low activity are results reliable.
  - Low bleed increases the signal-to-noise ratio, but if any of the analyte is adsorbed by active sites in the column, the results are flawed.
  - If the column is well deactivated but the bleed is high, some of the signal generated by the analytes is smothered by the bleed signal. Again, the results are flawed.
Agilent J&W Ultra Inert Columns – Innovation

• Only GC columns proven to deliver on the promise of column inertness and column bleed.
• Built on top of the existing Agilent J&W GC/MS columns with added column inertness.
• New QC testing for modern applications
  – A unique, very demanding test probe mixture to test columns individually
  – Worse case scenario testing to ensure column inertness performance
  – Raising the bar for GC column QC testing
• Critical for analysis of active compounds, trace level samples, and screening of unknown samples

<table>
<thead>
<tr>
<th>Features</th>
<th>Advantages</th>
<th>Benefits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unique QC testing procedure with demanding Ultra Inert test probe mixture</td>
<td>Consistent column inertness performance</td>
<td>More predictable and reliable results</td>
</tr>
<tr>
<td>Highest column inertness</td>
<td>Better peak shape</td>
<td>More accurate peak identification, more accurate quantification</td>
</tr>
<tr>
<td></td>
<td>Greater S/N ratio</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Minimum compound loss or degradation</td>
<td></td>
</tr>
<tr>
<td></td>
<td>More analysis without maintenance</td>
<td>Reduced costs and instrument downtime</td>
</tr>
<tr>
<td>Exceptionally low column bleed</td>
<td>Greater S/N ratio and increased sensitivity for all detectors</td>
<td>Lower detection limit, reduced detector contamination, and reliable compound identification</td>
</tr>
<tr>
<td></td>
<td>Faster baseline stabilization</td>
<td>Minimized conditioning time and increased sample throughput</td>
</tr>
<tr>
<td>Support of 0.18mm ID configuration</td>
<td>Higher sample throughput</td>
<td>More sample analysis in less time</td>
</tr>
</tbody>
</table>

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The Quality of the Ultra Inert Column Series Exceeds my Wildest Dream

“[Agilent’s] **breakthroughs** in *surface pretreatments* and **improvements** in *surface deactivation* came much more rapidly than I had anticipated. The quality of the new **Ultra Inert series** of columns exceed my wildest dreams.”

“I am satisfied that customers with the most demanding analyses of active analytes can have confidence that the DB-5ms and HP-5ms Ultra Inert Columns will provide the highest level of performance.”

**Walt Jennings**

**Professor Emeritus, University of California**
Every Column Individually Test – Ensured Quality

- Performance summary sheet shipped with each column with QC testing results against the Ultra Inert test probe mixture.
Same Selectivity – No Method Re-Development

- DB-5ms Ultra Inert columns have the same selectivity as their DB-5ms counterparts
- HP-5ms Ultra Inert columns have the same selectivity as their HP-5ms counterparts

Retention Index of 1-Methylnaphthalene

- DB-5ms Ultra Inert
  - Retention Index: 1371.8

- DB-5ms
  - Retention Index: 1324.8

Retention Index of 1-Undecanol

- DB-5ms Ultra Inert
  - Retention Index: 1324.9

- DB-5ms
  - Retention Index: 1324.8
Application Examples

- Semi Volatile Analysis
- Brominated Fire Retardants
- Pesticides in Orange Oil
- PAHs
- PBDEs
Semi Volatile Analysis

**GC:** Agilent 6890N/5975B MSD

**Sampler:** Agilent 7683B, 5.0 µL syringe (Agilent part # 5188-5246), 1.0 µL splitless injection, 5 ng on column

**Carrier:** Helium constant flow 30 cm/s

**Inlet:** Split/splitless; 260% C, 53.7 ml/min. total flow, purge flow 50 ml/min. on at 0.5 min., gas saver off

**Inlet Liner:** Deactivated single taper w glass wool (Agilent part # 5183-4647)

**Column:** DB-5ms Ultra Inert 30m x 0.25mm x 0.25µm (Agilent part # 122-5532UI)

**Oven:** 40% C (1 min) to 100%C (15% C/min), 10% C to 210% C (1 min), 5% C/min. to 310% C (8 min)

**Detection:** MSD source at 300% C, quadrupole at 180% C, transfer line at 290% C, scan range 50-550 AMU

1. N-nitrosodimethylamine
2. Aniline
3. 1,4 dichlorobenzene-D4
4. Benzoic acid
5. Naphthalene- D8
6. Acenaphthene-D10
7. 2,4-dinitrophenol
8. 4-nitrophenol
9. 2-methyl-4,6-dinitrophenol
10. pentachlorophenol
11. 4-aminobiphenyl
12. Penanthrene-D10
13. Benzidine
14. Chrysene-D12
15. 3,3'-dichlorobenzidine
16. Benzo [b] fluoroanthene
17. Benzo [k] fluoroanthene
18. Perylene-D12
“Large Mix” 5 ng on Column AccuStandard 8270 Mixes 1,2,3,4a,4b,5 &6 (93 Compounds) Select compound highlighted

1. n-Nitrosodimethylamine
2. 2-methyl pyridine
3. Benzidine
4. Flouranthene
5. Benzo (g,h,i) perylene

**GC/MSD Conditions**

**Column:** DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm part # 122-5532UI

**Carrier:** He 30 cm/sec constant flow

**Oven:** 40% C (1min) to 100% C (15 % C/min), 10 % C /min to 210% C (1min), 5 % C/min to 310% C (8 min)

**Inlet:** splitless 260 % C purge flow 50 % ml/min at 0.5 min, gas saver 80 ml/min on at 1 minute

**MSD:** transfer line 290 % C, source 300 % C, quad 180 % C
Pesticides and Fire Retardants (US EPA 527)

GC/MSD Conditions

Sample: Pesticide/PBDE standards 1 ng with 5 ng IS/SS on column

Column: DB-5MS Ultra Inert 30m x 0.25mm x 0.25um (Agilent part # 122-5532UI)

Carrier: Helium 52 cm/sec, constant flow

Oven: 60°C (1min) to 210°C (25°C/min), 20°C/min to 310°C (3 min)

Injection: Splitless, 250°C, purge flow 50 ml/min at 1 min, gas saver 80 ml/min on at 3 min

MSD: Transfer Line 290°C, Source 300°C, Quad 180°C

1. 1,2-Dimethyl-2-nitrobenzene
2. Acenaphthalene-D10
3. Dimethoate
4. Atrazine
5. Propazine
6. Anthracene-D10
7. Vinclozoline
8. Prometryne
9. Bromacil
10. Malathion
11. Thiazopyr
12. Dursban
13. Bendiocarb
14. Parathion
15. Terbus sulfone
16. Bioallethrin
17. Oxychlordane
18. Fenamiphos
19. Nitrophen
20. Norflurazone
21. Kepone
22. Hexazinone
23. Triphenyl phosphate
24. Bifenthrin
25. Chrysene-D12
26. BDE-47
27. Mirex
28. BDE-100
29. BDE-99
30. Perylene-D12
31. Fenvalerate
32. Esfenvalerate
33. Hexabromobiphenyl
34. BDE-153

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Drugs of Abuse

Column: DB-Sms Ultra Inert 30 m x 0.25 mm x 0.25 µm (Agilent part # 122-5532UI)
Carrier: Helium 43.8 cm/sec constant flow
Oven: 120% C (2min) 20% C/min to 180% C (6 min hold), 18% C/min to 270% C (2min),
25% C/min to 325% C (2 min)
Inlet: split 30:1, ~ 1 ng on column 250% C, single taper liner (Agilent # 5181-3316)
MSD: transfer line 300% C, source 280% C, quad 200% C, full scan m/z 50-450

1. Nicotine
2. Phenmetrazine
3. Ibuprofen
4. Butabarbital
5. Amobarbital
6. Secobarbital
7. Caffeine
8. Benzphetamine
9. Hexobarbital
10. Tropacocaine
11. Phenobarbital
12. Procaine
13. L-cocaine
14. Chlorcytizone
15. Codine
16. Diazepam
17. Oxymorphone

Time (min)
### Bezodiazepines

- **Column:** DB-5ms Ultra Inert 122-5532UI 30m x 0.25 mm x 0.25 μm
- **Carrier:** Hydrogen, 53 cm/sec, constant flow
- **Flow Program (mL/min):**
  - 1.6 for 11 min
  - 1.6 to 2.4 at 60 mL/min hold 2 min
  - 2.4 to 5.0 at 50 mL/min hold 9 min
- **Oven:** 170 °C for 3.2 min
  - 170-250 °C at 24.7 °C/min, hold 5.3 min
  - 250-280 °C at 18.6 °C/min, hold 4.0 min
  - 280-325 °C at 50.0 °C/min, hold 4 min
- **Injection:** Pulsed Splitless, 280 °C
  - 20 psi pulse pressure for 0.38 min
  - 50 mL/min purge at 0.40 min
- **Detector:** Direct Connect liner G1544-80730 FID, 350 °C
- **Sample:** 5-10 ng on column

#### Table

<table>
<thead>
<tr>
<th>No.</th>
<th>Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Medazepam</td>
</tr>
<tr>
<td>2</td>
<td>Halazepam</td>
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<tr>
<td>3</td>
<td>Oxazepam</td>
</tr>
<tr>
<td>4</td>
<td>Lorazepam</td>
</tr>
<tr>
<td>5</td>
<td>Diazepam</td>
</tr>
<tr>
<td>6</td>
<td>Desalkyl Aurazepam</td>
</tr>
<tr>
<td>7</td>
<td>Nordazepam</td>
</tr>
<tr>
<td>8</td>
<td>Clobazam</td>
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<tr>
<td>9</td>
<td>Oxazolam</td>
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<tr>
<td>10</td>
<td>Temazepam</td>
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<tr>
<td>11</td>
<td>Flunitrazepam</td>
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<td>12</td>
<td>Bromozepam</td>
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<td>13</td>
<td>Prazam</td>
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<td>14</td>
<td>Lormetazepam</td>
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<td>15</td>
<td>Nitrazepam</td>
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<td>16</td>
<td>Chlordiazepoxide</td>
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<td>Clonazepam</td>
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<td>Demoxepam</td>
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<td>19</td>
<td>Estazolam</td>
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<tr>
<td>20</td>
<td>Alprazolam</td>
</tr>
<tr>
<td>21</td>
<td>Trazolam</td>
</tr>
</tbody>
</table>

For Research Use Only. Not for use in diagnostic procedures.
Pesticides in Orange Oil

Analysis was carried out on the Agilent 7890A/5975 GC/MS or 7890A/7000 GC/MS/MS equipped with either a 7683 or 7683B Series ALS, split/splitless injection port and triple-axis detector. An Agilent J&W DB-Sms Ultra Inert 15 m x 0.25 mm x 0.25 um column (Agilent part # 122-5512UI) was used. The initial GC oven temperature was 70°C, which was held for 0.67 minutes. The oven was then ramped by 75°C/minute to 150°C, held for 0 minutes and ramped by 9°C/minute to 200°C and held for 0 minutes before ramping by 24°C/minute to 280°C and holding for 3 minutes. A six-minute post-run at 320°C was used. Pressure was held constant at 10 psi throughout the run and a split ratio of 10:1 for a 1uL injection. An open ended 4 mm helical liner was used (Agilent #5188-5396). The inlet temperature was 250°C and transfer line was set to 280°C. In the case of both detectors the source temperature was set to 300°C and the analyzer to 180°C.

Phenanthrene-D10 (2 ppm)  
2,4 D Ethyl ester

Chlorpyrifos

Ethion

Tetradifon

S/N for quantitating transition m/z 356 → 229
Signal to Noise ratio: 400:1

200 ppb Tetradifon in Orange Oil (1:10 split)

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PAH Analysis

GC/MSD Conditions

Sample: 10ug/ml PAH Standard
Column: DB-5ms Ultra Inert 30m x 0.25mm x 0.25um (Agilent part # 122-5532UI)
Carrier: Helium 45cm/sec, constant flow
Oven: 55°C (1min) to 320°C (25°C/min), hold 3 min
Injection: Pulsed splitless, 300°C, 40psi until 0.2 min, purge flow 30ml/min at 0.75 min
Gas saver 80ml/min on at 3 min
MSD: Transfer Line 280°C, Source 300°C, Quad 180°C

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benz(a)anthracene
10. Chrysene
11. Benz(b)fluoranthene
12. Benz(k)fluoranthene
13. Benz[a]pyrene
14. Indeno[1,2,3-cd]pyrene
15. Dibenzo[a,h]anthracene
16. Benzo[g,h,i]perylene

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PBDE Analysis

**GC/MS conditions**

Column: DB-5ms Ultra Inert 15 m × 0.25 mm × 0.25 µm (Agilent part # 122-5512UI)

Carrier: Carrier Helium 72 cm/s, constant flow

Oven: 150 to 325 °C (17 °C/min), hold 5 min

Injection: Pulsed splitless; 325 °C, 20 psi until 1.5 min, purge flow 50 mL/min at 2.0 min

MSD: Source at 300 °C, Quadrupole at 150 °C, transfer line at 300 °C, scan range 200–1000 amu

Total ion chromatogram (SIM mode) of a **0.005-ng** (BDEs -47, -100, -99, -154, -153, -183, and -205) and **0.025-ng** (BDE-209) on-column loading

Enlarged section of BDE-209 with excellent sensitivity for trace level analysis
PBDE Analysis

**GC/MS conditions**
- **Column:** DB-5ms Ultra Inert 15 m × 0.25 mm × 0.25 µm (Agilent part # 122-5512UI)
- **Carrier:** Carrier Helium 72 cm/s, constant flow
- **Oven:** 150 to 325 °C (17 °C/min), hold 5 min
- **Injection:** Pulsed splitless; 325 °C, 20 psi until 1.5 min, purge flow 50 mL/min at 2.0 min
- **MSD:** Source at 300 °C, Quadrupole at 150 °C, transfer line at 300 °C, scan range 200–1000 amu

Linearity is excellent across the range studied (0.5 ng/mL to 1,000 ng/mL, except for BDE-209 at 2.5 to 1,000 ng/mL range), giving R² values of 0.997 or greater in all cases and demonstrating highly inert surface of the column.
We Have the Most Inert Column, What about the Rest of the Flow Path??

Ultra Inert Liners
Basic Drug Suitability

**Higher response**

*Agilent UI single taper liner with wool (p/n 5190-2293)*

**Peaks:**
1. Oxycodone
2. Temazepam
3. Flunitrazepam
4. Heroin
5. Nitrazepam
6. Clonazepam
7. Alprazolam

Drug of abuse are shown on GC/MS SIM chromatograms 5 ng of checkout standards on column
Ultra Inert GC Inlet Liners Objective:

Equivalent or **Better** performance for:

Response Levels:  Highly inert surface for recovery at  *trace levels*

Robustness:  **Stability** of deactivation over time

Reliability:  **Reproducibility** and linearity

*Even when containing glass wool, Agilent **Ultra Inert GC liners** provide a robust, reproducible and reliable inert flow path for the analysis of difficult, active compounds at trace levels.*
Reliability / Quality Assurance:
Ultra Inert Liner Certificate of Performance

Lot to Lot Liner Reproducibility assured:
Each deactivation lot is Certified to ensure consistent and efficient coverage using both acidic and basic probes at trace (2 ng) levels on column

Certificate with every liner is printed on a label ready to peel and stick into analysts’ laboratory notebooks for easier compliance.

Traceability:
Deactivation Lot number is on Certificate Liner lot number (and part number) is permanently etched on glass
Take Home Message

• Raising the bar and setting a new industry standard for column inertness QC testing

• Best columns available for reactive analytes and trace analysis
  – Carefully selected Ultra Inert test mix for consistent column inertness
  – Excellent performance over a wide range of applications
  – Quality innovation to ensure the right answer the 1st time
  – Selectivity remains the same for consistent predictable separation
  – Low bleed profiles minimize interferences

• New Inert Liners to keep the entire flowpath INERT

• The bottom line
  – Highest and most consistent inertness performance

www.agilent.com/chem/ultrainert
References


Agilent/J&W Technical Support

800-227-9770 (phone: US & Canada)*

* Select option 3..3..1

866-422-5571 (fax)

email: gc-column-support@agilent.com

www.agilent.com/chem