Turning the Heat up on Gas Chromatography

How to Successfully Perform High Temperature GC Applications without Breaking a Sweat

Vanessa Abercrombie
GC Applications Chemist
How do you prepare for high temp GC?

➢ What temperature do you need to go up to?
➢ What supplies do you need?
➢ Are we using the right detector?
Temperature Range and Applications

- **Semi-volatiles**
  - Range: 100 °C to 350 °C

- **Volatile**
  - Range: -10 °C to 200 °C

- **Finished Petroleum Products**
  - Range: 200 °C to 350 °C

- **Industrial Chemicals**
  - Range: 40 °C to 400 °C

- **Flavor and Fragrances**
  - Range: 40 °C to 280 °C

- **Food Products**
  - Range: 40 °C to 280 °C

- **Crude Oil Residues**
  - Range: 280 °C to 400 °C

- **Waxes**
  - Range: 400 °C to 450 °C

- **Sterols**
  - Range: 400 °C to 450 °C

- **Triglycerides**
  - Range: 380 °C to 450 °C

**If you aren’t going above 350°C, you don’t need a high temperature column**
The Mystery of the MAOT

Max Temperature Range of the STATIONARY PHASE is independent of the CAPILLARY TUBING

\[ k = \frac{t_{\text{compound}} - t_{\text{methane}}}{t_{\text{methane}}} \]

\[ RI = 100 \times \left[ n + \frac{\log(t'_{r(\text{unknown})}) - \log(t'_{r(n)})}{\log(t'_{r(N)}) - \log(t'_{r(n)})} \right] \]
Gas Clean Filters: The First Line of Defense Against Leaks

P/N CP17973

www.agilent.com/chem/gasclean

New Smart Sensor
High temp + Leaks = Dead Column
Air Leaks = Increase in Column Bleed at High Temperatures
It’s what’s on the inside that counts….  
Max temperature of phase and how it degrades
Initial Checkout Test Mix

<table>
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<tr>
<th>Peak</th>
<th>Name</th>
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<tbody>
<tr>
<td>0</td>
<td>Methane</td>
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<tr>
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<td>Decane</td>
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<tr>
<td>2</td>
<td>1-Octanol</td>
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<tr>
<td>3</td>
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</tr>
<tr>
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</tr>
<tr>
<td>5</td>
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</tr>
<tr>
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</tr>
<tr>
<td>7</td>
<td>Tridecane</td>
</tr>
<tr>
<td>8</td>
<td>Methyl Decanoate</td>
</tr>
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</table>

**Column**: 30m x 0.25 mm x 0.10 μm

**Carrier**: Helium, constant flow, 1 mL/min

**Oven**: 90 °C (30 min), ramp 20 °C/min to 400 °C (60 min)

**Inlet**: MMI Split mode, split ratio 50:1, 300°C
Brand X-5ht Peak Symmetry Degradation

40 Hours at 400°C

Peak Name
0 Methane
1 Decane
2 1-Octanol
3 2,6-Dimethylphenol
4 2,6-Dimethylaniline
5 Naphthalene
6 1-Decanol
7 Tridecane
8 Methyl Decanoate

Graphs showing the degradation of various compounds over time at 400°C.
Column Efficiency Over 120 hours at 400°C

Brand X-5ht
30m x 0.25 mm x 0.10 µm

Agilent J&W DB-5ht
30m x 0.25 mm x 0.10 µm

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Turning up the Heat on Gas Chromatography
Column Efficiency at 430°C

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<td>0</td>
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<td>8</td>
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</table>

Tridecane

Brand X-5ht

Agilent J&W DB-5ht

Agilent Publication 5994-1013EN
Phase Degradation Increases Retention

$$k = \frac{t_{\text{compound}} - t_{\text{methane}}}{t_{\text{methane}}}$$

120 Hours at 400°C

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<tr>
<td>7</td>
<td>Tridecane (Highlighted)</td>
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Brand X-5ht

Agilent J&W DB-5ht
“Potholes” Created as the Phase Degrades
Raw Fused Silica exposed….

The more heat you add the more “potholes” you create
Thicker film will last longer…. But will eventually degrade at 400°C.

30m x 0.25 mm x 0.25 μm

Brand X-5ht

80 Hours at 400°C

60 Hours at 400°C

40 Hours at 400°C

20 Hours at 400°C

Initial

Tridecane

Agilent J&W DB-5ht

Brand X-5ht
The outside matters too….
Let’s talk about substrate
Columns by Temperature Limit

Don’t take fused silica columns above 400°C!

“Polyimide coatings, which originated in the aerospace industry, can tolerate temperatures of up to about 400 °C for brief periods…”

Don’t let this happen to you…..

Don’t Touch!!!!!!

Ahhhhh!!!!!
Competitor Fused Silica doesn’t always “take the heat”

Just because a company says their column can run at or above 400°C, doesn’t mean it can……..
Competitor Fused Silica doesn’t always “take the heat”

Agilent J&W DB-5ht

Brand Y
After 25 hours at 400°C
Agilent Publication 5994-1013EN
Can you tell which one is the Fused Silica and Which is the UltiMetal Column?

VF-5ht Fused Silica

VF-5ht UltiMetal

Tridecane

J&W VF-5ht Fused Silica

J&W VF-5ht UltiMetal

Agilent Publication 5994-1385EN
What is the best way to get heavy compounds off a column?

- Apply Heat
- Increase Column Flow
- Decrease Column Length
- Decrease Film Thickness
- Increase Diameter
Short, Thin Film Columns Elute Compounds Faster

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DB-5ht
Flow: 1 mL/min
15m x 0.25 mm x 0.10 μm

Flow: 1.7 mL/min
30m x 0.25 mm x 0.10 μm

Flow: 1 mL/min
15m x 0.25 mm x 0.10 μm

Flow: 1.7 mL/min
30m x 0.25 mm x 0.25 μm
Metal Columns Are Easy to Cut Properly

"Glass Infused" Metal Column

DB-HT-SimDis ProSteel

DB-HT-SIMDIS
5m x 0.535 mm x 0.15 μm

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<thead>
<tr>
<th>Peak</th>
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</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4-Chloro-3-Methylphenol</td>
</tr>
<tr>
<td>2</td>
<td>Tridecane</td>
</tr>
<tr>
<td>3</td>
<td>Dicyclohexylamine</td>
</tr>
<tr>
<td>4</td>
<td>Acenaphthene</td>
</tr>
<tr>
<td>5</td>
<td>1-Dodecanol</td>
</tr>
<tr>
<td>6</td>
<td>Pentadecane</td>
</tr>
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Agilent

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DE.5289814815
Lower bleed with Agilent ProSteel Columns

Column: Brand X-Metal SimDis
Agilent J&W DB-HT SimDis ProSteel
5m x 0.535 mm x 0.15 µm

Carrier: Helium, constant flow, 18 mL/min

Oven: 40 °C (0 min), Ramp 10°C/min to 430 °C (20 min)

Inlet: MMI Splitless mode, 100°C (0.5 min), Ramp 10°C/min to 430 °C

C50/C52 Resolution: 3.36

Brand X SimDis
Δ = 87.0 pA

J&W DB-HT SimDis
Δ = 17.3 pA

Agilent Publication 5994-1385EN
All the Polywax DB-HT SIM DIS

Polywax 500
Polywax 655
Polywax 1000

Agilent Publication 5994-1385EN
What do you do if you need to go above 400°C

- **Advanced Green Septa**
  - Long Life Septa
  - 35°C
- **Graphite/Vespel ferrules**
  - 35°C
- **Fluorocarbon O-rings**
  - 35°C
- **Ultra Inert liners**
  - 35°C
- **BTO Septa**
  - 350°C
- **Graphite ferrules**
  - 400°C
- **Ultimetal ferrules**
  - 450°C
- **Graphite seals**
  - 450°C
- **Standard liners**
  - 450°C

High Temp Applications
Advantages of MMI and “Cold” Splitless

- Larger injection volumes with lower detection limits
  - Lower solvent expansion
  - Less risk of back flash issues
- No needle discrimination
  - Improved recovery of high boiling compounds
- Less thermal exposure of compounds
  - Improved recovery of thermo-labile compounds
  - Less degradation

Lower detection limits → Better accuracy of data
Temperature Range, Applications, & **Detectors**

**Semi-volatiles**
- 100 °C to 350 °C

**Volatiles**
- -10 °C to 200 °C

**Finished Petroleum Products**
- 350 °C

**Industrial Chemicals**
- 40 °C

**Flavor and Fragrances**
- 40 °C

**Food Products**
- 40 °C

**Crude Oil Residues**
- 400/450 °C

**Waxes**
- 400 °C

**Polymer additives**
- 400 °C

**Dyes**
- 380 °C

**Sterols**
- 380 °C

**Triglycerides**
- 380 °C

**MSD/ FID/ ECD/ TCD/ FPD/ SCD**

**FID/SCD**
Just because you can do High Temp GCMS…. Should you?

GC Oven Max Temp 450 °C

Ion Source Max Temp 350 °C

Quads Max Temp 150 °C

V/G Ferrules Max Temp 350 °C

Do you really need Mass Spec?

- Shrink after thermal cycles creating leaks
- Leaks cause filament to burn out faster
- Condensation in Quads…..
Loss of Sensitivity & Loss of Inertness

Brand X-5ht over 45 hours at 400 °C
30m x 0.25 mm x 0.25 μm

Turning up the Heat on Gas Chromatography
MS UI Columns are better for Mass Spec than HT Columns

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<thead>
<tr>
<th>Peak</th>
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<tbody>
<tr>
<td>1</td>
<td>Propionic Acid</td>
</tr>
<tr>
<td>2</td>
<td>1-Octene</td>
</tr>
<tr>
<td>3</td>
<td>n-Octane</td>
</tr>
<tr>
<td>4</td>
<td>1,3-Propanediol</td>
</tr>
<tr>
<td>5</td>
<td>4-Methylpyridine</td>
</tr>
<tr>
<td>6</td>
<td>n-Nonane</td>
</tr>
<tr>
<td>7</td>
<td>Trimethylphosphate</td>
</tr>
<tr>
<td>8</td>
<td>n-Propylbenzene</td>
</tr>
<tr>
<td>9</td>
<td>1-Heptanol</td>
</tr>
<tr>
<td>10</td>
<td>3-Octanone</td>
</tr>
<tr>
<td>11</td>
<td>n-Decane</td>
</tr>
</tbody>
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Brand X-5ht
After 45 hours at 400°C

J&W DB-5ht

J&W DB-5MS UI

Turning up the Heat on Gas Chromatography
Summary

➢ Agilent J&W High temperature phase and Fused Silica are stable for extended time at 400°C

➢ If going to operate above 400°C use Deactivated Stainless Steel columns such as ProSteel and UltiMetal GC Columns

➢ Use the right consumables for high temperature applications
  ➢ Graphite
  ➢ Ultimetal ferrules
  ➢ BTO Septa
  ➢ Use regular liners

➢ High Temp Mass Spec is NOT Recommended
  ➢ Use Ultra Inert Mass Spec Columns for difficult Mass Spec Analysis
For More Information on High Temperature GC Columns

When the Heat Is On, Will Your GC Column Deliver?
Agilent high-temperature GC columns

Ready to lower the running costs of your high-temperature analysis?
Agilent J&W high-temperature GC columns feature a special polymer that can operate in temperatures of up to 450 °C. They deliver these cost-saving advantages:
- Less maintenance: Lower your downtime costs for high-performance with low bleed and the highest signal-to-noise ratios.
- Reduced maintenance: A proprietary stabilization technology minimizes detector contamination and stabilization time.
- Longer lifetime: Agilent high-temperature GC columns combine robustness with advanced surface treatment to maximize performance and column life.

For applications above 450 °C, Agilent J&W UltiDur and ProBond® GC columns offer added durability and ruggedness—even under extreme conditions. And they’re easy to cut, too.

Live webinar: Turning up the Heat on Gas Chromatography
What does “maximum operating temperature” really mean? What can happen to your GC column when this temperature is exceeded? How can you protect your GC column from oxygen contamination? You’ll learn the answers to these questions and more.

The difference is clear
Recently, we compared the performance of several high-temperature GC columns under the most extreme conditions.

www.agilent.com/chem/high-temp

5994-1013EN

Technical overview
Impact of Temperature on the Efficiency of High-Temperature GC Columns

Download
PDF / 1.1 MB

Technical overview
Successful High-Temperature GC Applications with Agilent J&W Deactivated Stainless Steel GC Columns

Download
PDF / 1.2 MB

Poster
Examination of the Efficiency on High-Temperature GC Columns and Strategies for Successful High-Temperature Applications

Download
PDF / 0.6 MB
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

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<thead>
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