It’s Peak Season for Great Peak Shape

Tips and tricks on troubleshooting in GC chromatography

Mark Sinnott
Alexander Ucci
6 August, 2020
“Everything Was Just Fine... and Then This Happened!”

“How do I troubleshoot?”

Track your actions/log book:

- Changed column, liner, septum, syringe, etc.
- Injected samples, other method, etc.
- Carried out maintenance, cut column, inlet flush, etc.

Logic

Something changed (slowly or suddenly)

= Something is different

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Logical Troubleshooting

Troubleshooting starts with isolating the problem.
• There are five basic areas from where problems can arise:
  - Injector
  - Flow
  - Column
  - Detector
  - Electronics
Or…
  - A combination of these

Knowing what can and cannot cause the symptom is the key, and most importantly **DON'T PANIC!**
Common Peak Shape Issues

• **Peak tailing** – flow path or activity
• **Bonus peaks** – in sample or back flash (carry-over)
• **Split peaks** – injector problems, mixed solvent
• **No peaks** – wasn’t introduced, wasn’t detected
• **Response changes** – activity, injector discrimination, detector problem
• **Peak fronting** – overload or solubility mismatch, injector problems
• **Shifting retention** – leaks, column aging, contamination, or damage
• **Loss of resolution** – separation decreasing, peak broadening
• **Baseline disturbances** – column bleed, contamination, electronics
• **Noisy or spiking baseline** – electronics or contaminated detector
• **Quantitation problems** – activity, injector, or detector problems
Peak Tailing

**Compound ID**

1. Methane
2. Decanal
3. Propionic acid
4. Ethylene glycol
5. Heptadecane
6. Aniline
7. Methyl dodecanoate
8. 2-Chlorophenol
9. 1-Undecanol
10. Nonadecane
11. 2-Ethylhexanoic acid
12. Ethyl maltol

**Injector or column is active**

- Reversible adsorption of active compounds (-OH, -NH, -SH)

**Flow problem**

- Dead volume, obstruction, poor installation, or severe column contamination

**Miscellaneous** - overloading of PLOT columns, co-elution, polarity mismatch between phase, solute or solvent, and some compounds always tail

*Tip = Inject a light hydrocarbon, should not tail unless flow path problem.*
Agilent Inert Flow Solution

Modified Agilent J&W DB-WAX UI mix on DB-WAX UI, 122-7032UI

*Every column is tested individually

<table>
<thead>
<tr>
<th>Compound ID</th>
<th>Compound Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>*</td>
<td>Methane</td>
</tr>
<tr>
<td>1</td>
<td>5-Nonanone</td>
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<tr>
<td>2</td>
<td>Decanal</td>
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<tr>
<td>3</td>
<td>Propionic acid</td>
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<td>Ethylene glycol</td>
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<td>5</td>
<td>Heptadecane</td>
</tr>
<tr>
<td>6</td>
<td>Aniline</td>
</tr>
<tr>
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<td>Methyl dodecanoate</td>
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<td>12</td>
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</tr>
</tbody>
</table>

Brochure 5991-6709EN
Agilent Inert Flow Solution

Agilent Ultimetal Plus inlet weldment, shell and transfer lines

Agilent Ultra Inert inlet liner

Agilent Ultimetal Plus ferrules

Agilent Ultimetal Capillary Flow Technology Devices, Ultimate union

Agilent Ultra Inert gold seal

Agilent J&W Ultra Inert GC column

Agilent Ultimetal Plus– TCD, FPD, NPD/FID jets

5990-8532EN brochure
Bonus or Ghost Peaks

Contamination in injector, column or flow (carrier gas)

- Carry-over from a backflash or previous sample
- Bad tank of gas or traps have expired
- Septum bleed

**Tip:** Run a blank run…it should be blank!
What Are These Repeating Peaks?

Septa contamination in wash vials or inlet liners can be diagnosed by looking for siloxane polymers in your total ion chromatogram. Each peak in the chromatogram corresponds to a cyclized (ring structure) siloxane molecule. These molecules fragment with very similar patterns.

Example spectrum:
Multiple Injections from the Same Vial: Siloxanes!

Run 1
Run 6
Does Your Baseline Look Like This? Do You See Extra Peaks?
If your target ions are buried beneath matrix peaks, it might be time to trim the column or do sample clean-up.

The Matrix

- 500 ppb ethenoprox in black tea
- 10 ppb linalool in shampoo

...(or improve your sample cleanup)
The Importance of Sample Cleanup

For sample cleanup help, please contact us!  spp-support@agilent.com
Split Peaks

Injector (poor sample introduction)
- Injecting the sample twice (somehow?)
- Mixed sample solvent (polarity difference)
- Sample in syringe needle (manual inject)

Injector (activity)
- Breakdown (not really a split peak, two peaks)
- Sample degradation in injector

Volatility
- High boilers dropping out on cold spots
- Transfer line temperatures
- Unions or fittings not tracking column temperature

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No Peaks

- Detector (not on, or not operational)
- Injector (not working)
- Plugged syringe/plunger not moving
  - Wrong injector (or detector)
  - Huge leak (older systems)
  - No carrier gas flow

Not the column unless…
  - Broken column or no column
Peak Response
All change in size

Injector
- Leaky syringe
- Split ratio set incorrectly
- Wrong purge activation time
- Septum purge flow too high
- Injector temperature too low*

Detector (response problem)
- Settings or flows changed
- Electronics failing

*Tip: Ask is it all of them or some of them, if all then injector or detector
Injector or column is active/contaminated

- Irreversible adsorption of active compounds (-OH, -NH, -SH)

Decomposition of sample

- Temperature change – Discrimination
- Evaporation from sample
Change in Response: Pyraclostrobin in Spinach on Run 65
Change in Response: Pyraclostrobin in Spinach on Run 1 vs Run 65

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Change in Response: Pyraclostrobin in Spinach with New Liner
Peak Fronting
Shark fin-shaped or just slight

- Column (contaminated)
  - Overload (more pronounced with large solute and phase polarity differences)

- Injector
  - Compound very soluble in injection solvent (need retention gap)
  - Mixed sample solvent

- Other
  - Co-elution
  - Breakdown
Retention Time Shift

Injector
- Leak in the septum
- Change in injection solvent
- Large change in sample concentration

Flow
- Change in gas velocity

Column
- Contamination
- Damaged stationary phase
- Loss of stationary phase
- Change in temperature
# Thermal Stability and Retention Time Shifting on Standard WAX Column

<table>
<thead>
<tr>
<th>Peak</th>
<th>Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methanol</td>
</tr>
<tr>
<td>2</td>
<td>Benzene</td>
</tr>
<tr>
<td>3</td>
<td>Toluene</td>
</tr>
<tr>
<td>4</td>
<td>Ethylbenzene</td>
</tr>
<tr>
<td>5</td>
<td>p-Xylene</td>
</tr>
<tr>
<td>6</td>
<td>m-Xylene</td>
</tr>
<tr>
<td>7</td>
<td>o-Xylene</td>
</tr>
</tbody>
</table>

**Initial BTEX Injection**
- 50 hours at 280°C
- 25 hours at 280°C

Application note 5991-9035EN
Initial Injection

- 100 hours at 280°C
- 75 hours at 280°C
- 50 hours at 280°C
- 25 hours at 280°C

Application Note 5991-9035

**Peak Compound**

- 1 Methanol
- 2 Benzene
- 3 Toluene
- 4 Ethylbenzene
- 5 p-Xylene
- 6 m-Xylene
- 7 o-Xylene
Loss of Resolution

Resolution is a function of separation and peak width.
Loss of Resolution - Separation Decrease (RT's change)

Column
  - Different column temperature
  - Contamination (more phase?)
  - Matrix components coeluting

Flow
  - Change in velocity?

Separation

Peak width
Loss of Resolution - Peak Broadening (RT's unchanged)

Flow
- Make-up gas

Column
- Contamination
- Phase degradation

Injector (efficiency)
- Settings, liner, installation, etc.
Peak Broadening: Omethoate in Avocado in Run 1
Peak Broadening: Omethoate in Avocado in Run 65
Peak Broadening: Omethoate in Avocado in Run 1 versus Run 65
Peak Broadening: Recover Peak Shape with New Liner
Peak Broadening: The Case of the Wrong Liner
Baseline Disturbances
Sudden changes, wandering, or drifting

Drifting/wandering/weird disturbances

Column or detector
- Not fully conditioned or stabilized (electronics)
- Contamination

Flow
- Changes in carrier and/or detector gas flows
- Valves switching, leaks
Noisy Baseline

Flow
- Contaminated gas
- Incorrect detector settings

Column
- Bleed if at high temperature
- In detector flame (poor installation)

Detector
- Air leak - ECD, TCD
- Electronics malfunction
Spiking Baseline

Detector
- Particles entering the detector
- Random: poor connection
- Regular: nearby "cycling" equipment (electronics)

Application note 5991-2975EN
Quantitation Problems

Detector
• Poor stability (electronics) or baseline disturbances (contamination)
• Outside detector's linear range or wrong settings
• Integration parameters

Activity (adsorption) in injector or column

Injector
• Technique, settings, conditions
• Syringe worn

Other
• Co-elution
• Matrix effects
• Sample evaporation – leaky vials
• Sample decomposition
What is Not Caused by a Column?

Not responsible

• Peaks
  - Any reproducible sharp chromatographed peak
• Siloxanes (even though it looks like bleed spectrally)
• Degradation product peaks: Endrin Aldehyde, endrin ketone, DDE, DDD…
• Carry-over of sample compounds
• Splitting of peaks
Troubleshooting Tools

Bleed profile (non-injection): baseline problems
Inject a nonretained peak: peak shape problems
Test mix: all problems
Isolate the components: all problems

Condensation test: baseline problems
Jumper tube test: baseline problems
Generating a Bleed Profile

Produce when the column is new (for future reference) when there is a baseline problem

(Simply remove syringe from ALS)

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.
Inject a Nonretained Compound to Check Flow Path

Used to check flow path

Potential explanations:
• Injector or septum leak
• Too low of a split ratio
• Liner problem
  - (broken, leaking, misplaced)
• Column position in injector and detector
Test Mix – Make your Own!

A test mix is used to determine how “good” the column is, or if the problem is related to the chemical properties of the analytes.

It is simplest to use your own standard.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrocarbons</td>
<td>Efficiency Retention</td>
</tr>
<tr>
<td>Alcohols</td>
<td>Activity</td>
</tr>
<tr>
<td>FAME's, PAH's</td>
<td>Retention</td>
</tr>
<tr>
<td>Acids</td>
<td>Acidic Character Activity</td>
</tr>
<tr>
<td>Bases</td>
<td>Basic Character Activity</td>
</tr>
</tbody>
</table>

**Test Conditions**

- **Inlet:** Split (250°C)
- **Detector:** FID(320°C)
- **Flow:** 37.3 cm/sec (1.8 mL/min)
- **Carrier gas:** Hydrogen
- **Holdup compound:** Methane (0.671-min)
- **Temperature program:** Isothermal (110°C)
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 (continental US only as of now)
- Custom standard solutions including our new online custom quoting tool, enabling customers to upload recipe formulations to and to modify the recipe before submitting it
  - Tool will allow customers to see the quote pricing instantly and allow them to check quote pricing based on quantity range
- Rigorously tested and manufactured under ISO 9001, ISO 17025, and ISO 17034 certifications
- Sample preparation materials, columns, supplies, instrumentation, and reference materials from a single source
Agilent J&W DB-624 Column

QC Test Mix

Column: Agilent J&W DB-624  
30 m x 0.53 mm id, 3.0 µm

Carrier: Helium at 40 cm/sec  
measured at 35 °C

Injector: Direct, 260 °C

Detector: FID, 300 °C

Oven: 35 °C for 1.50 min  
30 °C/min to 65 °C for 10 min

1. 1,2-Dichloropropane  
2. Octane  
3. Tetrachloroethylene  
4. Chlorobenzene  
5. Nonane

Time (min.)

2.71 7.43 10.92 12.49 17.42 20.78
Example of Column Contamination

Agilent J&W DB-624 QC Test Mix
After 75 injections of oily sample

Time (min.)

<table>
<thead>
<tr>
<th>Time</th>
<th>Value</th>
</tr>
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<tbody>
<tr>
<td>2.21</td>
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<tr>
<td>3.30</td>
<td>1.4e4</td>
</tr>
<tr>
<td>6.03</td>
<td>1.3e4</td>
</tr>
<tr>
<td>9.26</td>
<td>1.2e4</td>
</tr>
<tr>
<td>10.46</td>
<td>1.1e4</td>
</tr>
<tr>
<td>14.40</td>
<td>1.0e4</td>
</tr>
<tr>
<td>17.86</td>
<td>9.0e3</td>
</tr>
</tbody>
</table>

*Temperature program: 35°C hold 1.50 min // 30°/min to 65°C, hold 10 min*

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Example of Column Contamination

*Before column rinse and bake
Temperature program // 35 °C hold 1.50 min // 30° C/min to 65 °C, hold 10 min

Removed $1\frac{1}{2}$ m from injector end
Example of Column Contamination

QC test mix to upper temperature limit

1 1/2 m removed*

*Before column bake
Temperature program // 35 °C, hold 1.50 min // 30 °C/min to 65 °C, hold 15 min // 20 °C/min to 260 °C, hold 50 min

We have more semivolatile contamination!
Condensation Test

Used to isolate the cause of*:

- Erratic baselines
- Ghost peaks or carry-over

*Use when problems are worse after periods of GC non-use
Condensation Test

Procedure:

• Leave GC at 40-50 °C for > 8 hours

• Blank run

• Repeat a blank run immediately after the first blank run is complete

• Compare the two blank runs
Condensation Test

Results

First blank run is worse

• Contaminants (from injector, lines, traps or carrier gas) carried into the column

• Blank runs the same: *contaminants are not strongly focused on the front of the column*
Jumper Tube Test

Purpose

• Helps to locate the source of contamination or noise

• Isolates GC components
Jumper Tube Test

Isolate the detector

• Remove column from the detector
• Cap detector and turn on
• Blank run
Jumper Tube Test

Isolation of detector – results:

Detector OK

Detector is the problem
Jumper Tube Test

Isolate the injector

- Connect the injector and detector
  - 1-2 meters deactivated fused silica tubing
- Turn on carrier gas
- Blank run
Jumper Tube Test

Isolate the injector – results:

Injector OK

Injector, lines or carrier gas contaminated
Jumper Tube Test

Isolate the column

• Re-install the column

• Set up as before

• Blank run
Jumper Tube Test

Isolate the column – results:

• Problem returns? It’s the column

• Problem gone? Previous leak, solid debris, or installation problem
Troubleshooting Example
Problem: No Peaks with Semivolatiles Checkout Mixture

Everyone needs to have a reference checkout sample that they can use to confirm whether their system is OK.

What my TIC looked like:

What my TIC should look like:
Problem: No Peaks with Semivolatiles Checkout Mixture

What could cause this?

- The wrong vial was injected
- The sample has degraded
- The inlet is leaking
- The column is damaged
Problem: No Peaks with Semivolatiles Checkout Mixture

What could cause this?

- The wrong vial was injected: **Sequence and vial checked, no problem found**
- The sample has degraded
- The inlet is leaking
- The column is damaged
Problem: No Peaks with Semivolatiles Checkout Mixture

What could cause this?

- The wrong vial was injected: **Sequence and vial checked, no problem found**
- The sample has degraded: **A new vial of standard was used, no difference observed**
- The inlet is leaking
- The column is damaged
Problem: No Peaks with Semivolatiles Checkout Mixture.

What could cause this?

- The wrong vial was injected: **Sequence and vial checked, no problem found**
- The sample has degraded: **A new vial of standard was used, no difference observed**
- The inlet is leaking: **A tune was performed. O₂, N₂, and H₂O levels were normal**
- The column is damaged

![Graph of Counts vs. Acquisition Time](image-url)
Problem: No Peaks with Semivolatiles Checkout Mixture

What could cause this?

- The wrong vial was injected: Sequence and vial checked, no problem found
- The sample has degraded: A new vial of standard was used, no difference observed
- The inlet is leaking: A tune was performed. O₂, N₂, and H₂O levels were normal
- The column is damaged: “Well, I guess I need to replace my column”
Problem: No Peaks with Semivolatiles Checkout Mixture

What could cause this?

- The wrong vial was injected: **Sequence and vial checked, no problem found**
- The sample has degraded: **A new vial of standard was used, no difference observed**
- The inlet is leaking: **A tune was performed. O2, N2, and H2O levels were normal**
- The column is damaged: Well, I guess I need to replace my column

**WAIT**

Test (a few more things) before you replace!
Follow a Logical Troubleshooting Procedure!

Start

Step 1: What does a working GC/MS look like?

Step 1: Half-split the problem

Step 1: Make repairs, as necessary

Step 1: Put the system back together

Step 1: Develop steps to prevent re-occurrence

Finish
Troubleshooting Step 1: What is the “Working System”? What are the components of the GC/MS system (follow the sample flow-path)
• Agilent 7693A autosampler + 10 μL syringe
• Agilent 7890B GC
• Agilent MultiMode Inlet (with CO₂ cryo)
• Agilent J&W HP-5ms UI, 30 m x 0.25 mm x 0.25 μm
• Agilent 5977A Series Extractor GC/MSD
Troubleshooting Step 1: What is the “Working System”? 

Compare your current data to known good data, when possible. Use over-lay to zero-on on differences

- How does your background compare to normal?

- Does the problem occur for every run, every analyte, every method? Only affects certain samples/analytes/Instruments?

- Are the peaks smaller or larger than normal?

- Is the peak shape gaussian, or are the peaks splitting, tailing, or saturated?
Compare your current data to known good data, when possible.

- How does your background compare to normal?
  
  Background looked a LOT bigger than peaks in the good TIC

- Does the problem occur for every run, every analyte, every method?
  
  Only affects certain samples/analytes?
  
  Occurring on all checkout sample runs attempted

- Are the peaks smaller or larger than normal?
  
  Definitely smaller

- Is the peak shape gaussian, or are the peaks splitting, tailing, or saturated?
  
  Let’s find out
Troubleshooting Step 1: What is the “Working System”?

Compare your current data to known good data. Now, the data is much clearer, and the background is not significantly higher.

Signals in separate scales:

- **Good, previous run from April**
  - Counts vs. Acquisition Time (min)
  - Counts: $8.0 \times 10^6$

- **Bad, recent run**
  - Counts vs. Acquisition Time (min)
  - Counts: $8.0 \times 10^5$
Troubleshooting Step 1: What is the “Working System”?

Compare your current data to known good data. Now, the data is much clearer, and the background is not significantly higher.

Signals with linked Y axis:

Similar background profiles

8.0 x 10^6

8.0 x 10^6

Bad, recent run
Think of a set of tests that will break the system into smaller pieces.

1. Try a new sample.
2. Tune the MS to half-split the detector from the GC.
3. Perform a manual injection with a new syringe to split autosampler and inlet/column.
What does a working GC/MS look like?

Half-split the problem

Make repairs, as necessary

Put the system back together

Develop steps to prevent re-occurrence

Think of a set of tests that will break the system into smaller pieces.

1. Try a new sample.

2. Tune the MS to half-split the detector from the GC.

3. Perform a manual injection with a new syringe to split autosampler and inlet/column.
Think of a set of tests that will break the system into smaller pieces

1. Try a new sample.
2. Tune the MS to half-split the detector from the GC.
3. Perform a manual injection with a new syringe to split autosampler and inlet/column.
Let’s focus on the autosampler and syringe:

While sample was new, what is the solvent? Dichloromethane

What kind of syringe? Agilent 10 µL syringe, 23-26s/42/cone (G4513-80204)

Does the autosampler work? Autosampler turns and moves plunger up and down

Does the syringe pull up liquid? No, it doesn’t

We may have found the problem!
Troubleshooting Step 3: Make the Repair

Replace the syringe with a 10 μL PTFE tipped plunger syringe (G4513-80203) – a much easier repair than venting and changing the column.

PTFE tipped syringes are more chemically resistant and offer a reduced chance of carry over and longer syringe lifetime.

Proper syringe maintenance must still be performed. Clean and refill syringe wash vials frequently.

Beware highly concentrated samples and samples with particulates (organic material, salts, etc.)
Troubleshooting Step 4: Put the System Back Together

What happened with a new syringe?

SUCCESS!
Have a Good Troubleshooting Story? Let Us Know!

Please call or email us today to share a troubleshooting success story or if you need help troubleshooting!
Troubleshooting Tips

1. Isolate the problem – half-split the system into its component parts
   (blank run, inject unretained compound, jumper tube test)
2. Change only one variable at a time
3. Compare before/after chromatograms
   (Peak shape, response, retention, baseline rise, background, look for trends, etc.)
4. Utilize technical support
Remember

Complete system = carrier gas + injector + column + detector + data system

• Multiple cause and effect
• Do not change too many variables at once
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
“Everything was Just Fine and then this Happened!”
“How do I go about Troubleshooting?”

Logic = Something changed (slowly or sudden) = Something is different

Track events- log book
- Changed column, liner, septum, syringe, etc.
- Injected samples, other method, etc.
- Did maintenance, cut column, inlet flush, etc.
Hexane blanks (testing vial storage over time)

Hexane Solvent Impurities

Immediate
1 day
1 week
If GC/MS was off for 1+ week (no carrier gas flow)...

System “open” for 1+ week

TIC looks okay (I think). How does it compare to a previous run of the same sample?
If GC/MS was off for 1+ week (no carrier gas flow)… zoom out

“Open system” TIC is ~10x lower than good run in the previous month. What happens if we replace the column and line...
If GC/MS was off for 1+ week (no carrier gas flow)...

“Open system” TIC is ~10x lower than good run in the previous month. What happens if we replace the column and liner?

System “open” for 1+ week (Dec)
System under vacuum (Oct)
Recover peak response with new column and liner

Try a new liner and re-conditioning column first. If response doesn’t recover, a new column may be required.

System “open” for 1+ week (Dec)
System under vacuum (Oct)
New column, liner, gold seal (Dec)
Brand X-5ht Peak Symmetry Degradation

40 Hours at 400°C

<table>
<thead>
<tr>
<th>Peak</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Methane</td>
</tr>
<tr>
<td>1</td>
<td>Decane</td>
</tr>
<tr>
<td>2</td>
<td>1-Octanol</td>
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<tr>
<td>3</td>
<td>2,6-Dimethylphenol</td>
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<td>4</td>
<td>2,6-Dimethylaniline</td>
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<td>5</td>
<td>Naphthalene</td>
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<tr>
<td>6</td>
<td>1-Decanol</td>
</tr>
<tr>
<td>7</td>
<td>Tridecane</td>
</tr>
<tr>
<td>8</td>
<td>Methyl Decanoate</td>
</tr>
</tbody>
</table>

Tridecane Tailing Factor

Brand X-5ht

DB-5ht
Column Efficiency Over 120 hours at 400°C

<table>
<thead>
<tr>
<th>Peak</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Methane</td>
</tr>
<tr>
<td>1</td>
<td>Decane</td>
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<tr>
<td>2</td>
<td>1-Octanol</td>
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<tr>
<td>3</td>
<td>2,6-Dimethylphenol</td>
</tr>
<tr>
<td>4</td>
<td>2,6-Dimethylaniline</td>
</tr>
<tr>
<td>5</td>
<td>Naphthalene</td>
</tr>
<tr>
<td>6</td>
<td>1-Decanol</td>
</tr>
<tr>
<td>7</td>
<td>Tridecane</td>
</tr>
<tr>
<td>8</td>
<td>Methyl Decanoate</td>
</tr>
</tbody>
</table>

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
Column Efficiency at 430°C

<table>
<thead>
<tr>
<th>Peak</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Methane</td>
</tr>
<tr>
<td>1</td>
<td>Decane</td>
</tr>
<tr>
<td>2</td>
<td>1-Octanol</td>
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Agilent J&W DB-5ht

Brand X-5ht
**Phase Degradation Increases Retention**

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

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**120 Hours at 400°C**

**Initial**

**Tridecane "k"**

**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
Recover peak shape with new liner (black)
Recover peak shape with new liner (black)