

# 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?

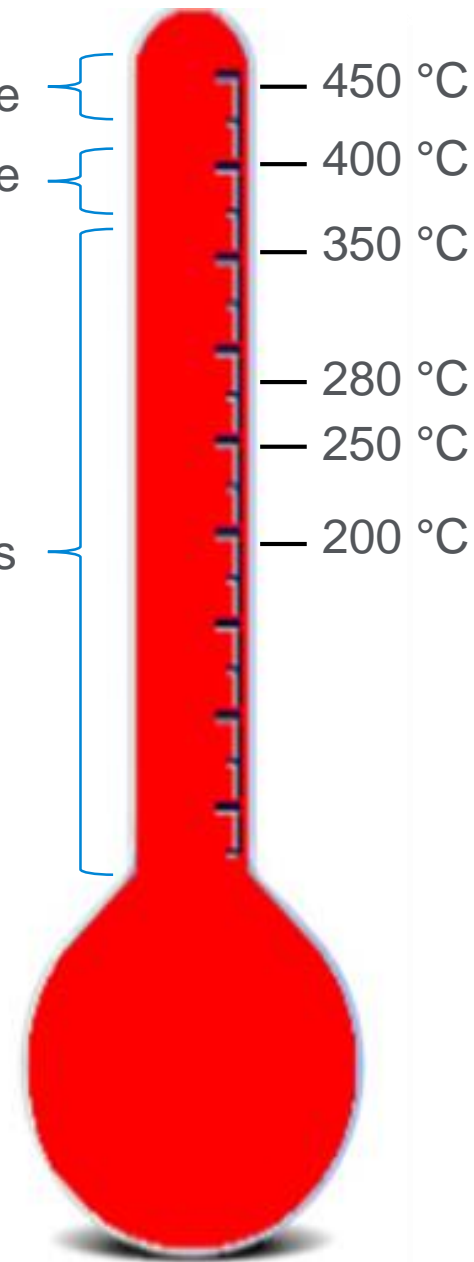


Extreme high temperature

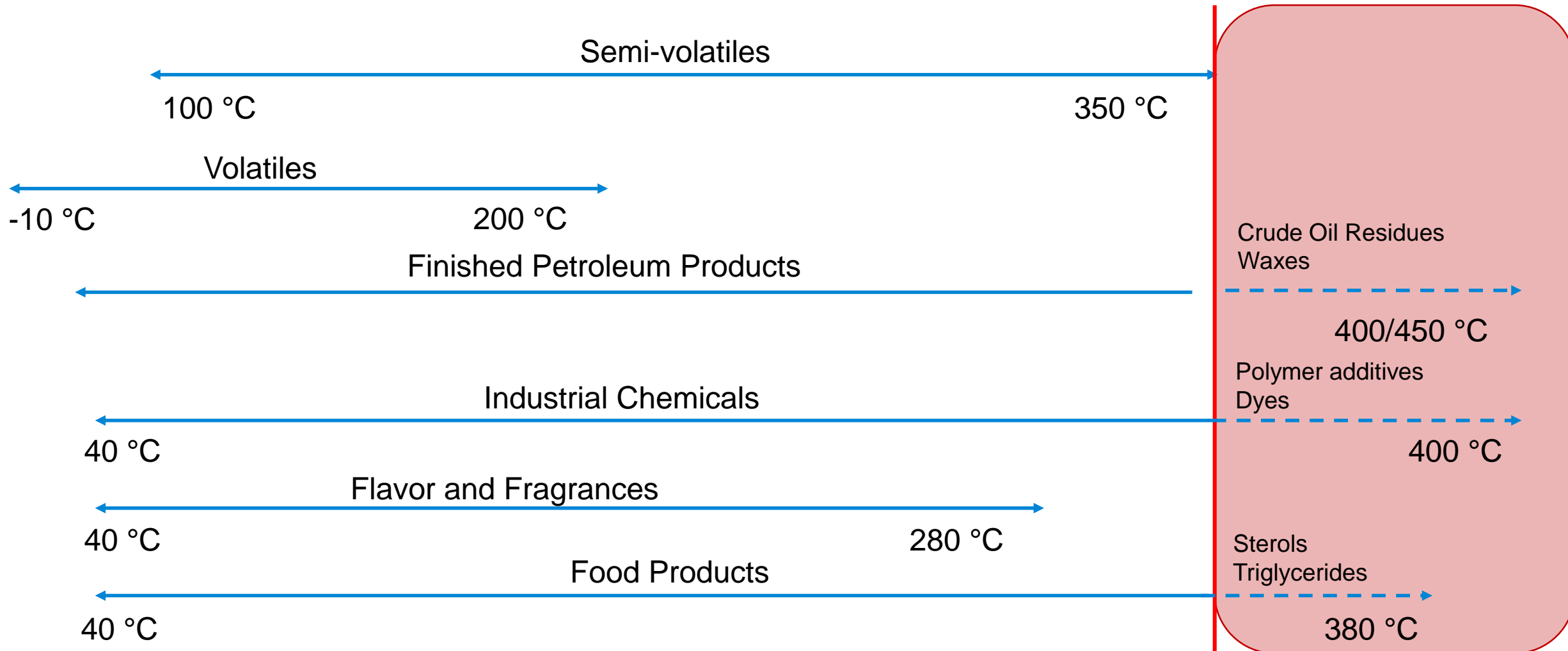
High temperature



Traditional GC temperatures



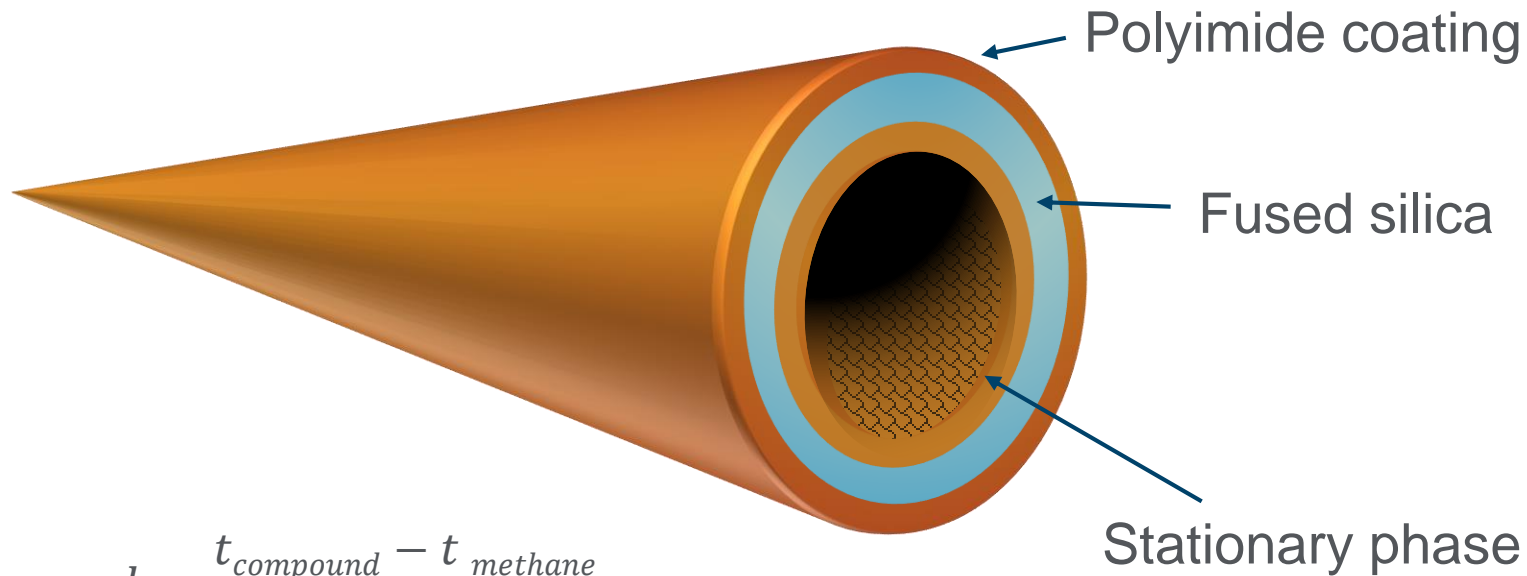
# Temperature Range and Applications



**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



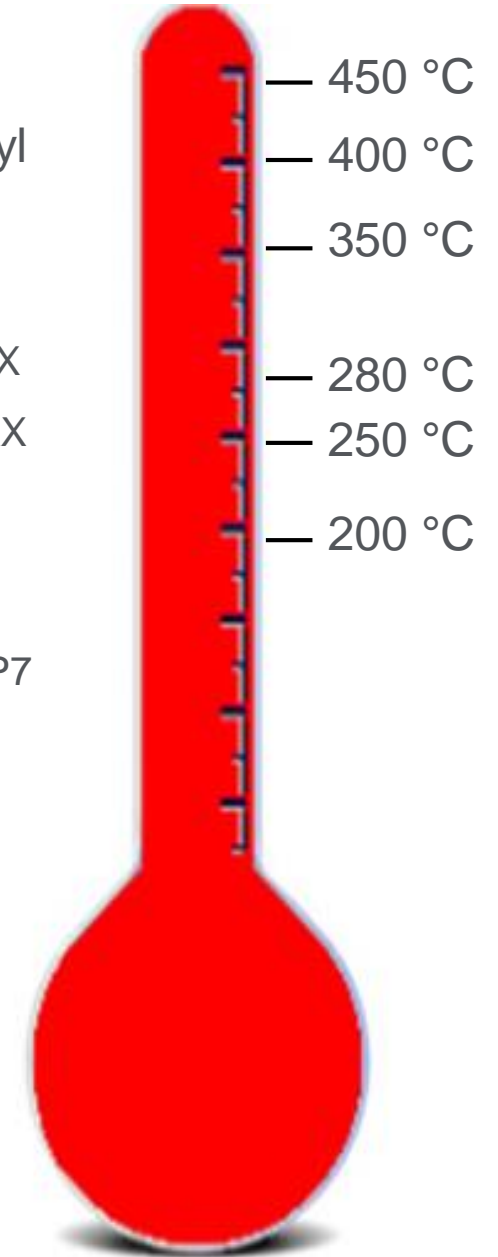
$$k = \frac{t_{\text{compound}} - t_{\text{methane}}}{t_{\text{methane}}}$$

$$RI = 100 \times \left[ n + \frac{\log(t'_{r(\text{unknown})}) - \log(t'_{r(n)})}{\log(t'_{r(N)}) - \log(t'_{r(n)})} \right]$$

PDMS  
5% Phenyl

DB-HeavyWAX  
Traditional WAX

Carboplot P7



# Gas Clean Filters: The First Line of Defense Against Leaks

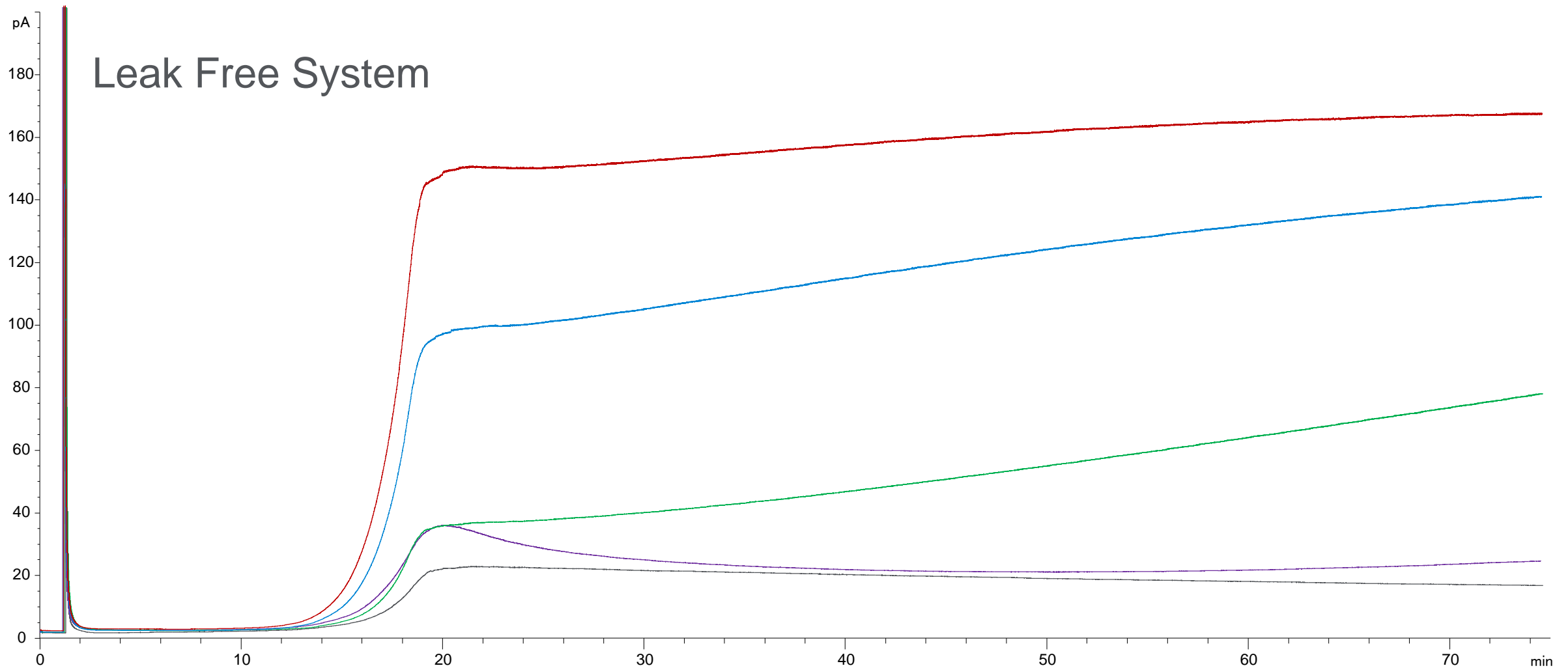


P/N CP17973

[www.agilent.com/chem/gasclean](http://www.agilent.com/chem/gasclean)

# High temp + Leaks = Dead Column

## Air Leaks = Increase in Column Bleed at High Temperatures



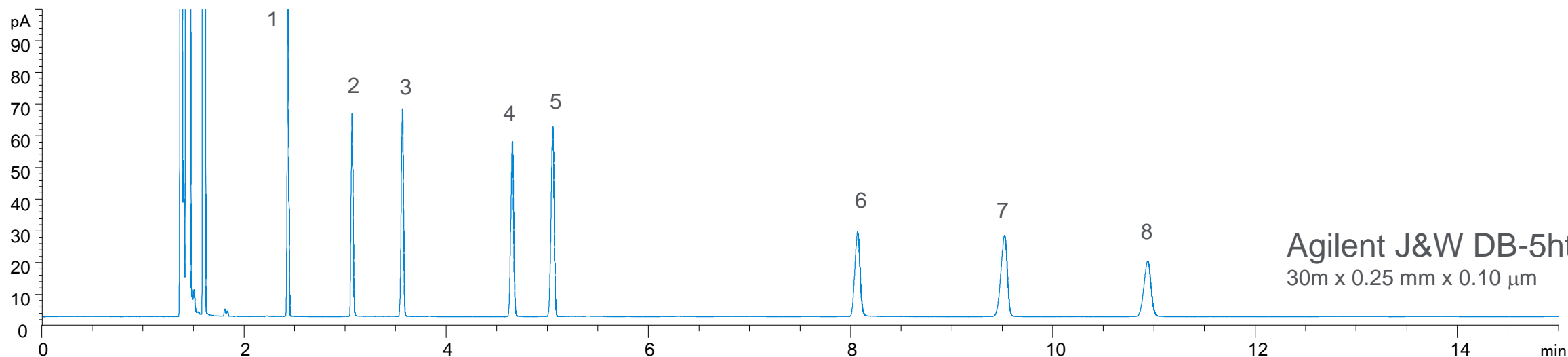
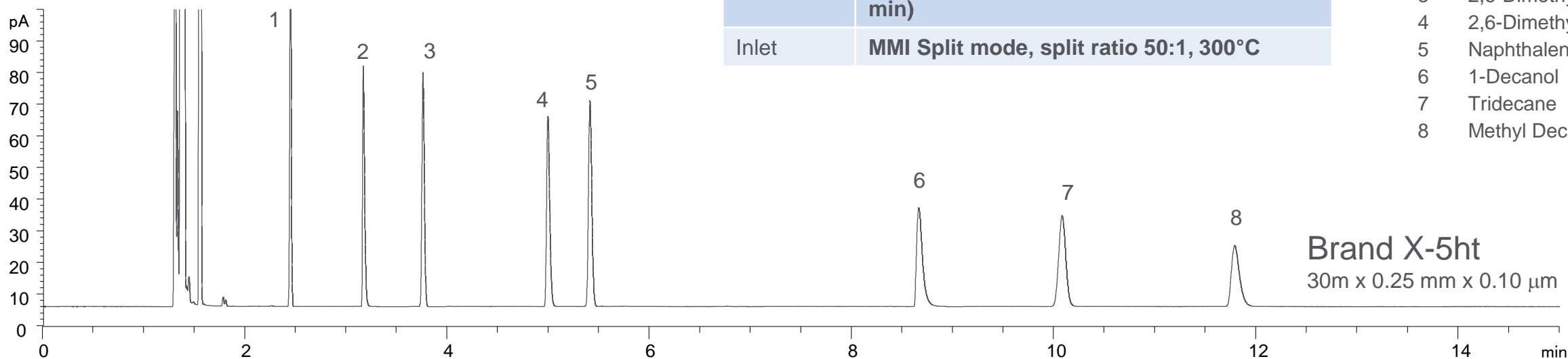
It's what's on the inside that counts....

Max temperature of phase and how it degrades

# Initial Checkout Test Mix

Column	30m x 0.25 mm x 0.10 $\mu$ m
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

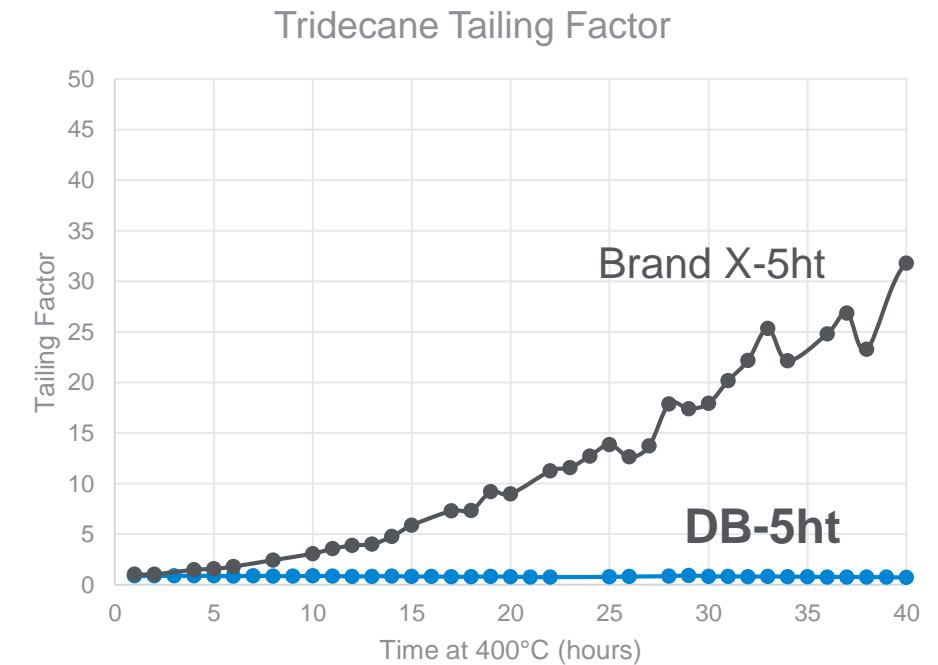
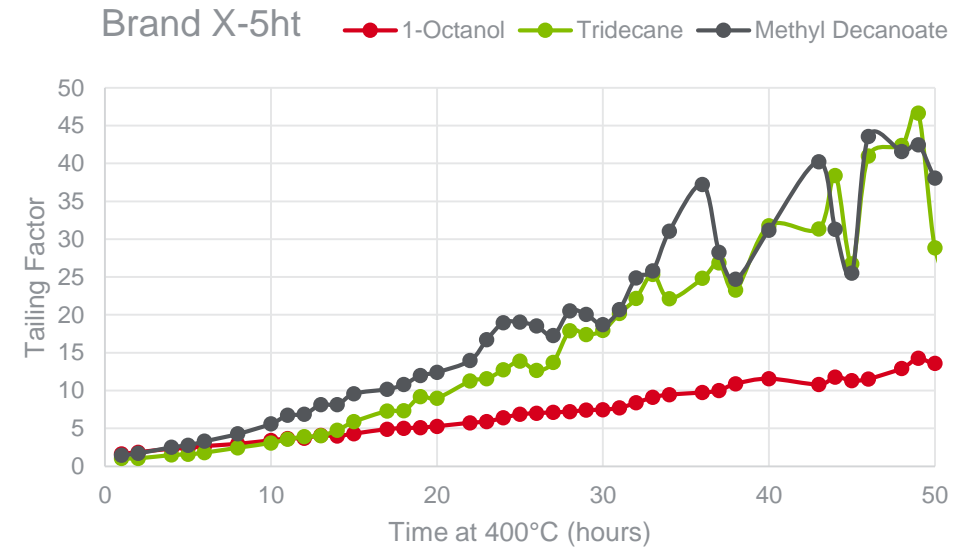
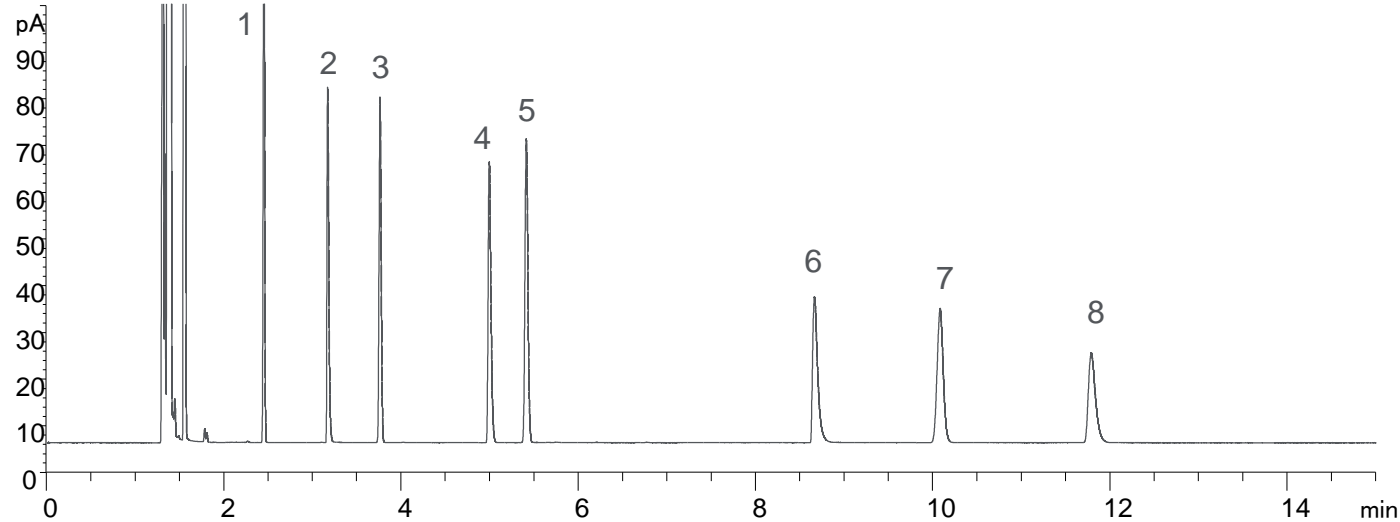
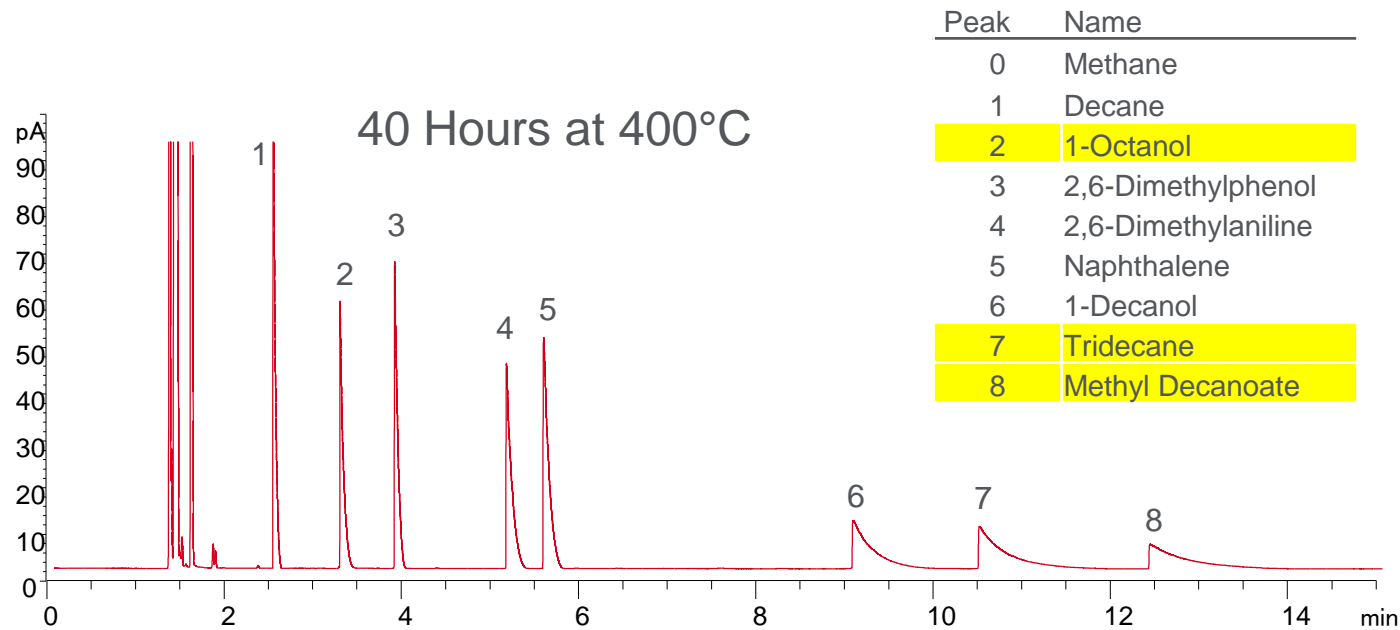
Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate



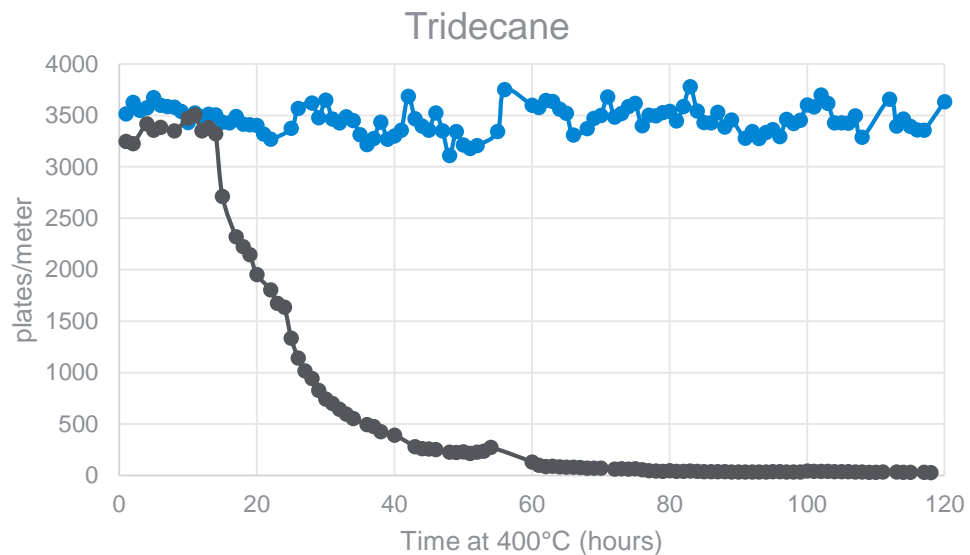
Agilent Publication 5994-1013EN



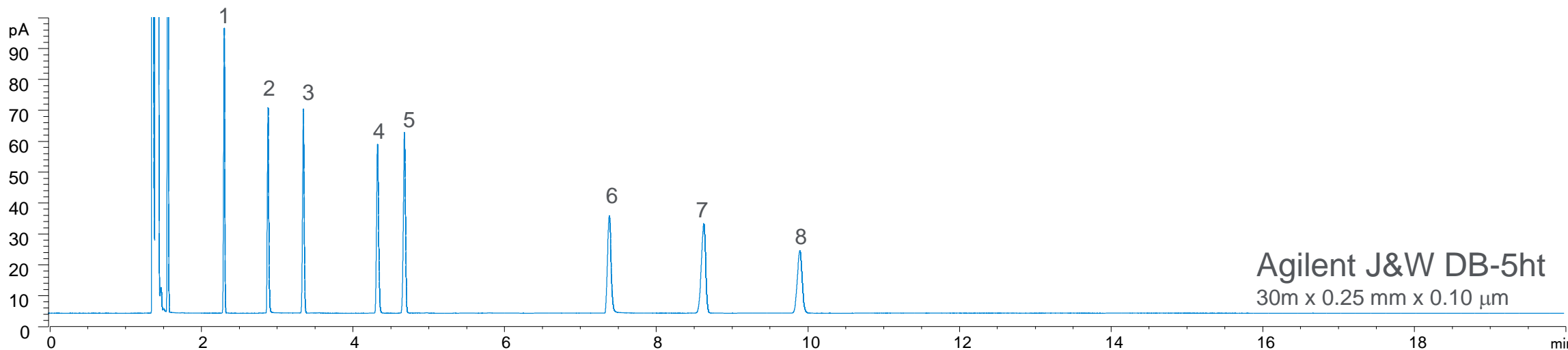
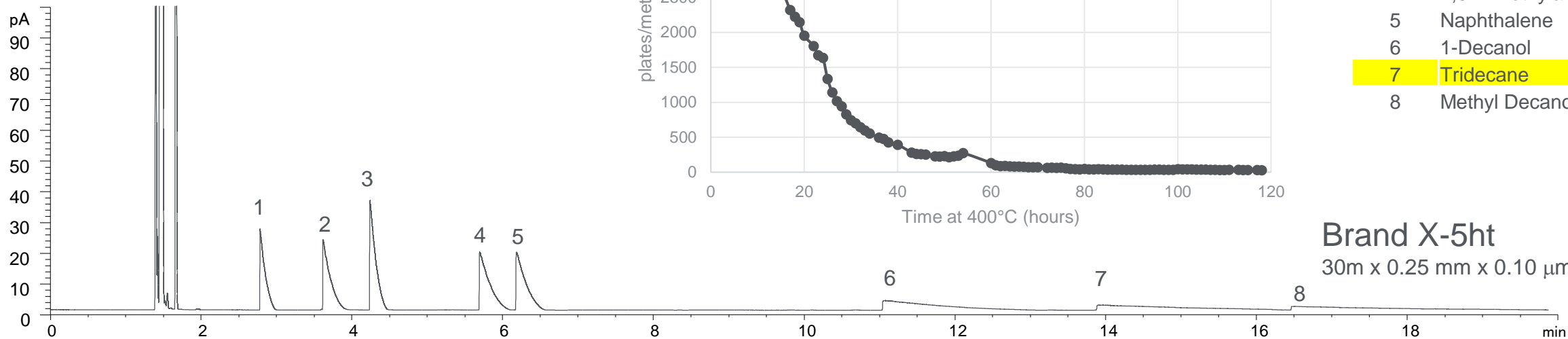
# Brand X-5ht Peak Symmetry Degradation



# Column Efficiency Over 120 hours at 400°C

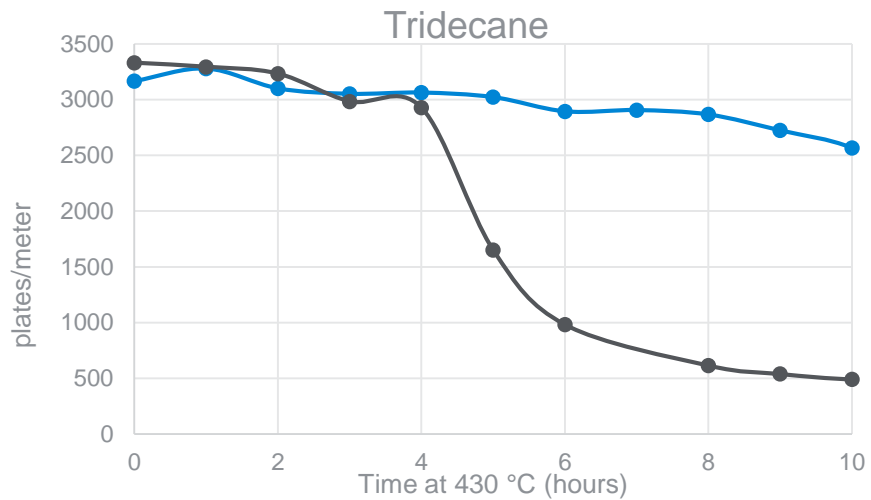
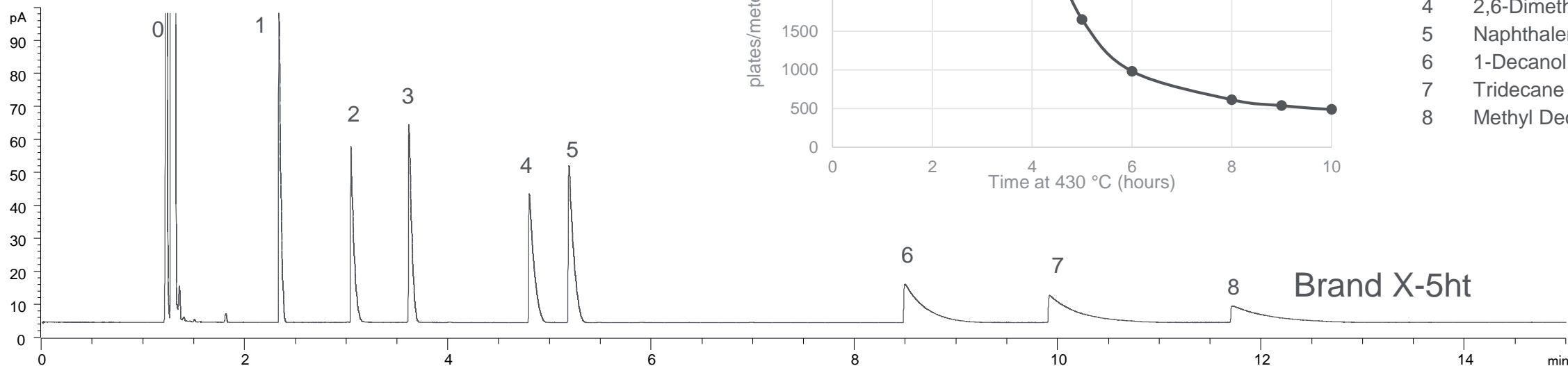


Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate

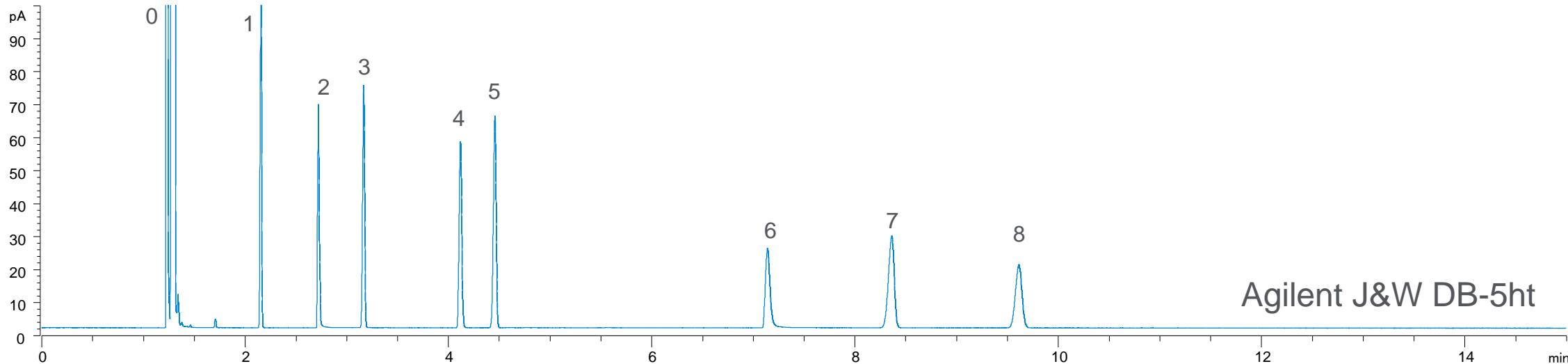


Agilent Publication 5994-1013EN

# Column Efficiency at 430°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

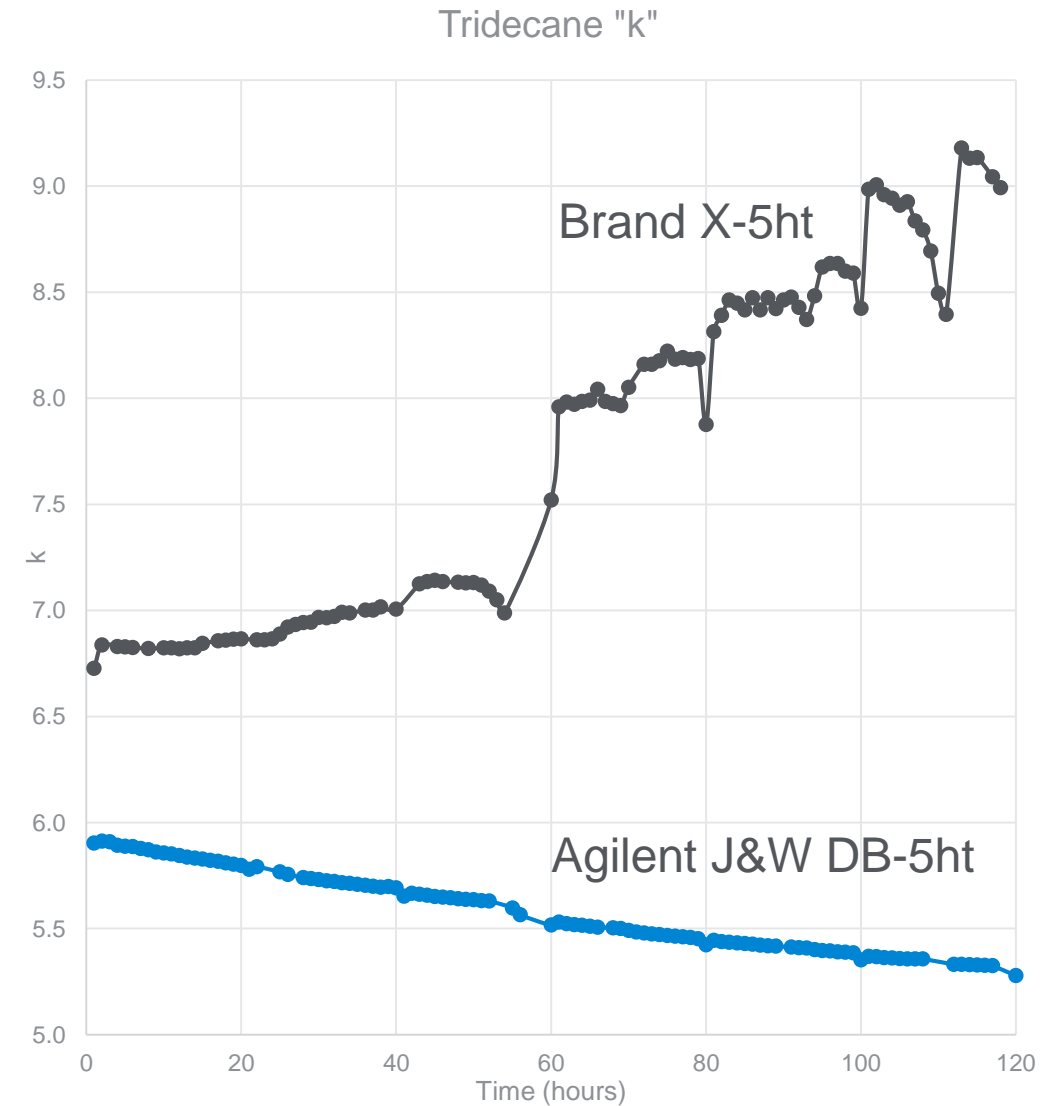
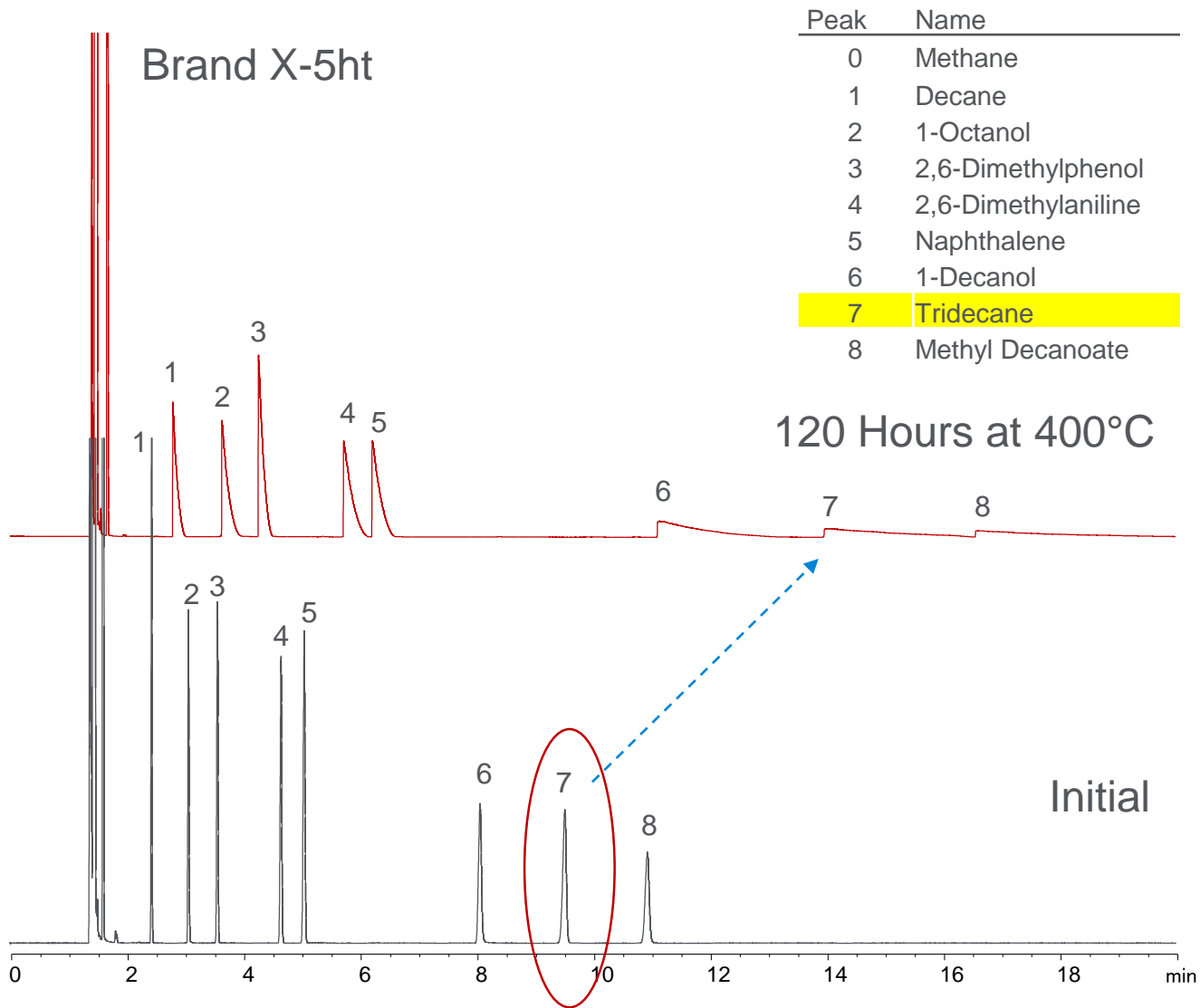


Agilent J&W DB-5ht

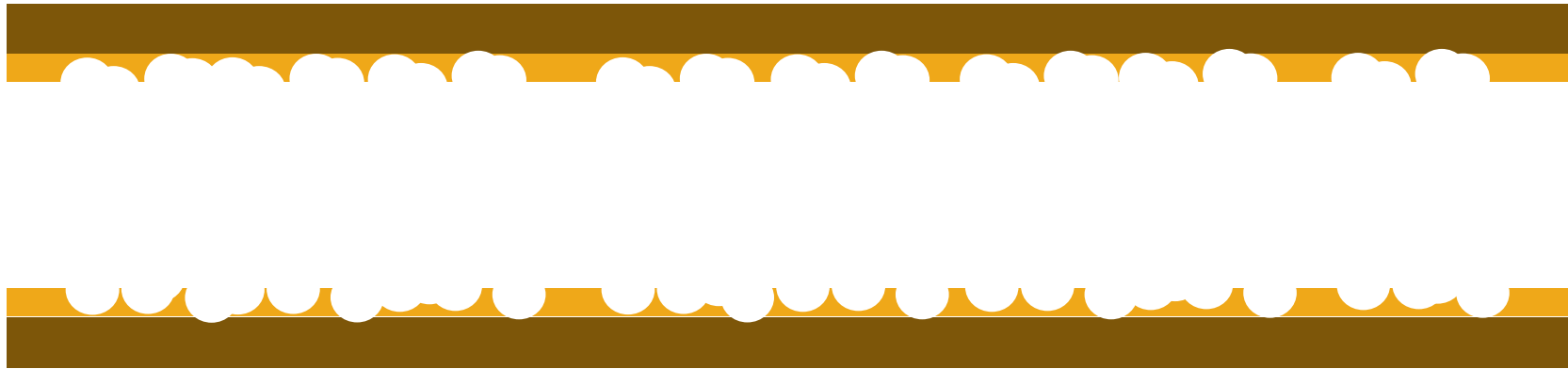
Agilent Publication 5994-1013EN

# Phase Degradation Increases Retention

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



# “Potholes” Created as the Phase Degrades Raw Fused Silica exposed.....

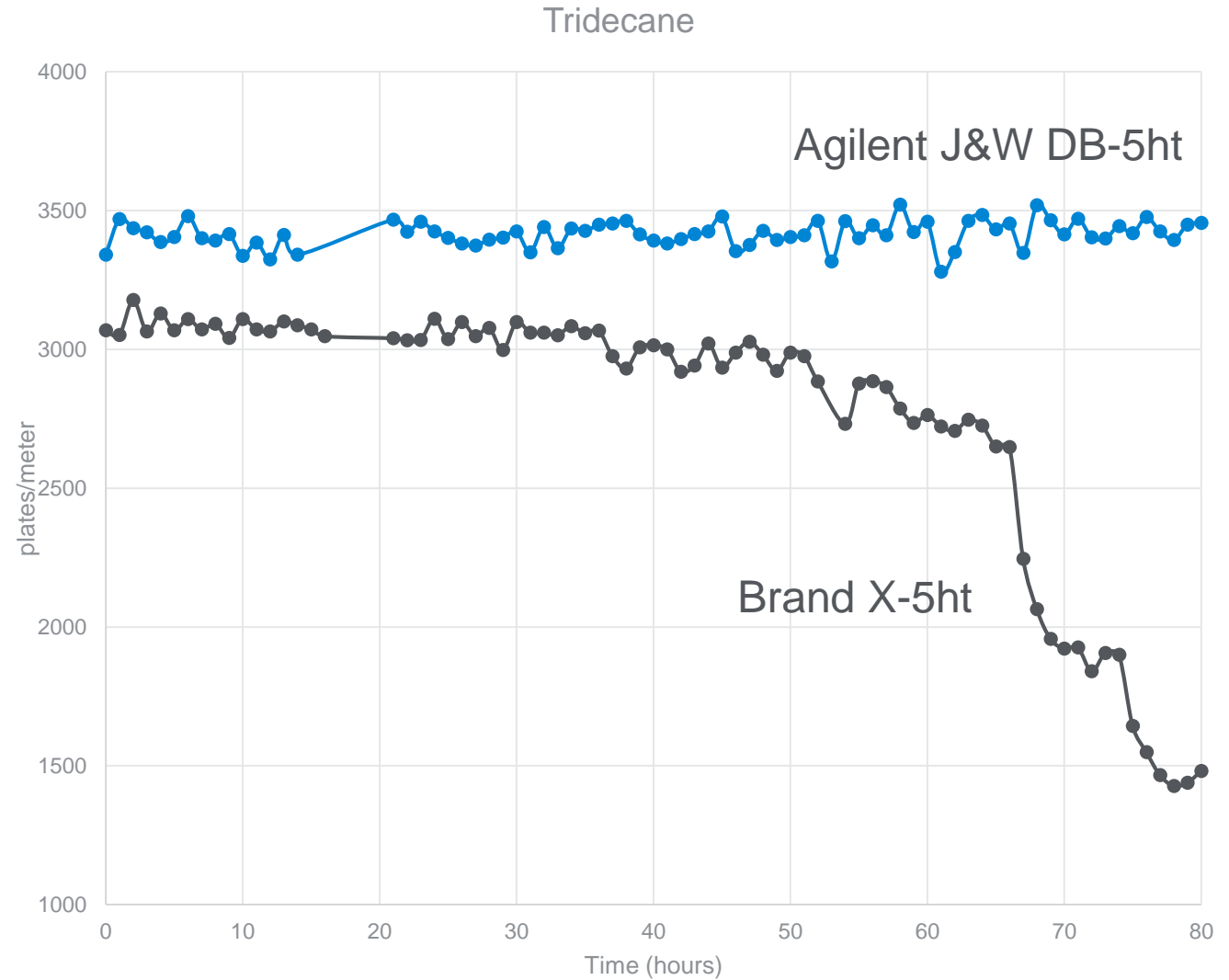
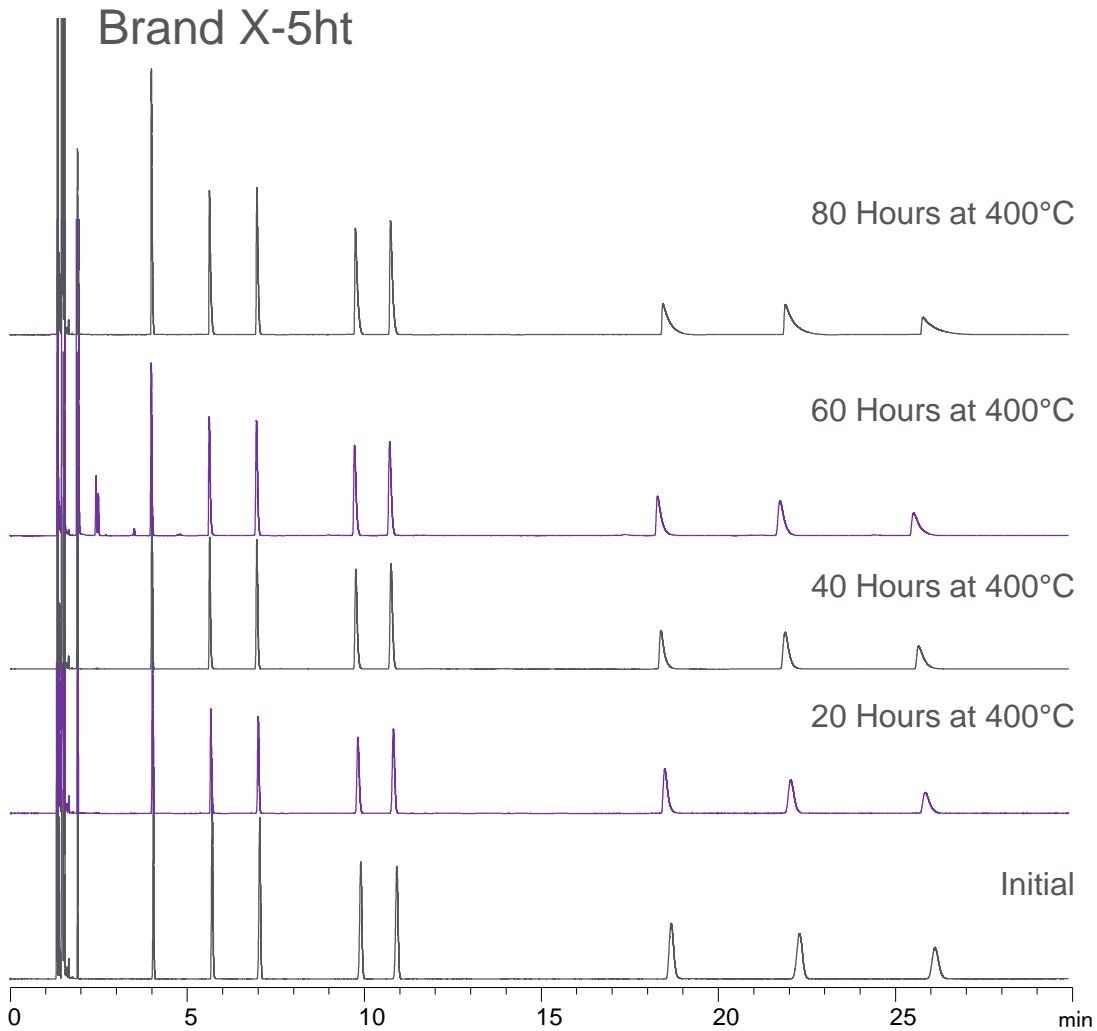


The more heat you add the more “potholes” you create



# Thicker film will last longer.... But will eventually degrade at 400°C

30m x 0.25 mm x 0.25  $\mu\text{m}$



# The outside matters too....

## Let's talk about substrate



# Columns by Temperature Limit

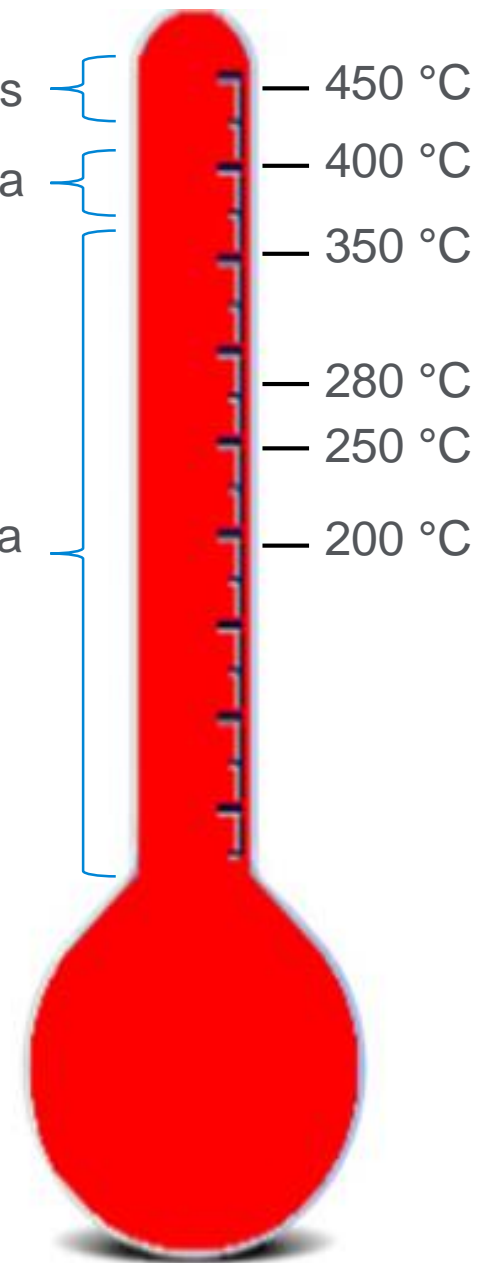


**Don't take fused silica columns above 400°C!**

Deactivated stainless steel columns

High temperature fused silica

Regular fused silica



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

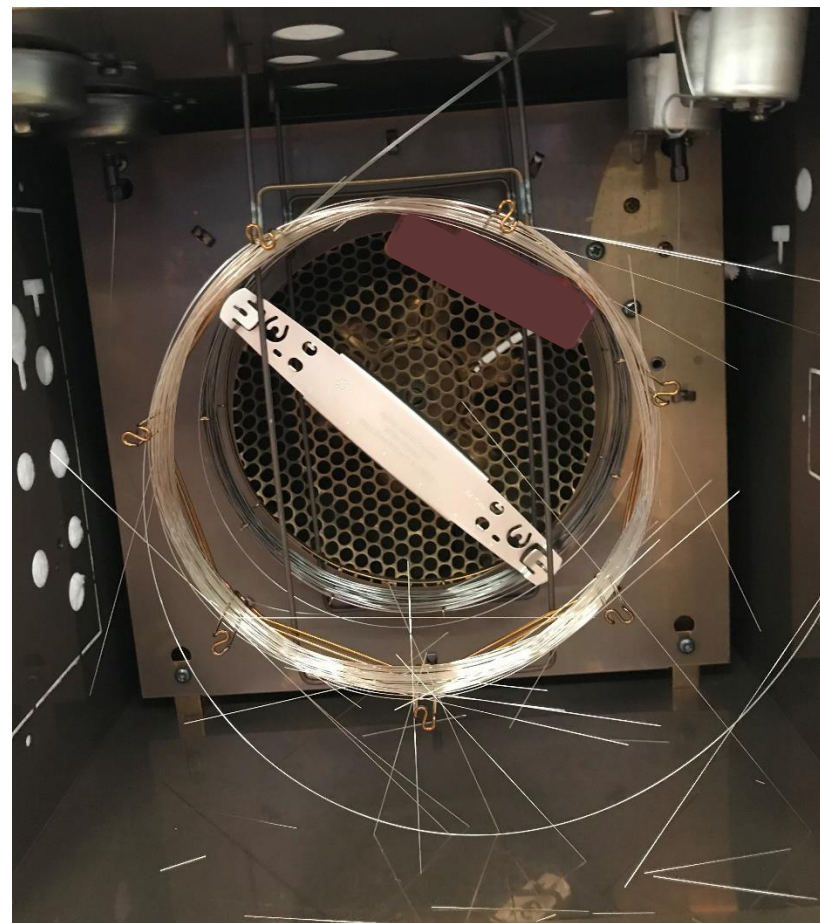
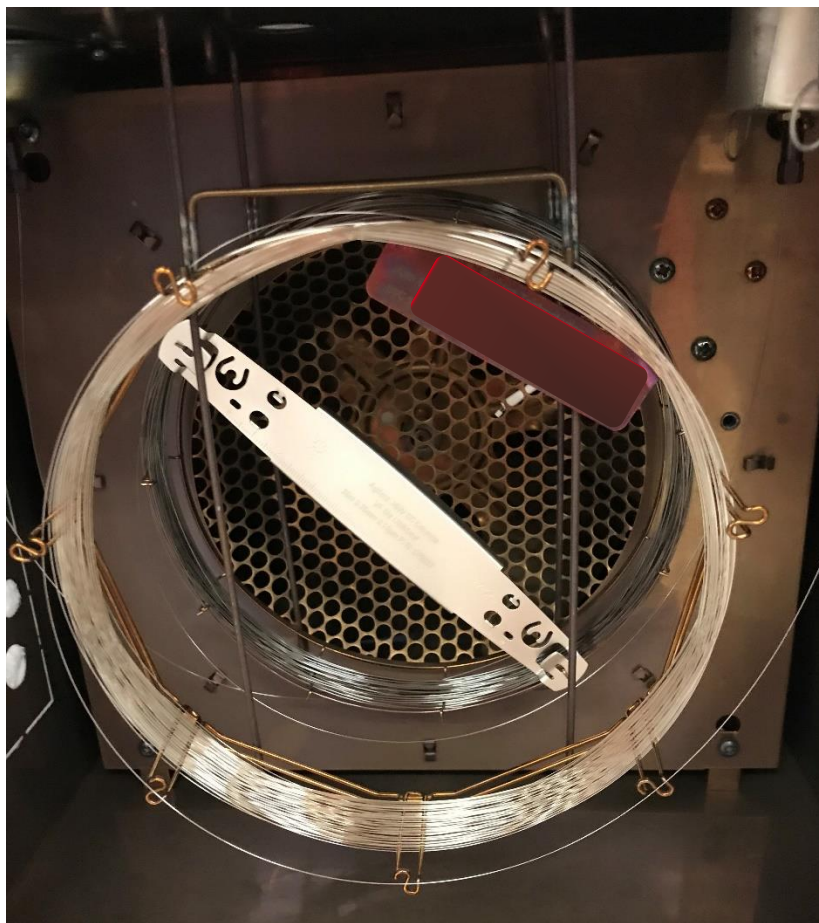
John V. Hinshaw, “The Making of a Column” LCGC Europe, Volume 19, Issue 2, pg 93–98, Feb 01, 2006



# Don't let this happen to you.....

Don't Touch!!!!!!

Ahhhhh!!!!

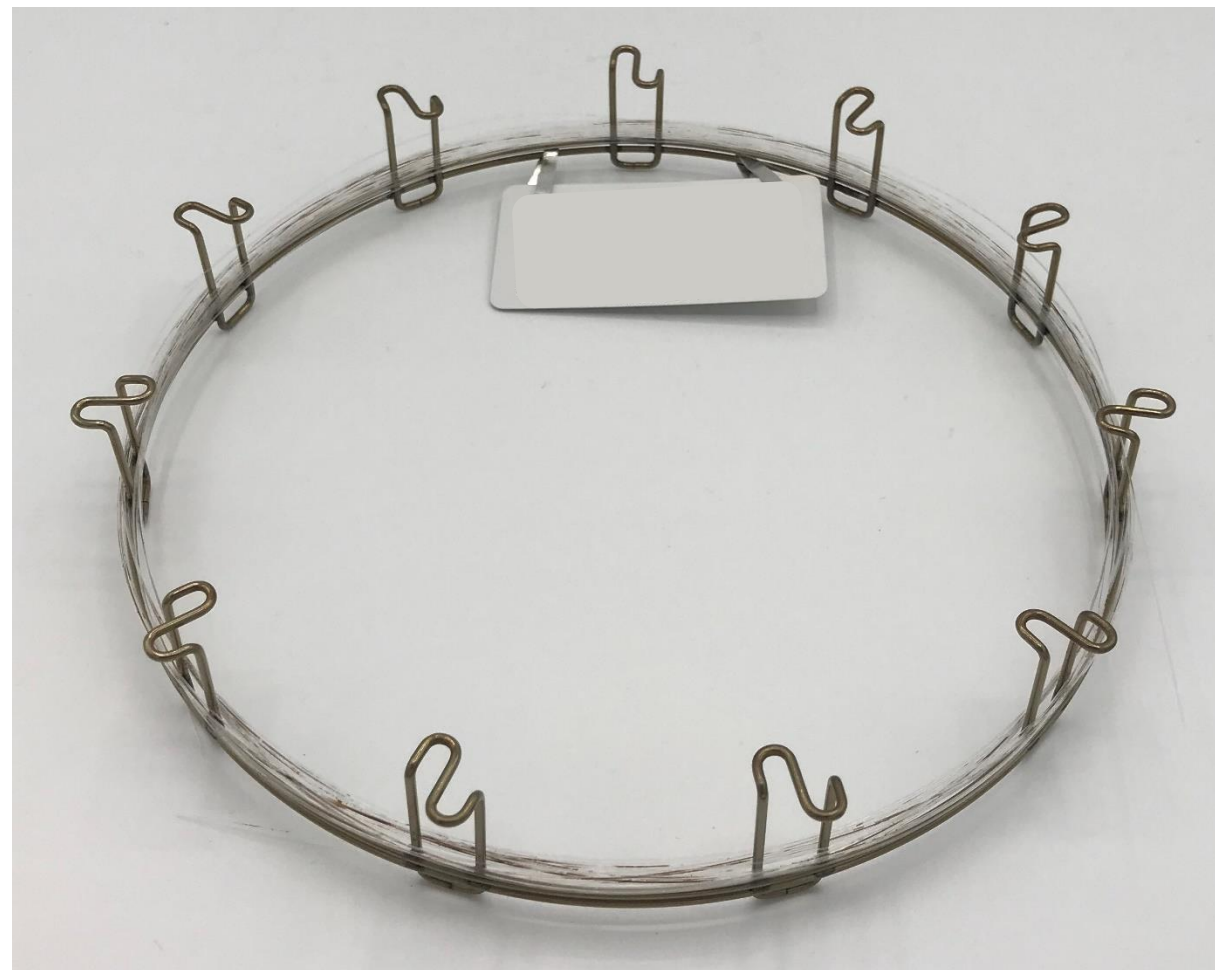


# Competitor Fused Silica doesn't always "take the heat"

Just because a company says their column can run at or above 400°C, doesn't mean it can.....



Brand Y  
After 20 hours at 400°C

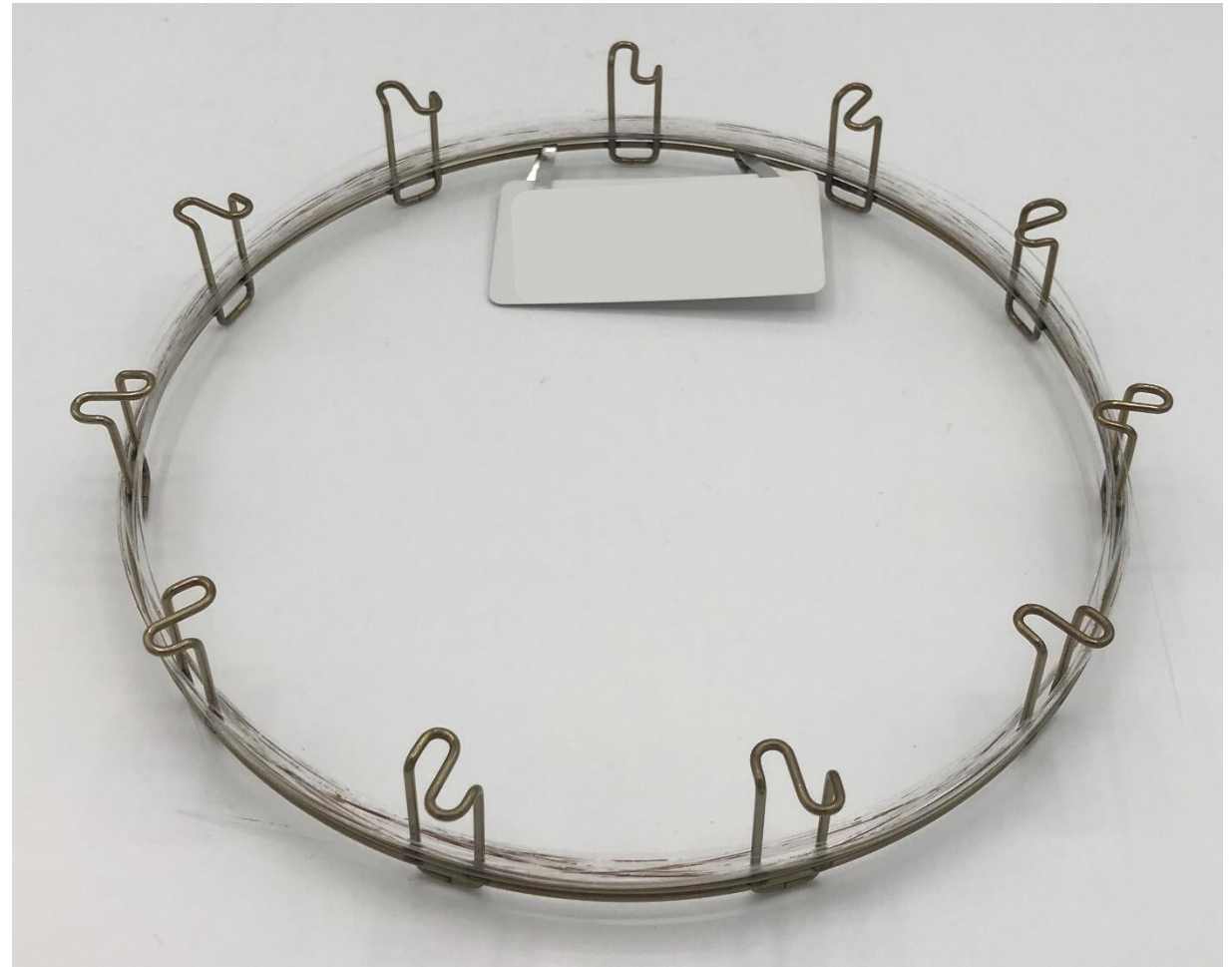


Brand Y  
After 25 hours at 400°C  
Agilent Publication 5994-1013EN

# Competitor Fused Silica doesn't always "take the heat"

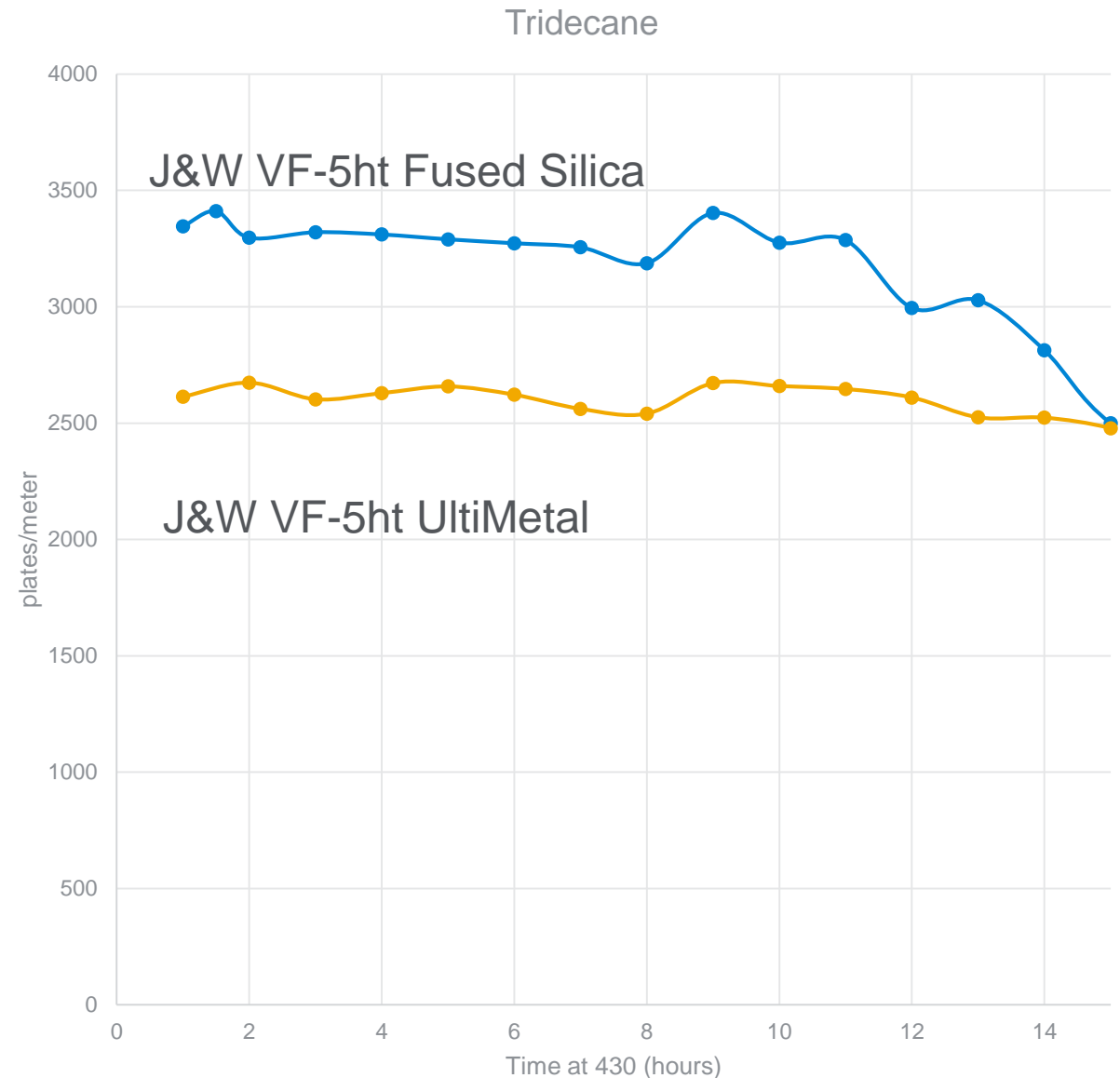
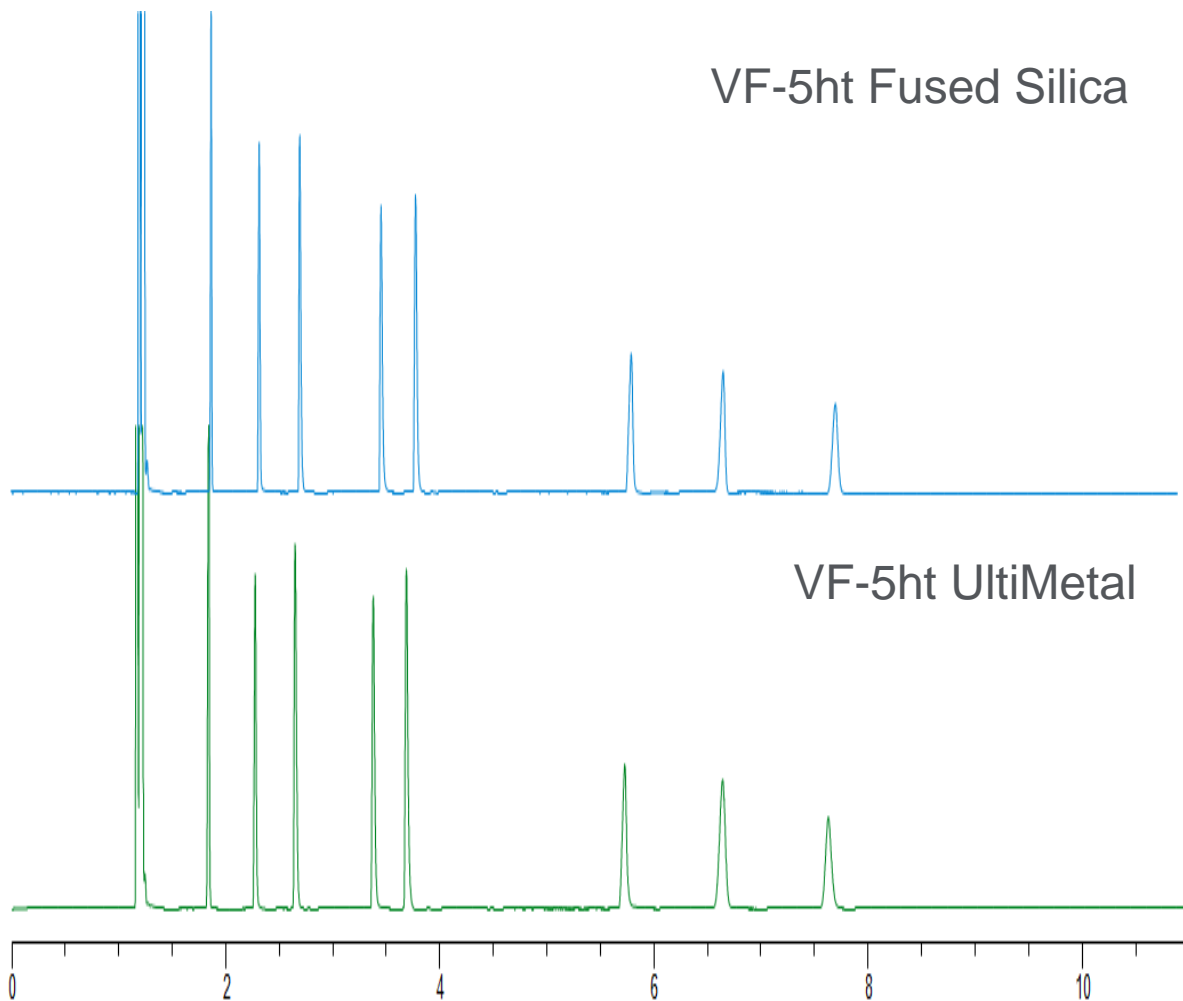


Agilent J&W DB-5ht



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?



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

The screenshot displays the Agilent Technologies Method Translator software interface. The window title is "Agilent Technologies Method Translator". On the left, there are three radio buttons: "Speed gain" (selected), "Translate", and "Best Efficiency". The "Speed gain" value is set to 2.5784. Below these are "Isothermal" (selected) and "Ramps" radio buttons, with a "Ramps" count of 1. The "Pressure Units" are set to "PSI".

The main area is divided into "Original Method Parameters" and "Calculated Method Parameters". Both sections show "Gas" as He (original) and H2 (calculated). A red box highlights the "Length (m)", "Inner Diameter (μm)", and "Film Thickness (μm)" parameters in the original method section. The "Final Temp (°C)" value of 35 in the "Ramps" table is also circled in red.

Parameter	Original Method Parameters	Calculated Method Parameters
Length (m)	30 m	50 m
Inner Diameter (μm)	320 μm	420 μm
Film Thickness (μm)	0.25 μm	0.15 μm
Phase Ratio	319.25	699.25
Inlet Pressure (gauge)	7 psi	6 psi
Outlet Flow (mL/min)	1.4856 mL/min	4.8781 mL/min
Average Velocity (cm/s)	25.465 cm/sec	49.962 cm/sec
Outlet Pressure (abs)	14.696 psi	14.696 psi
Holdup Time	1.9635 min	1.6679 min
Outlet Velocity (cm/s)	31.819	60.65

#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		35	

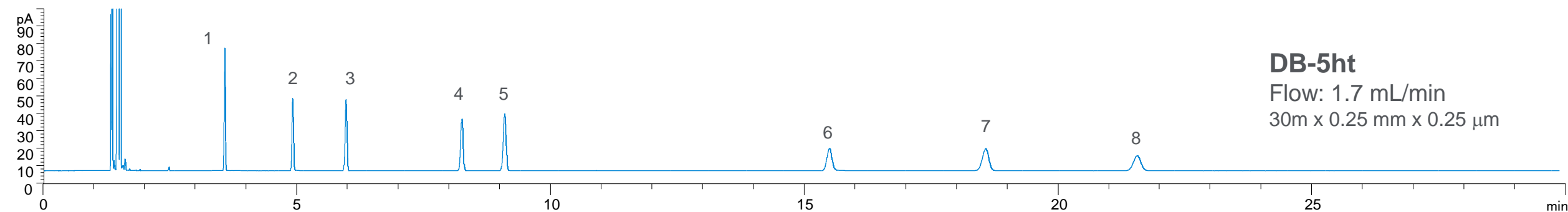
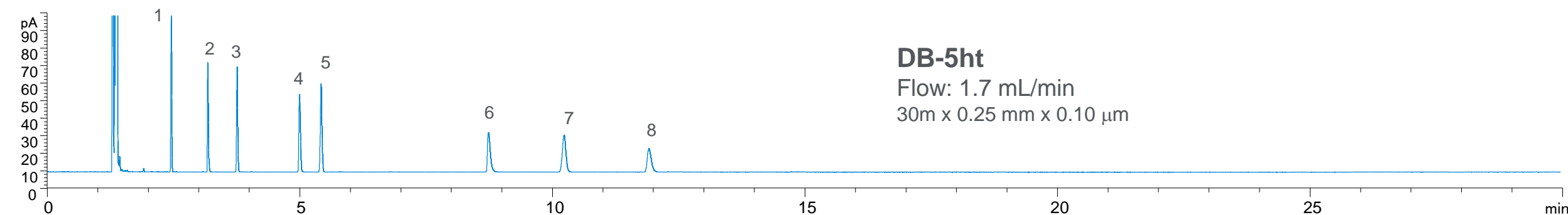
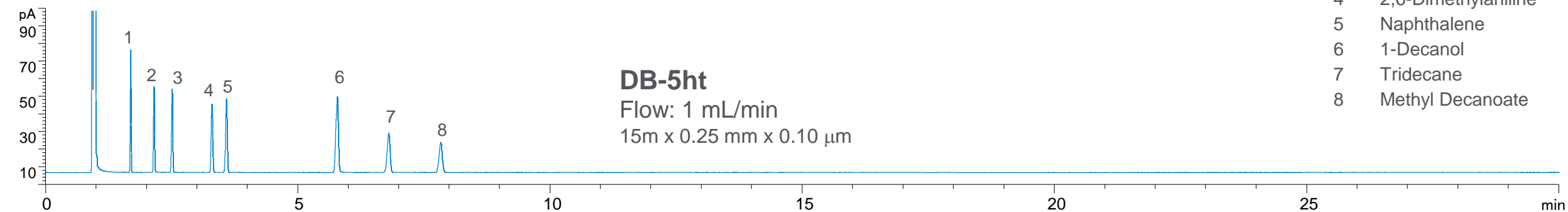
#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		35	0.00

Original Column Capacity: 2.48  
Translated Column Capacity: 2.89

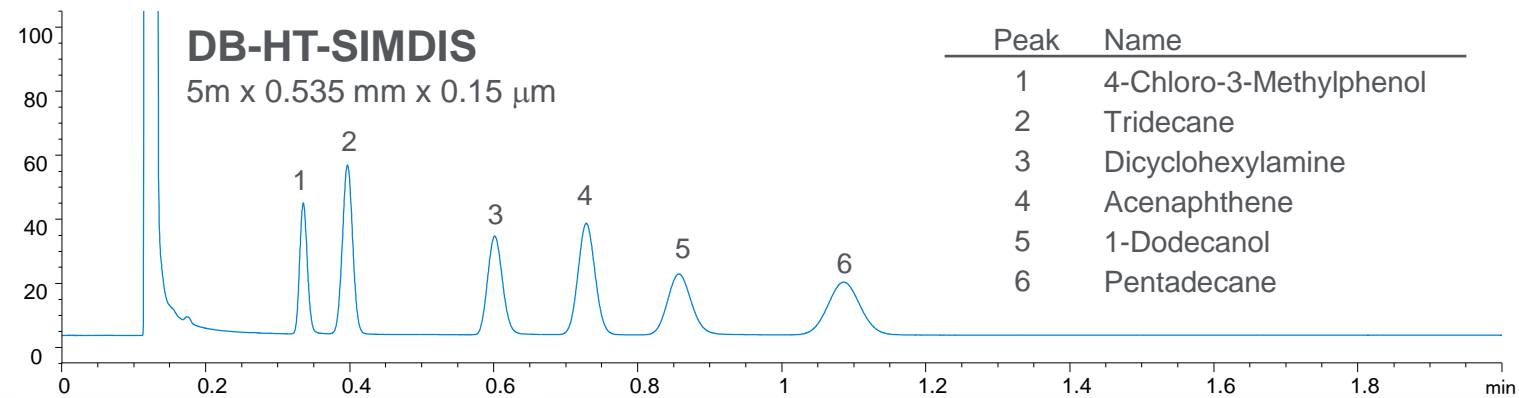
The column capacity of the translated method is 116% of the original column capacity. You may need to adjust your injection volume.

# Short, Thin Film Columns Elute Compounds Faster

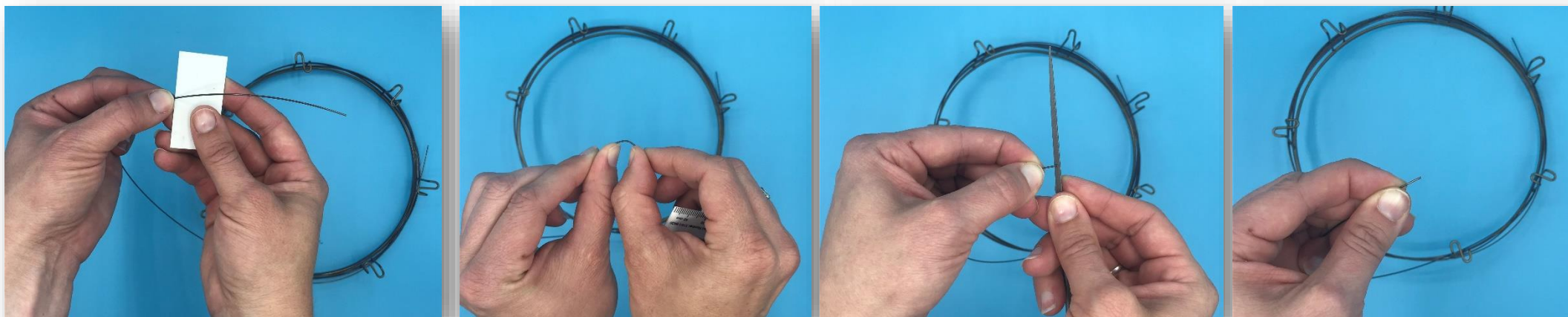
Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate



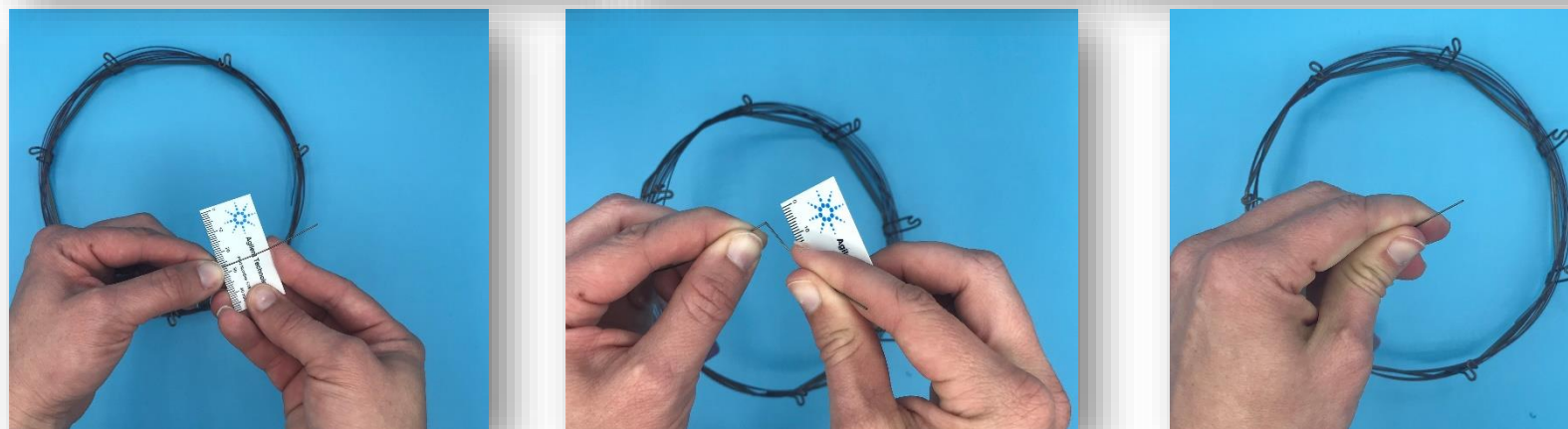
# Metal Columns Are Easy to Cut Properly



“Glass Infused”  
Metal Column

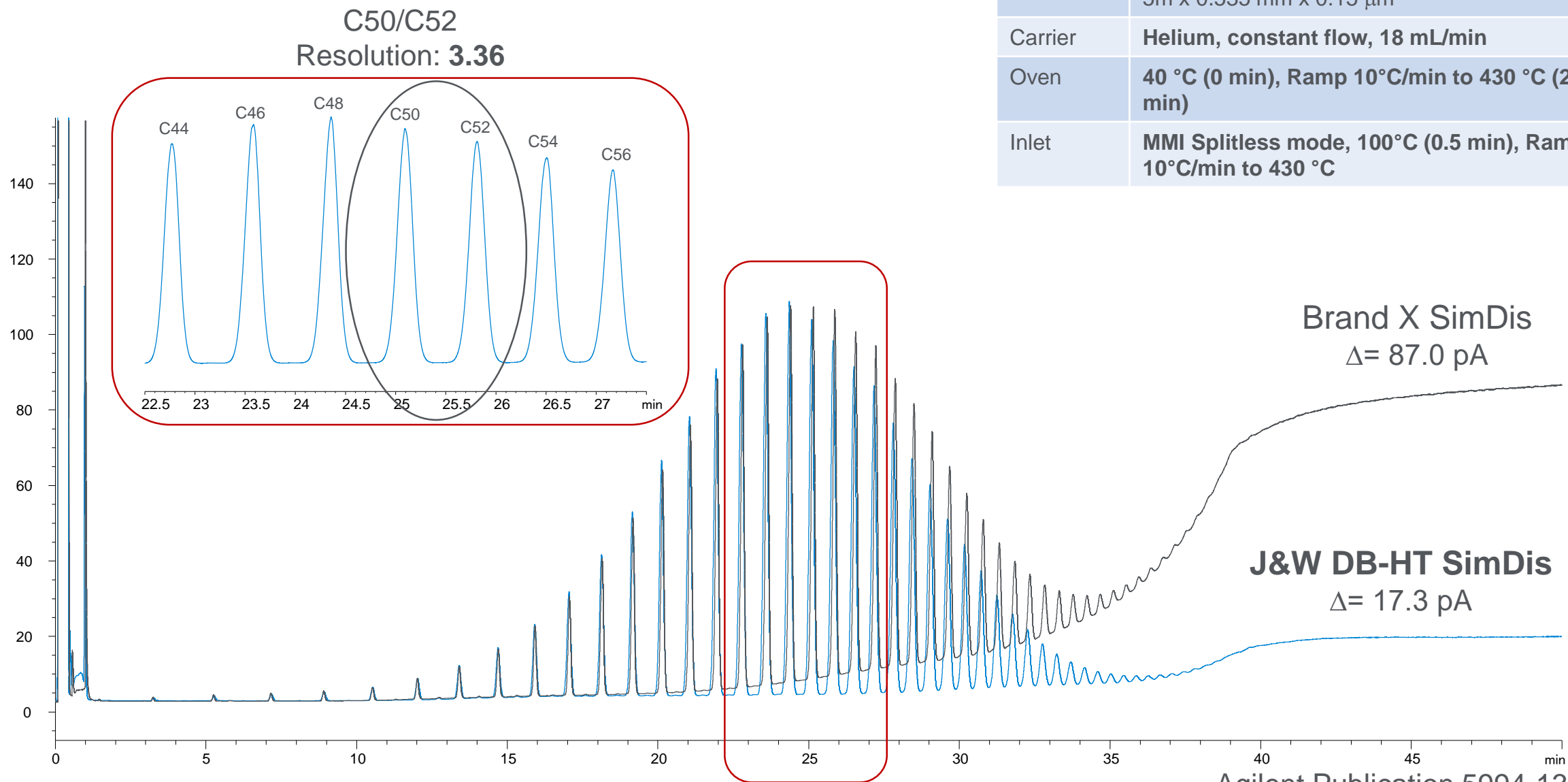


DB-HT-SimDis  
ProSteel



# Lower bleed with Agilent ProSteel Columns

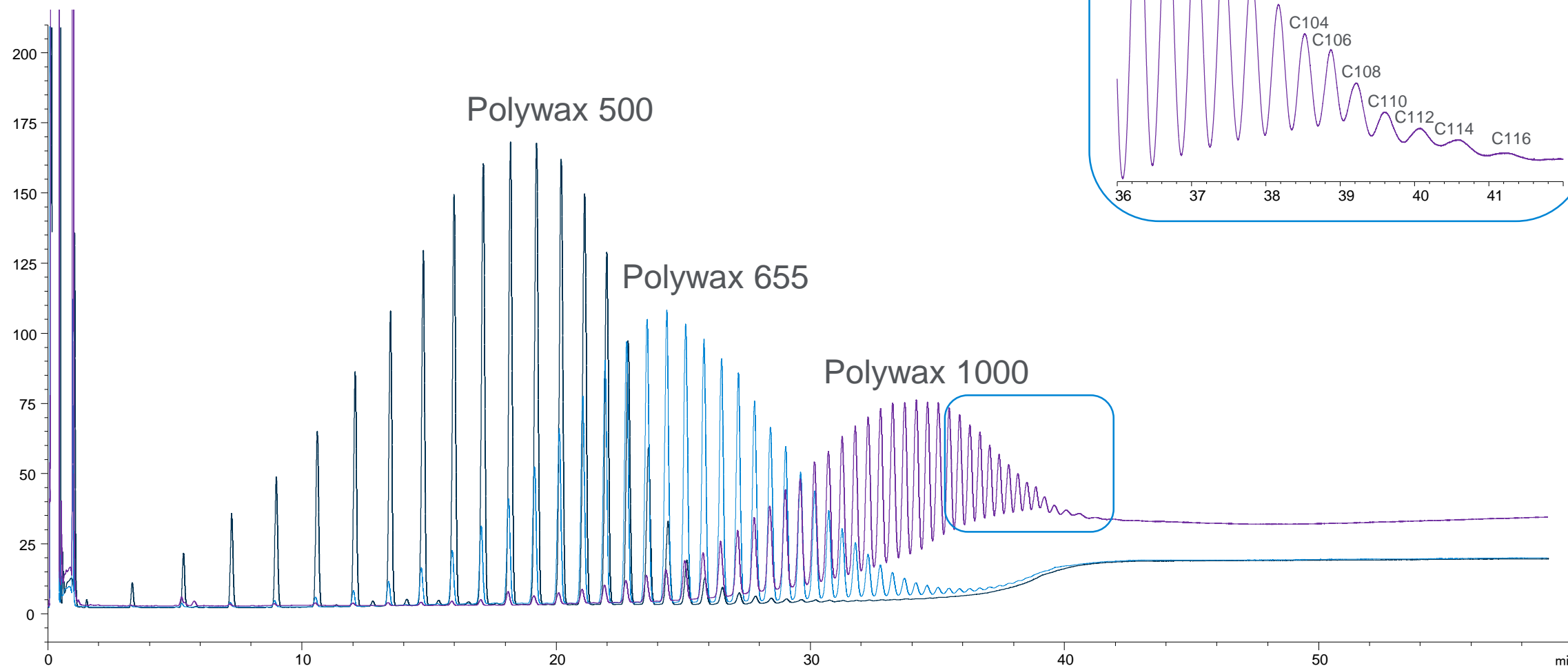
Column	Brand X-Metal SimDis Agilent J&W DB-HT SimDis ProSteel 5m x 0.535 mm x 0.15 $\mu$ m
Carrier	Helium, constant flow, 18 mL/min
Oven	40 °C (0 min), Ramp 10°C/min to 430 °C (20 min)
Inlet	MMI Splitless mode, 100°C (0.5 min), Ramp 10°C/min to 430 °C



Agilent Publication 5994-1385EN



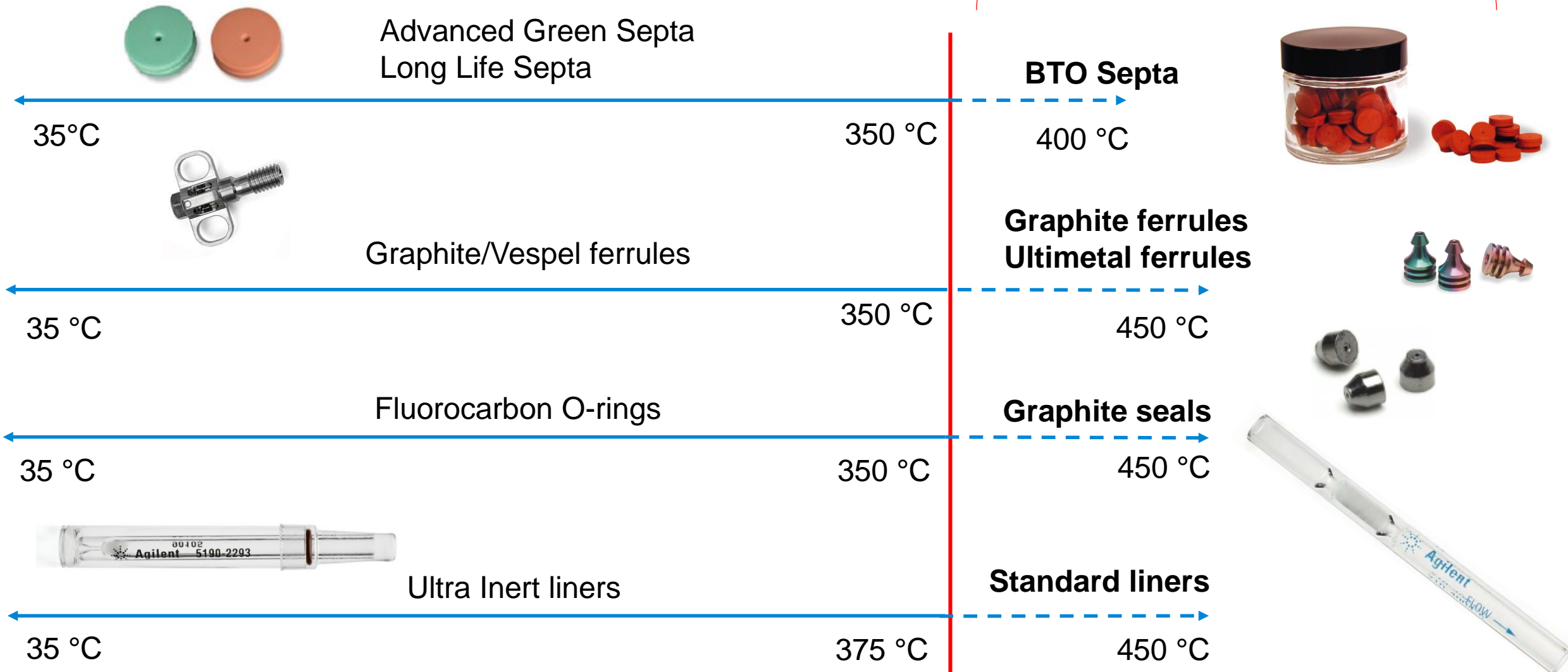
# All the Polywax DB-HT SIM DIS



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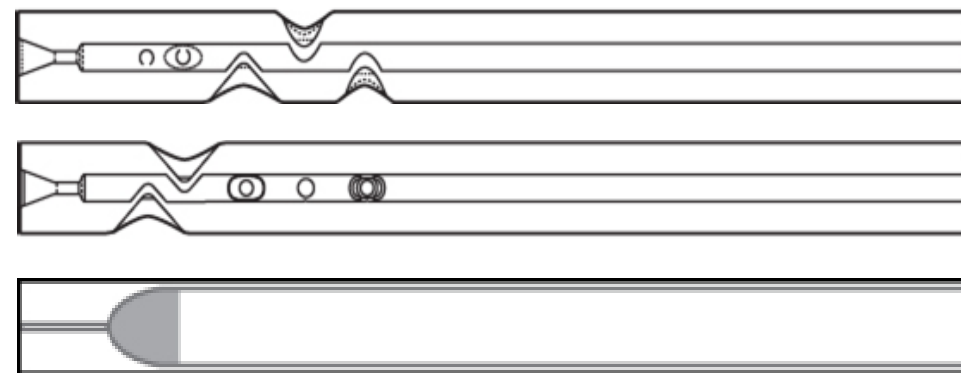
# What do you do if you need to go above 400°C

## High Temp Applications



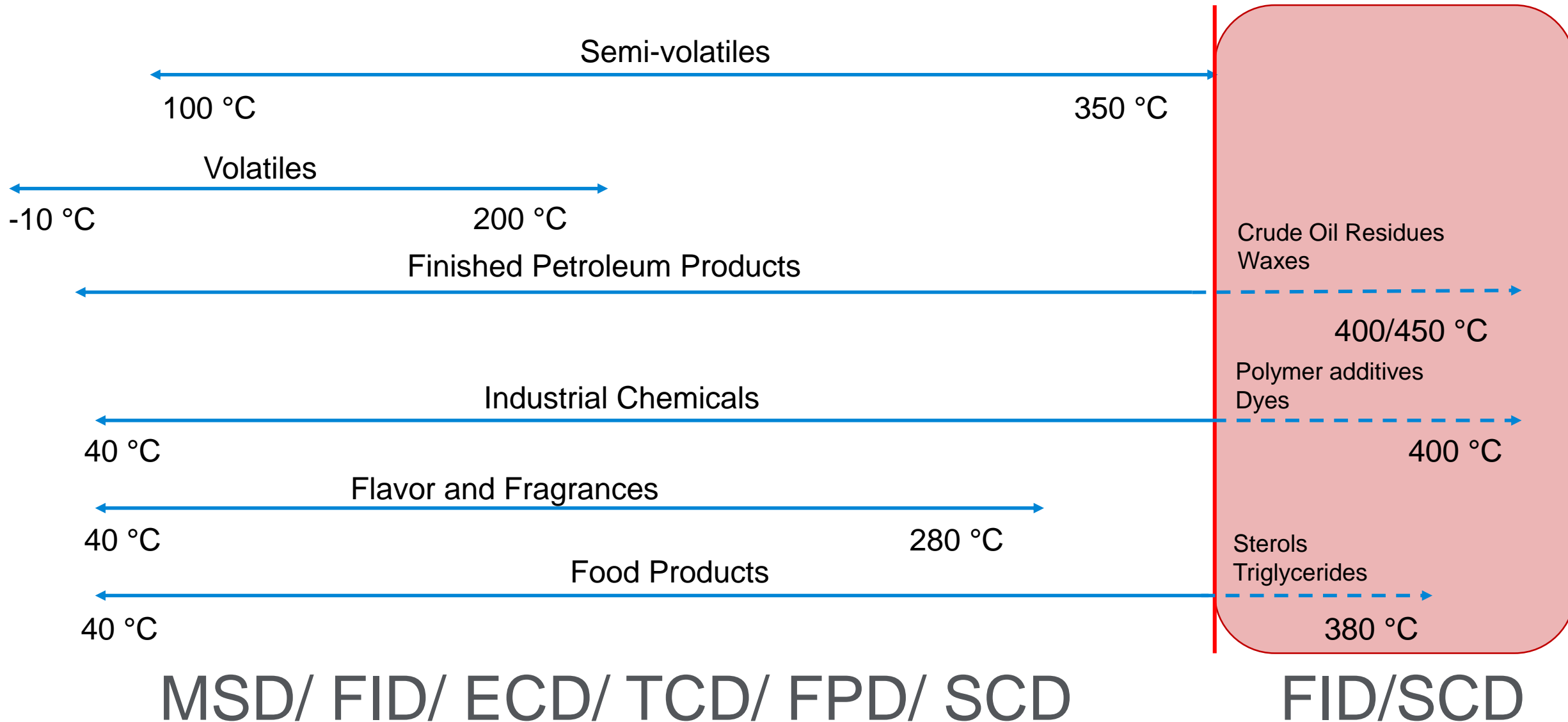
# Advantages of MMI and “Cold” Splitless

- ❑ 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

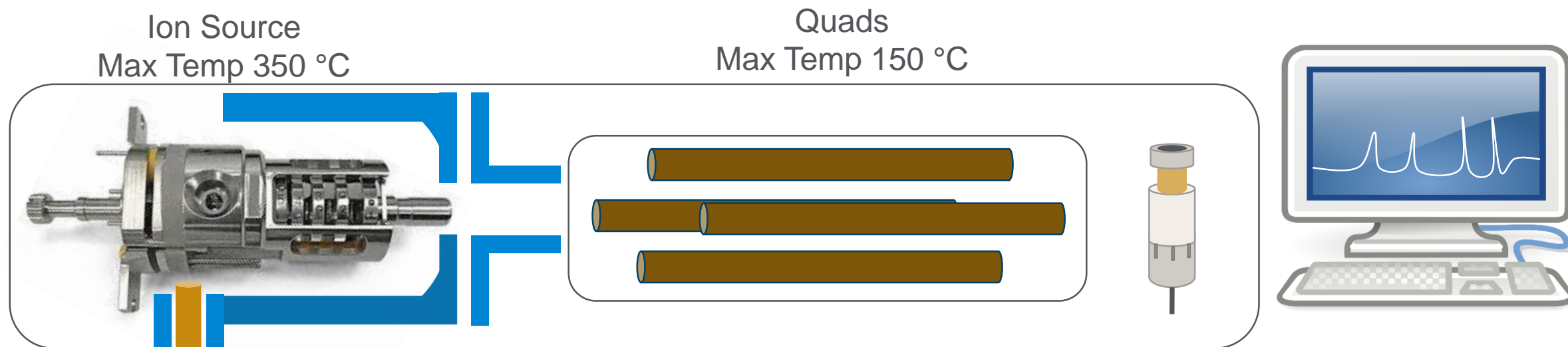


**Lower detection limits → Better accuracy of data**

# Temperature Range, Applications, & Detectors



# Just because you can do High Temp GCMS.... Should you?



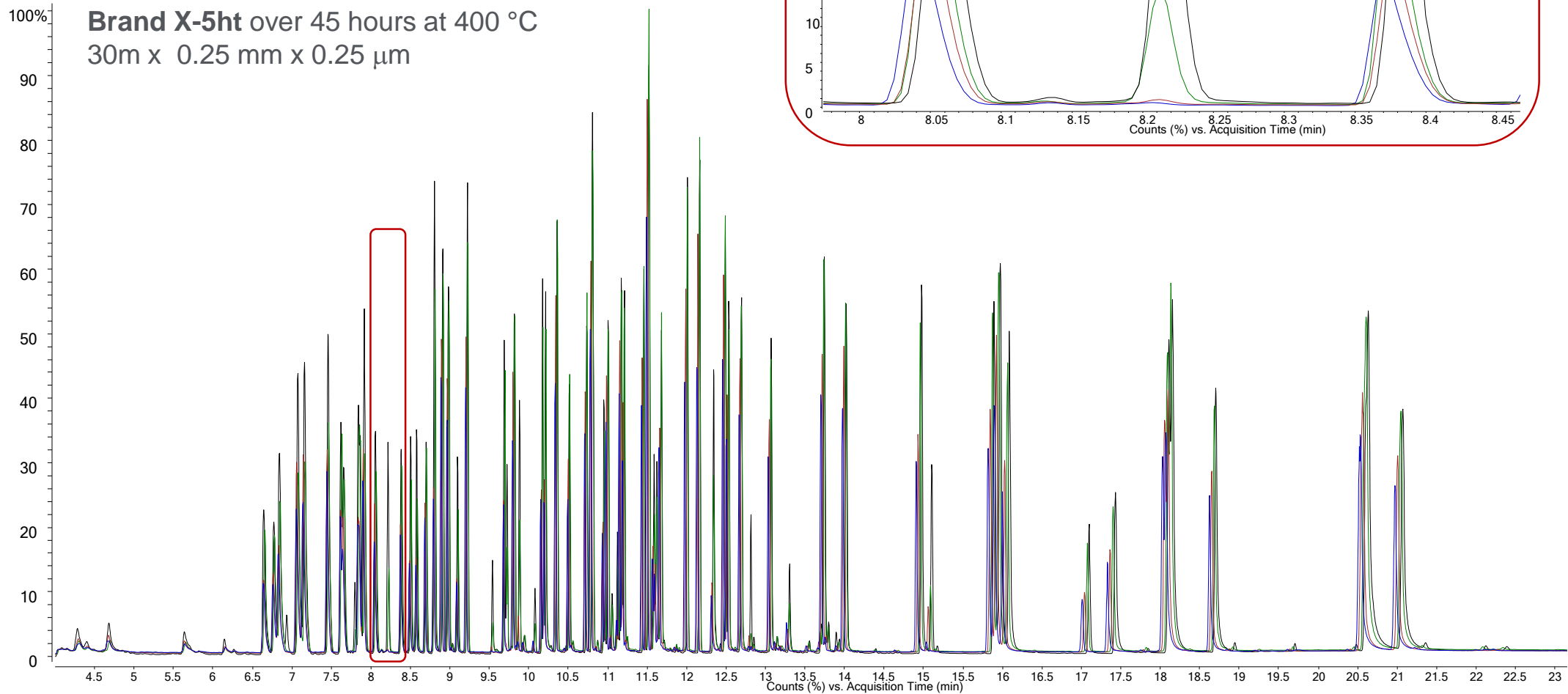
## Do you really need Mass Spec?

- Shrink after thermal cycles creating leaks
- Leaks cause filament to burn out faster
- Condensation in Quads.....

GC Oven Max Temp 450 °C

# Loss of Sensitivity & Loss of Inertness

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



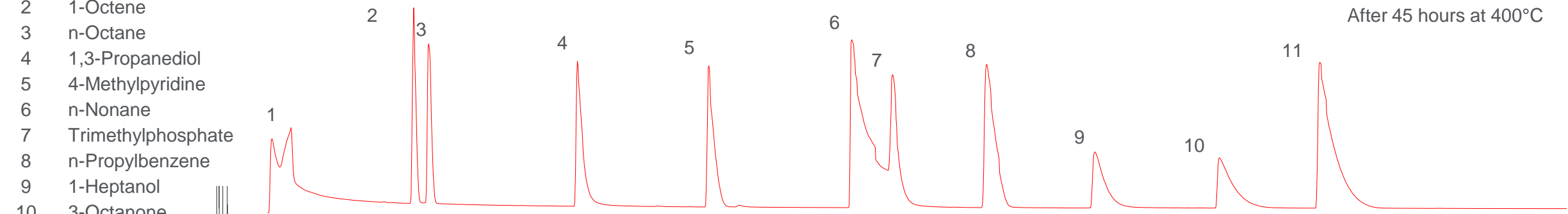
SVM-8270-1

Peak	Compound
1	N-Nitrosodimethylamine
2	Phenol
3	2-Chlorophenol
4	1,3-Dichlorobenzene
5	1,4-Dichlorobenzene
6	1,2-Dichlorobenzene
7	N-Nitrosodi-n-propylamine
8	o-Cresol
9	Bis(2-chloroisopropyl) ether
10	p-Cresol
11	Hexachloroethane
12	Nitrobenzene
13	Isophorone
14	2-Nitrophenol
15	2,4-Dimethylphenol
16	Bis(2-chloroethoxy)methane
17	2,4-Dichlorophenol
18	1,2,4-Trichlorobenzene
19	Naphthalene
20	4-Chloroaniline
21	Hexachlorobutadiene
22	4-Chloro-3-methylphenol
23	2-Methylnaphthalene
24	Hexachlorocyclopentadiene
25	2,4,6-Trichlorophenol
26	2,4,5-Trichlorophenol
27	2-Chloronaphthalene
28	2-Nitroaniline
29	Dimethyl phthalate
30	2,6-Dinitrotoluene
31	Acenaphthylene
32	3-Nitroaniline
33	Acenaphthene
34	2,4-Dinitrophenol
35	4-Nitrophenol
36	Dibenzofuran
37	2,4-Dinitrotoluene
38	Diethyl phthalate
39	Fluorene
40	4-Chlorophenyl phenyl ether
41	4-Nitroaniline
42	2-Methyl-4,6-dinitrophenol
43	Azobenzene
44	4-Bromophenyl phenyl ether
45	Hexachlorobenzene
46	Pentachlorophenol
47	Phenanthrene
48	Anthracene
49	Carbazole
50	Di-n-butyl phthalate
51	Fluoranthene
52	Pyrene
53	Butyl benzyl phthalate
54	Benzo[a]anthracene
55	Chrysene
56	Bis(2-ethylhexyl) phthalate
57	Di-n-octyl phthalate
58	Benzo[b]fluoranthene
59	Benzo[k]fluoranthene
60	Benzo[a]pyrene
61	Indeno(1,2,3-cd)pyrene
62	Dibenzo[a,h]anthracene
63	Benzo[ghi]perylene

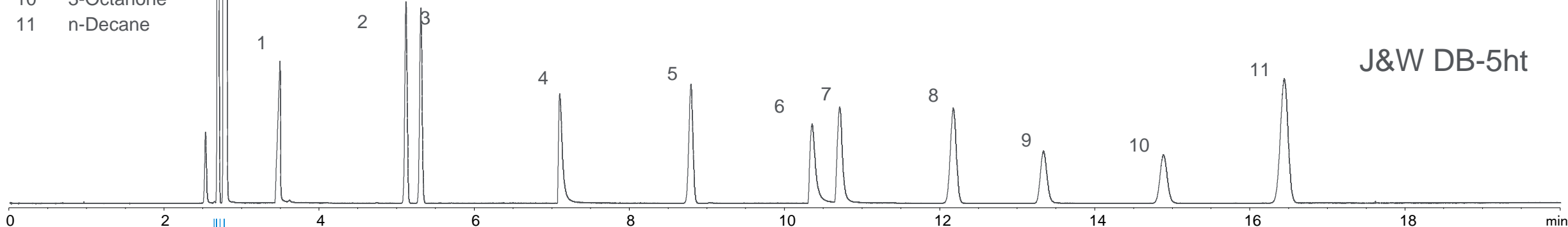
# MS UI Columns are better for Mass Spec than HT Columns

Peak	Name
1	Propionic Acid
2	1-Octene
3	n-Octane
4	1,3-Propanediol
5	4-Methylpyridine
6	n-Nonane
7	Trimethylphosphate
8	n-Propylbenzene
9	1-Heptanol
10	3-Octanone
11	n-Decane

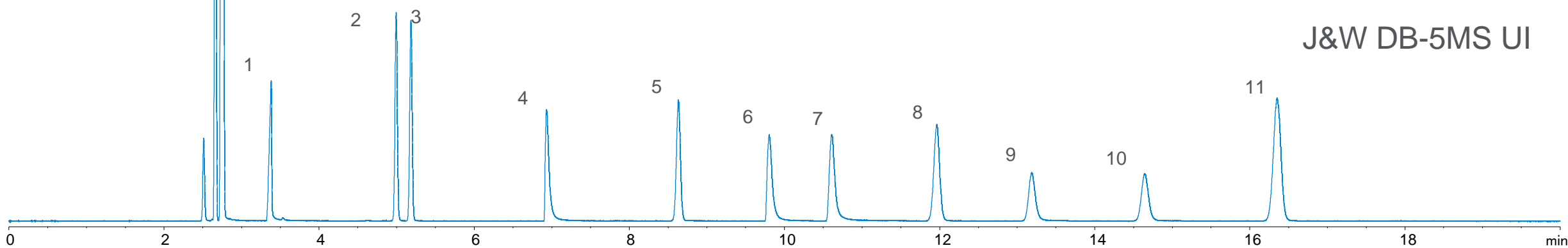
Brand X-5ht  
After 45 hours at 400°C



J&W DB-5ht



J&W DB-5MS UI



# Summary

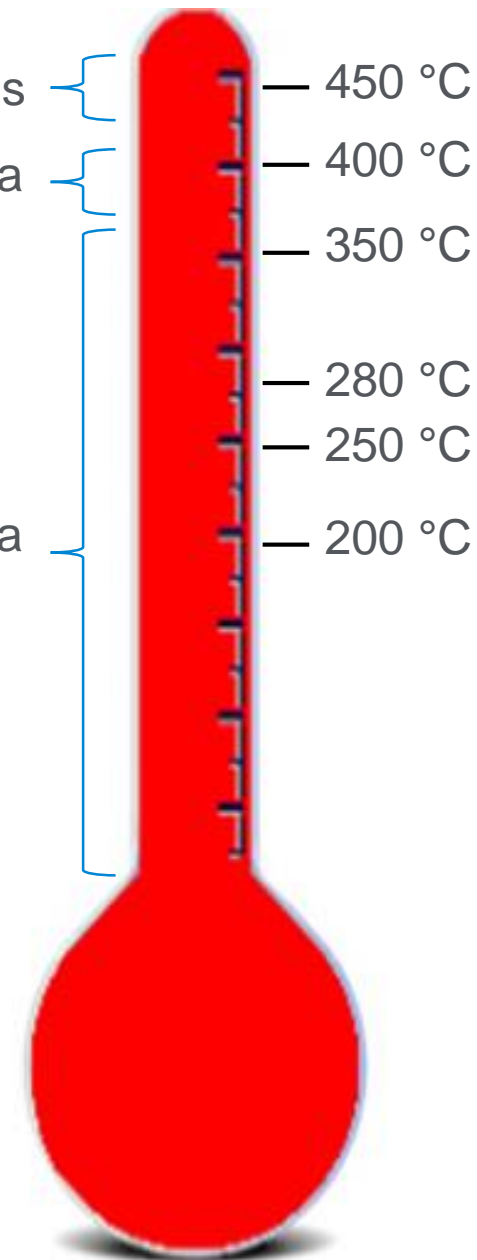
- Agilent J&W High temperature phase and Fused Silica are stable for extended time at 400°C
- If going to operate above 400°C use Deactivated Stainless Steel columns such as ProSteel and UltiMetal GC Columns
- Use the right consumables for high temperature applications
  - Graphite
  - UltiMetal ferrules
  - BTO Septa
  - Use regular liners
- High Temp Mass Spec is NOT Recommended
  - Use Ultra Inert Mass Spec Columns for difficult Mass Spec Analysis



Deactivated stainless steel columns


High temperature fused silica

Regular fused silica





# 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 polyimide that can operate in temperatures of up to 400 °C. They deliver these cost-saving advantages:

- Less rework. Lower your detection limits for high boilers with low bleed and the highest signal-to-noise ratios.
- Reduced maintenance. A proprietary stabilization technology minimizes detector contamination and stabilization time.
- Less downtime. Agilent high-temperature GC columns combine robustness with advanced surface deactivation to maximize performance and column life.

For applications above 400 °C, Agilent J&W UltraMetal and ProSteel GC columns offer added durability and ruggedness—even under extreme conditions. And they're easy to cut, too.

Learn how to go hot—and stay hot—with these resources

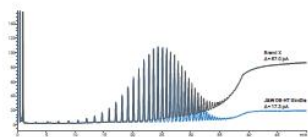
- In-depth column comparison tech overview
- High-temperature GC applications poster
- Metal GC columns tech overview
- High-temperature GC column brochure

[Request now](#)

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.

[Register now](#)



The difference is clear

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

[See the results](#)



## Performance and Durability for High-Temperature GC Columns

Agilent J&W high-temperature GC columns and consumables

Agilent CrossLab  
From Insight to Outcome



5994-1384EN



### Impact of Temperature on the Efficiency of High-Temperature GC Columns

5994-1013EN

## Technical overview

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

[Download](#)

PDF / 1.1 MB



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

5994-1385EN

## Technical overview

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

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

## Poster

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

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PDF / 0.6 MB

[www.agilent.com/chem/high-temp](http://www.agilent.com/chem/high-temp)

# 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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