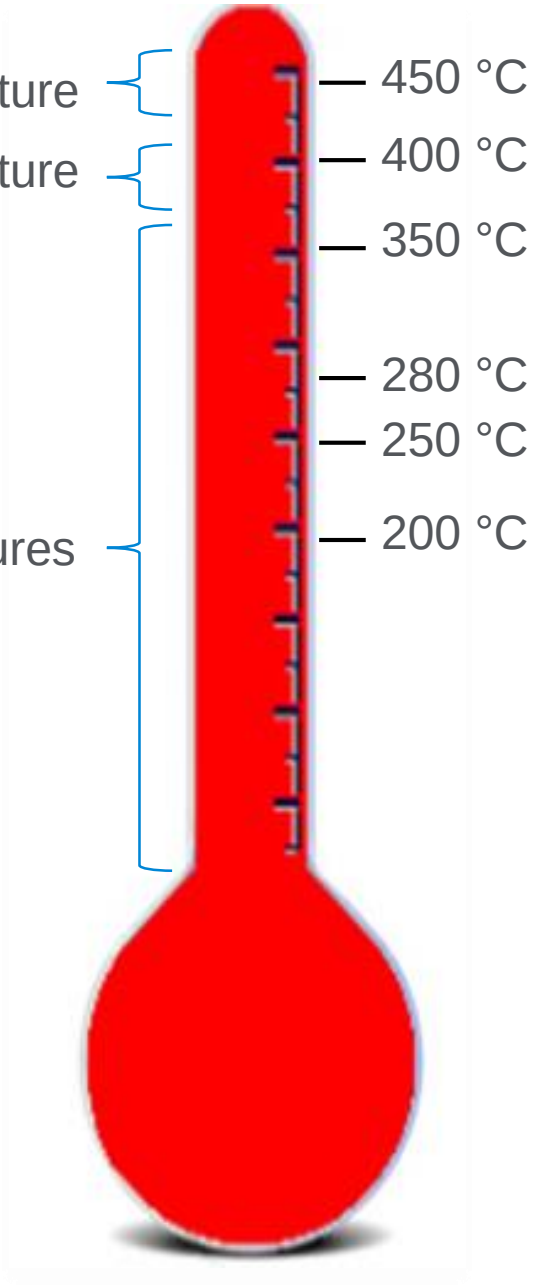
Turning the Heat up on Gas Chromatography

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

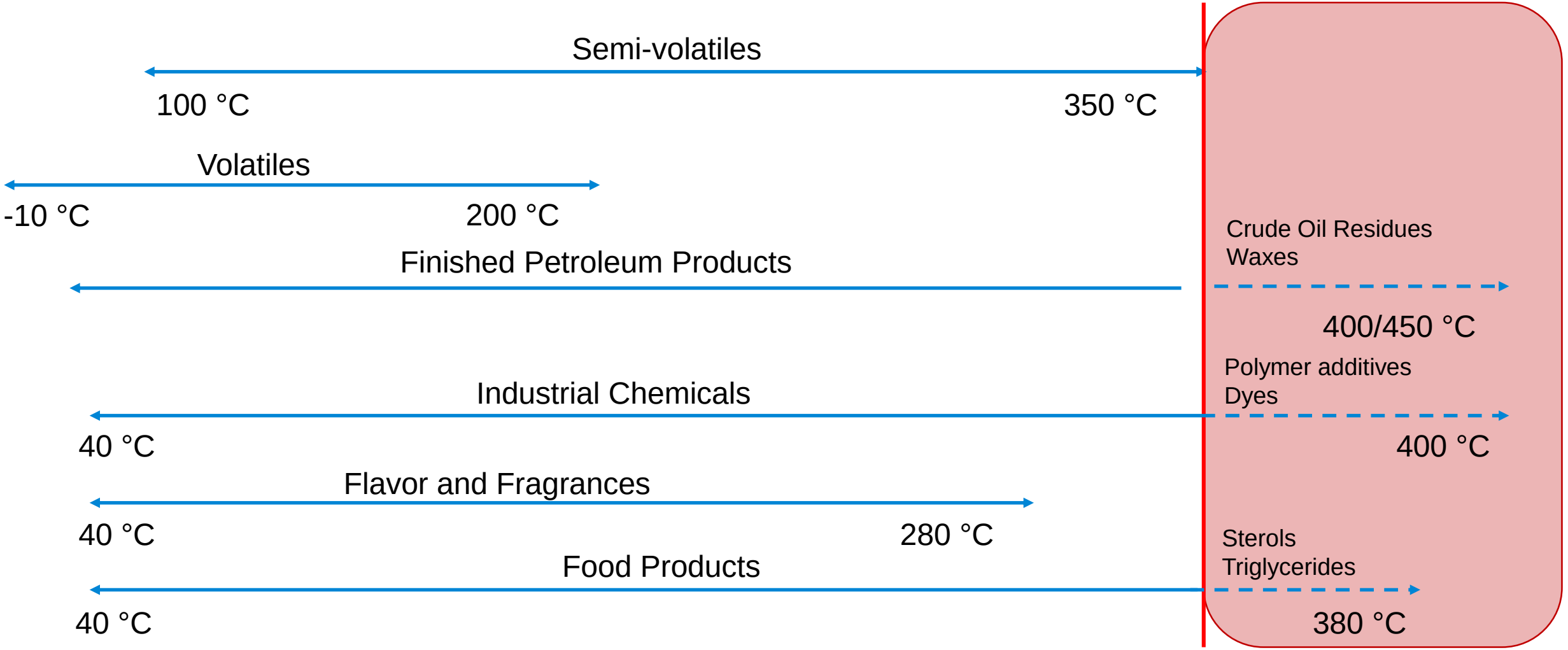
Vanessa Abercrombie
GC Applications Chemist

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?



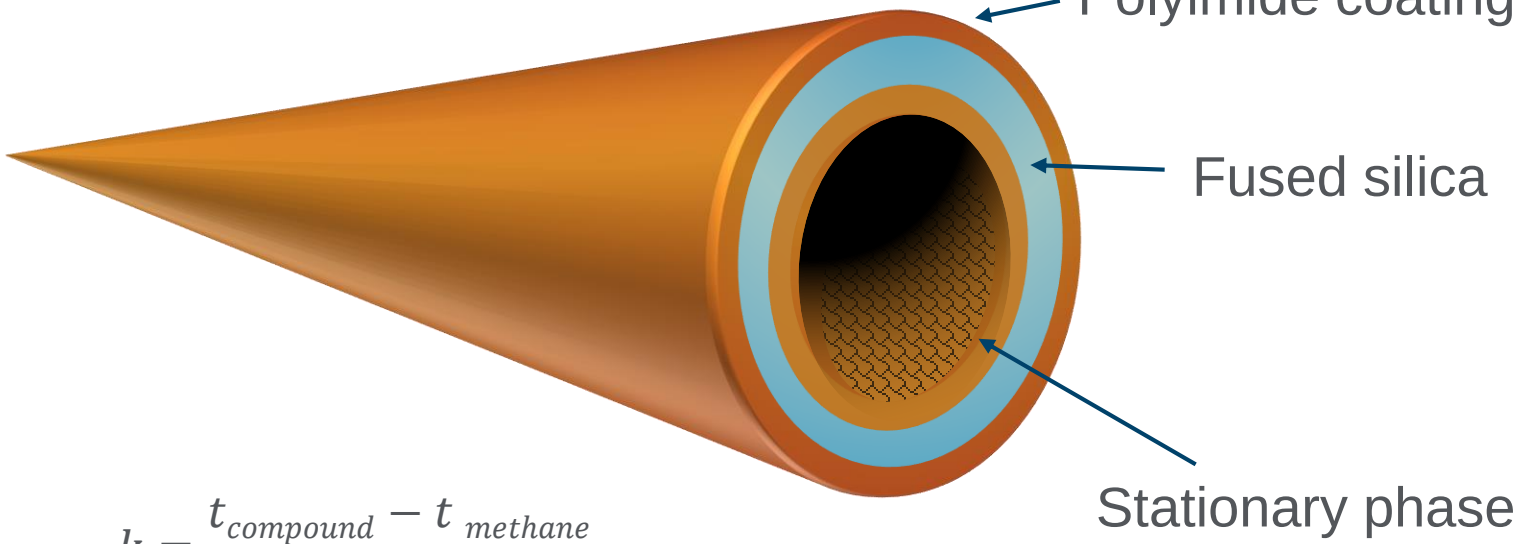
Temperature Range and Applications



If you aren't going above 350°C, you don't need a high temperature column

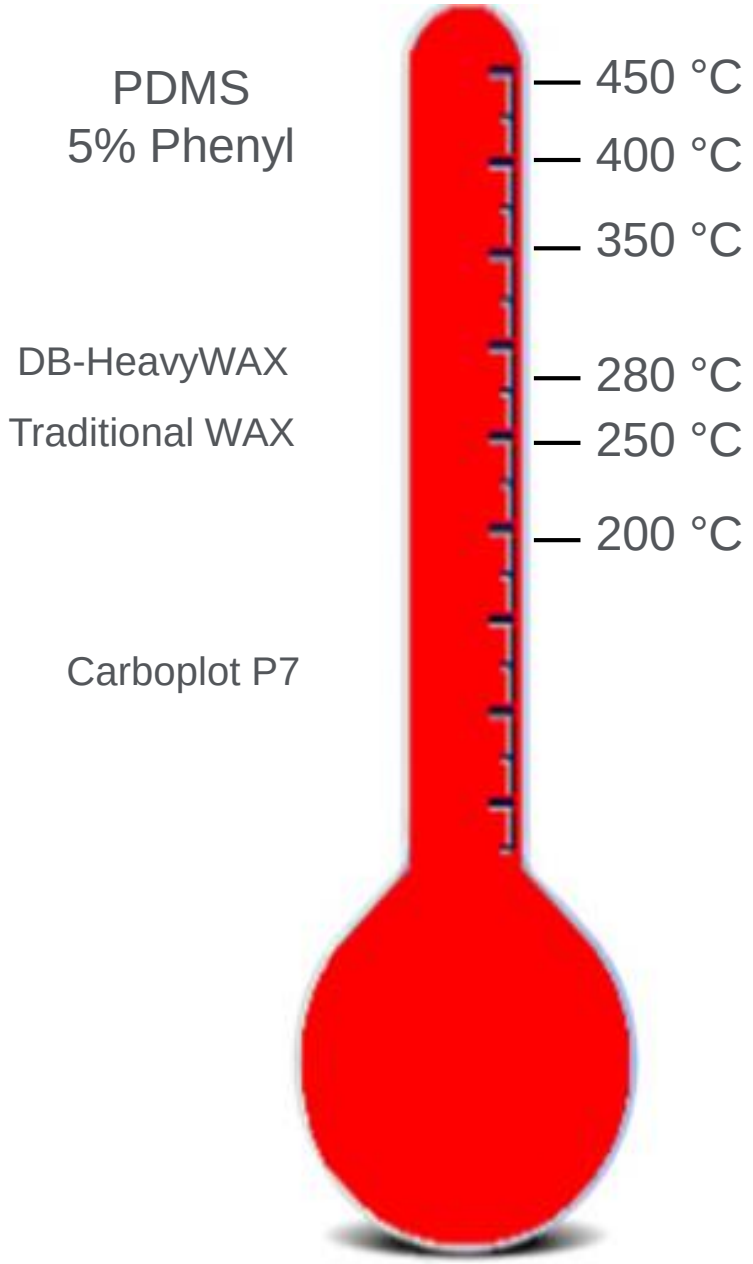
The Mystery of the MAOT

Max Temperature Range of the STATIONARY **PHASE** is <u>independent</u> of the CAPILLARY **TUBING**



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

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



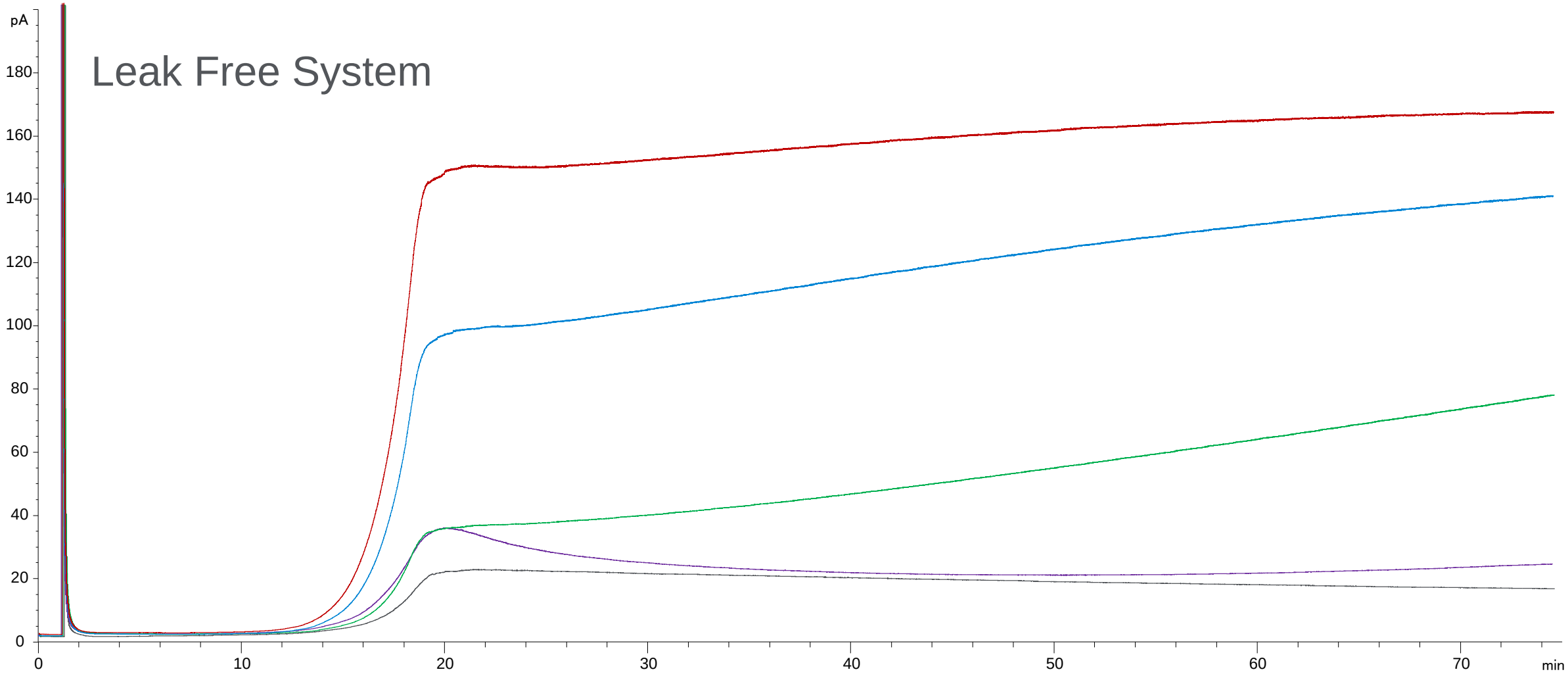
Gas Clean Filters:
The First Line of Defense Against Leaks

New Smart Sensor

P/N CP17973

www.agilent.com/chem/gasclean

High temp + Leaks = Dead Column
Air Leaks = Increase in Column Bleed at High Temperatures



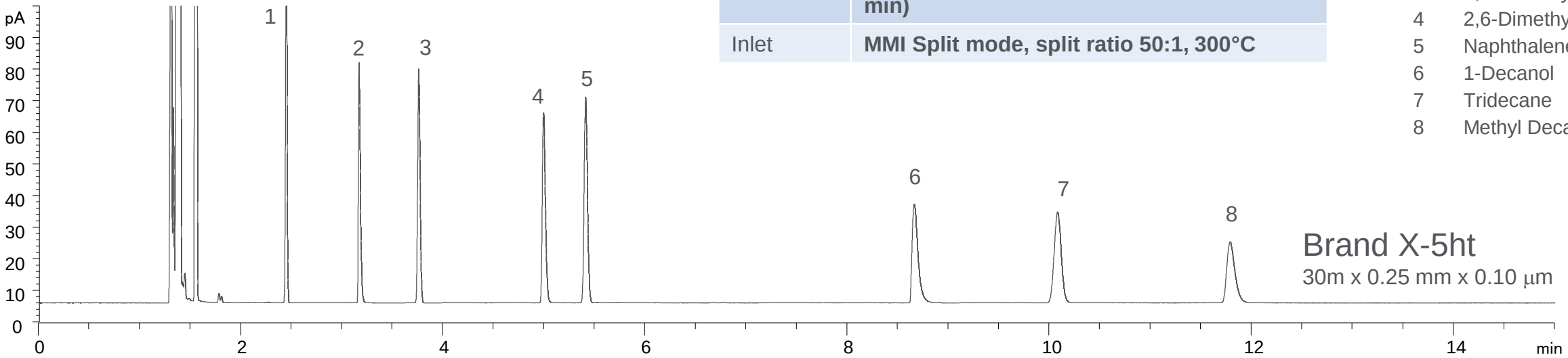
It’s what’s on the inside that counts….

Max temperature of phase and how it degrades

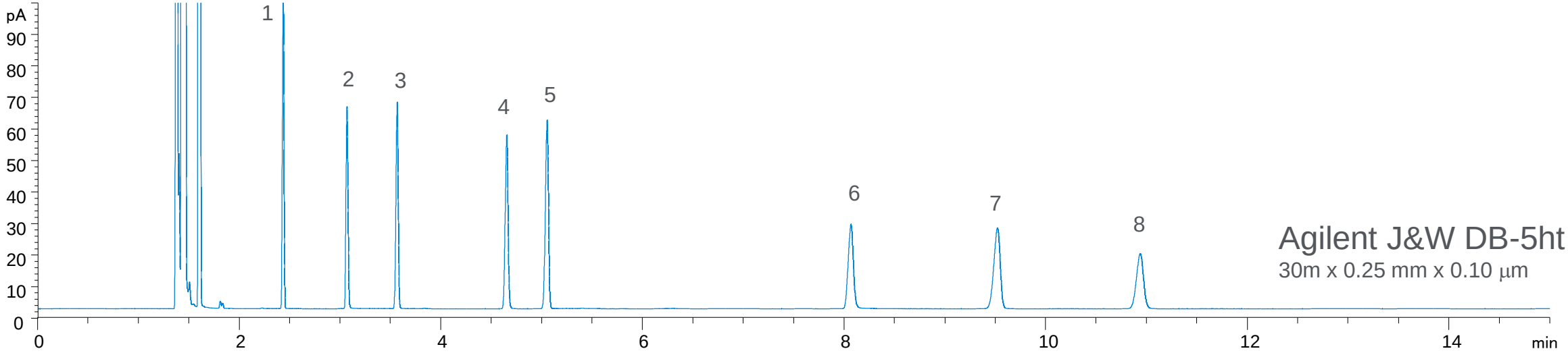
Initial Checkout Test Mix

Column	30m x 0.25 mm x 0.10 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**

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



Brand X-5ht
30m x 0.25 mm x 0.10 µm

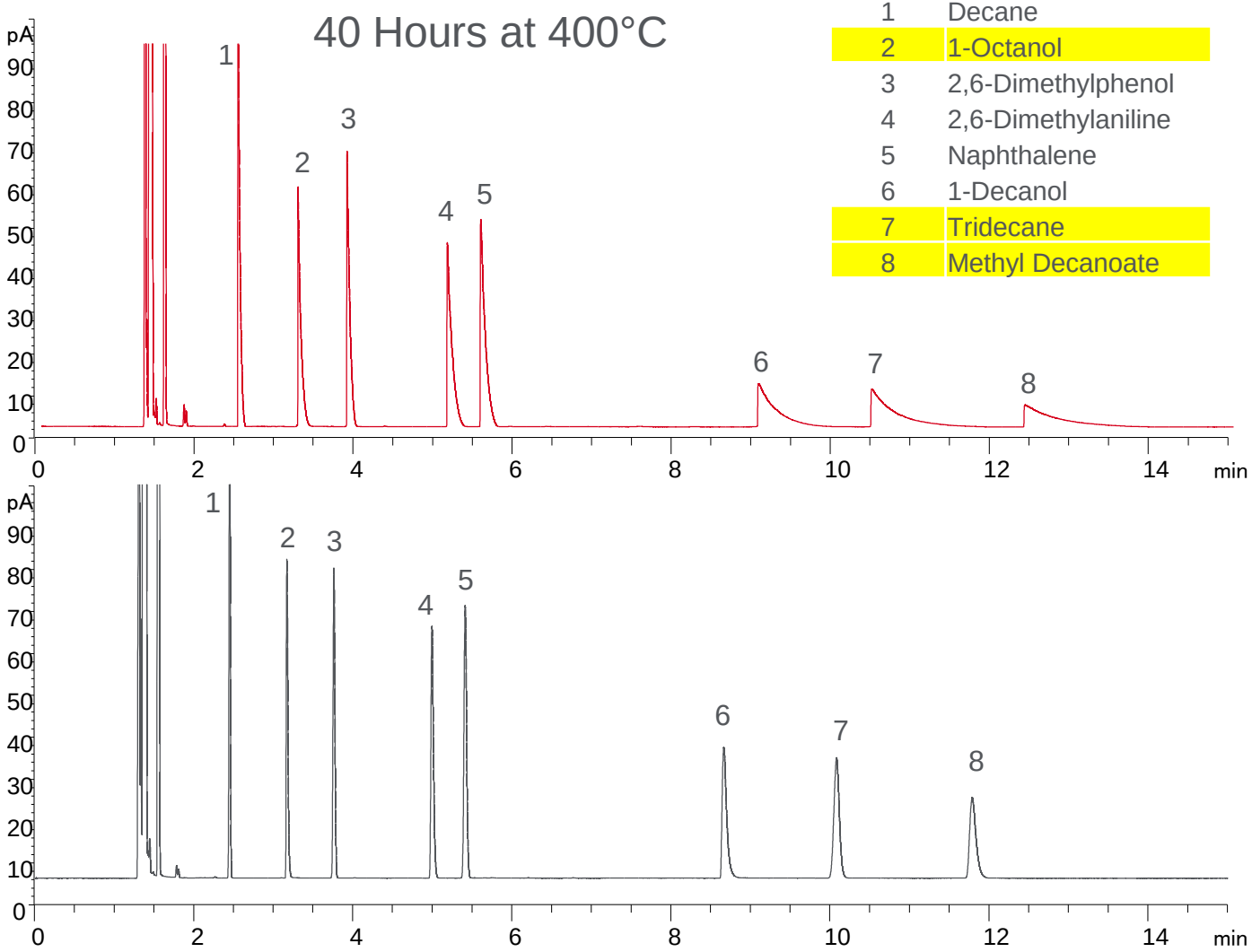


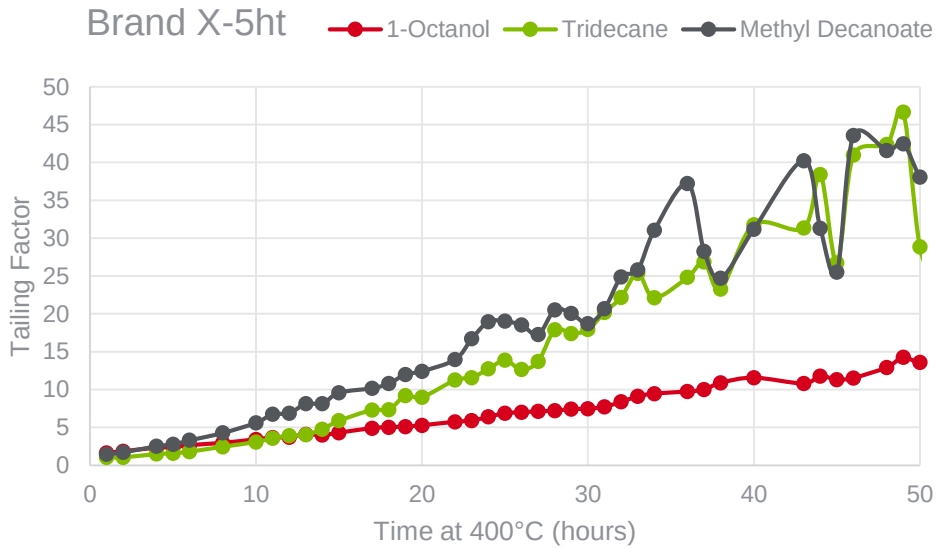
Agilent J&W DB-5ht
30m x 0.25 mm x 0.10 µm

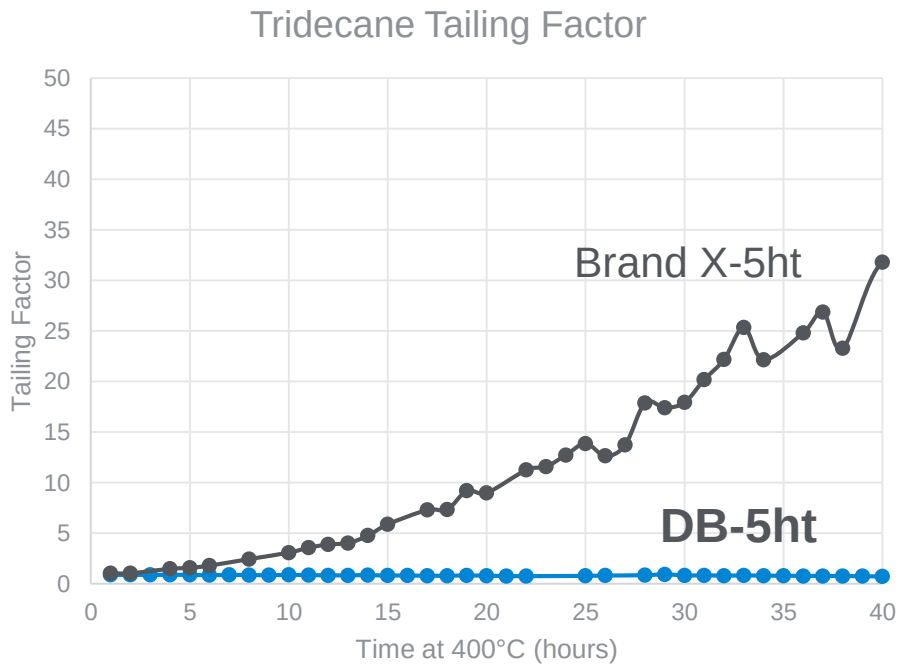
Agilent Publication 5994-1013EN

Brand X-5ht Peak Symmetry Degradation

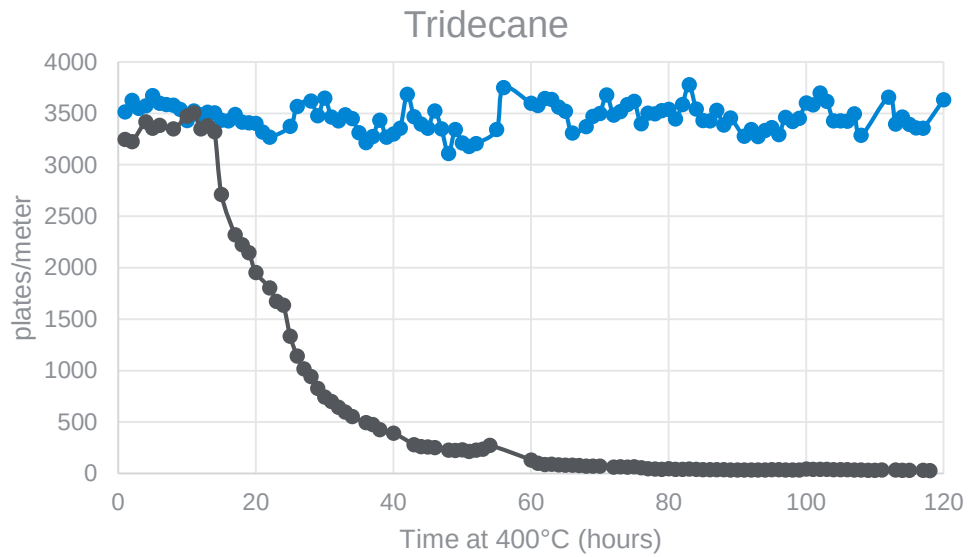
Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate







Column Efficiency Over 120 hours at 400°C



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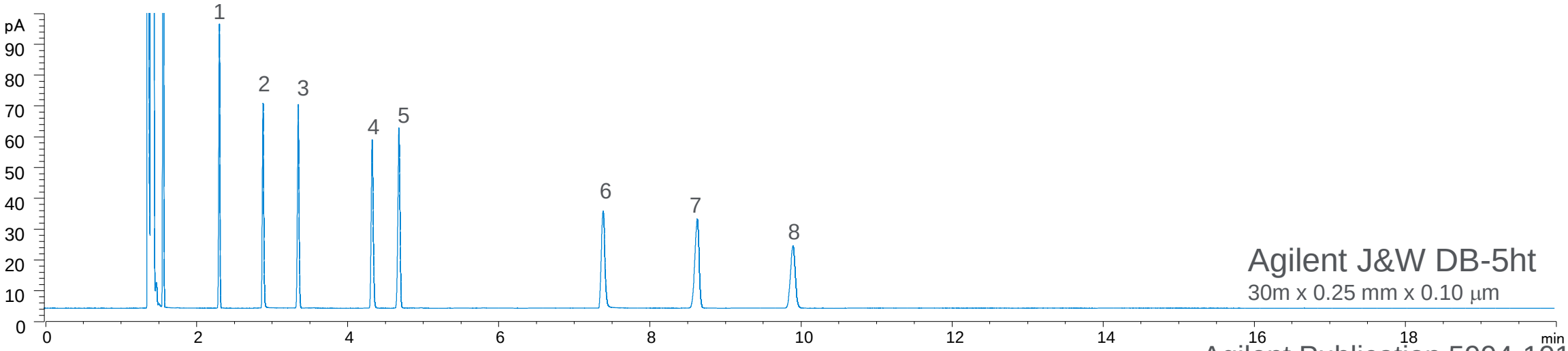
6	1-Decanol
7	Tridecane
8	Methyl Decanoate

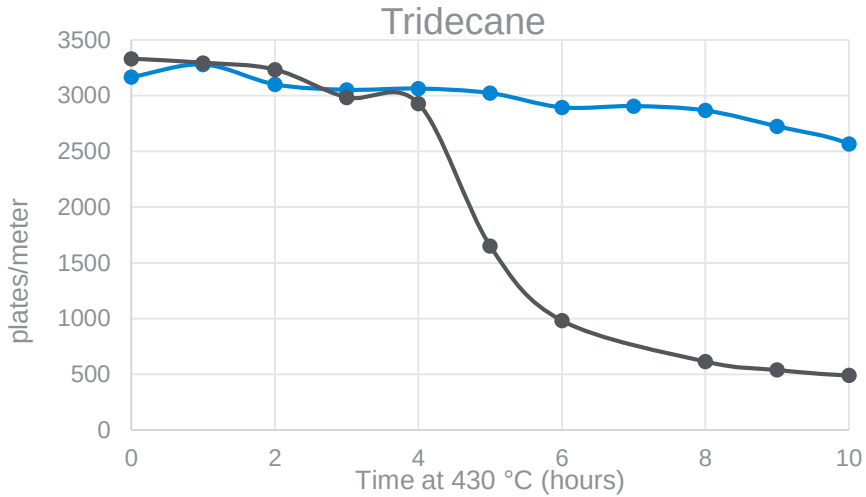
Brand X-5ht
30m x 0.25 mm x 0.10 µm



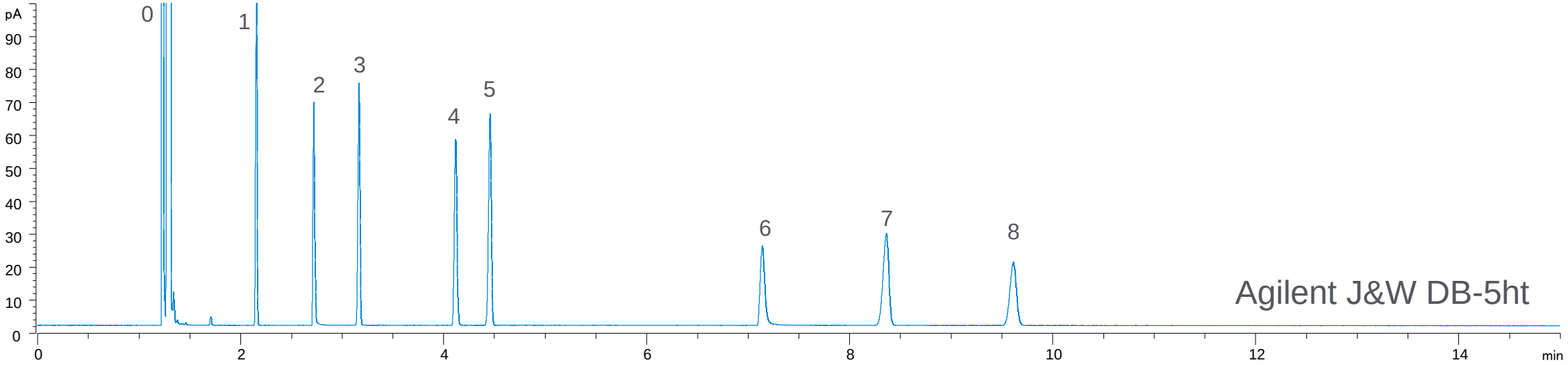
Agilent J&W DB-5ht
30m x 0.25 mm x 0.10 µm

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

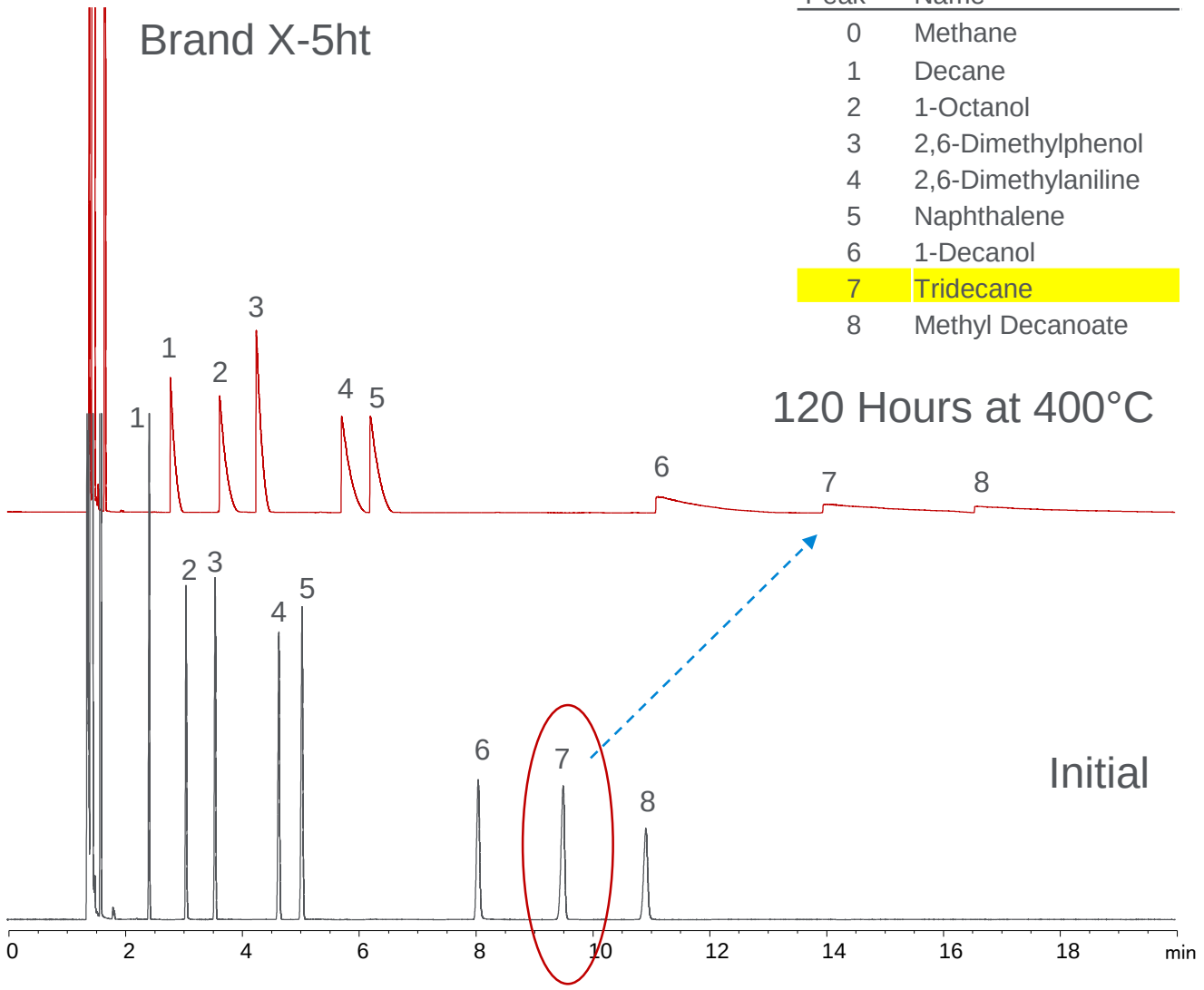


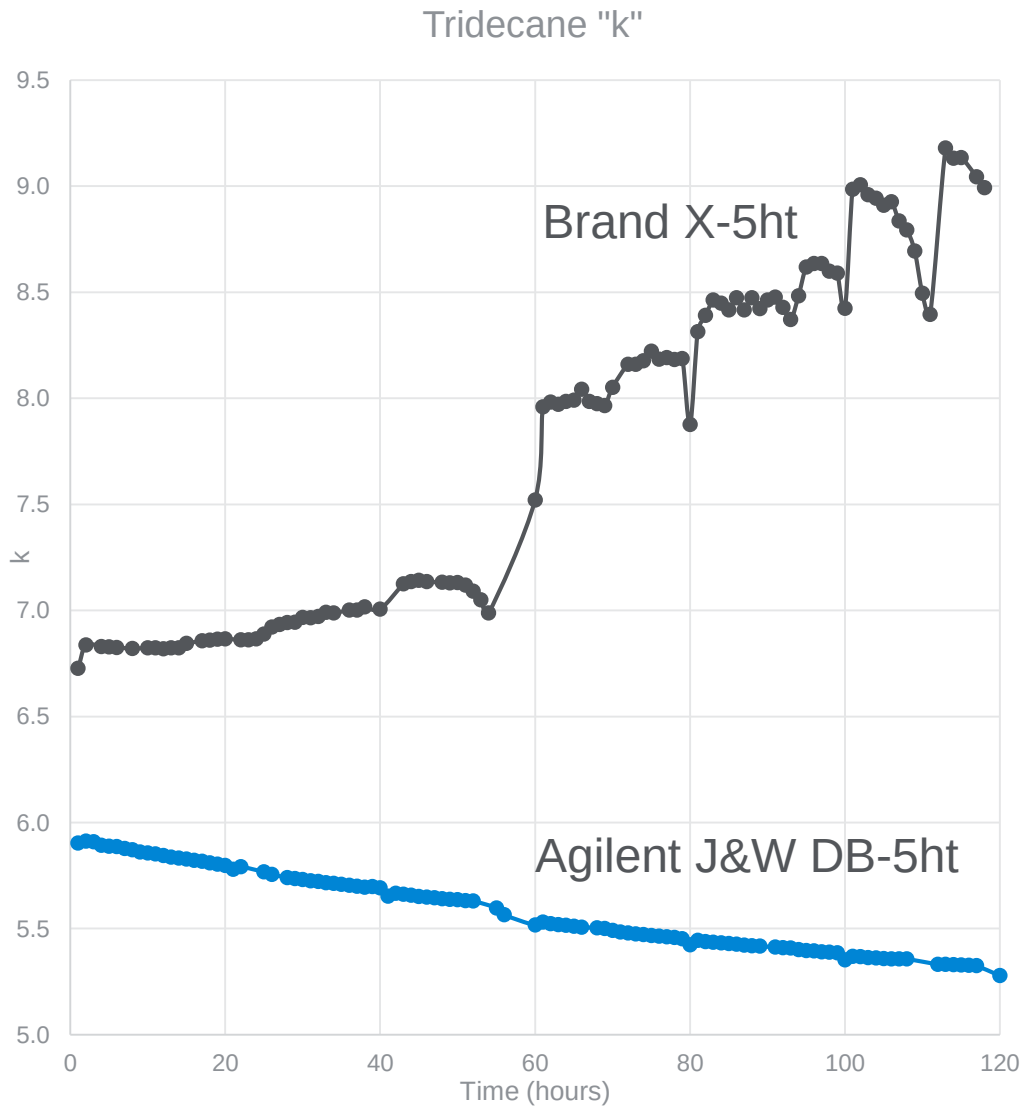
Agilent Publication 5994-1013EN

Phase Degradation Increases Retention

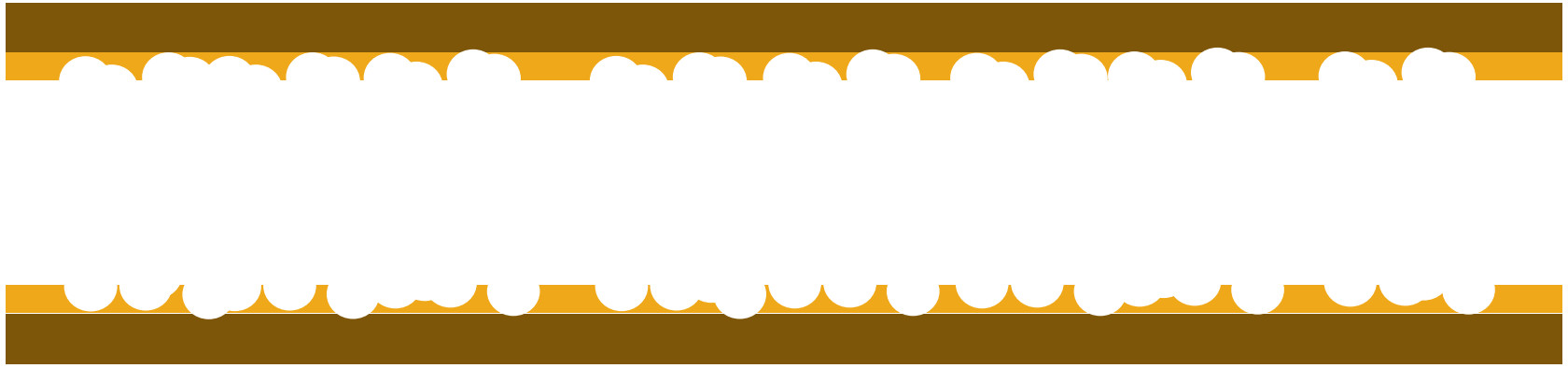
$$k = \frac{t_{compound} - t_{methane}}{t_{methane}}$$

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



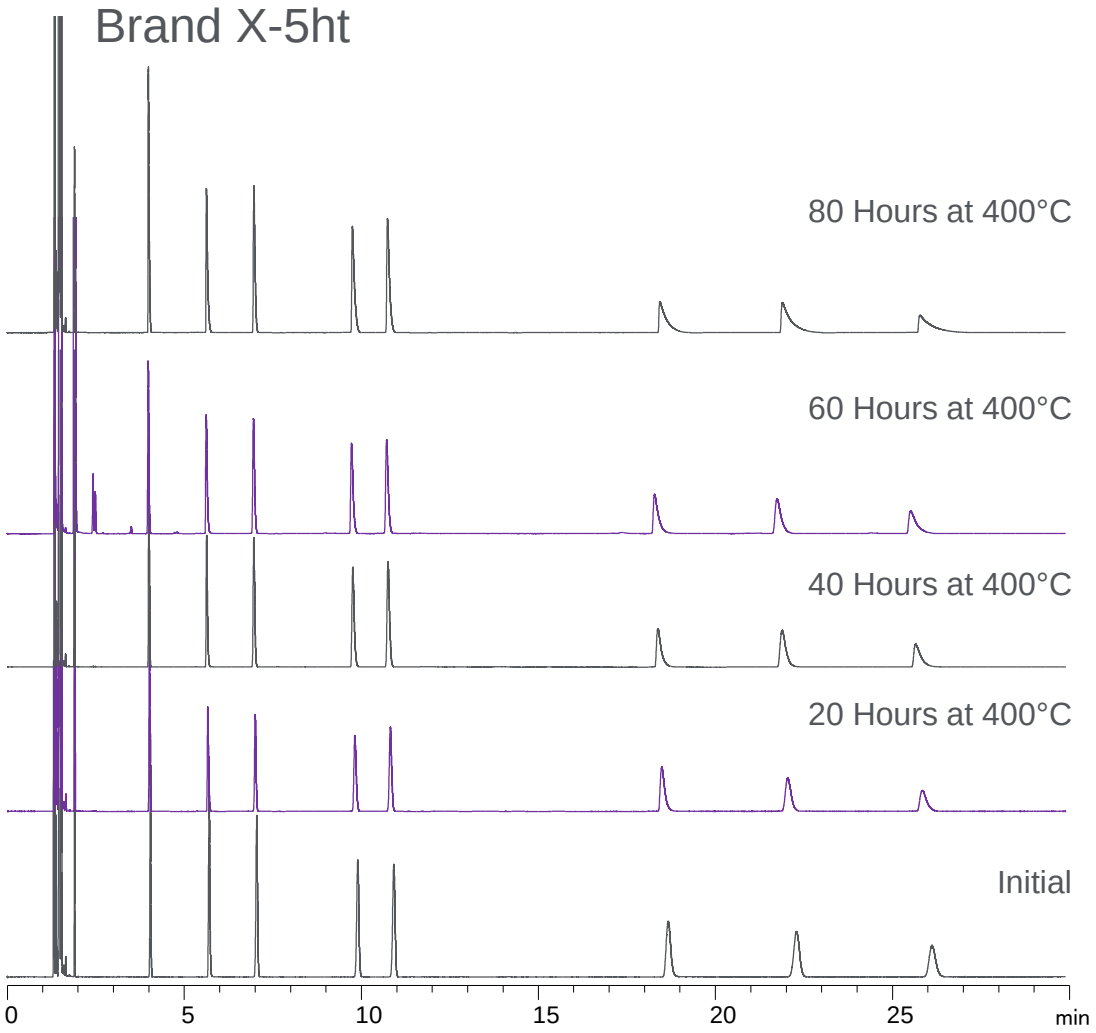


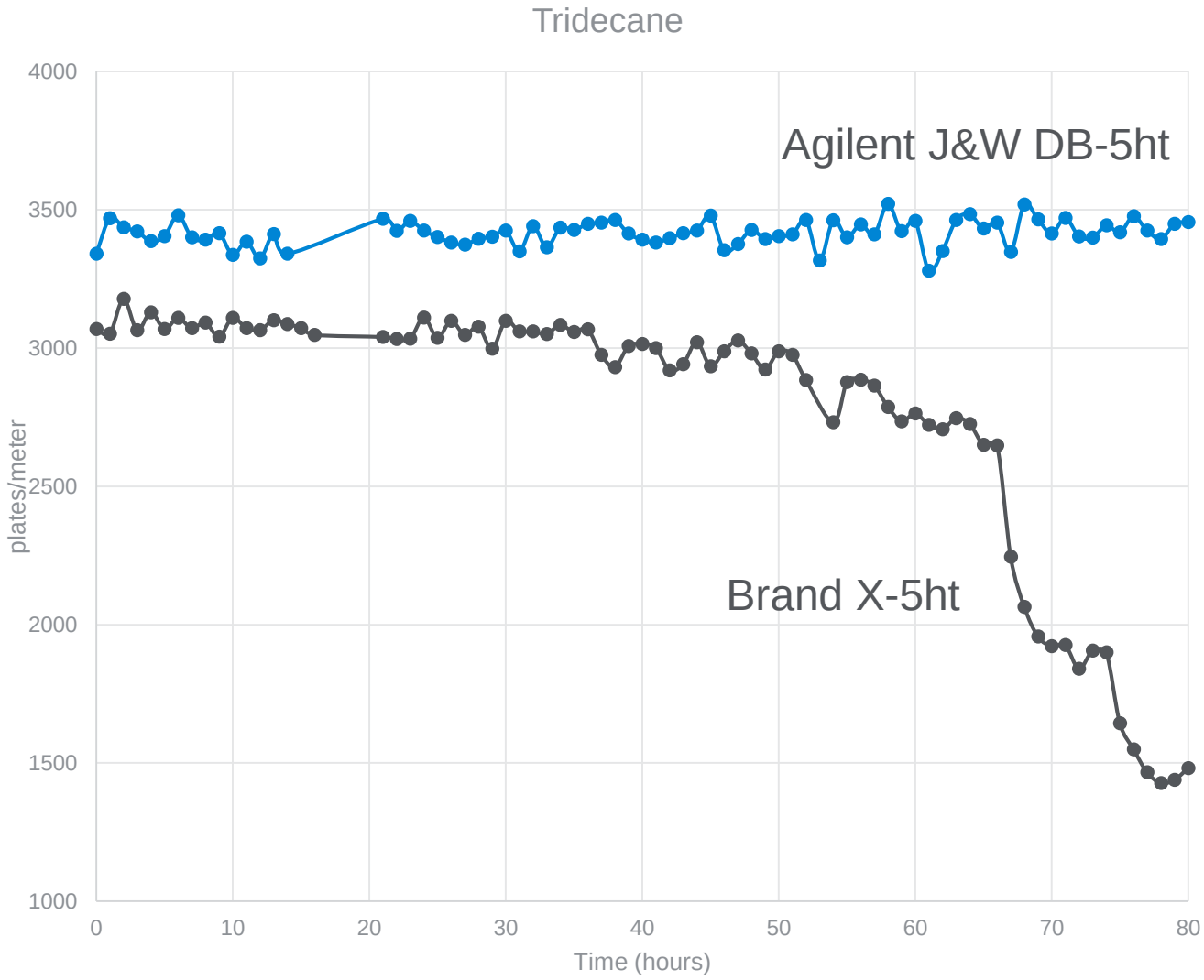
“Potholes” Created as the Phase Degrades
Raw Fused Silica exposed…..



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



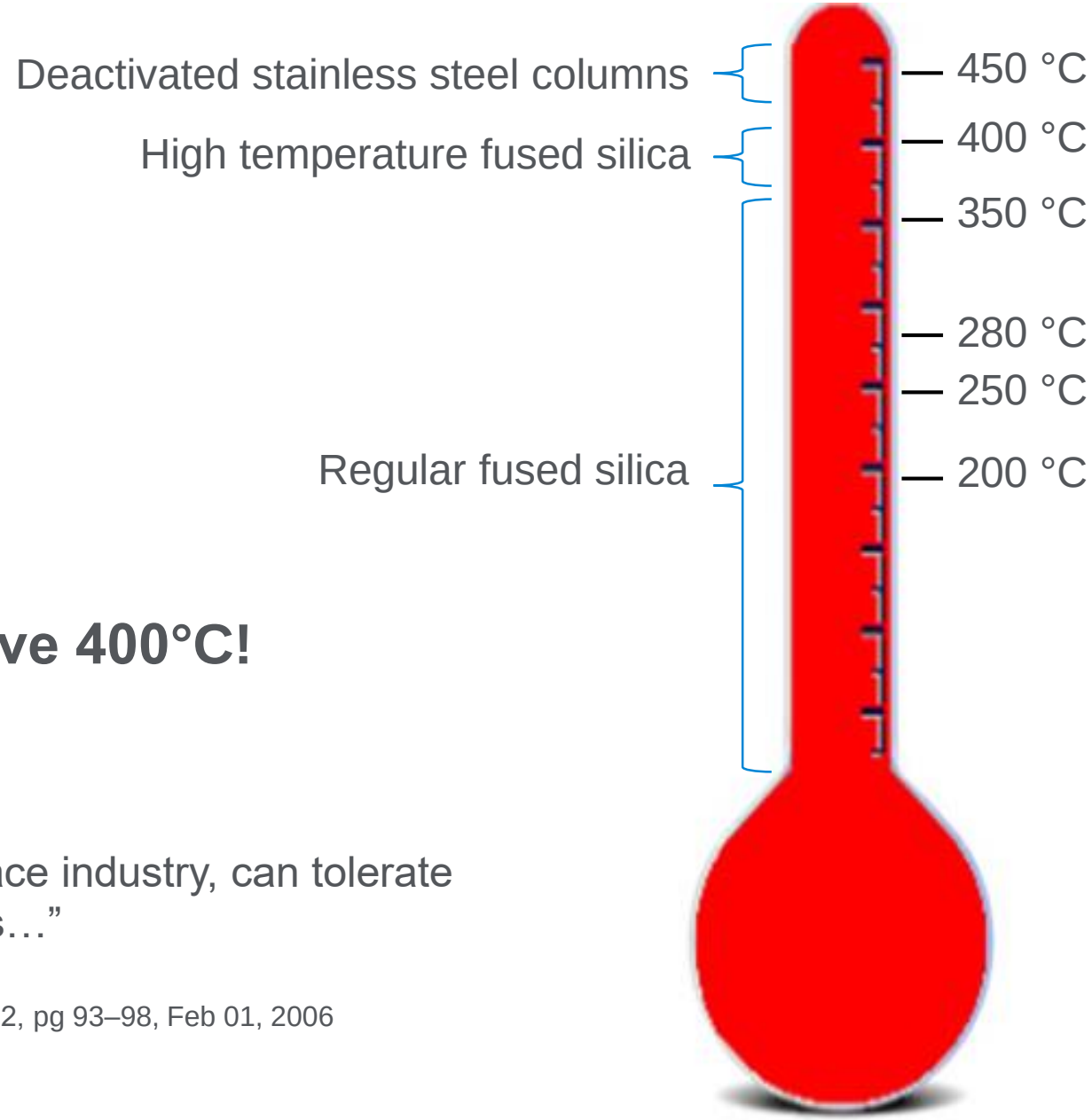


The outside matters too….

Let’s talk about substrate

Agilent

Columns by Temperature Limit



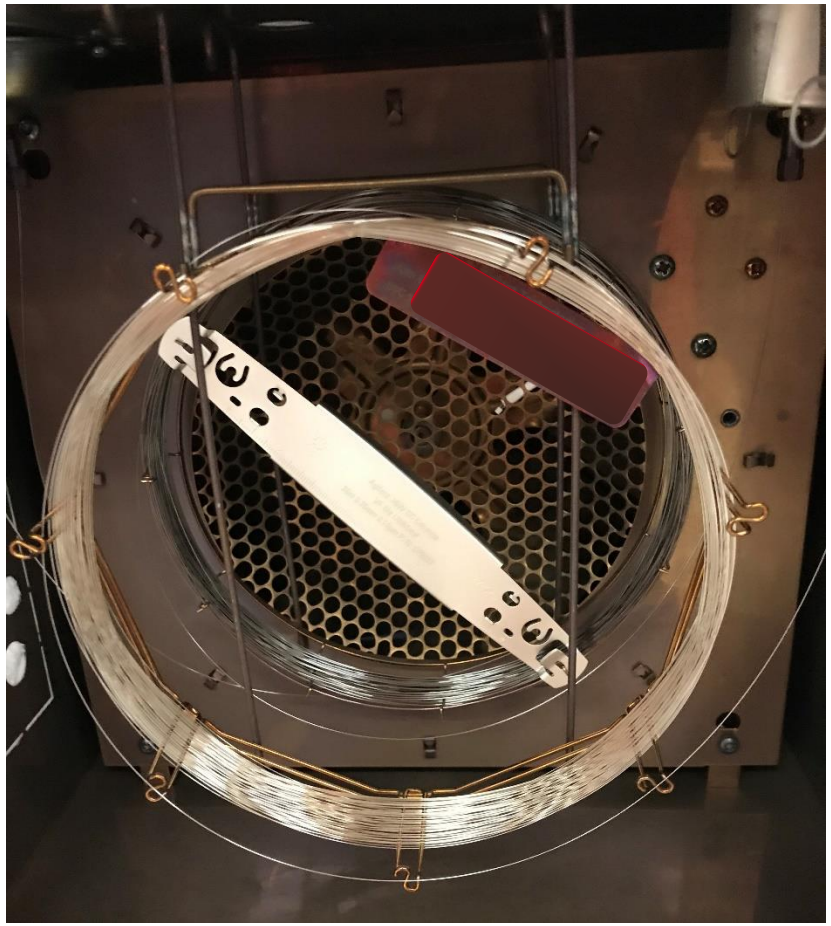
Don’t take fused silica columns above 400°C!

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

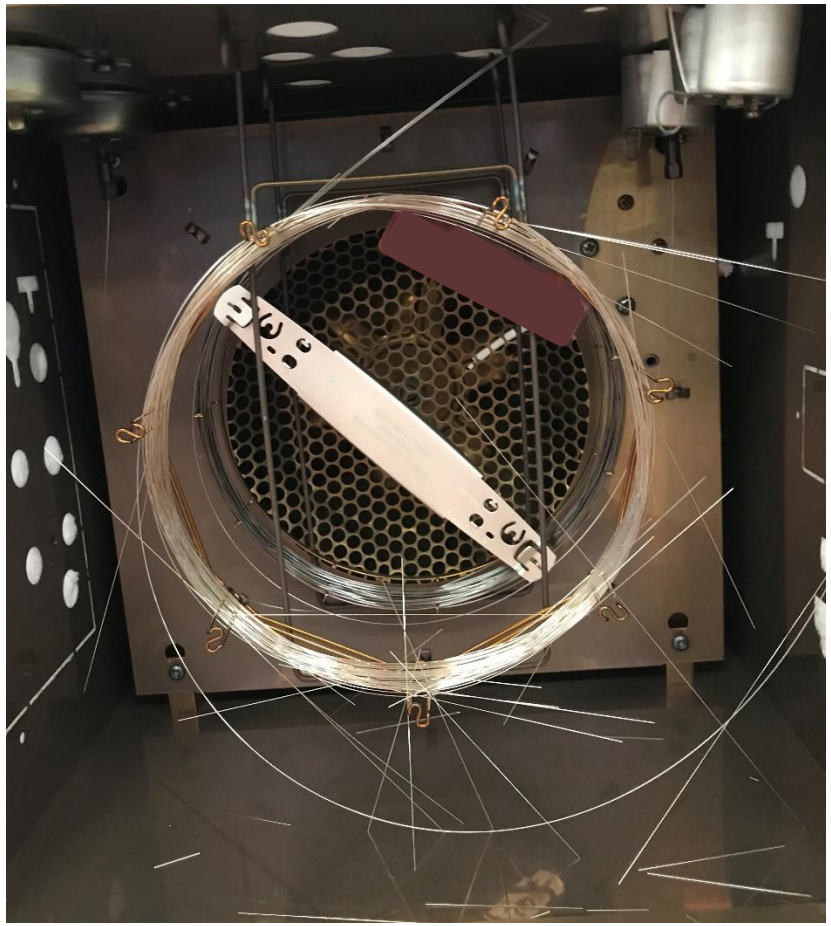
John V. Hinshaw, “The Making of a Column” LCGC Europe, Volume 19, Issue 2, pg 93–98, Feb 01, 2006

Don’t let this happen to you…..

Don’t Touch!!!!!!

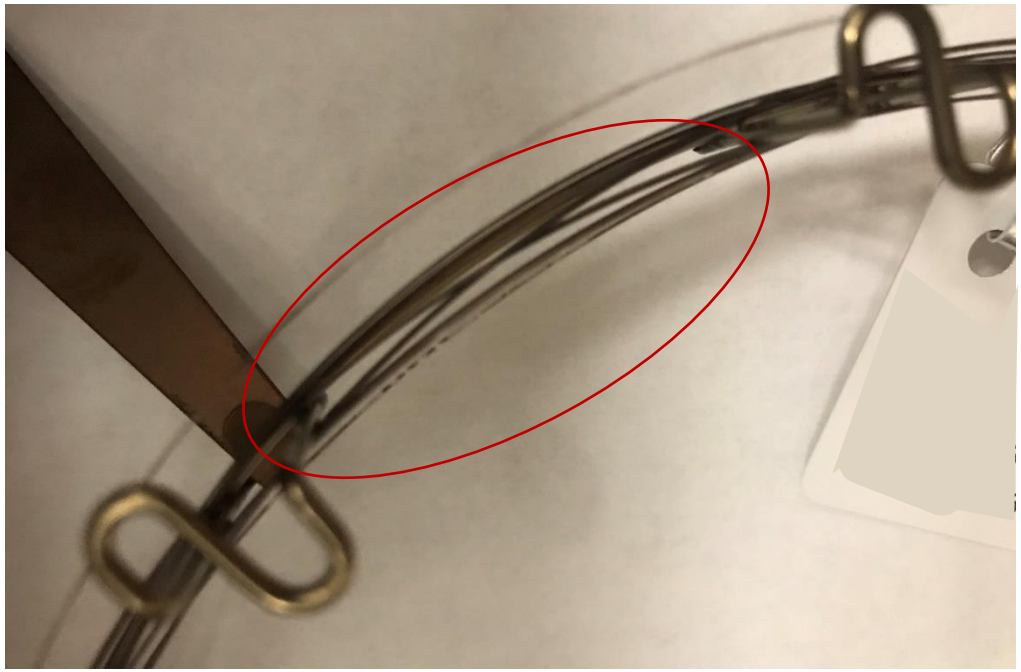


Ahhhhh!!!!

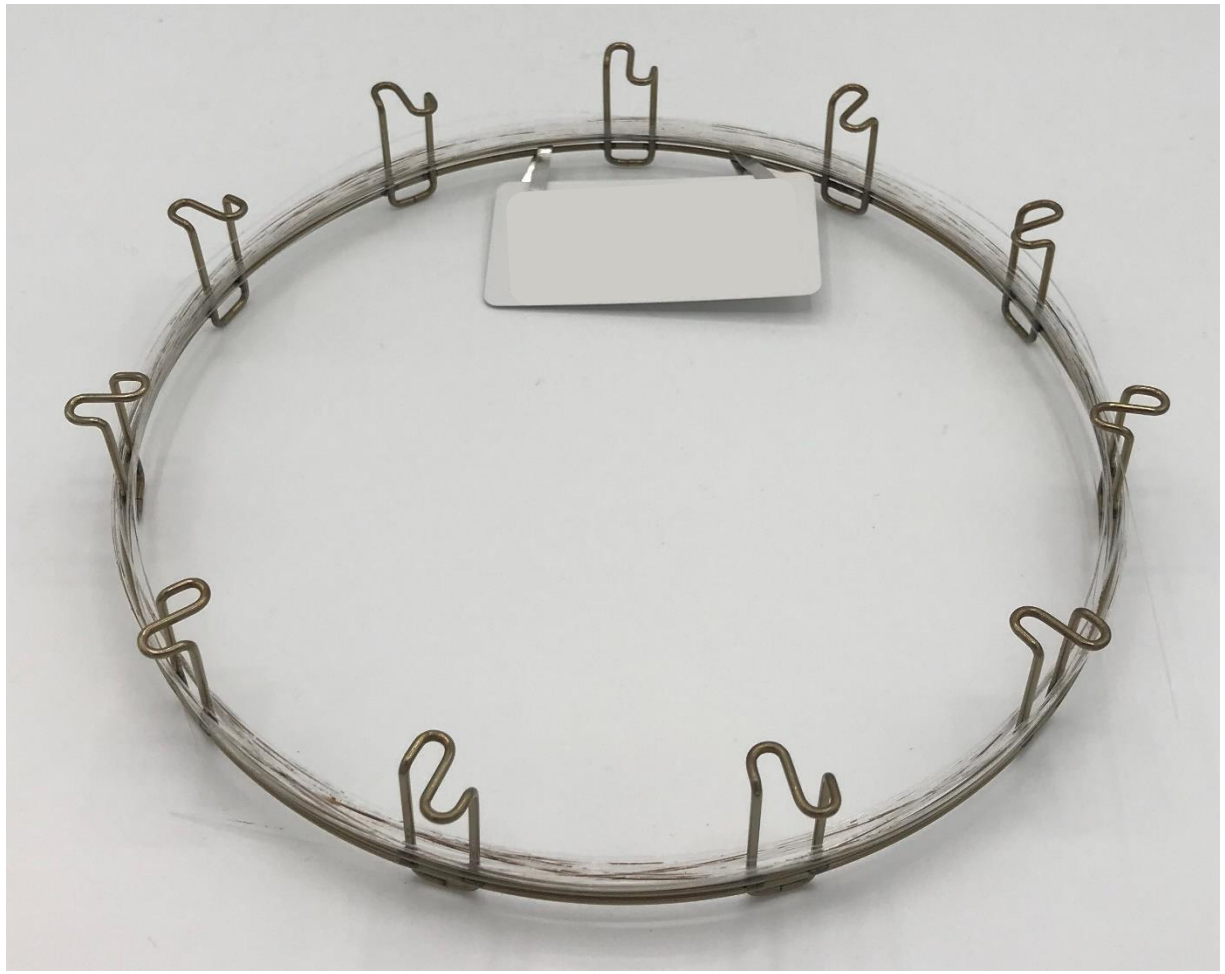


Competitor Fused Silica doesn’t always “take the heat”

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

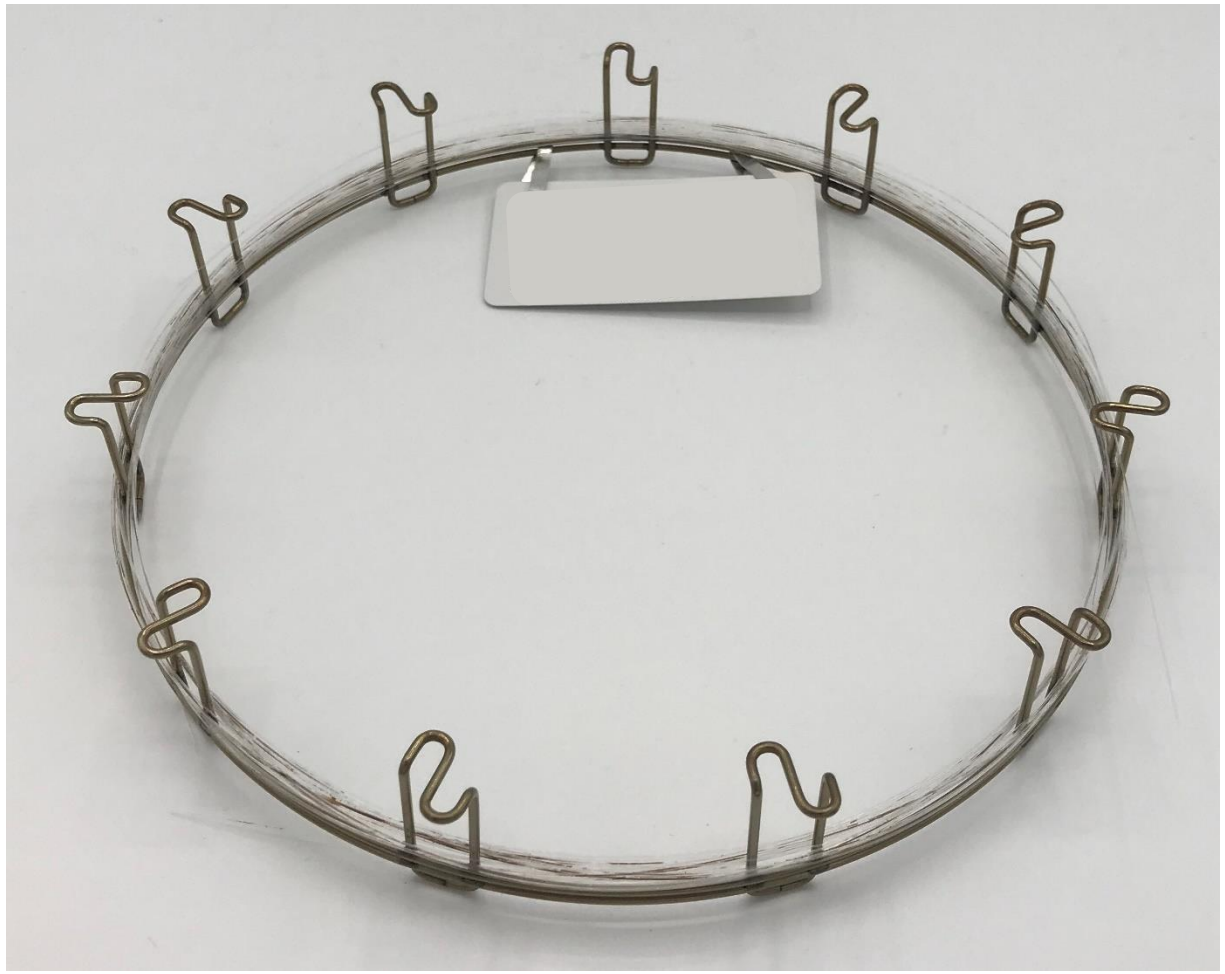


Brand Y
After 25 hours at 400°C

Agilent Publication 5994-1013EN

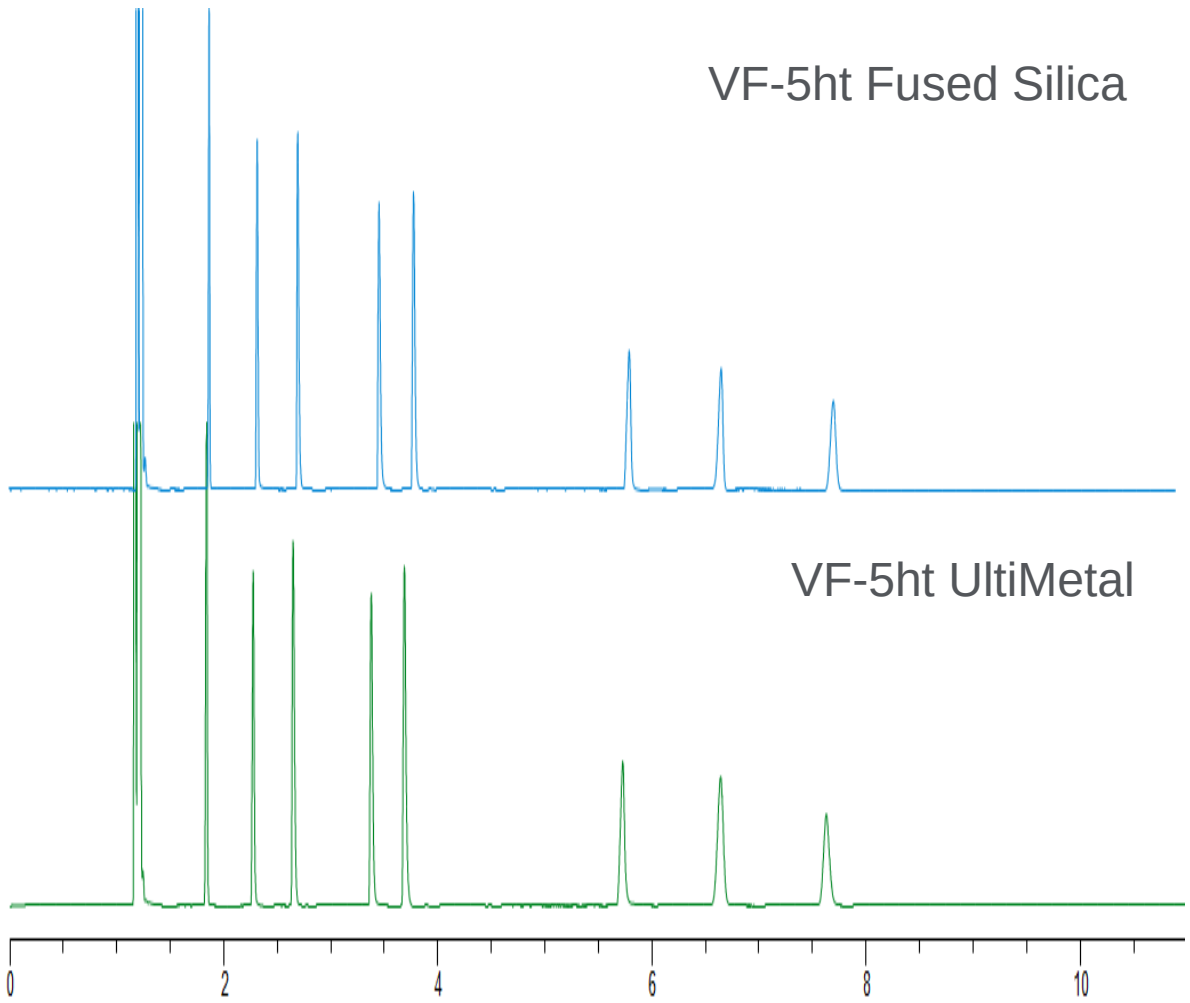
Competitor Fused Silica doesn’t always “take the heat”

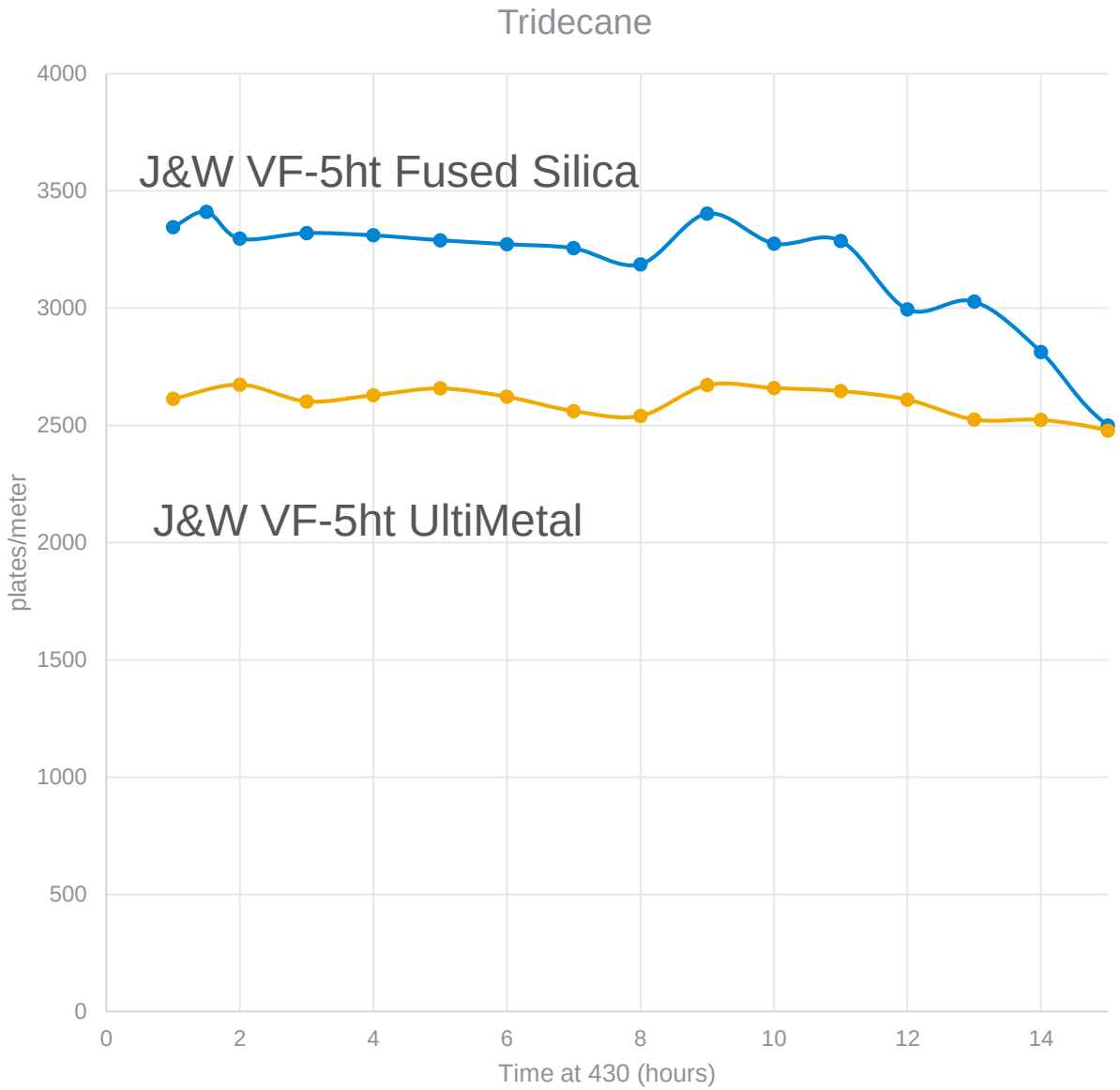
Agilent J&W DB-5ht



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?

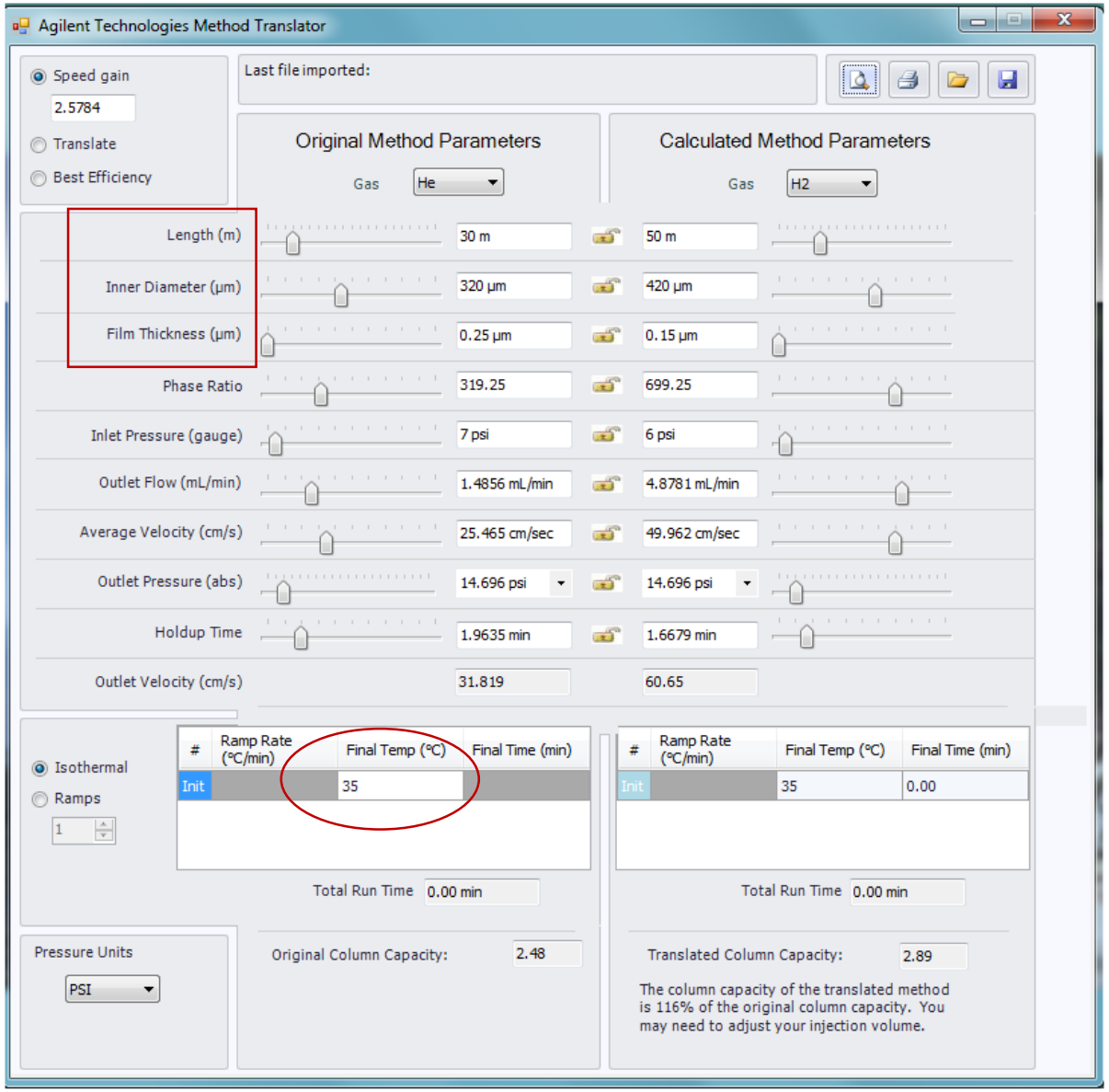




Agilent Publication 5994-1385EN

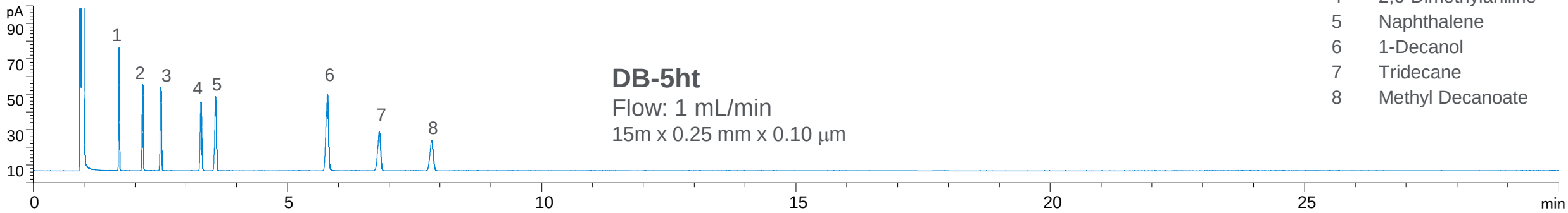
What is the best way to get heavy compounds off a column?

- ❑ Apply Heat
- ❑ Increase Column Flow
- ❑ Decrease Column Length
- ❑ Decrease Film Thickness
- ❑ Increase Diameter

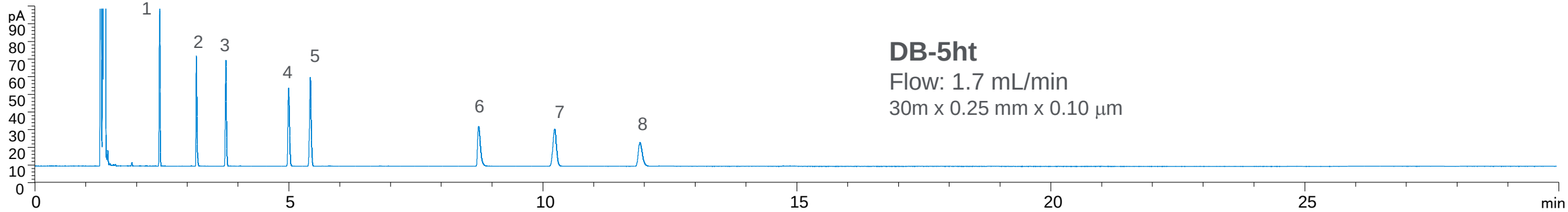


Short, Thin Film Columns Elute Compounds Faster

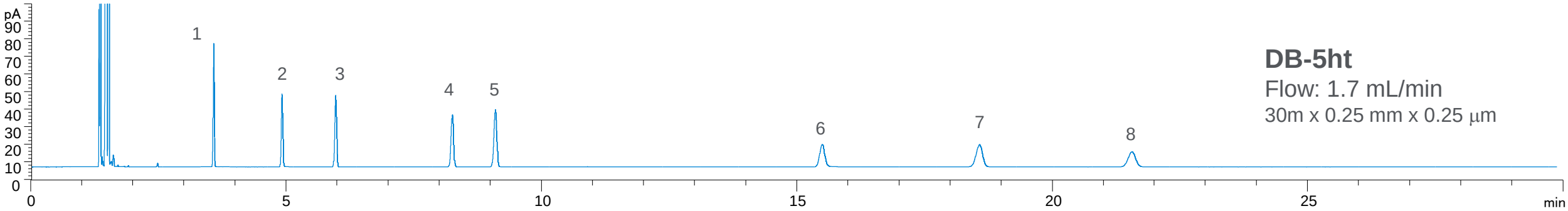
Peak	Name
0	Methane
1	Decane
2	1-Octanol
3	2,6-Dimethylphenol
4	2,6-Dimethylaniline
5	Naphthalene
6	1-Decanol
7	Tridecane
8	Methyl Decanoate



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

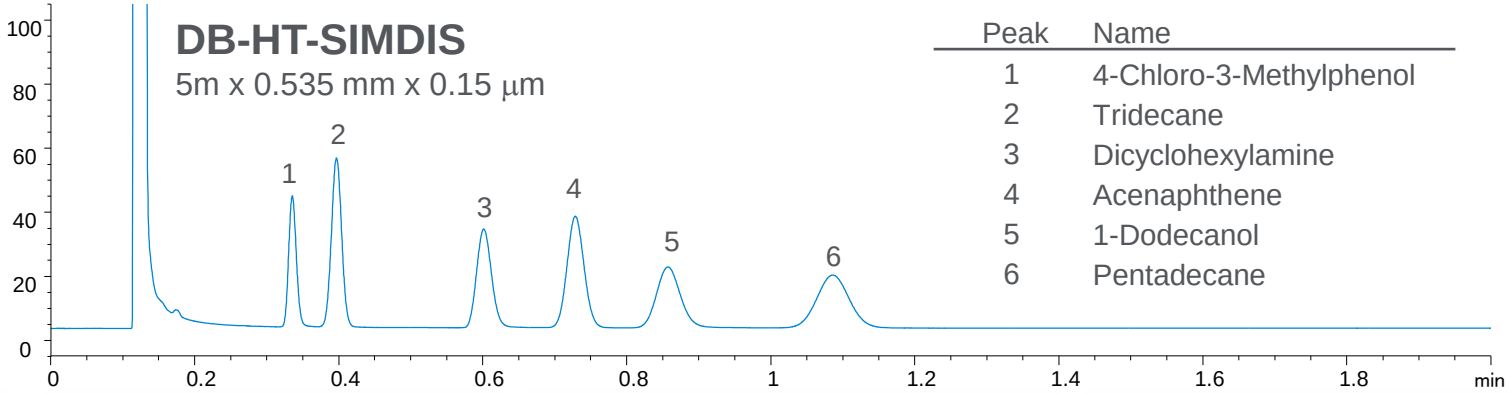


DB-5ht
Flow: 1.7 mL/min
30m x 0.25 mm x 0.10 µm



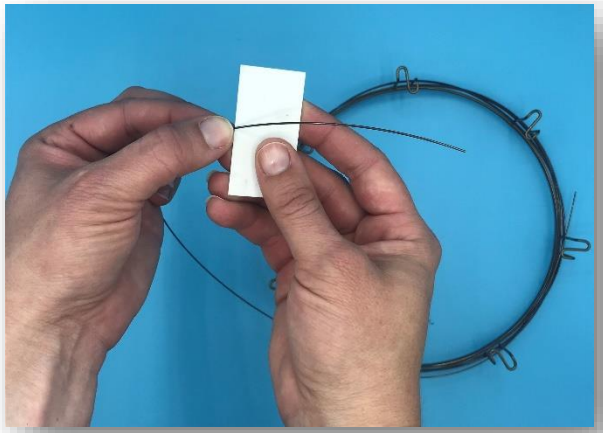
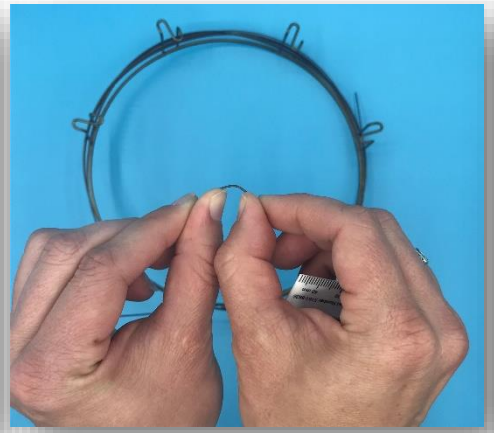
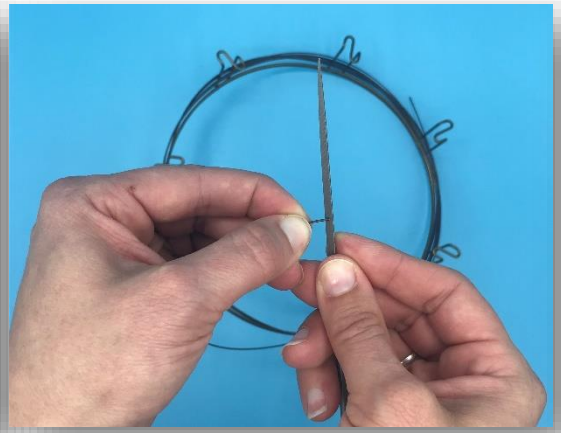
DB-5ht
Flow: 1.7 mL/min
30m x 0.25 mm x 0.25 µm

Metal Columns Are Easy to Cut Properly

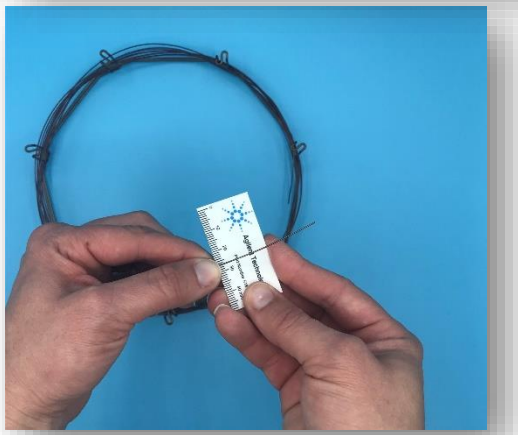
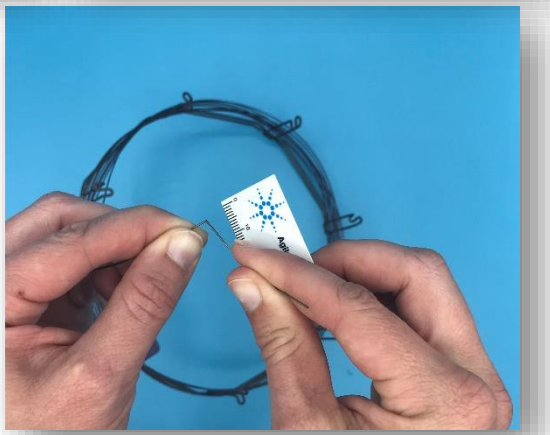
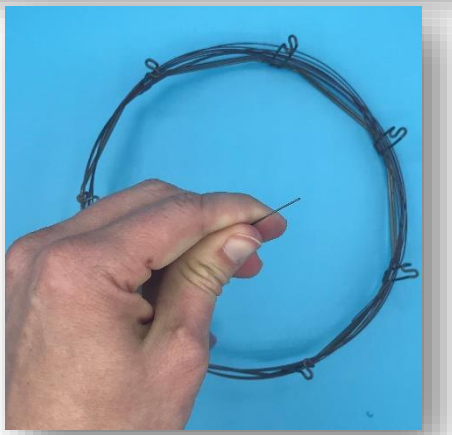


Peak	Name
1	4-Chloro-3-Methylphenol
2	Tridecane
3	Dicyclohexylamine
4	Acenaphthene
5	1-Dodecanol
6	Pentadecane

"Glass Infused" Metal Column

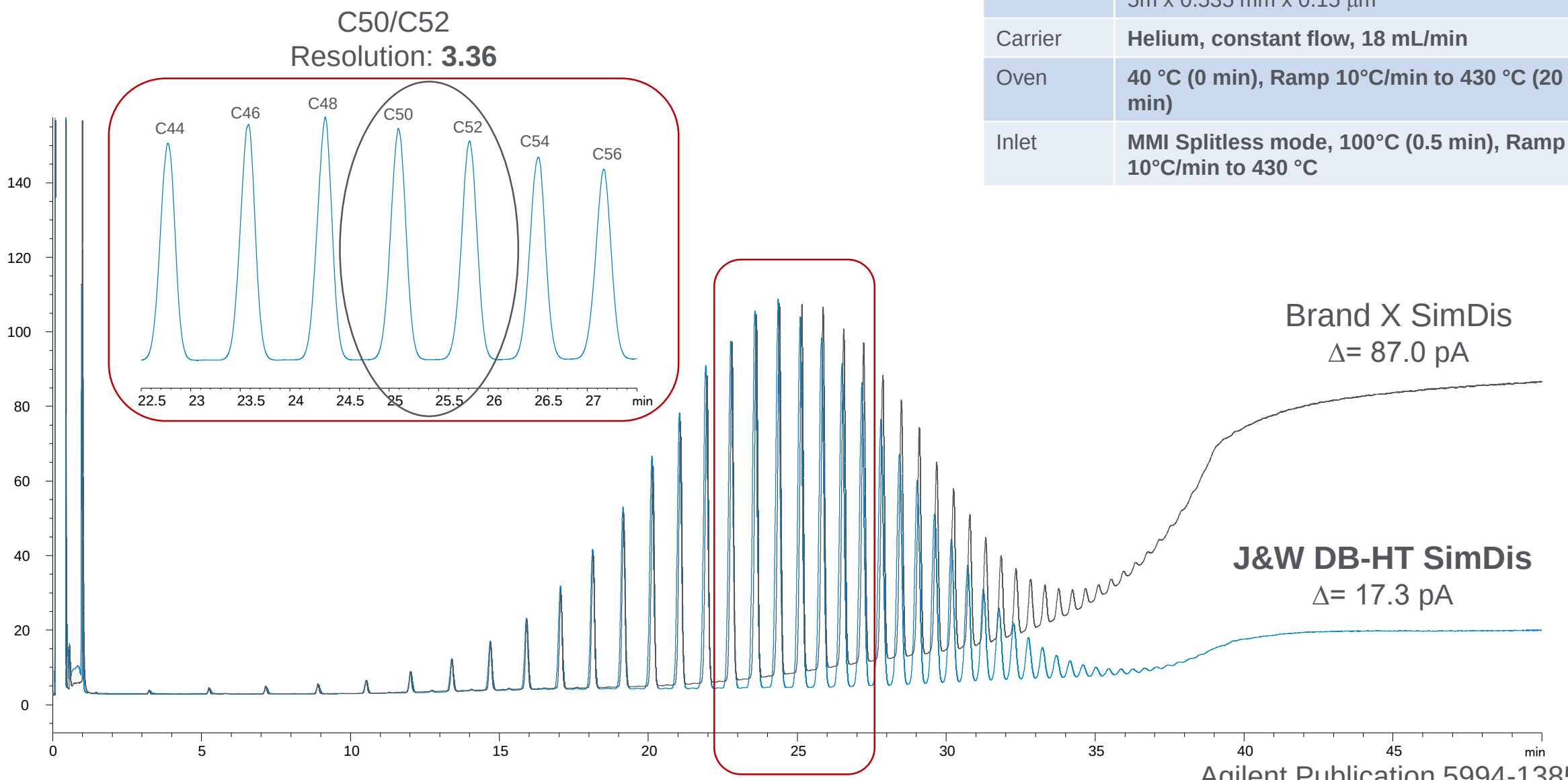





DB-HT-SimDis ProSteel

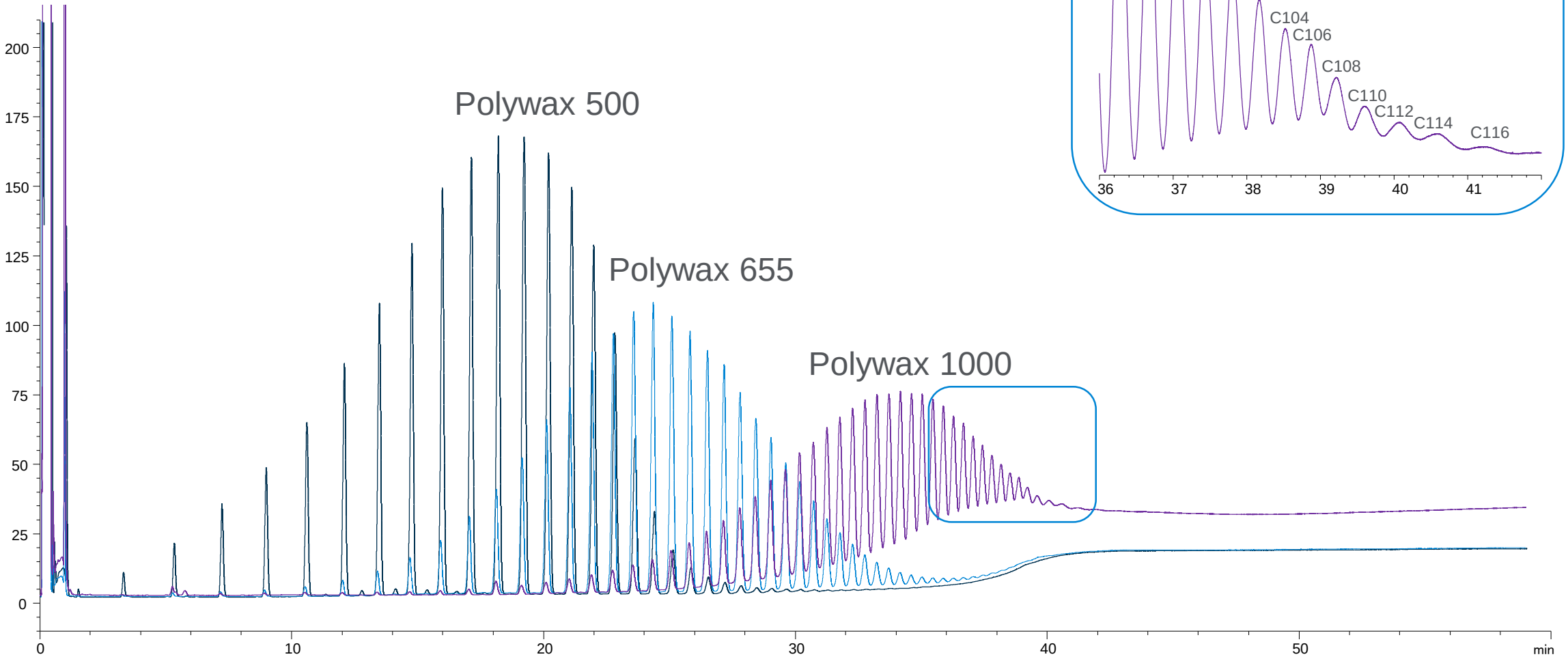
Lower bleed with Agilent ProSteel Columns

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



Agilent Publication 5994-1385EN

All the Polywax DB-HT SIM DIS



Agilent Publication 5994-1385EN

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

High Temp Applications

Advanced Green Septa
Long Life Septa

35°C — 350 °C

BTO Septa

400 °C

Graphite/Vespel ferrules

35 °C — 350 °C

Graphite ferrules
Ultimetal ferrules

450 °C

Fluorocarbon O-rings

35 °C — 350 °C

Graphite seals

450 °C

Ultra Inert liners

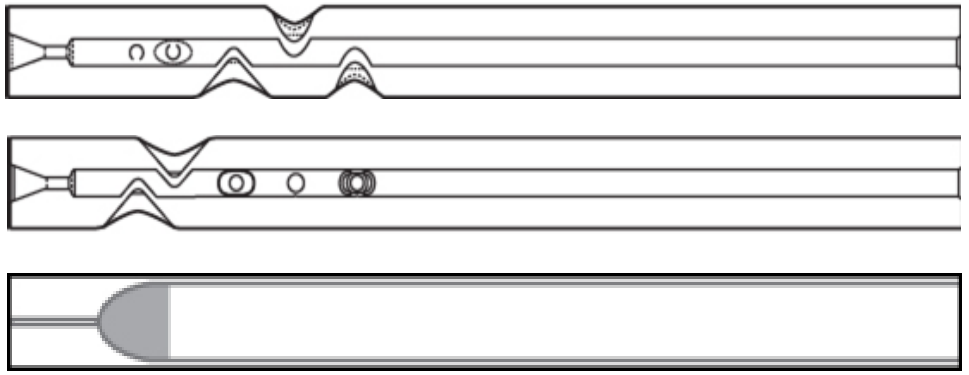
35 °C — 375 °C

Standard liners

450 °C

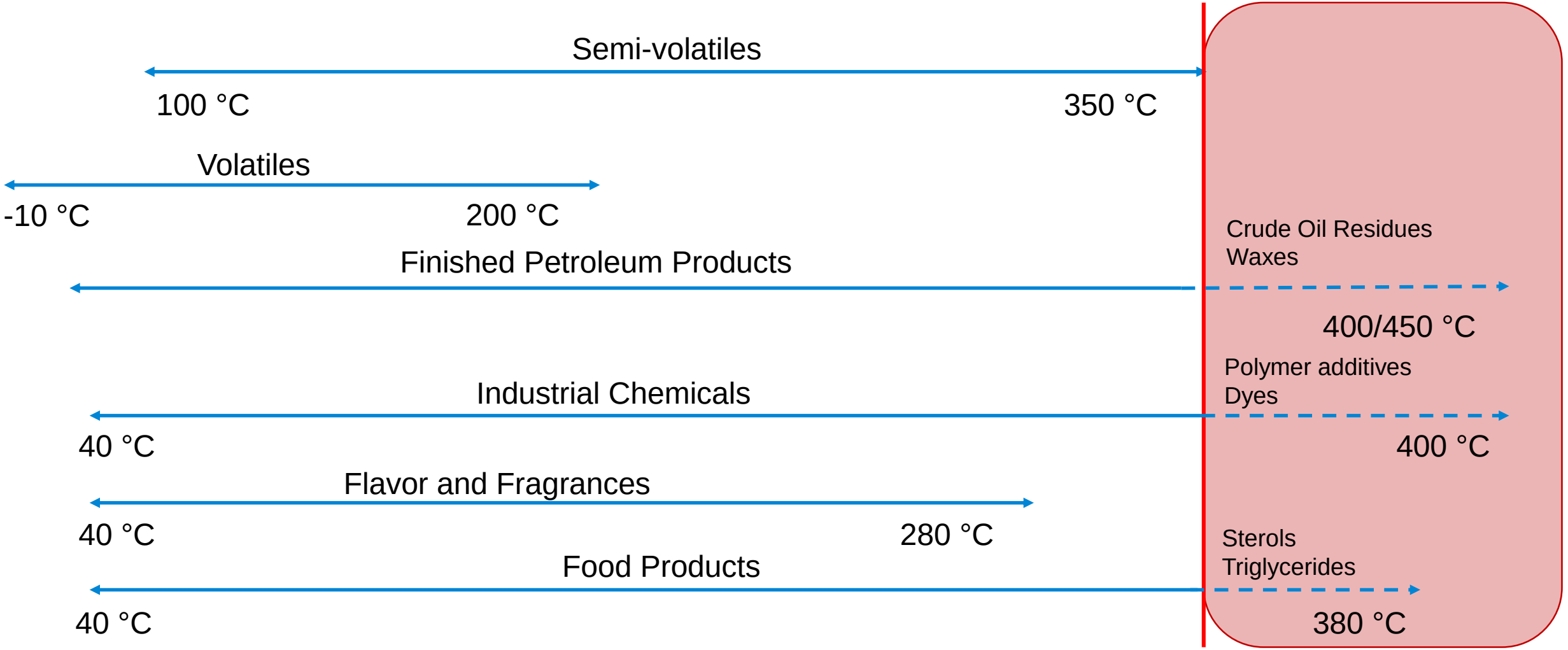
Advantages of MMI and “Cold” Splitless

- 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

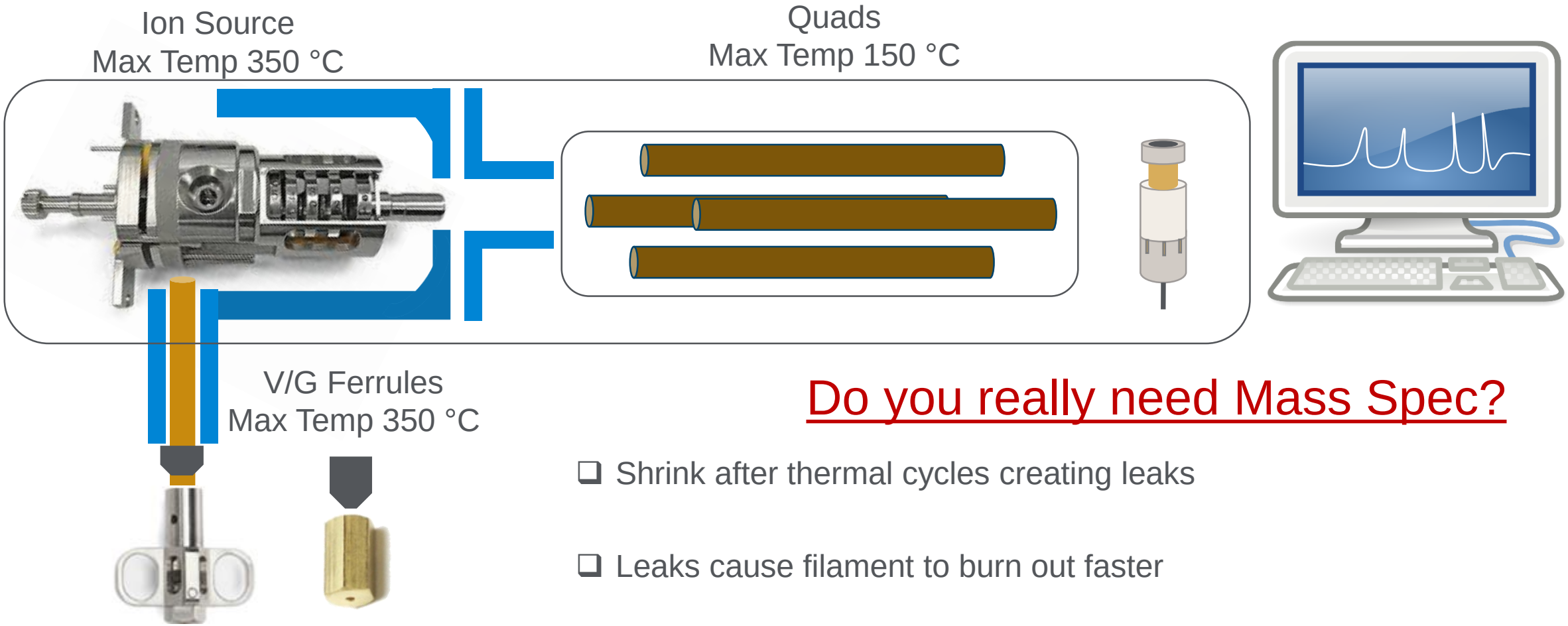
Temperature Range, Applications, & **Detectors**



MSD/ FID/ ECD/ TCD/ FPD/ SCD

FID/SCD

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

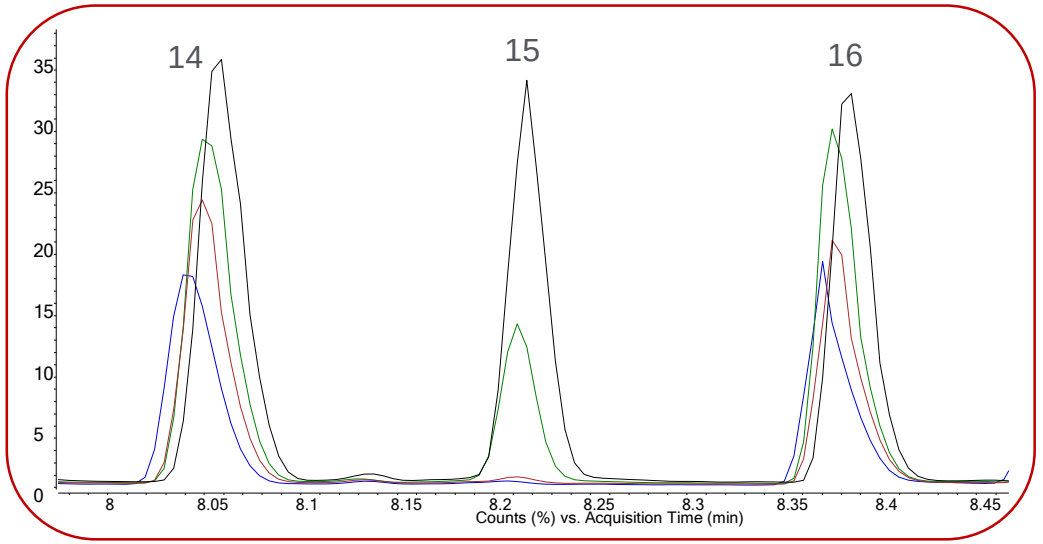


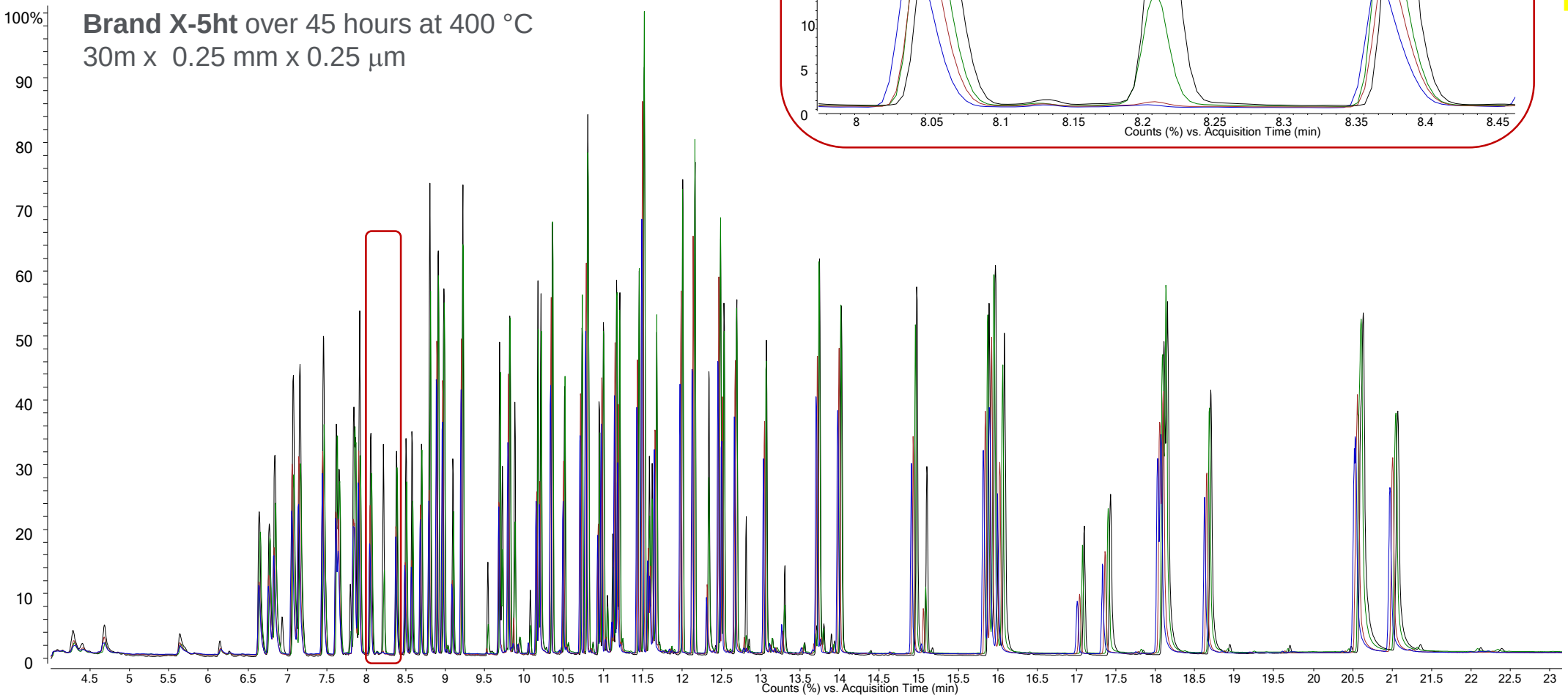
Do you really need Mass Spec?

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

Loss of Sensitivity & Loss of Inertness

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



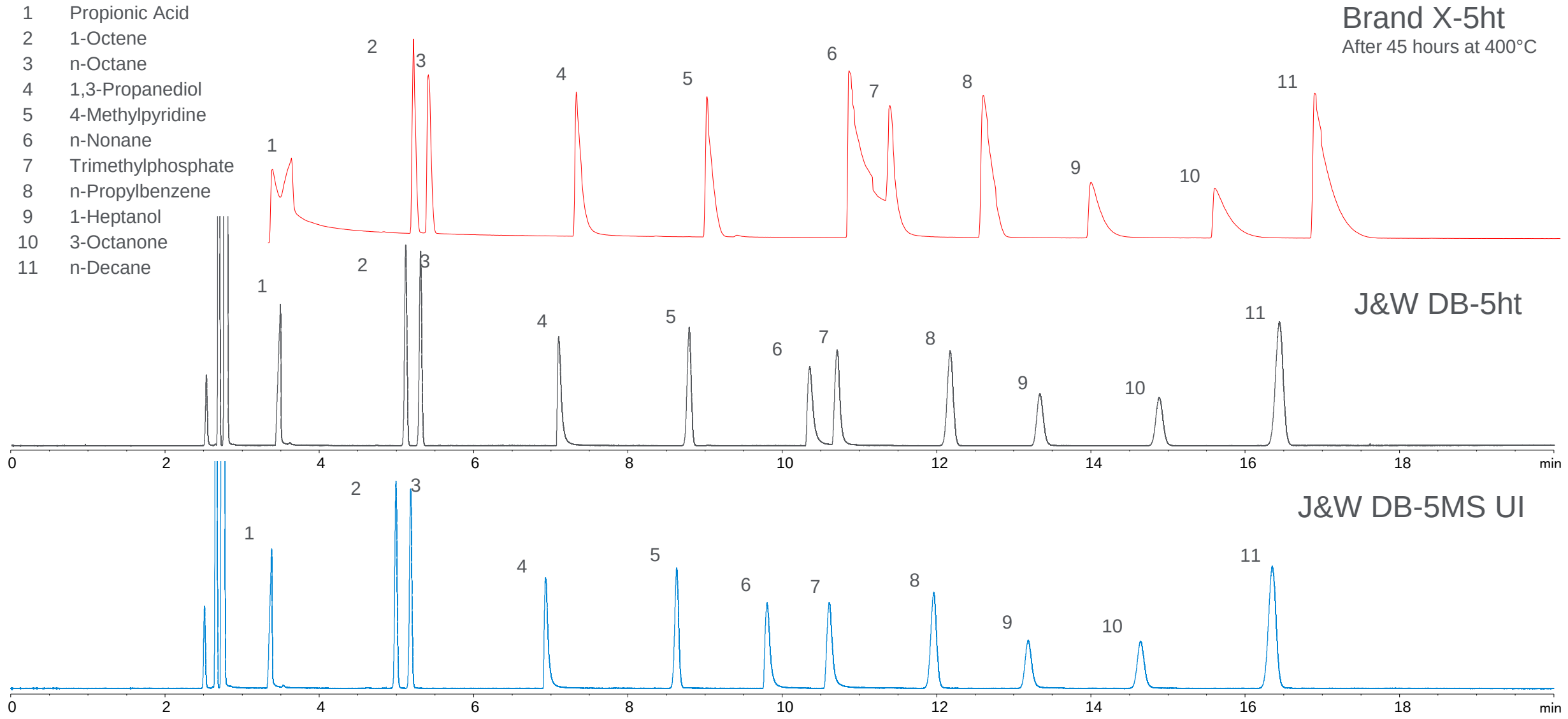


SVM-8270-1

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	Benz[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	Dibenz[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



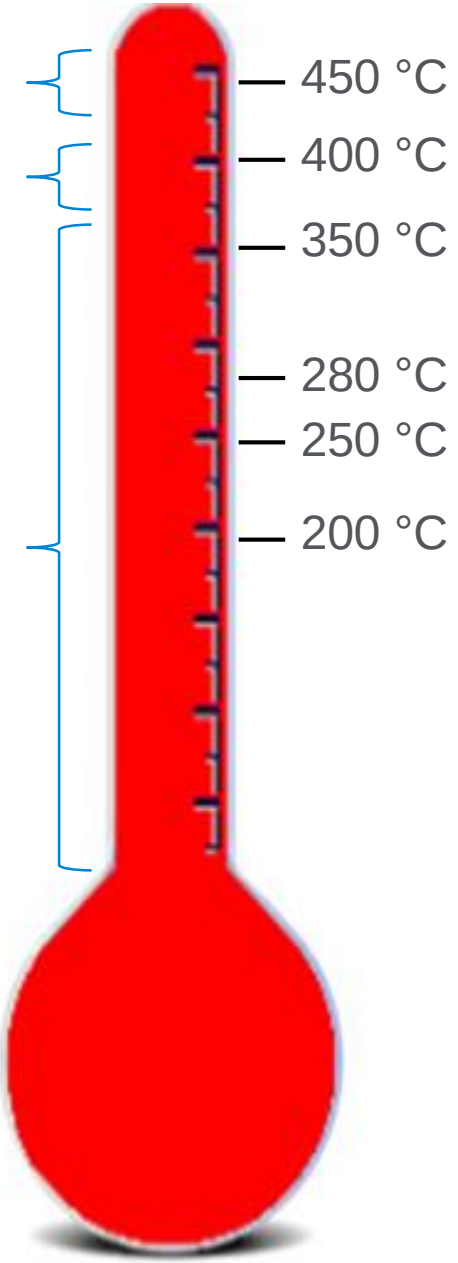
Summary

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

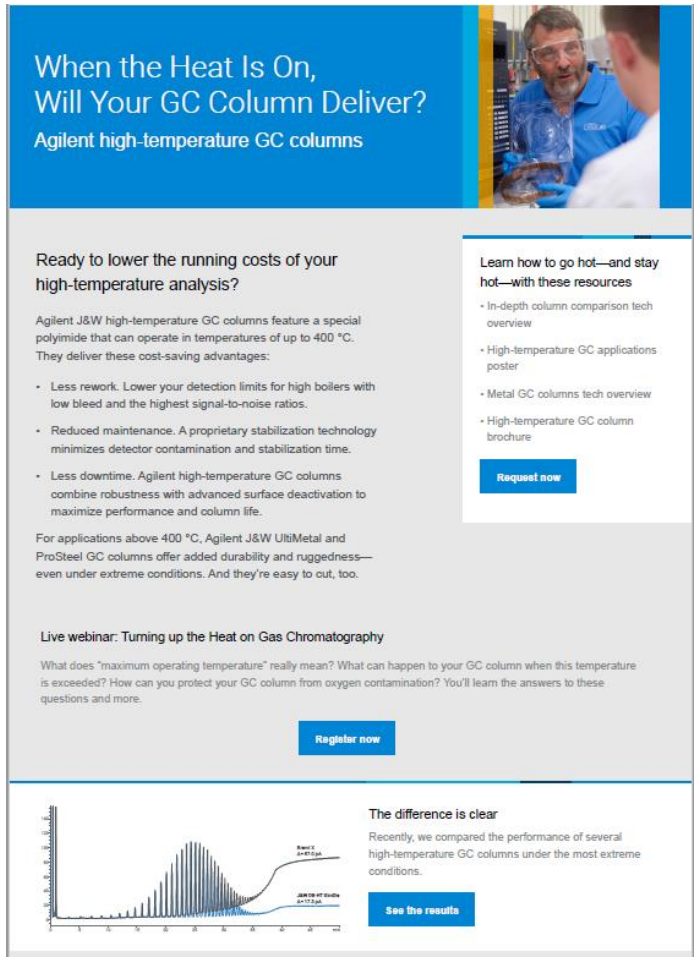
- If going to operate above 400°C use Deactivated Stainless Steel columns such as ProSteel and UltiMetal GC Columns

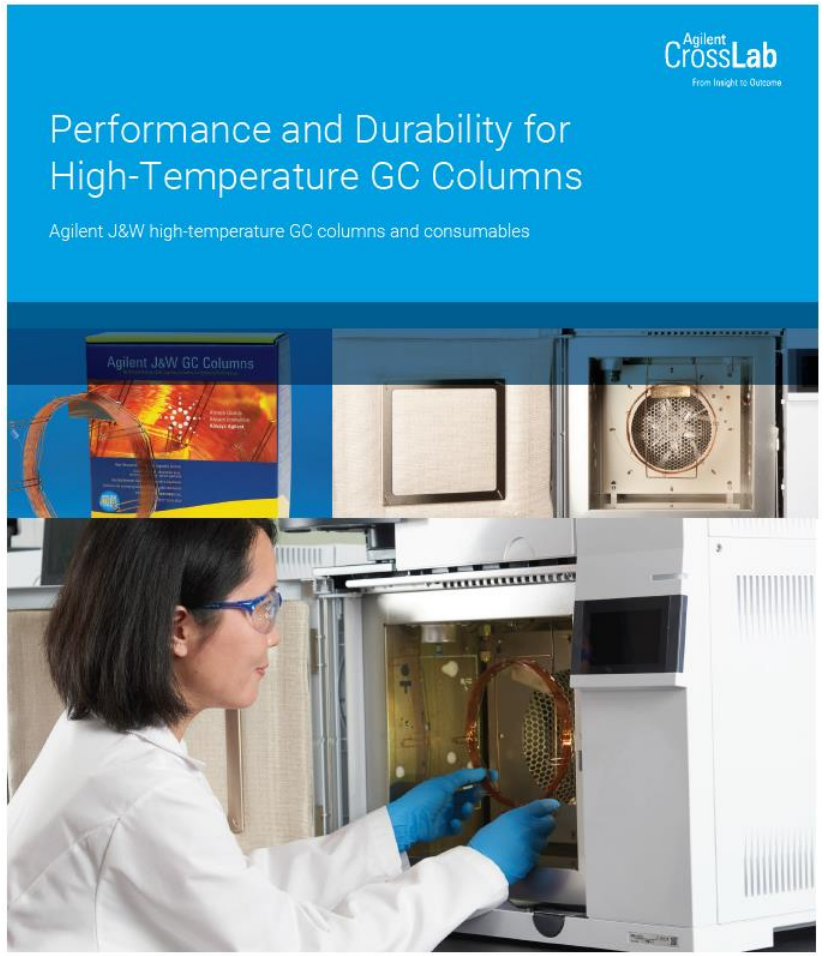
- Use the right consumables for high temperature applications
  - Graphite
  - Ultimetal ferrules
  - BTO Septa
  - Use regular liners

- 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





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5994-1013EN

Technical overview

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

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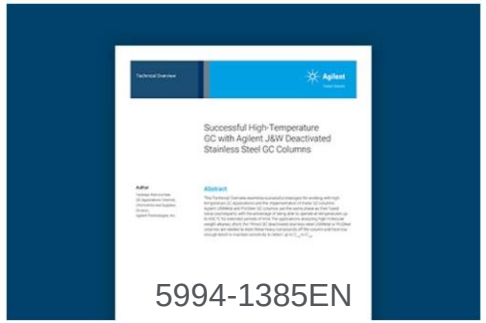

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Technical overview

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

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Poster

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

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