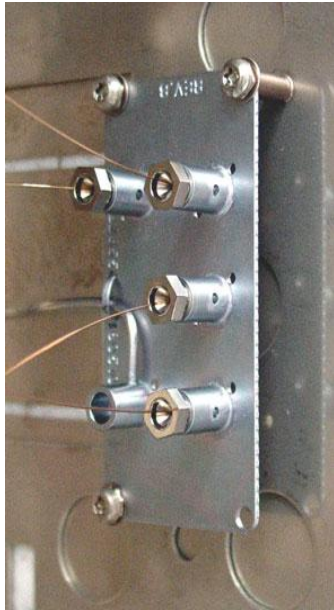
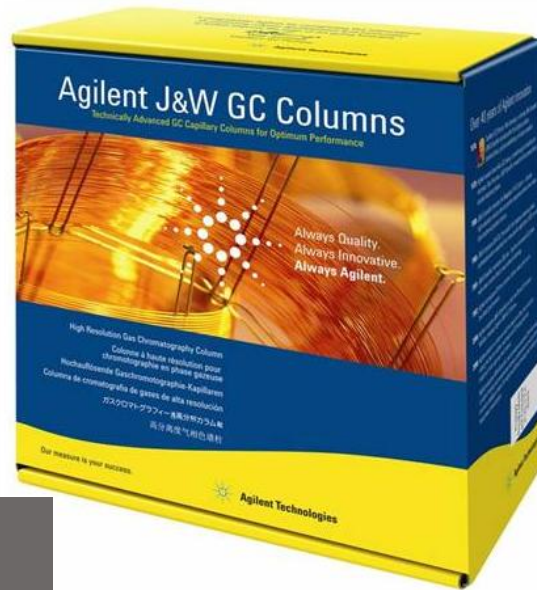
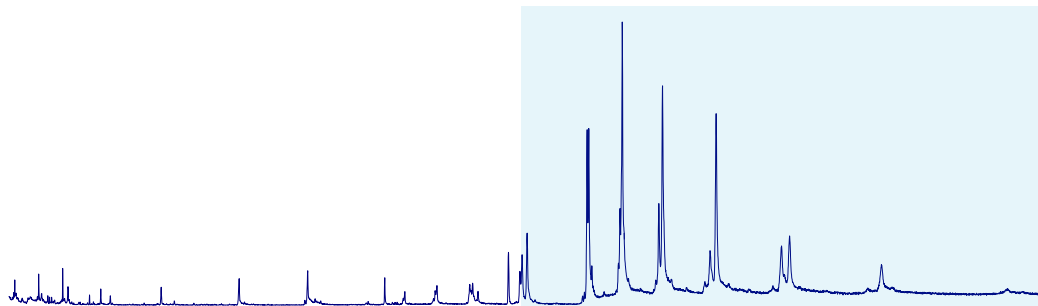


Alternate Carrier Gas Considerations and Faster GC Analysis



Faster GC Total Analytical Cycle Times – A Variety of Approaches



Included
in
HW/SW

“ALS Sample Overlap”
in ChemStation SW

7890 GC
Faster Cool-down

Column & Gas Selection,
Method Translation

New Devices
Significantly Faster GC
Analytical Cycle Times

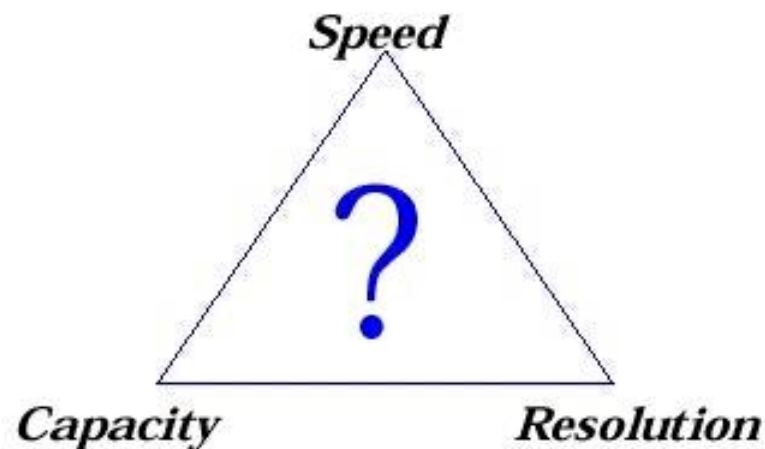
Capillary Flow Technology
(Backflush)

LTM Technology
(Rapid Heating)

LTM Technology
(Rapid Cooling)

Variables for Shortening GC Run Times

- Stationary Phase
- ★ • Carrier Gas: type and linear velocity
- ★ • Shorten Column Length
- ★ • Decrease Internal Diameter
- Temperature Programming



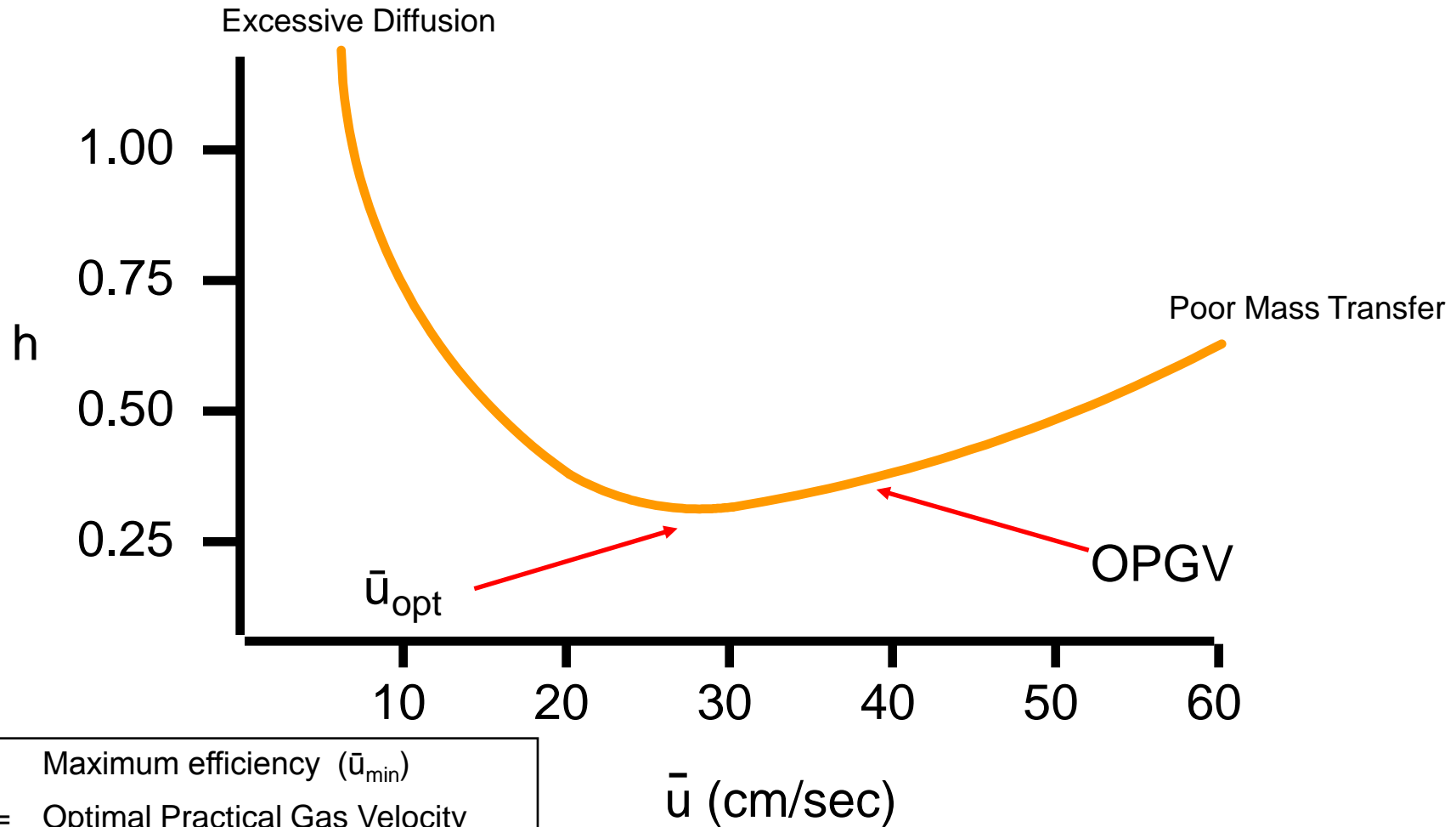
Carrier Gas

Affects resolution and retention time

Optimal range of velocities

Too low or high results in loss of resolution

van Deemter Curve



- \bar{u}_{opt} = Maximum efficiency (\bar{u}_{min})
- OPGV = Optimal Practical Gas Velocity
Maximum efficiency per unit time
(1.5 – 2 x \bar{u}_{opt})

Diffusion Constants for Dodecane @ 150°C

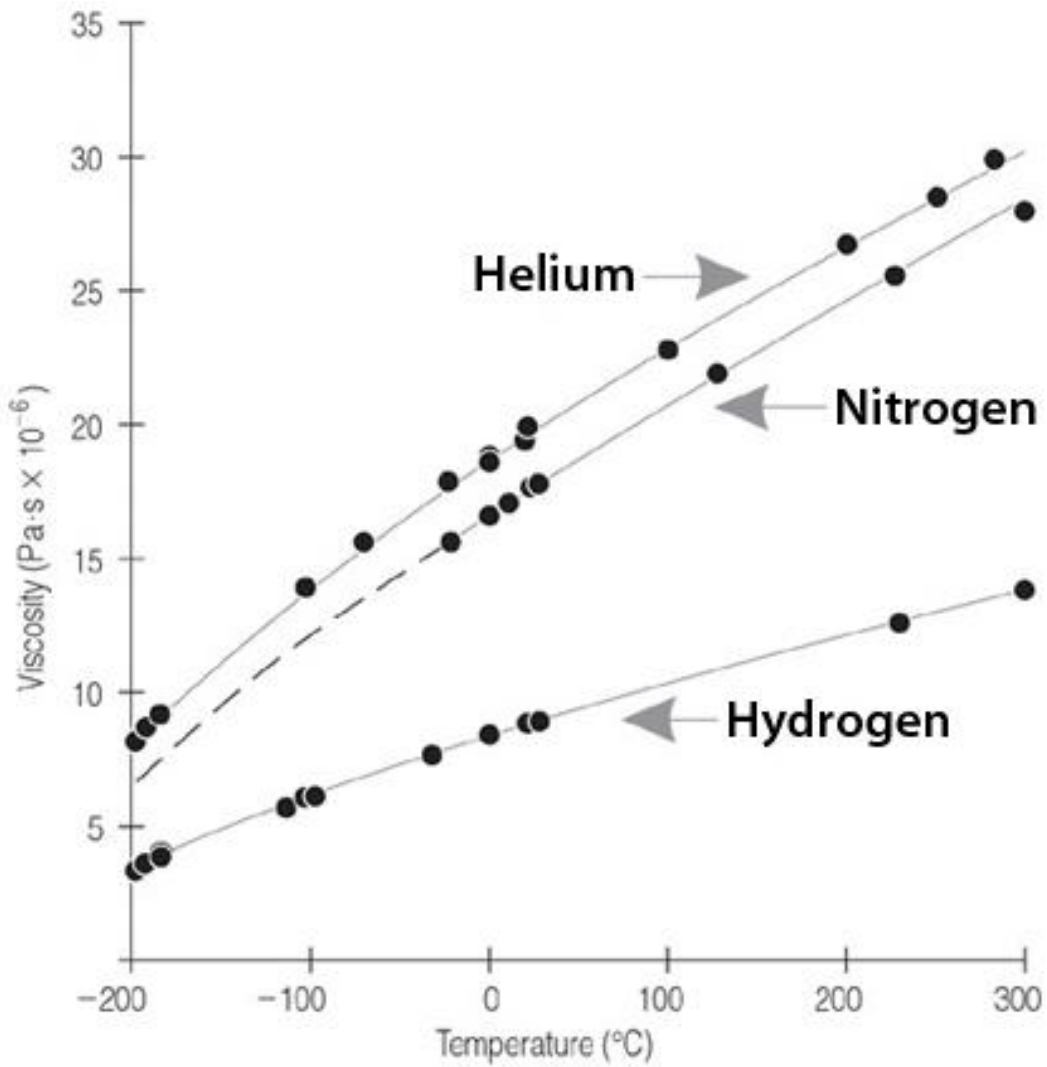
Nitrogen = 0.15 cm²/sec

Helium = 0.4 cm²/sec

Hydrogen = 0.6 cm²/sec

© *Walter Jennings, 1999*

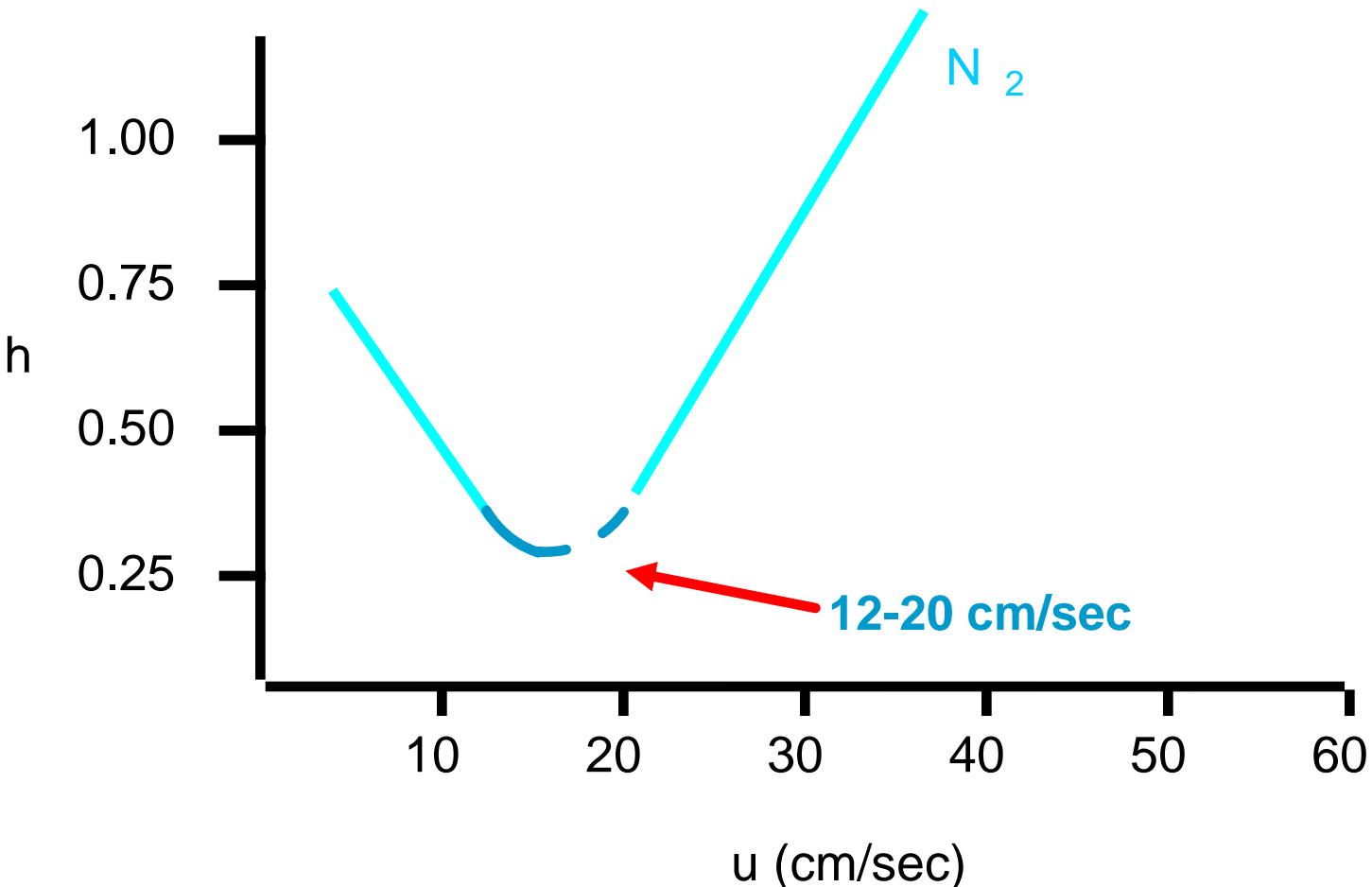
Gas Viscosity vs Temperature



J.V. Hinshaw, Column Connections, LCGC Asia Pacific, 12(2), 1100 (2009).

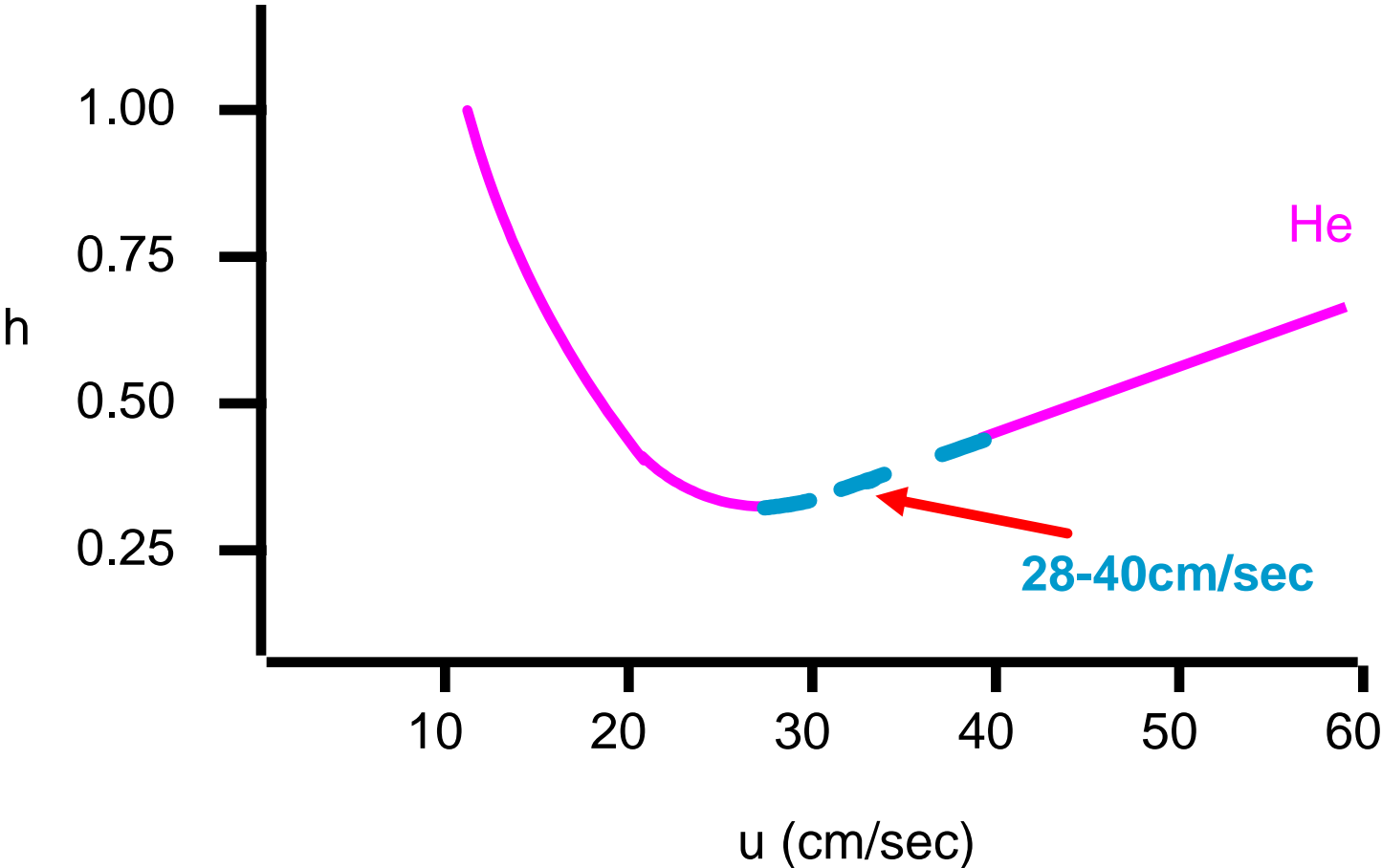
van Deemter Curve

Nitrogen



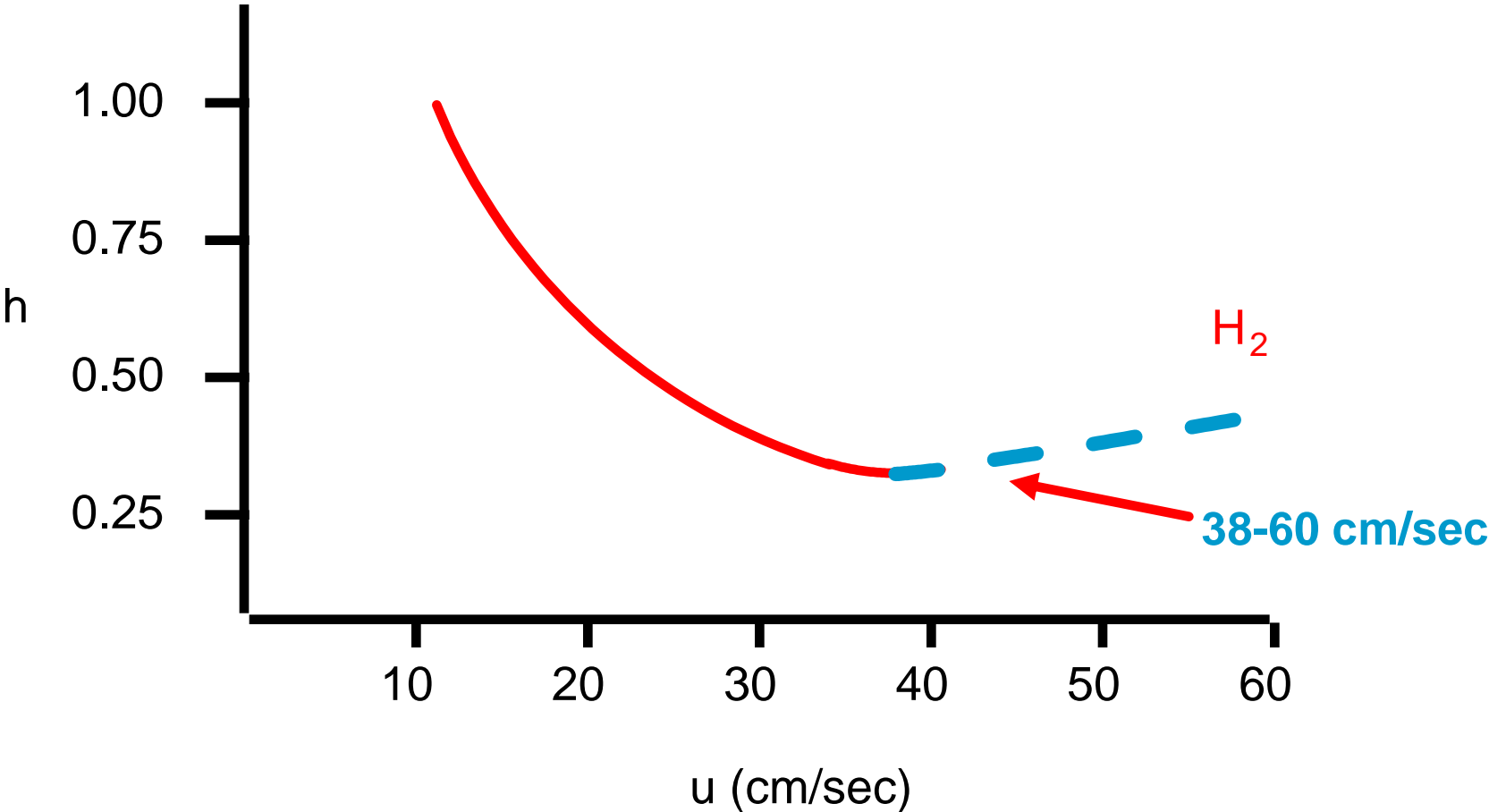
van Deemter Curve

Helium



van Deemter Curve

Hydrogen



Carrier Gas - Hydrogen Comments

Hydrogen is extremely diffusive in air

Difficult to reach explosive level of ~4 %

Most GC's flow regulated with safety shutdown

Spring loaded/Explosion ready doors

Hydrogen as Carrier – Contamination?

Contamination of GC flow modules and lines.

Hydrogen acts as a scrubber.

On the MS this typically looks like Hydrocarbon contamination

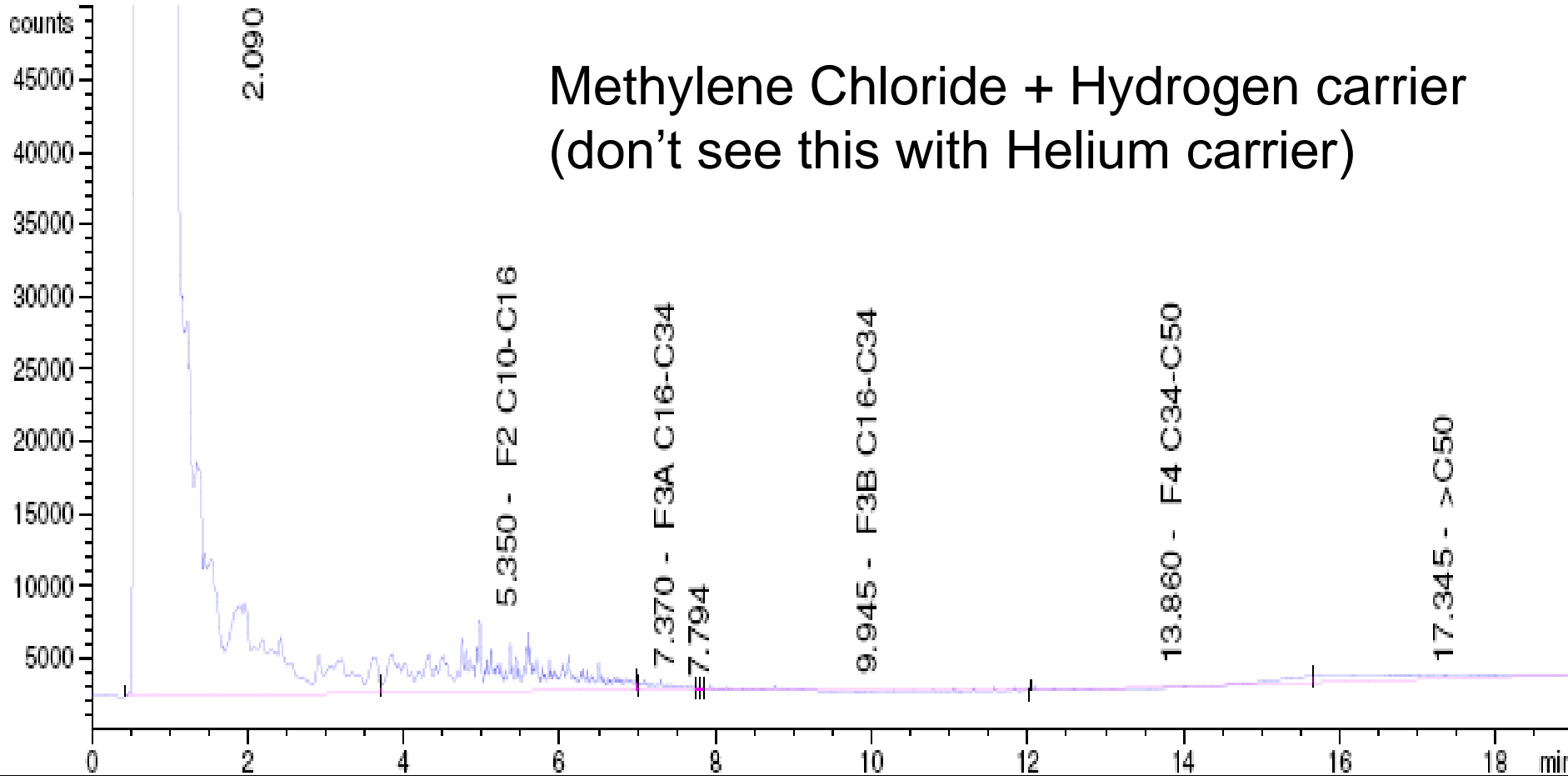
Most people report that it takes 2-4 weeks to clean out, depending on flows.

The FID only sees a high background for that time.

Other issues? $H_2 + Cl \text{ Solvent} + \text{Heat} = HCl?$

Contamination?! – scrubbing, vapor volume or HCl

FID1 A, (SEP2007\003F0601.D)



Methylene Chloride + Hydrogen carrier
(don't see this with Helium carrier)

Hydrogen as Carrier – MS and Lower Pressures

Hydrogen is more difficult for the MS vacuum system to pump away as compared to Helium.

Be careful not to get to a “negative” head pressure situation. (pulling instead of pushing flow is not good).

Trace level work on the MS requires low flow rates.

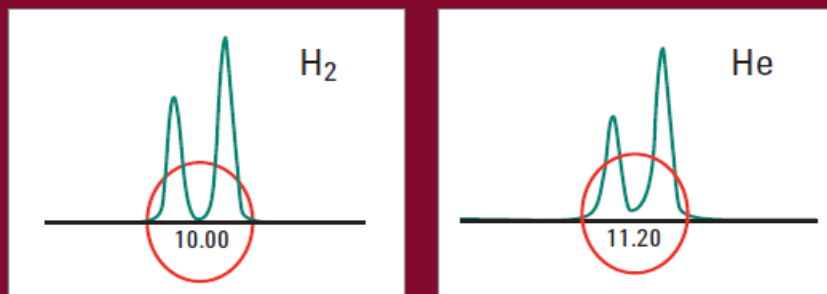
Must use smaller ID columns so you don't have higher flow rates. (length plays a role as well)

0.18mm ID or smaller is ideal for trace level MS work when using Hydrogen.

The Only GC-MS Engineered for Hydrogen Carrier Gas to Reduce Operating Costs

Use hydrogen to lower your cost per analysis

The new hydrogen signal-to-noise specification for the 5975C Series GC/MSD makes Agilent the first and only instrument manufacturer to certify the performance and safety of hydrogen as a carrier gas. In fact, hydrogen often provides faster analysis times and resolution greater than GC/MS systems operating in helium mode.

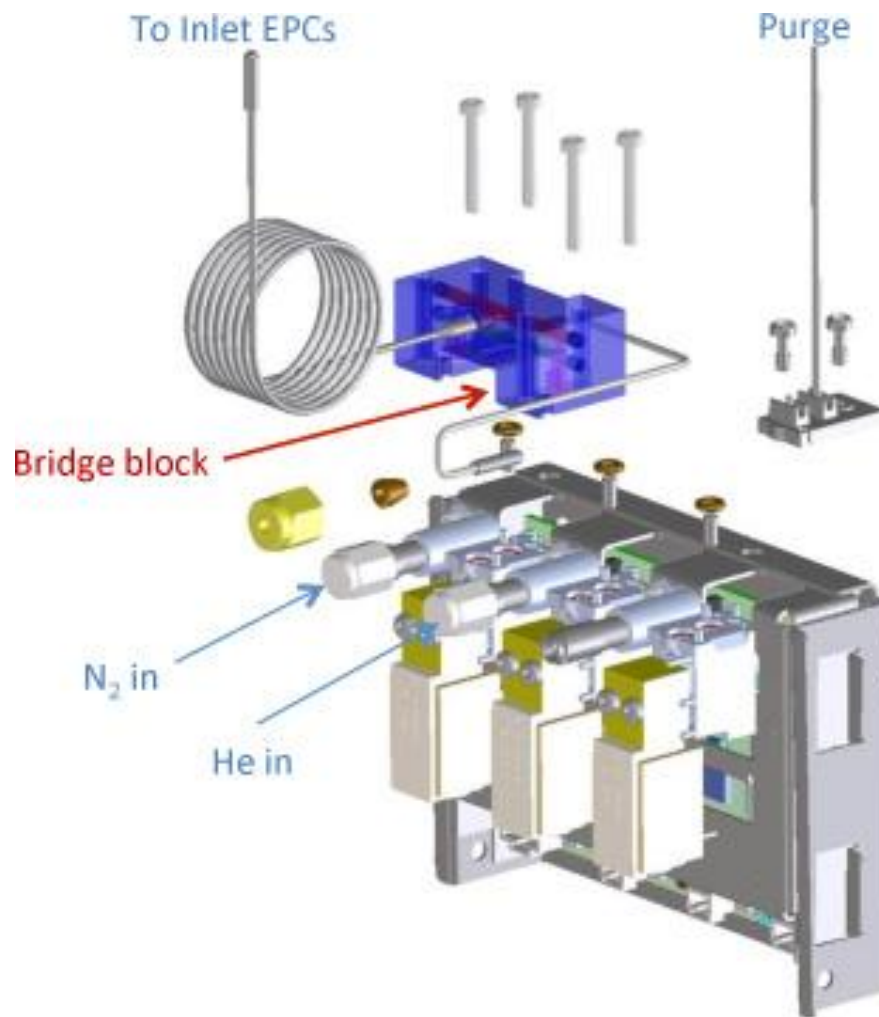


*Analysis of polyaromatic hydrocarbons using H₂ and He.
In this example, H₂ actually provides better resolution.*

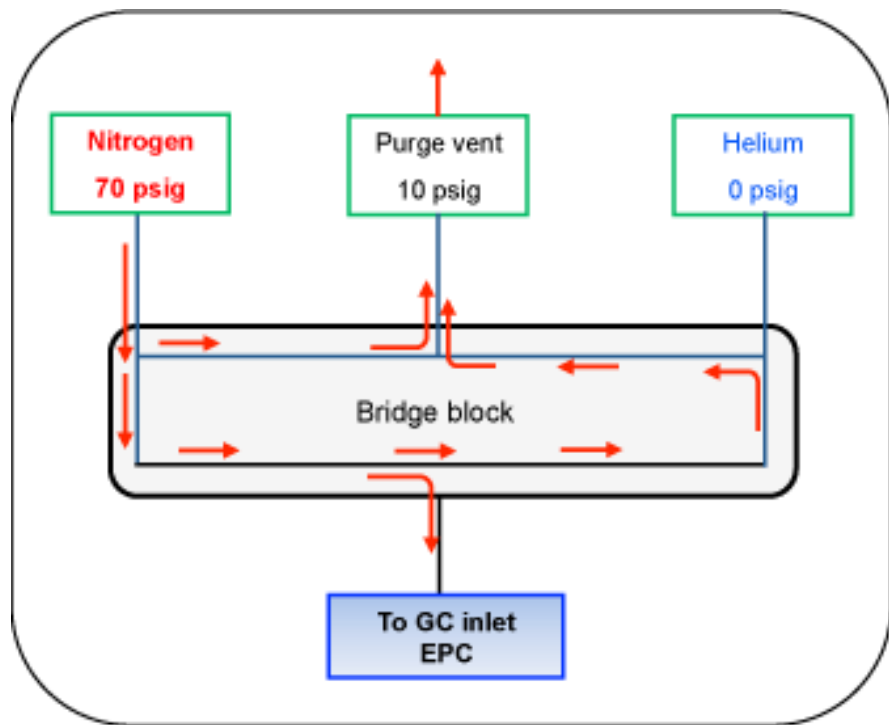
NEW Presentation!
**“Conversion of Agilent
EI GC/MSD Systems To
Hydrogen Carrier Gas”**
*(make a note on your
evaluation sheet to send)*

<http://www.chem.agilent.com/en-US/Promotions/pages/alternate-carrier-gas.aspx>

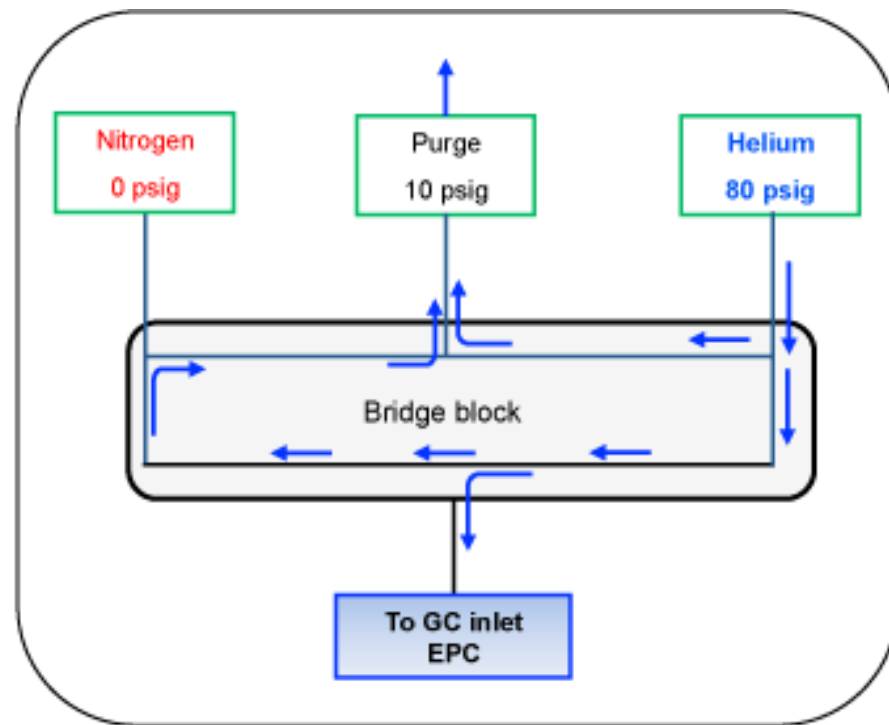
Programmable Helium Conservation Module



Programmable Helium Conservation Module



Helium savings mode
(N₂ on, He off)



Helium operation mode
(He on, N₂ off)

He to N2 Conversion (E-Max, Diesel)

GC Method Translation - EMAX.MXD

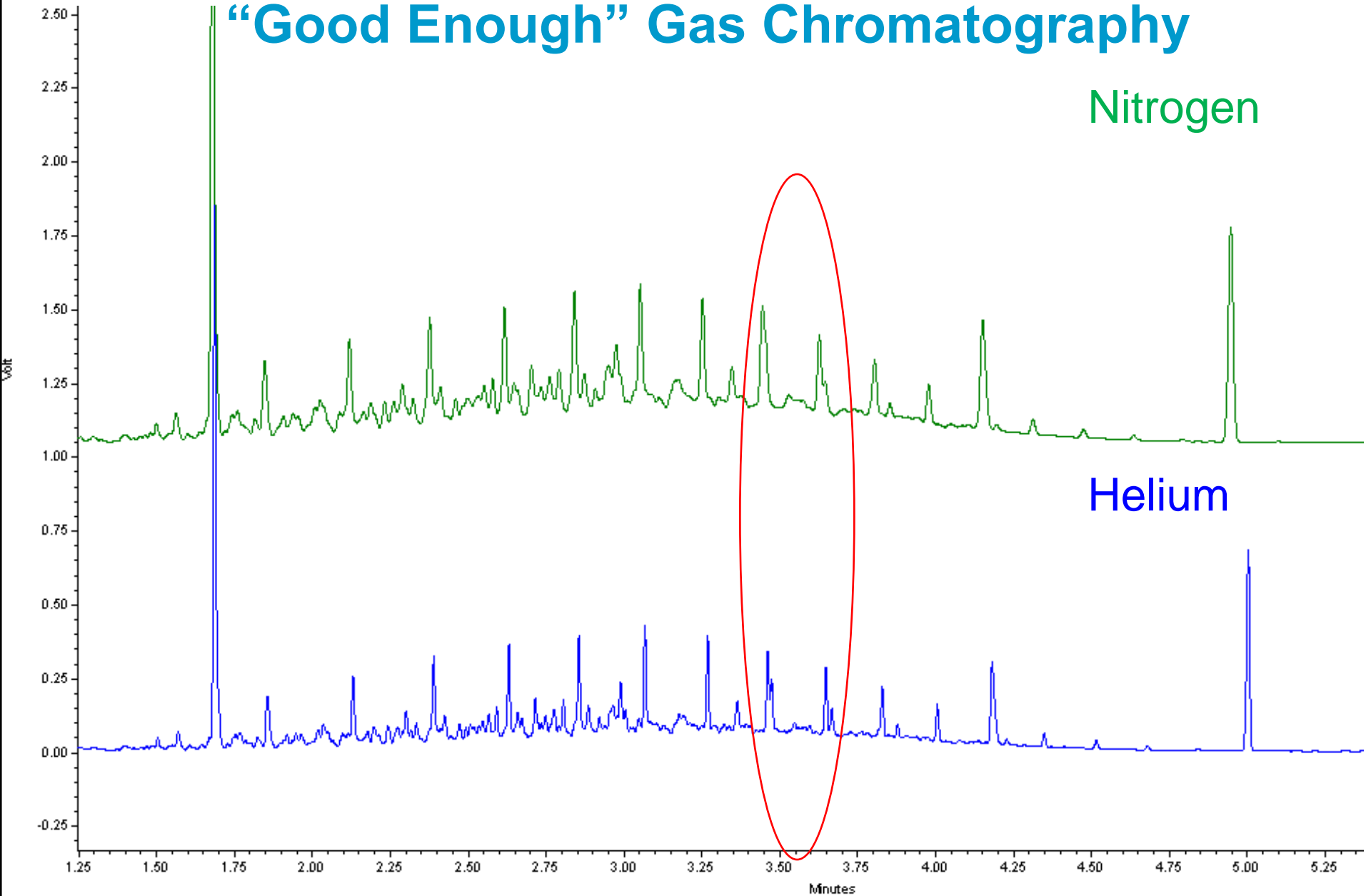
Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 1.00000**

	Original Method	Translated Method																								
Column																										
Length, m	30	<input checked="" type="checkbox"/> 30																								
Internal Diameter, μm	320	<input checked="" type="checkbox"/> 320																								
Film		<input type="radio"/> Unlock																								
Thickness, μm	0.25	<input type="radio"/> 0.250																								
Phase Ratio	320.0	<input checked="" type="radio"/> 320.0																								
Carrier Gas	Helium	<input type="checkbox"/> Nitrogen																								
Enter one Setpoint		<input type="radio"/> Unlock																								
Head Pressure, psi	19.733	<input type="radio"/> 17.579																								
Flow Rate, mLn/min	5	<input type="radio"/> 4.7486																								
Outlet Velocity, cm/sec	114.40	<input type="radio"/> 108.65																								
Average Velocity, cm/sec	64.95	<input type="radio"/> 64.95																								
Hold-up Time, min	0.769785	<input checked="" type="radio"/> 0.769785																								
Outlet Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																								
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																								
Oven Temperature 1-ramp Program																										
	<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th>$^{\circ}\text{C}/\text{min}$</th> <th>$^{\circ}\text{C}$</th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>55</td> <td>0.5</td> </tr> <tr> <td>60</td> <td>320</td> <td>5.08</td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		55	0.5	60	320	5.08	<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th>$^{\circ}\text{C}/\text{min}$</th> <th>$^{\circ}\text{C}$</th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>55</td> <td>0.500</td> </tr> <tr> <td>60.000</td> <td>320</td> <td>5.080</td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		55	0.500	60.000	320	5.080
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60.000	320	5.080																								
Initial Ramp 1																										
Sample Information None																										

“Good Enough” Gas Chromatography

Nitrogen

Helium



Column Dimensions

Diameter

Length

The Secrets Are In There

$$R_s = \frac{\sqrt{N}}{4} \left(\frac{k}{k+1} \right) \left(\frac{\alpha-1}{\alpha} \right)$$

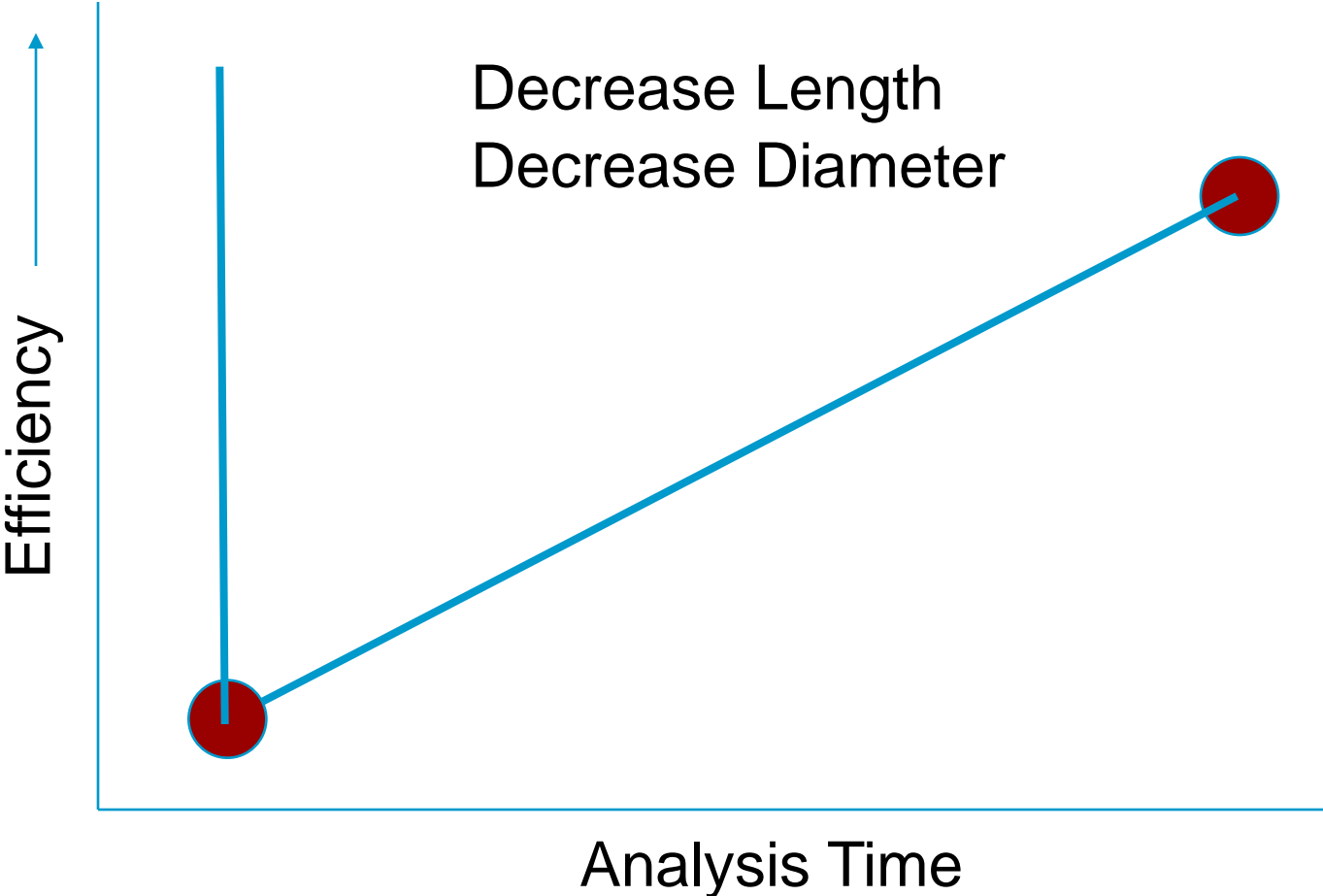
Efficiency	$N = f$ (gas, L , r_c)	L = Length
Retention	$k = f$ (T , d_f , r_c)	r_c = column radius d_f = film thickness
Selectivity	$\alpha = f$ (T , phase)	T = temperature

Column Diameter - Theoretical Efficiency

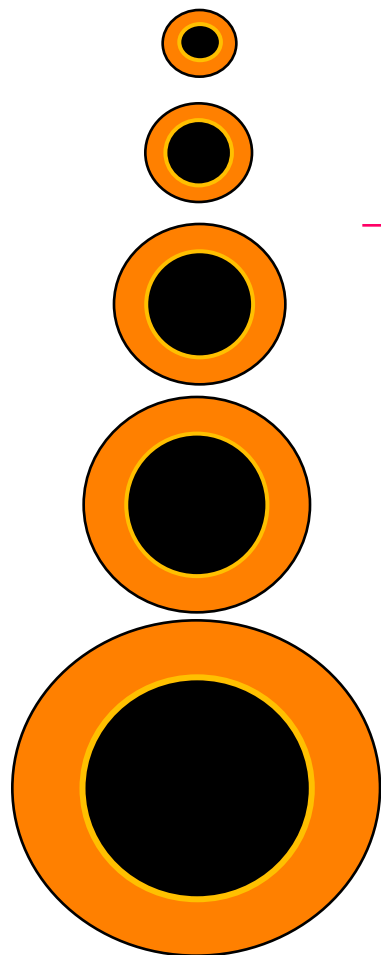
I.D. (mm)	n/m
0.05	23,160
0.10	11,980
0.18	6,660
0.20	5830
0.25	4630
0.32	3760
0.45	2840
0.53	2060

$k = 5$

Combining a change in Length with a change in Diameter



Column Diameter and Capacity



I.D. (mm)	Capacity (ng)
-----------	---------------

0.05	1-2
------	-----

1-2

0.10	6-13
------	------

6-13

0.18	25-55
------	-------

25-55

0.20	35-70
------	-------

35-70

0.25	80-160
------	--------

80-160

0.32	110-220
------	---------

110-220

0.45	600-800
------	---------

600-800

0.53	1000-2000
------	-----------

1000-2000

Like Polarity
Phase/Solute
0.25 μm film thickness

Changes in Column Dimensions, Gas Type or Velocity Require Changes in Temp Program Rates

GC Method Translation

Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 1.66552**

	Original Method	Translated Method																																										
Column																																												
Length, m	30	<input checked="" type="checkbox"/> 30																																										
Internal Diameter, μm	250.0	<input checked="" type="checkbox"/> 250.0																																										
Film																																												
Thickness, μm	0.25	<input type="radio"/> Unlock																																										
Phase Ratio	250.0	<input type="radio"/> 0.25																																										
		<input type="radio"/> 250.0																																										
Carrier Gas	Helium	<input type="checkbox"/> Hydrogen																																										
Enter one Setpoint																																												
Head Pressure, psi	9.023	3.106																																										
Flow Rate, mLn/min	1.2	1.5000																																										
Outlet Velocity, cm/sec	Very large	Very large																																										
Average Velocity, cm/sec	39.29	65.44																																										
Hold-up Time, min	1.27249	0.764023																																										
Outlet Pressure (absolute), psi	0	<input checked="" type="checkbox"/> 0																																										
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																																										
Oven Temperature 3-ramp Program																																												
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Ramp 3	3.331	220.00	3.002																																									
Sample Information	None																																											

GC Method Translation

Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 1.92318**

	Original Method	Translated Method																																										
Column																																												
Length, m	30	<input checked="" type="checkbox"/> 30																																										
Internal Diameter, μm	250.0	<input checked="" type="checkbox"/> 250.0																																										
Film																																												
Thickness, μm	0.25	<input type="radio"/> Unlock																																										
Phase Ratio	250.0	<input type="radio"/> 0.25																																										
		<input type="radio"/> 250.0																																										
Carrier Gas	Helium	<input type="checkbox"/> Hydrogen																																										
Enter one Setpoint																																												
Head Pressure, psi	9.023	<input type="radio"/> 5.860																																										
Flow Rate, mLn/min	1.2	<input checked="" type="radio"/> 2.0																																										
Outlet Velocity, cm/sec	Very large	Very large																																										
Average Velocity, cm/sec	39.29	75.57																																										
Hold-up Time, min	1.27249	0.661663																																										
Outlet Pressure (absolute), psi	0	<input checked="" type="checkbox"/> 0																																										
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																																										
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Ramp 2	1.923	60.00	7.800																																									
Ramp 3	3.846	220.00	2.600																																									
Sample Information	None																																											

Method Translation Software to the Rescue!



To Get the Software:

<http://www.chem.agilent.com/en-US/Technical-Support/Instruments-Systems/Gas-Chromatography/Pages/gcmethodtranslation.aspx>

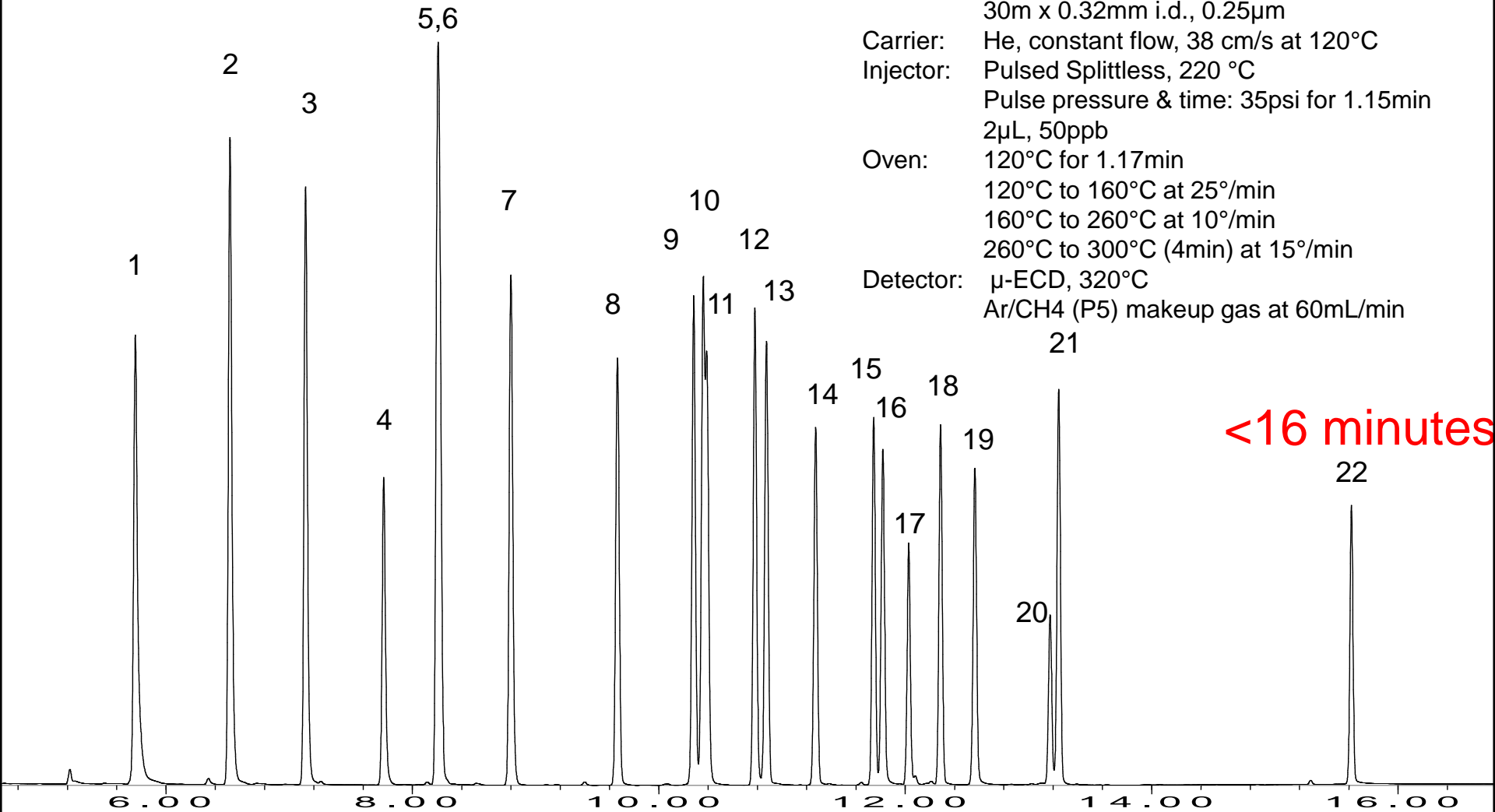
OR

Google → Agilent GC Method Translation Software

CLP-Pesticides - Original "Improved" Method

0.32mm I.D., Helium Carrier Gas

Column: DB-XLB
30m x 0.32mm i.d., 0.25 μ m
Carrier: He, constant flow, 38 cm/s at 120°C
Injector: Pulsed Splittless, 220 °C
Pulse pressure & time: 35psi for 1.15min
2 μ L, 50ppb
Oven: 120°C for 1.17min
120°C to 160°C at 25°/min
160°C to 260°C at 10°/min
260°C to 300°C (4min) at 15°/min
Detector: μ -ECD, 320°C
Ar/CH4 (P5) makeup gas at 60mL/min



Input Original Method Parameters

GC Method Translation - EPACLP.MXD

Criterion: Translate Only Best Efficiency Fast Analysis None Speed gain: 1.17407

Column

Length, m

Internal Diameter, μm

Film

Thickness, μm

Phase Ratio

Original Method

Length: 30

Internal Diameter: 320

Thickness: 0.25

Phase Ratio: 320.0

Translated Method

30

320

Unlock

0.25

320.0

Carrier Gas

Enter one Setpoint

Head Pressure, psi

Flow Rate, mLn/min

Outlet Velocity, cm/sec

Average Velocity, cm/sec

Hold-up Time, min

Outlet Pressure (absolute), psi

Ambient Pressure (absolute), psi

Original Method

Carrier Gas: Helium

Head Pressure: 12.786

Flow Rate: 2.0502

Outlet Velocity: 56.20

Average Velocity: 38

Hold-up Time: 1.31579

Outlet Pressure (absolute): 14.696

Ambient Pressure (absolute): 14.696

Translated Method

Helium

Head Pressure: 15.126

Flow Rate: 2.5600

Outlet Velocity: 70.17

Average Velocity: 44.61

Hold-up Time: 1.12070

Outlet Pressure (absolute): 14.696

Ambient Pressure (absolute): 14.696

Oven Temperature 3-ramp Program

	Ramp Rate	Final Temp.	Final Time
	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min
Initial		120	1.17
Ramp 1	25	160	0
Ramp 2	10	260	0
Ramp 3	15	300	4

Original Method

	Ramp Rate	Final Temp.	Final Time
	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min
Initial		120	1.17
Ramp 1	25	160	0
Ramp 2	10	260	0
Ramp 3	15	300	4

Translated Method

	Ramp Rate	Final Temp.	Final Time
	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min
Initial		120	0.997
Ramp 1	29.352	160	0.000
Ramp 2	11.741	260	0.000
Ramp 3	17.611	300	3.407

Sample Information None

New Column Dimensions, H2 Gas, Fast Analysis

GC Method Translation - EPACL.P.MXD

Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 2.34453**

	Original Method	Translated Method																																										
Column																																												
Length, m	30	<input type="checkbox"/> 20																																										
Internal Diameter, μm	320	<input type="checkbox"/> 177																																										
Film																																												
Thickness, μm	0.25	<input checked="" type="radio"/> Unlock																																										
Phase Ratio	320.0	<input type="radio"/> 0.18																																										
		<input type="radio"/> 245.8																																										
Carrier Gas	Helium	<input type="checkbox"/> Hydrogen																																										
Enter one Setpoint																																												
Head Pressure, psi	12.786	26.714																																										
Flow Rate, mLn/min	2.0502	1.7700																																										
Outlet Velocity, cm/sec	56.20	158.74																																										
Average Velocity, cm/sec	38	77.31																																										
Hold-up Time, min	1.31579	0.431144																																										
Outlet Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																																										
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																																										
Oven Temperature 3-ramp Program																																												
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Input NEW dimensions

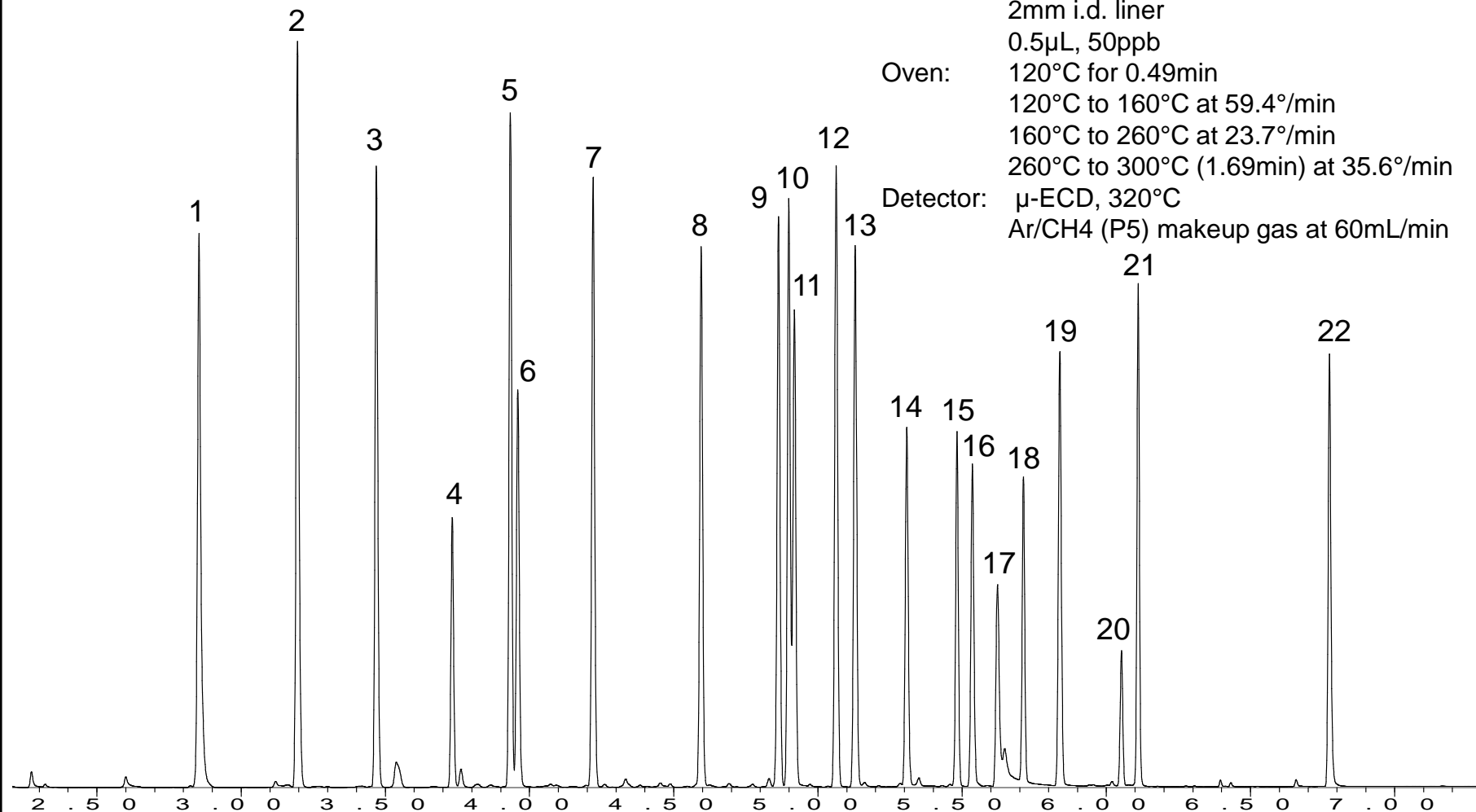
Close enough (got lucky)

New Velocity

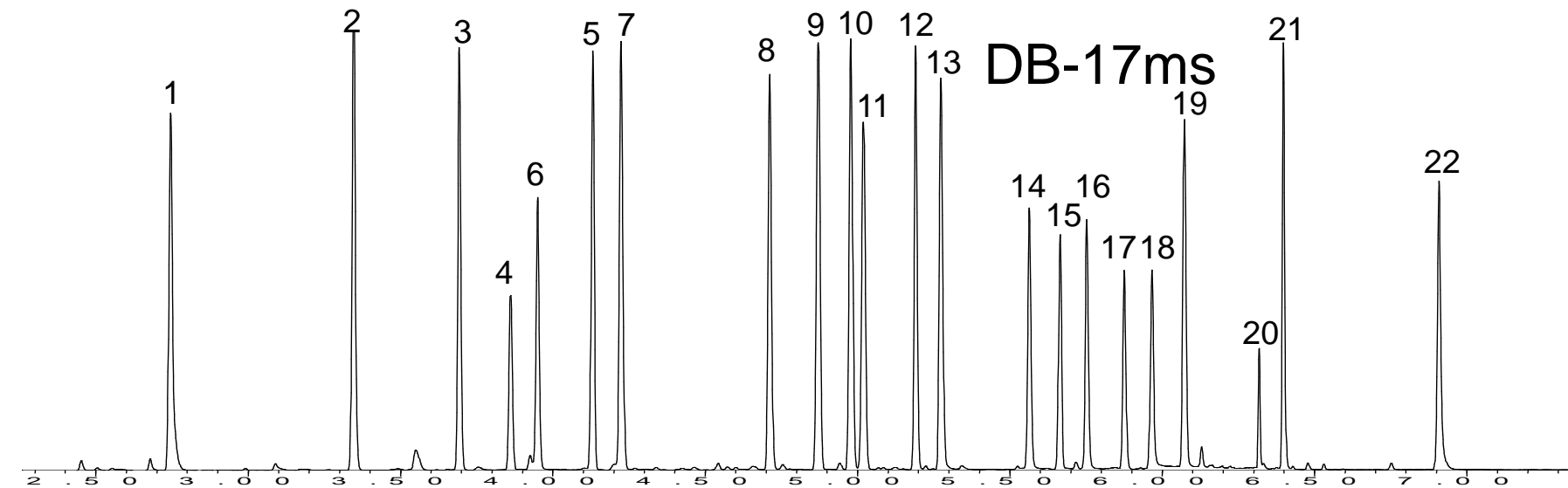
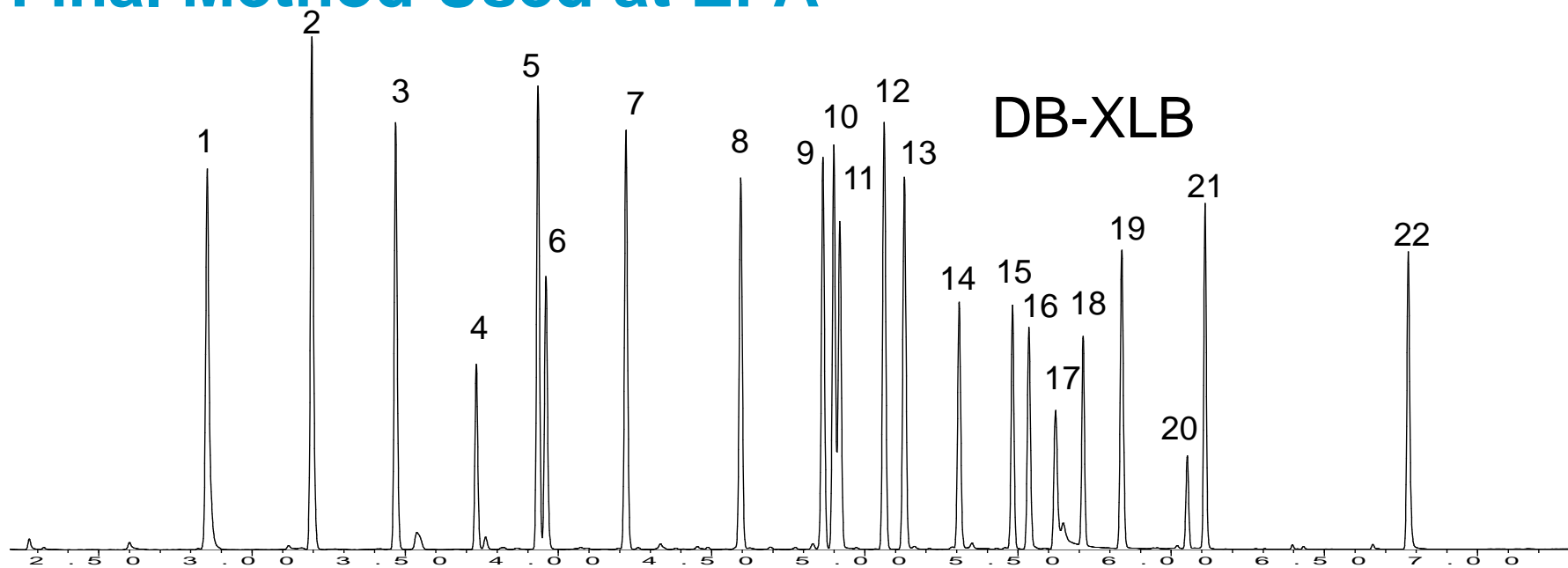
New Temp. Program

Final Method Used at EPA

Column: DB-XLB
20m x 0.18mm i.d., 0.18 μ m
Carrier: H₂, constant flow, 77.3cm/s at 120°C
Injector: Pulsed Splitless, 220 °C
Pulse pressure & time: 35psi for 0.5min
Flow ramp at 6.25min of 99mL/min² to 3mL/min
2mm i.d. liner
0.5 μ L, 50ppb
Oven: 120°C for 0.49min
120°C to 160°C at 59.4°/min
160°C to 260°C at 23.7°/min
260°C to 300°C (1.69min) at 35.6°/min
Detector: μ -ECD, 320°C
Ar/CH₄ (P5) makeup gas at 60mL/min

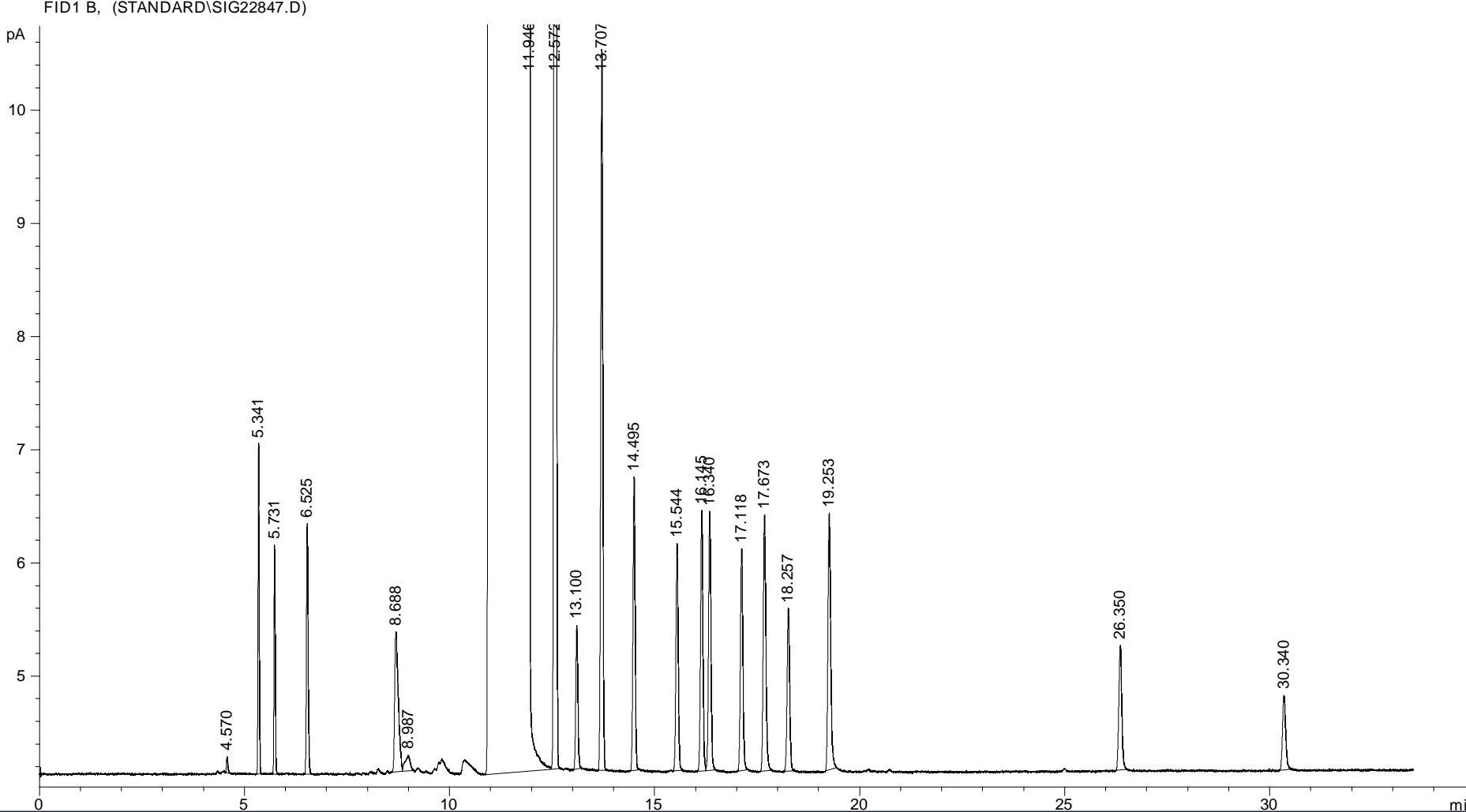


Final Method Used at EPA



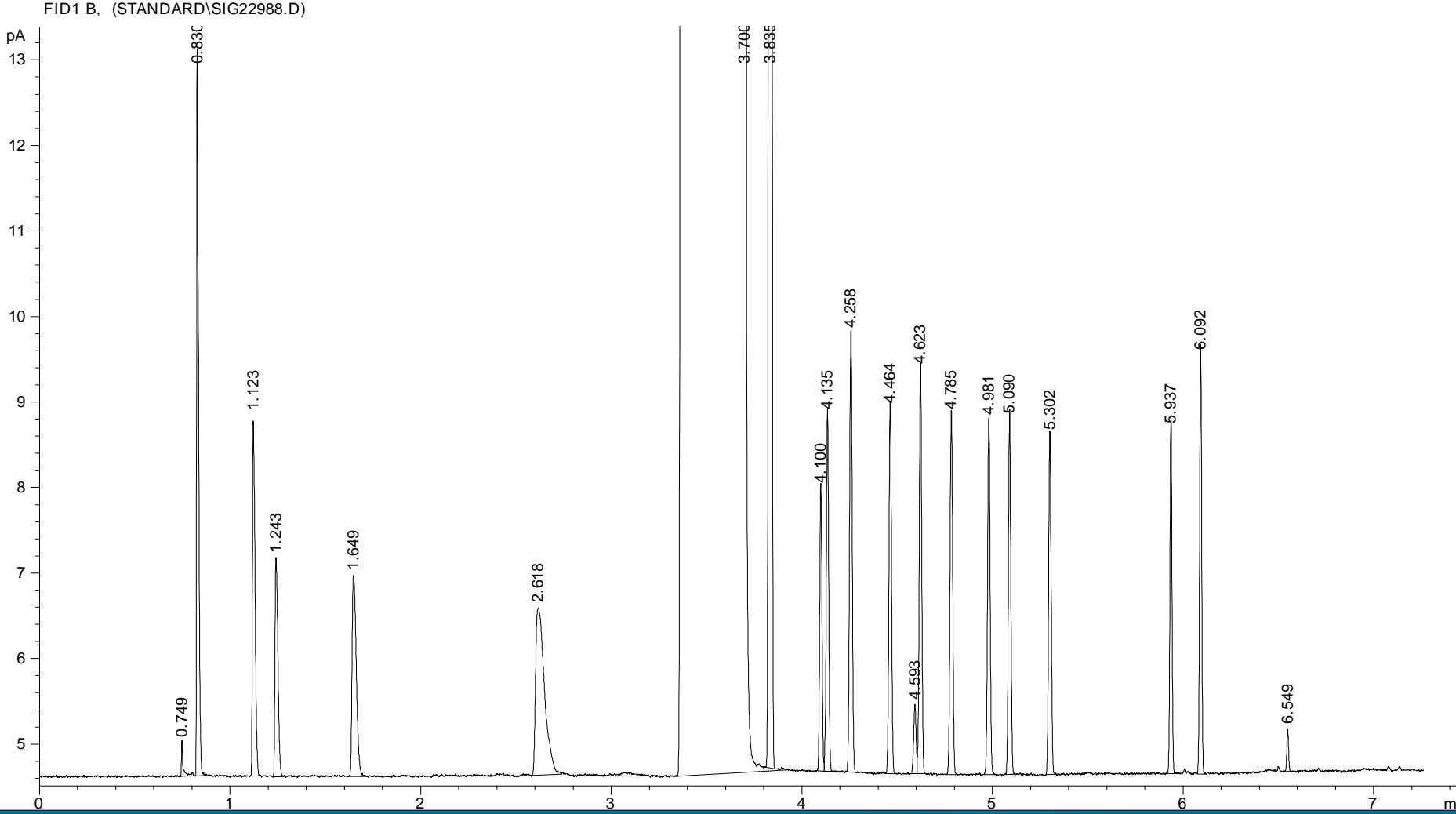
Cumene Analysis

-60m x 0.32mm ID (overkill)



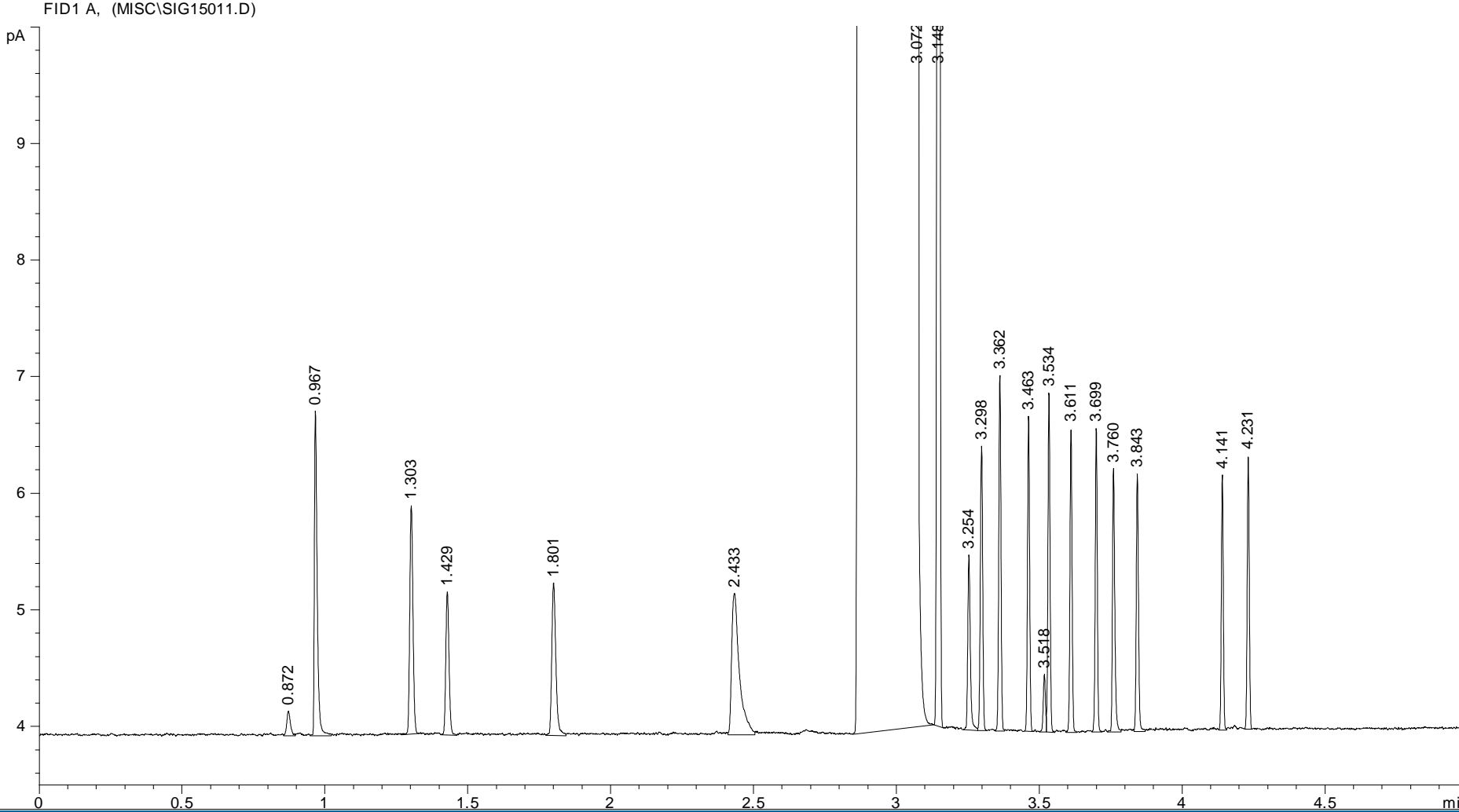
Cumene Analysis

-30m x 0.25mm ID



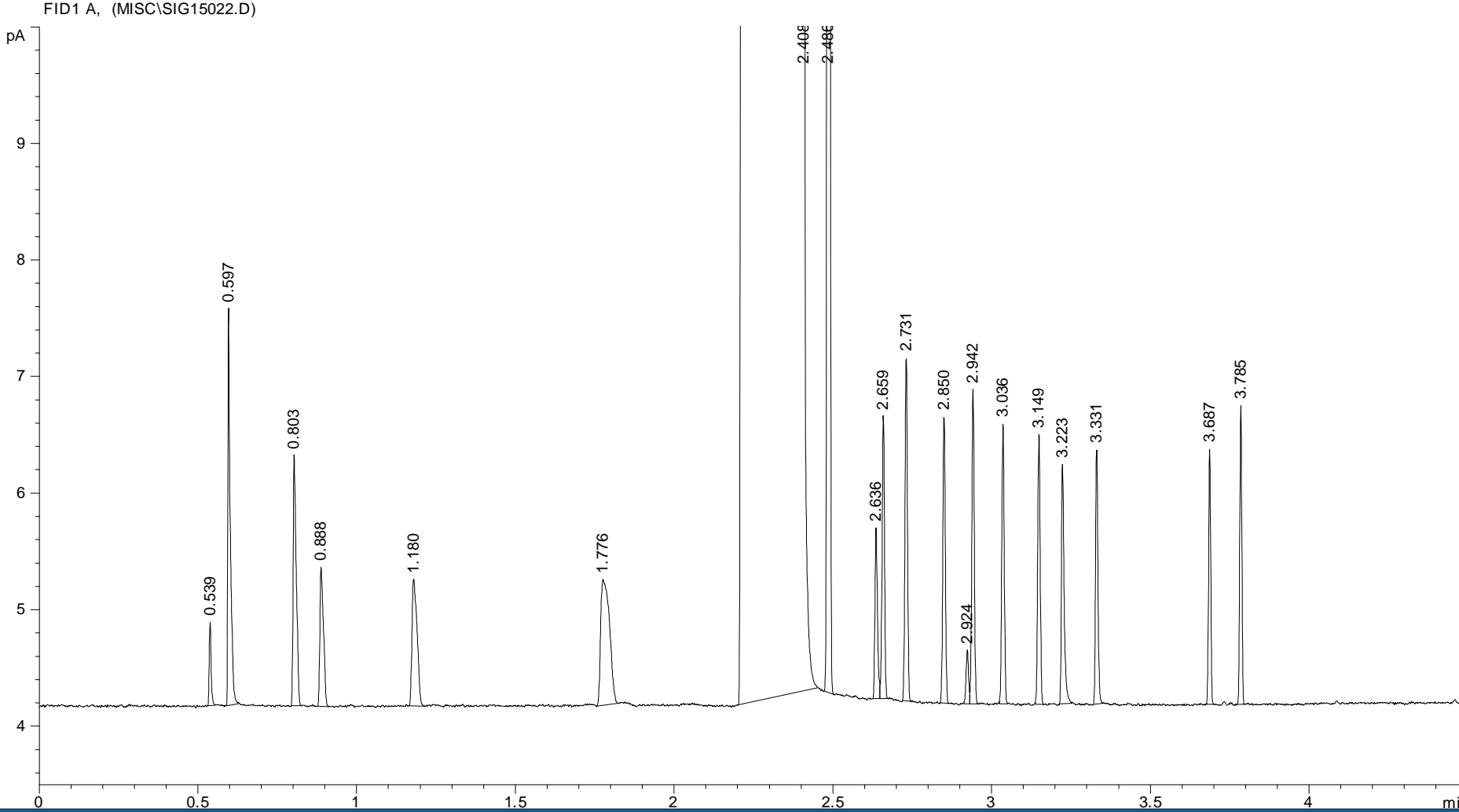
Cumene Analysis

-20m x 0.18mm ID



Cumene Analysis

-20m x 0.18mm ID, Hydrogen Carrier



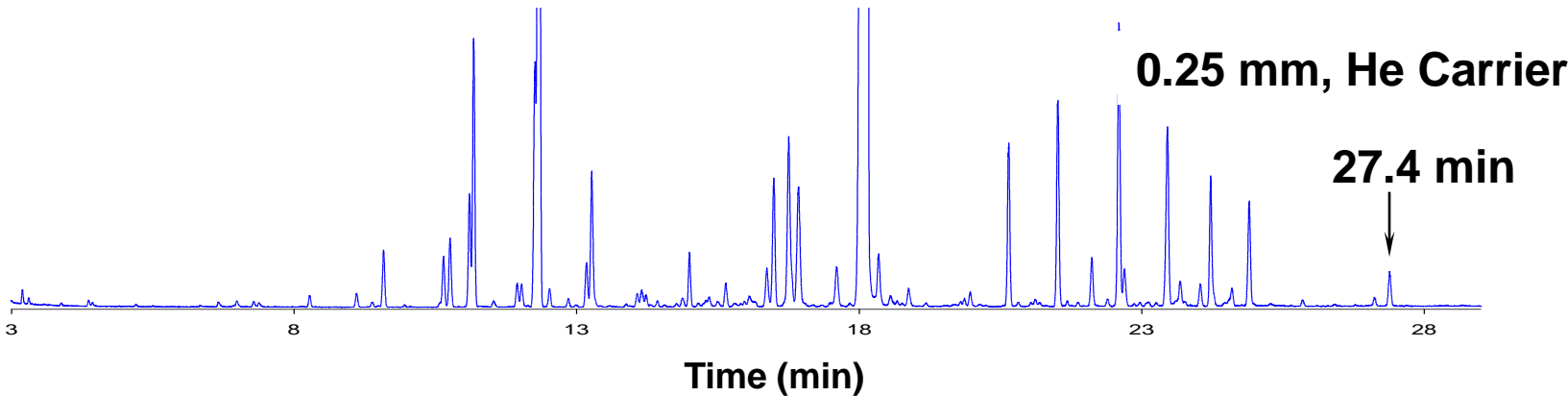
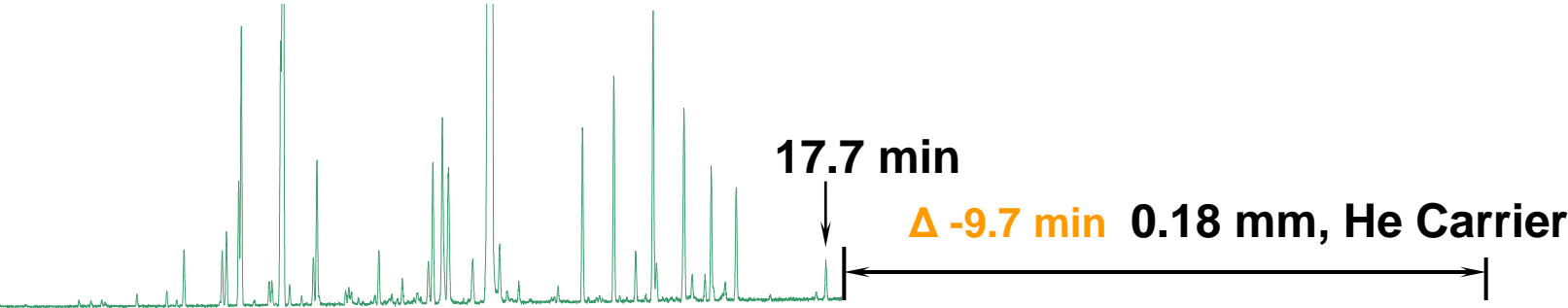
Food/Fragrance – Method translation

GC Method Translation

Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 1.55885**

	Original Method	Translated Method																								
Column																										
Length, m	30	<input type="checkbox"/> 20																								
Internal Diameter, μm	250.0	<input type="checkbox"/> 180																								
Film																										
Thickness, μm	0.250	<input type="radio"/> Unlock																								
Phase Ratio	250.0	<input type="radio"/> 0.180																								
		<input checked="" type="radio"/> 250.0																								
Carrier Gas	Helium	<input type="checkbox"/> Helium																								
Enter one Setpoint																										
Head Pressure, psi	0.563	5.698																								
Flow Rate, mLn/min	0.4833	0.3480																								
Outlet Velocity, cm/sec	Very large	Very large																								
Average Velocity, cm/sec	25.00	25.98																								
Hold-up Time, min	2.00000	1.28300																								
Outlet Pressure (absolute), psi	0	<input checked="" type="checkbox"/> 0																								
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																								
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Spearmint Oil on DB-1



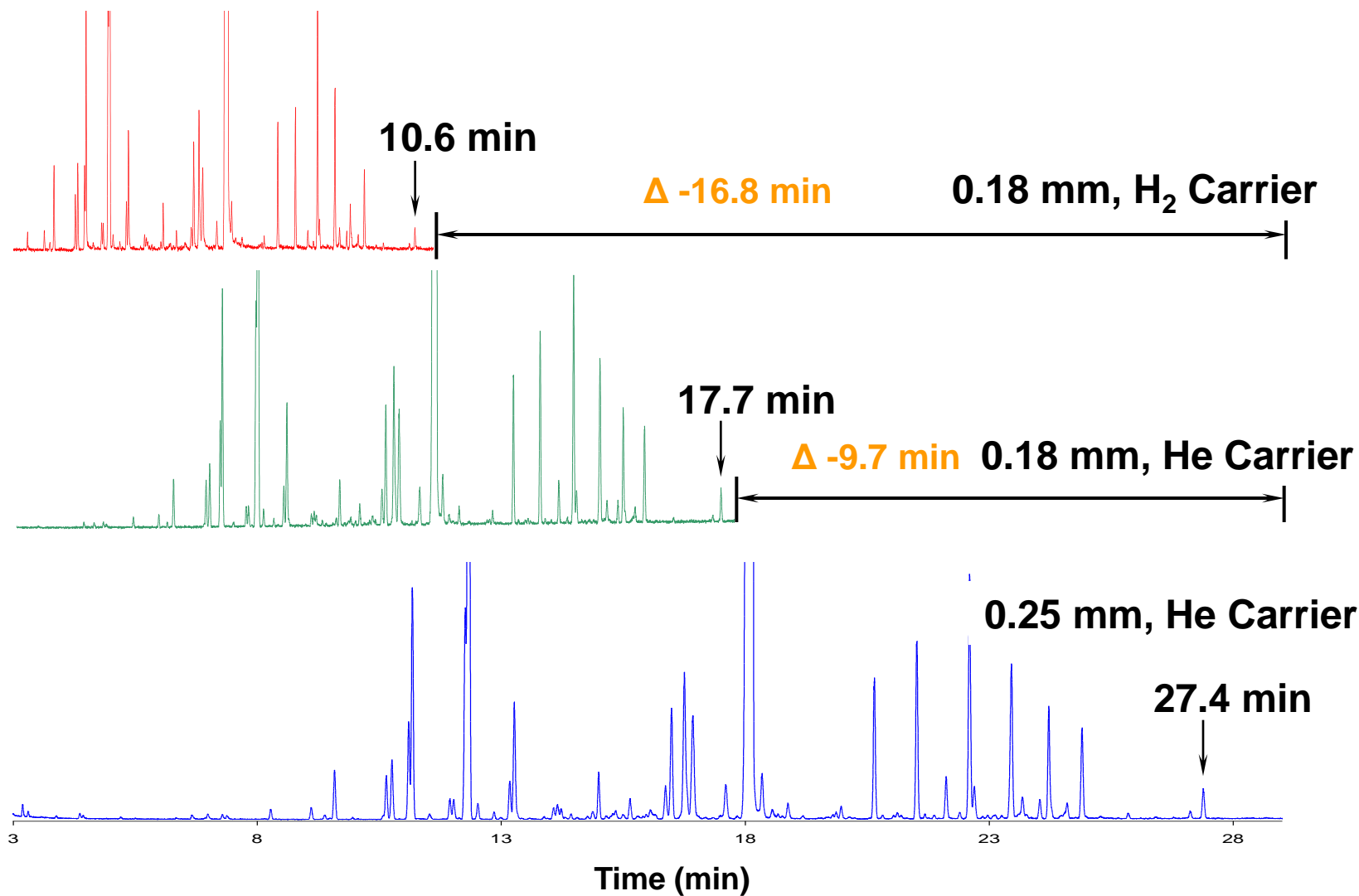
Food/Fragrance – Method translation, Hydrogen

GC Method Translation _ □ ×

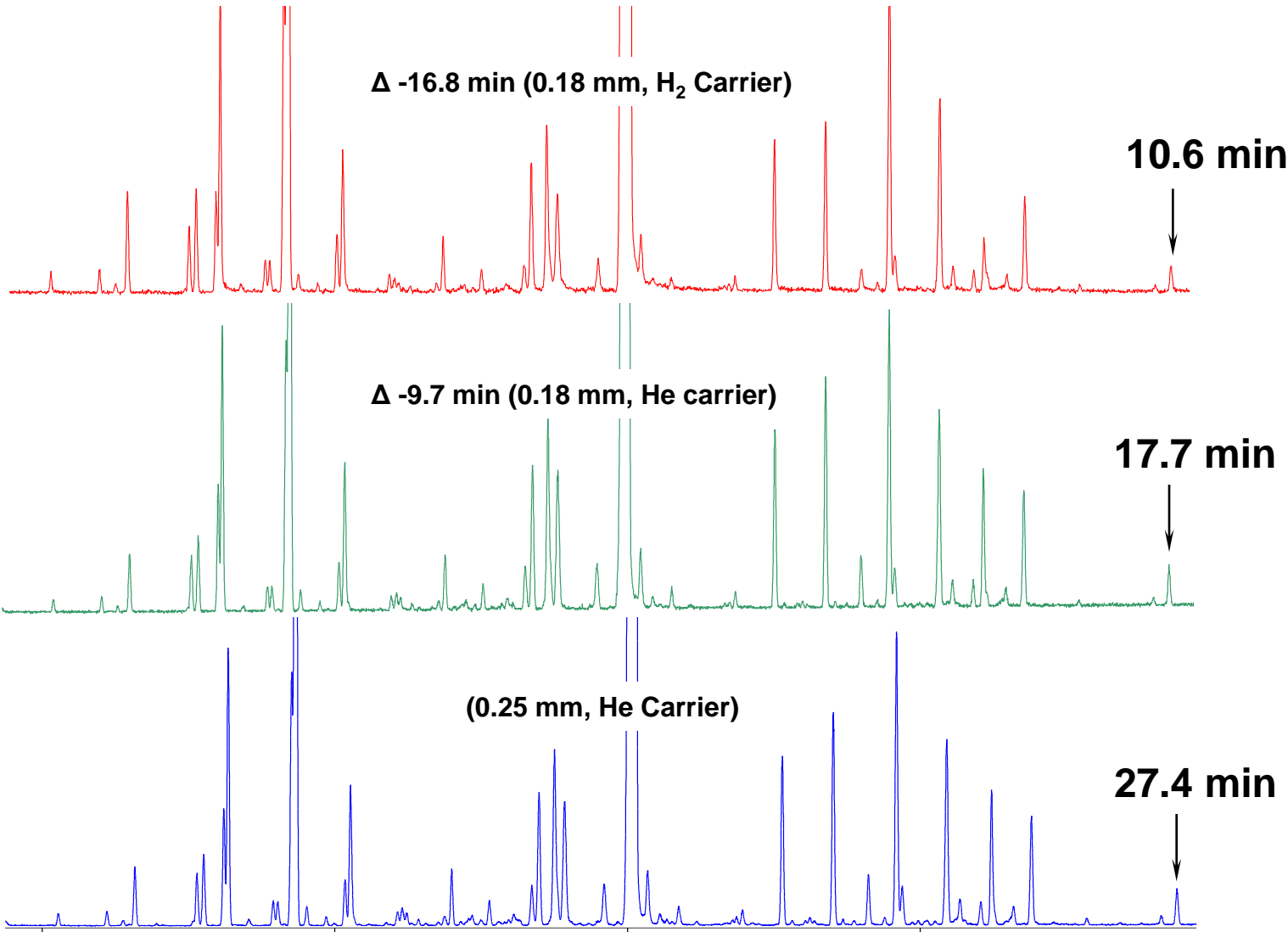
Criterion: Translate Only Best Efficiency Fast Analysis None Speed gain: 2.59618

	Original Method	Translated Method																								
Column																										
Length, m	30	<input type="checkbox"/> 20																								
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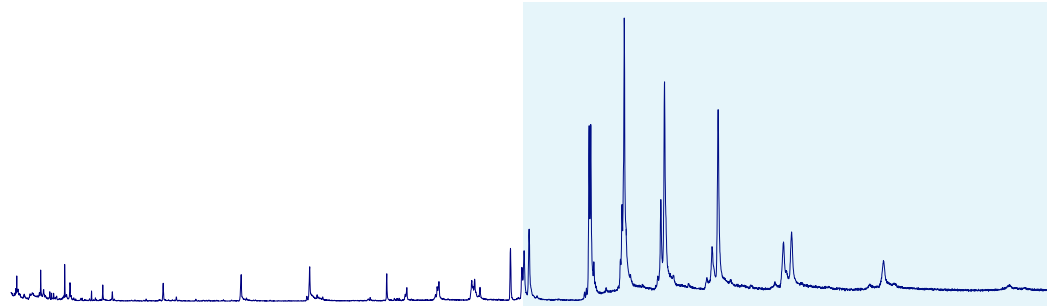
Spearmint Oil on DB-1, (App. Note 5989-7509EN)



Spearmint Oil on DB-1 – Resolution Check



Faster GC Total Analytical Cycle Times – A Variety of Approaches



Included
in
HW/SW

“ALS Sample Overlap”
in ChemStation SW

7890 GC
Faster Cool-down

Column & Gas Selection,
Method Translation

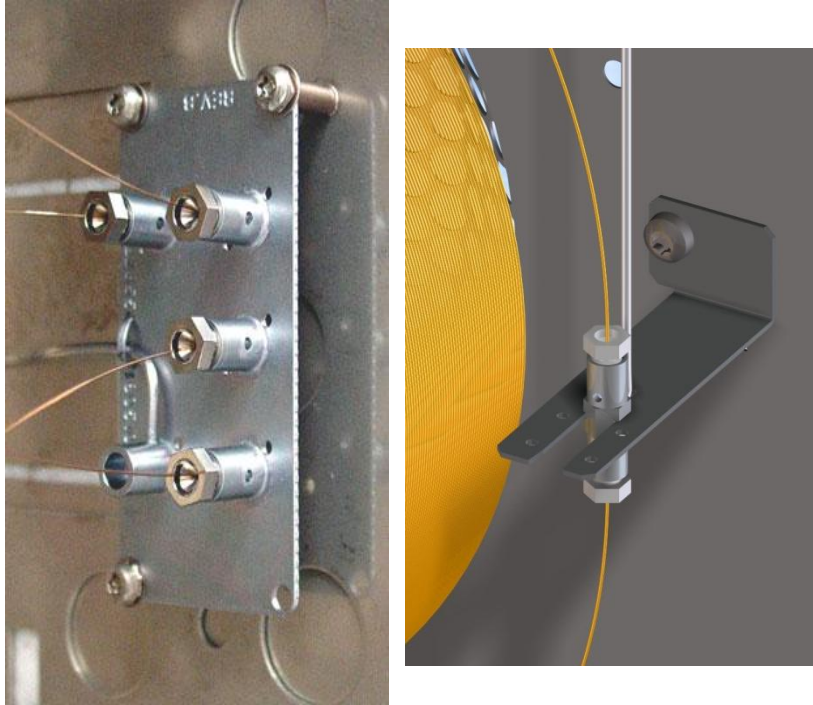
New Devices
*Significantly Faster GC
Analytical Cycle Times*

Capillary Flow Technology
(Backflush)

LTM Technology
(Rapid Heating)

LTM Technology
(Rapid Cooling)

New Devices for Faster Analyses



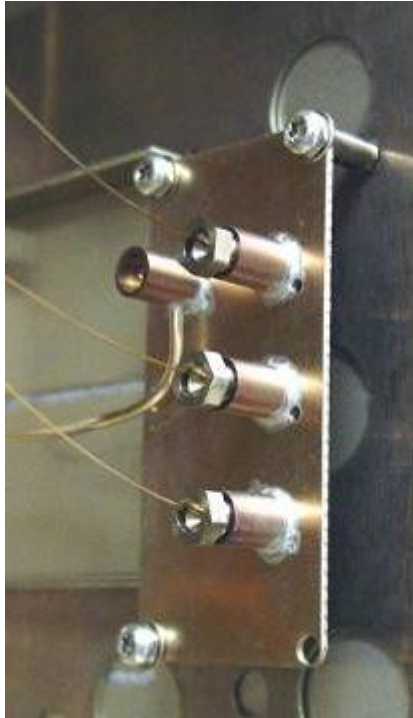
Capillary Flow Technology



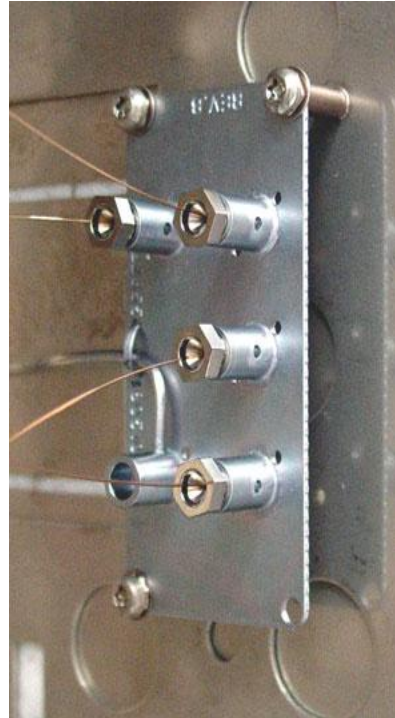
Low Thermal Mass Technology

Purged Capillary Flow Devices

**2-Way Splitter
with Makeup**



**3-Way Splitter
with Makeup**



Deans Switch

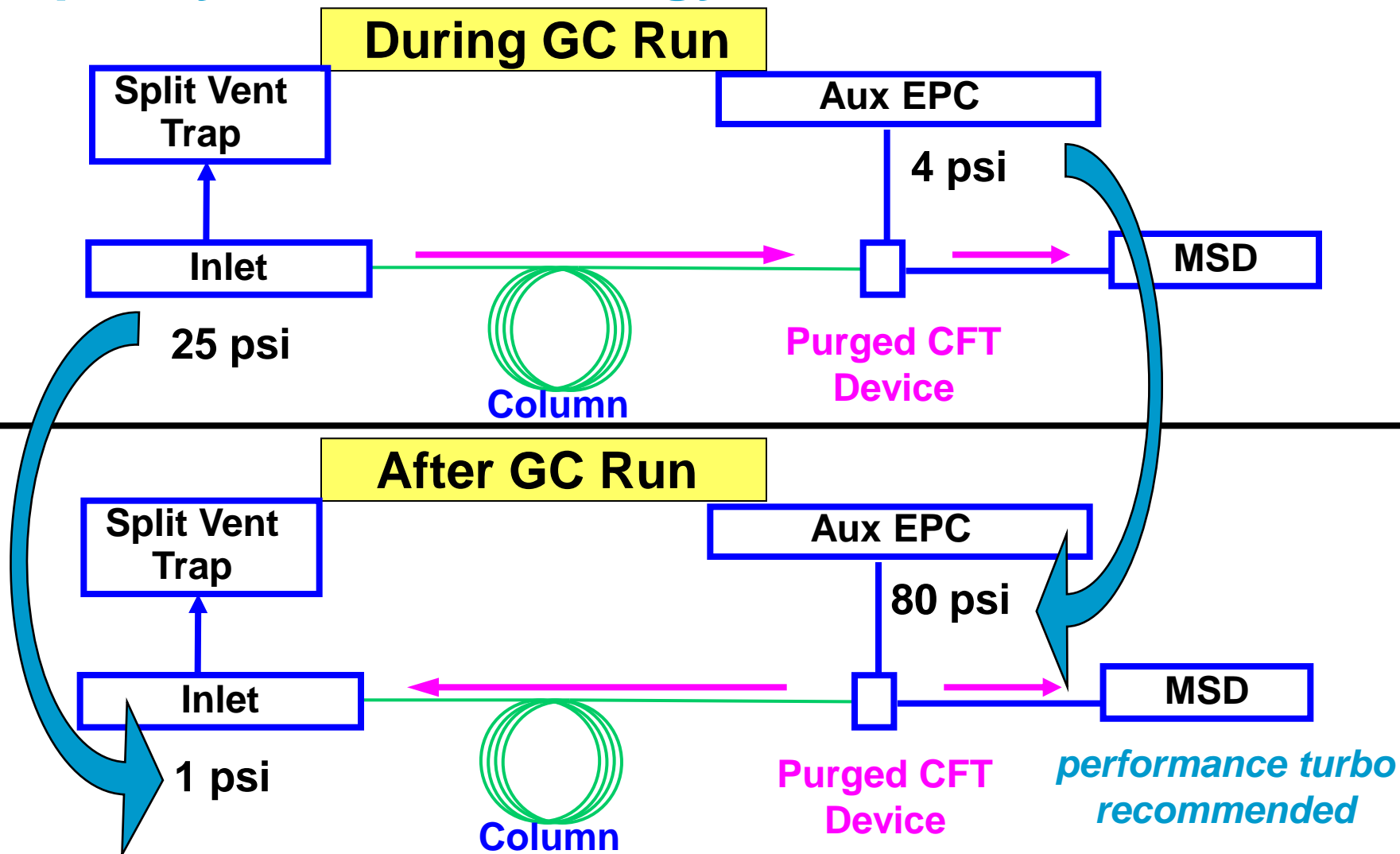


**Purged Union
(most recent)**



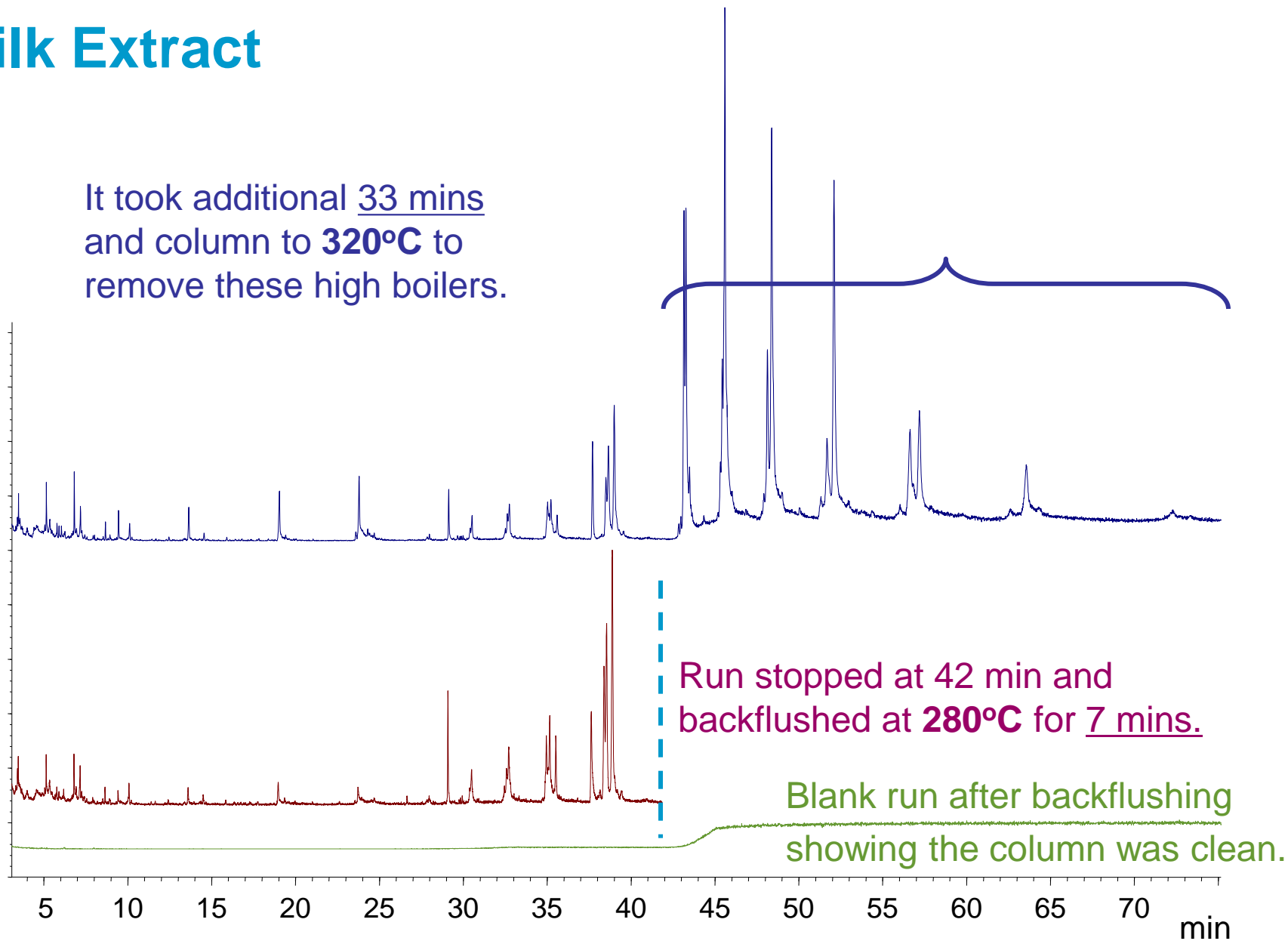
All Purged Capillary Flow Devices are Capable of Backflushing

Backflush with Purged Union or Any Purged Capillary Flow Technology Device



Milk Extract

It took additional 33 mins
and column to **320°C** to
remove these high boilers.



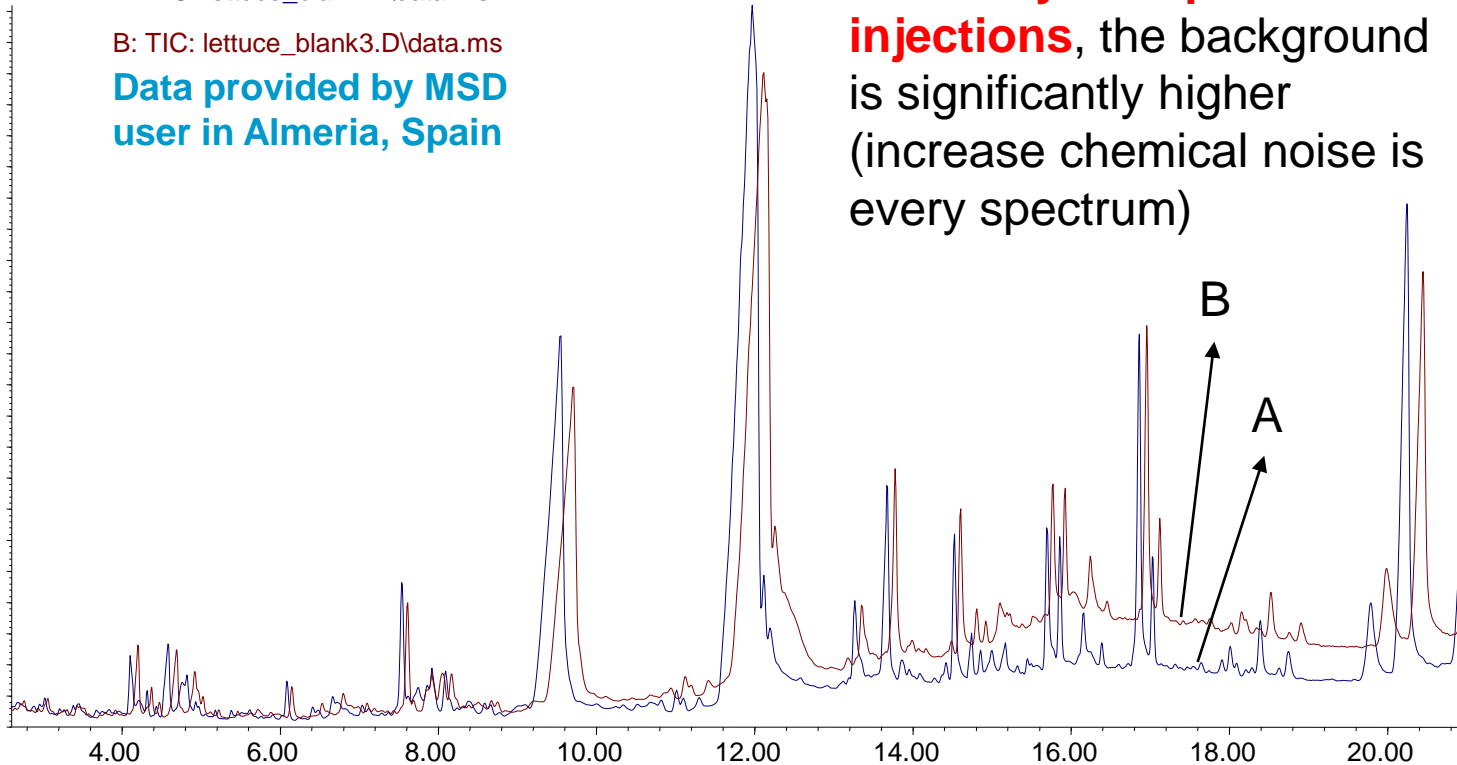
Without Backflush: A Serious Problem

Abundance

4.6e+07
4.4e+07
4.2e+07
4e+07
3.8e+07
3.6e+07
3.4e+07
3.2e+07
3e+07
2.8e+07
2.6e+07
2.4e+07
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
800000
600000
400000
200000
0

A: TIC: lettuce_blank.D\data.ms
B: TIC: lettuce_blank3.D\data.ms
Data provided by MSD
user in Almeria, Spain

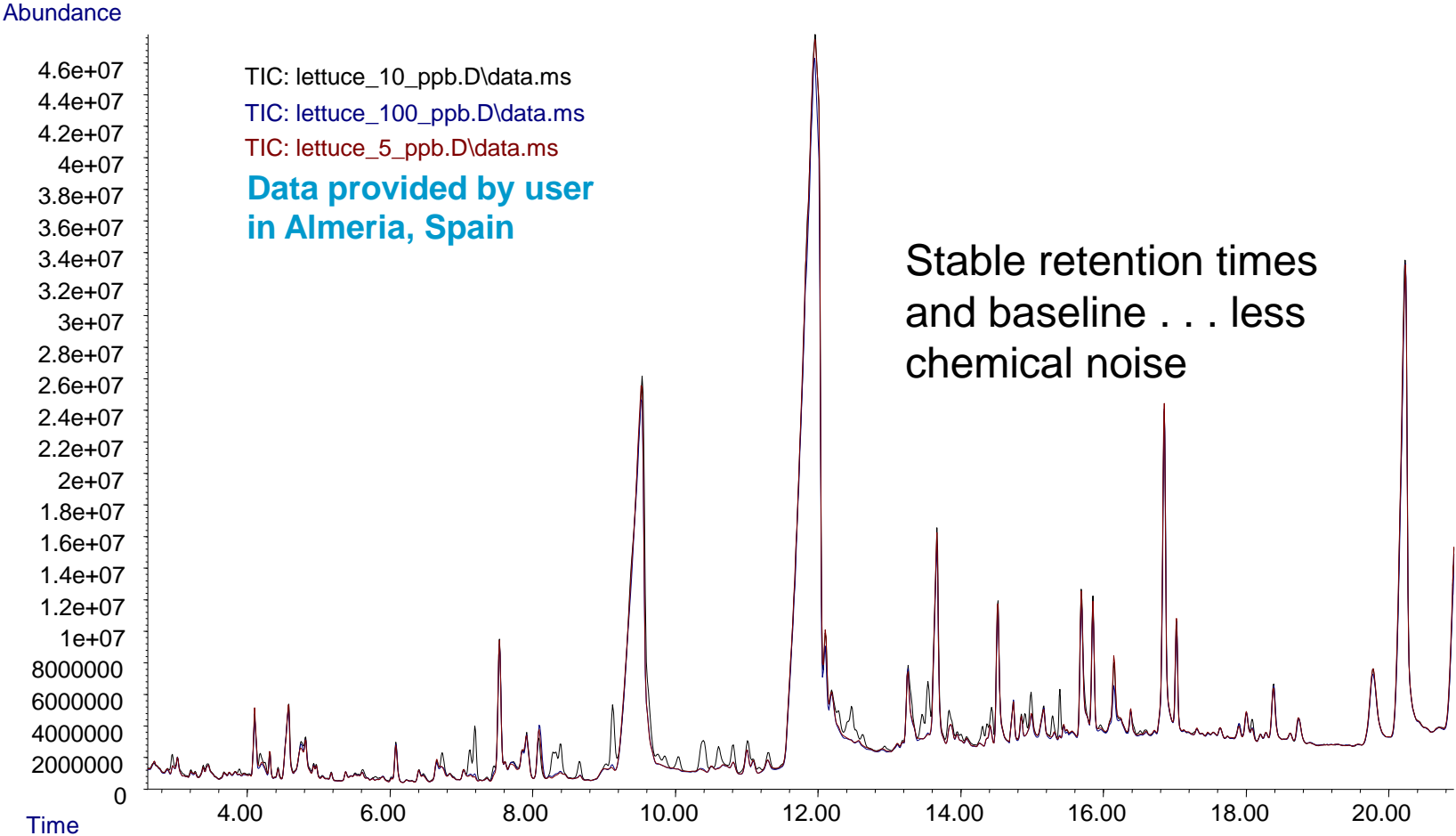
Time



After **only 3 10- μ L injections**, the background is significantly higher (increase chemical noise is every spectrum)

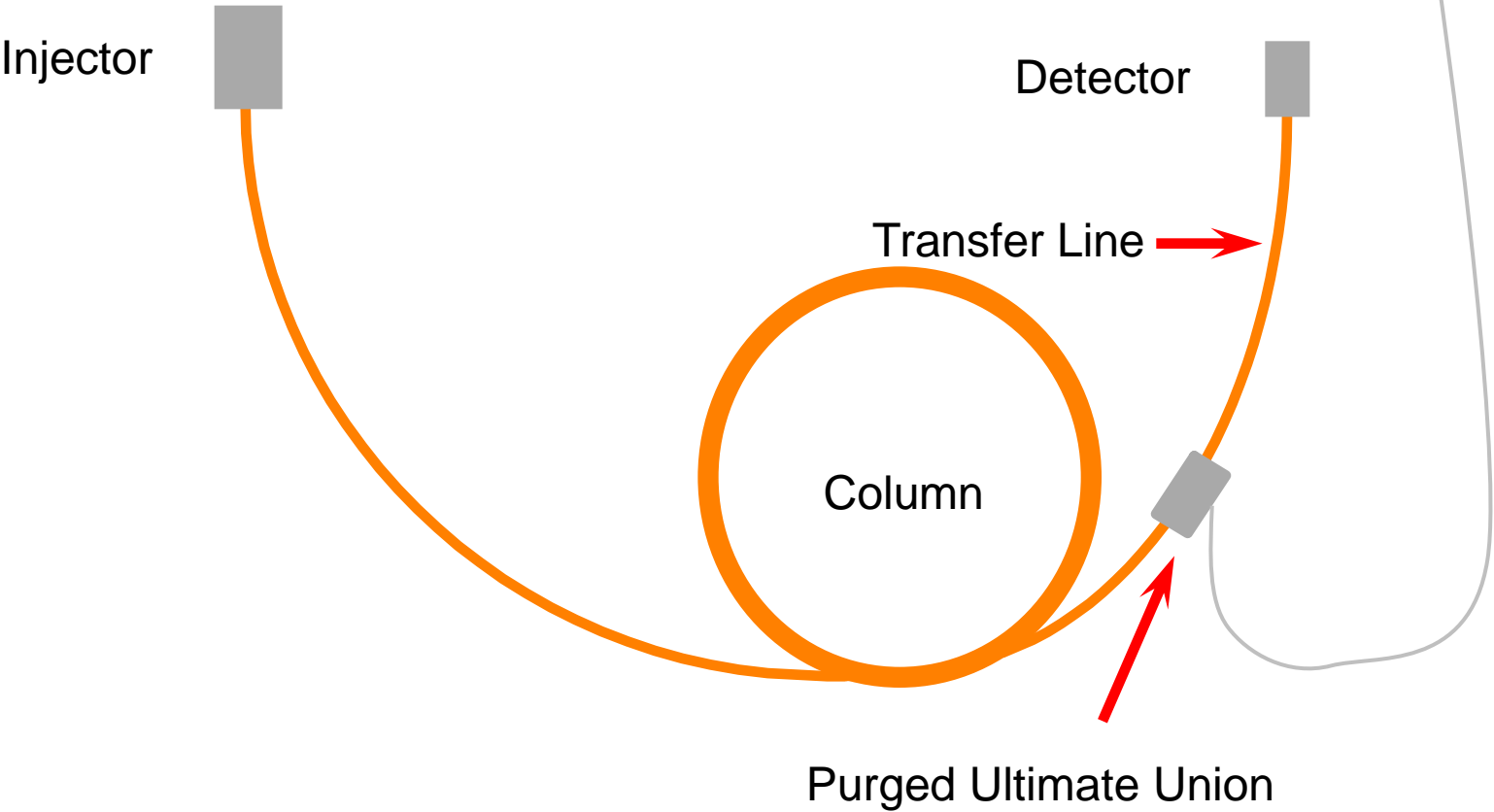
Overlay of two chromatograms of a blank extract injected BEFORE (A) and AFTER (B) three injections without backflush

With Backflush: No Increased Background (Less Spectral Noise) and Consistent Retention Times

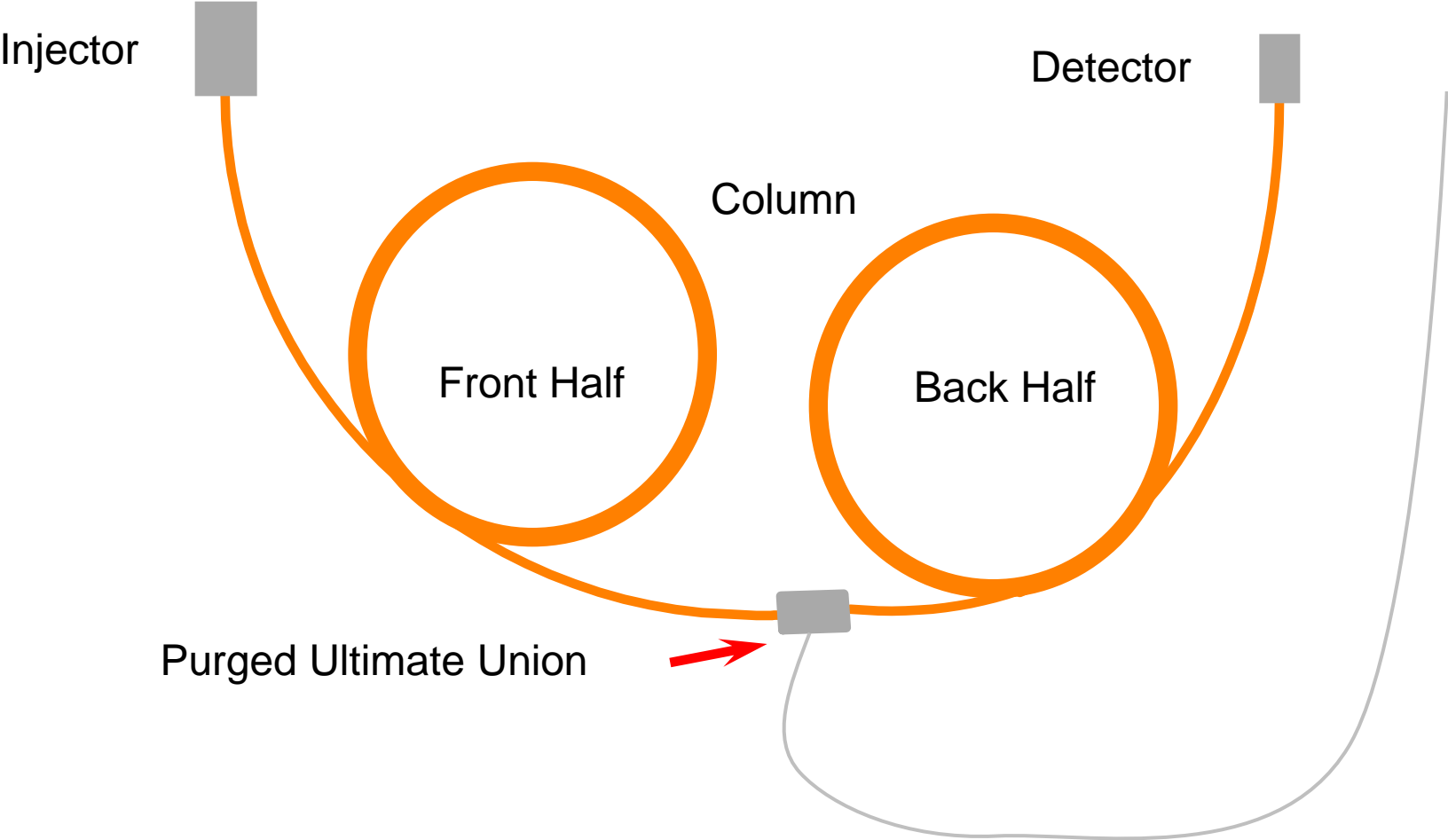


Overlay of three chromatograms of lettuce extract run with 2 min of back flush

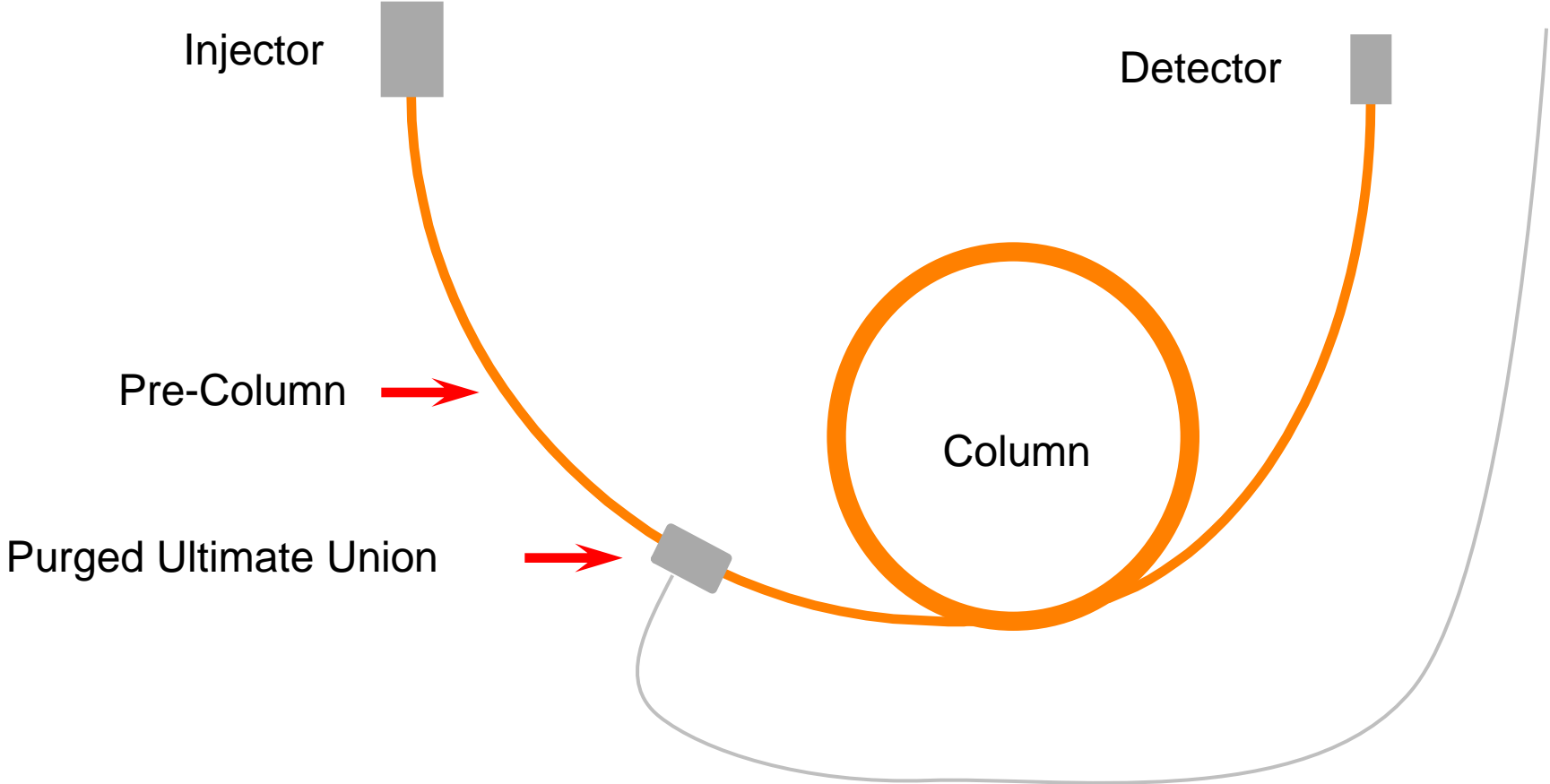
Post-Column Backflush



Mid-Column Backflush



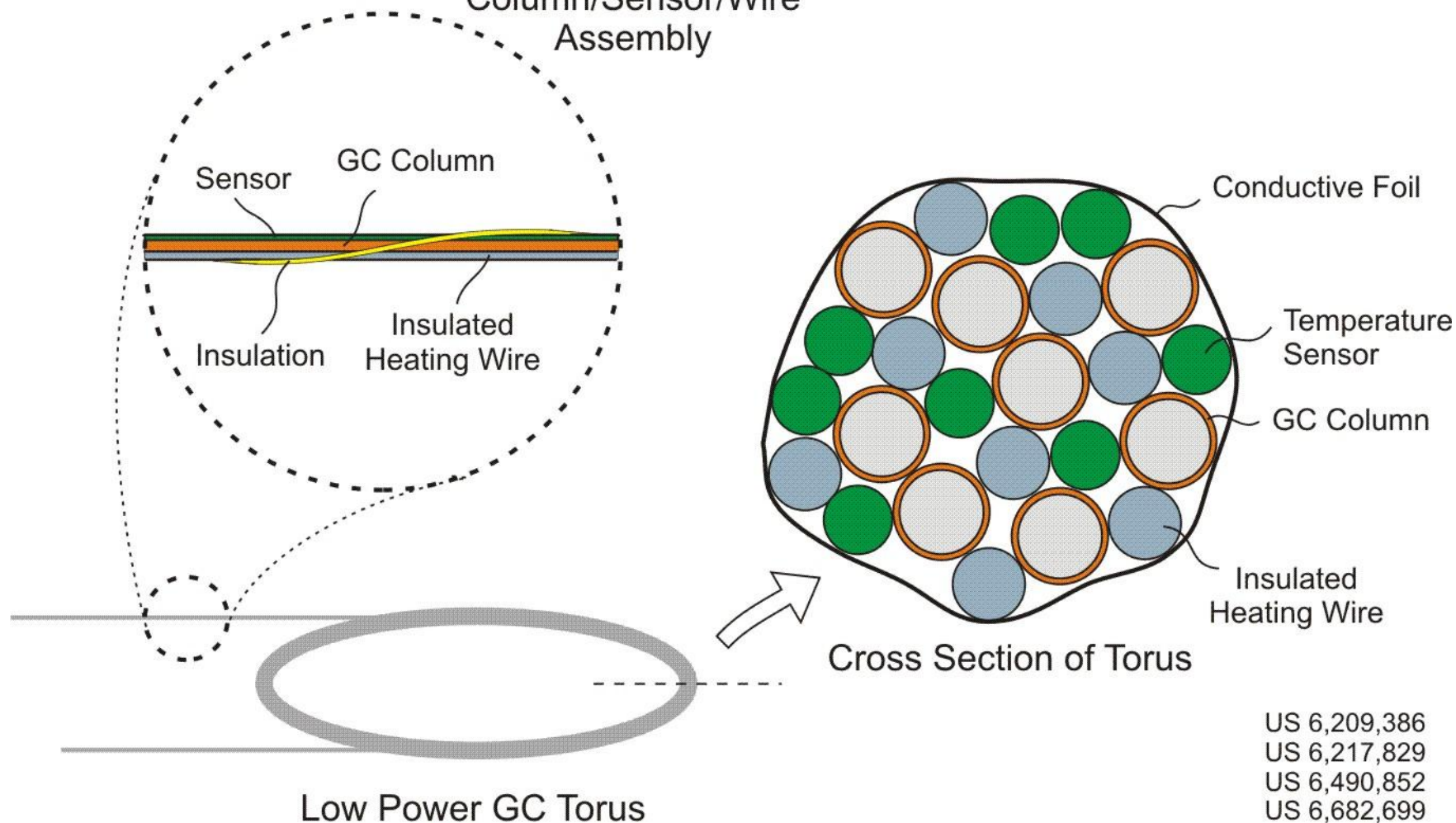
Pre-Column Backflush



“LTM” (Low Thermal Mass) Technology (Patented)

Directly heat/cool fused silica GC columns

Column/Sensor/Wire
Assembly



US 6,209,386
US 6,217,829
US 6,490,852
US 6,682,699

Interfacing LTM GC to an Agilent 7890 or 6890 GC

**GC oven door replaced with LTM-ready GC oven door (easy)
Use same GC injectors, detectors, autosamplers, software, ...**

Column Modules mount outside isothermal GC oven for fast heating and cooling

Independent and simultaneous temperature programming of 1-4 column modules

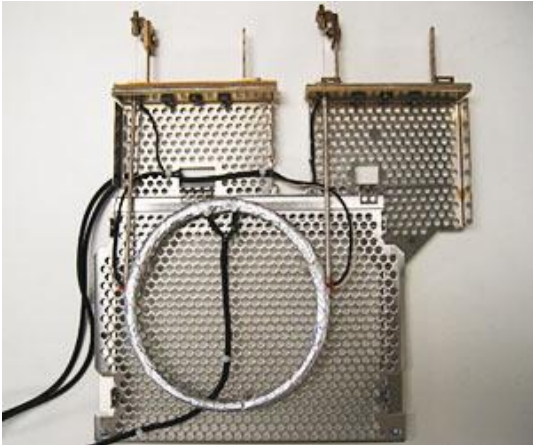
LTM Column Modules

LTM Control System
w/ Keypad User Interface
(LTM Control SW for ChemStation
Available from Agilent Nov/Dec'08)



Note: Front thermal shield removed to show LTM column modules
Entire thermal shield should always be in place during operation

A Closer Look



LTM Column Assembly

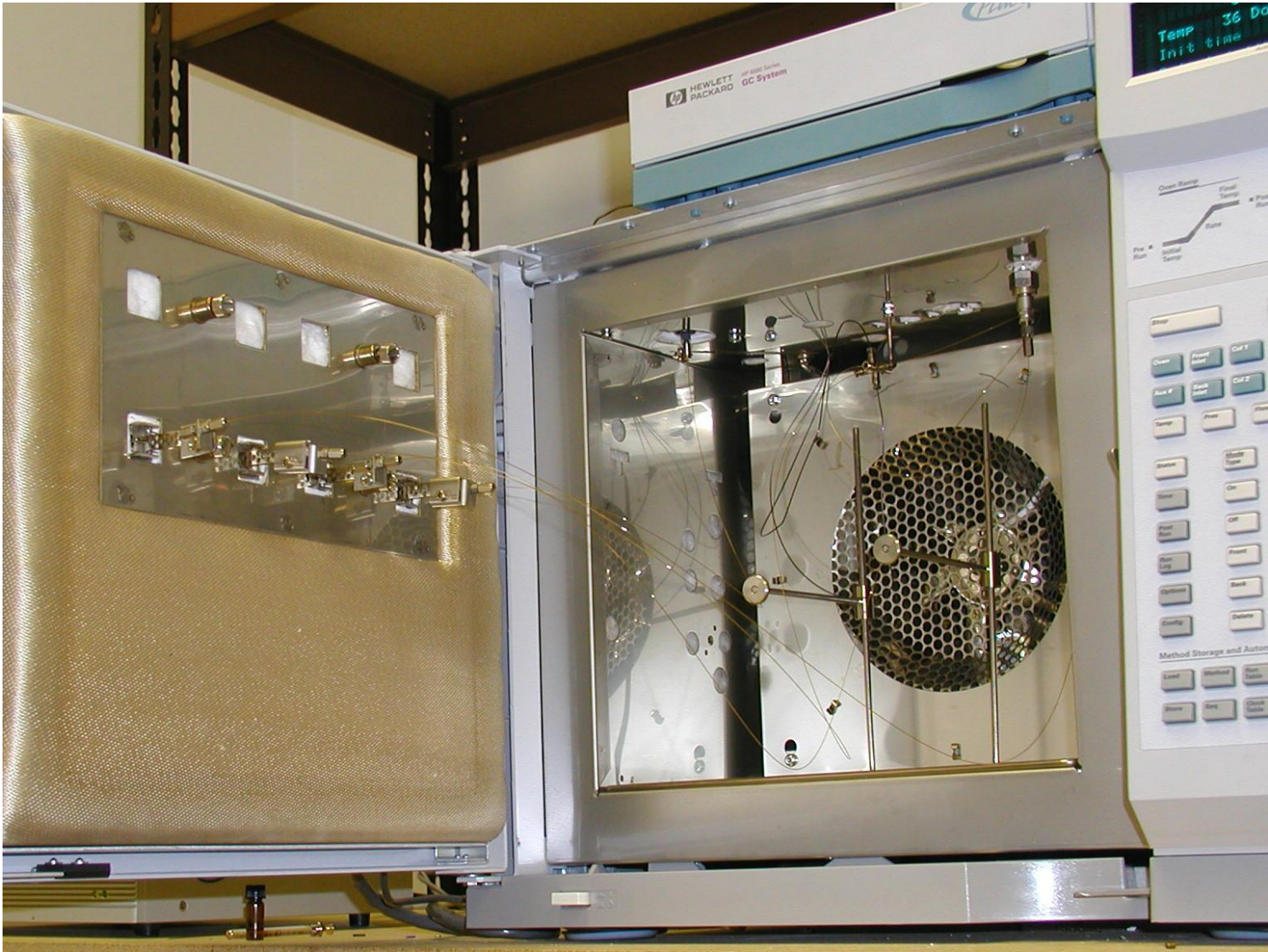


LTM Retrofit Door



LTM Column Module

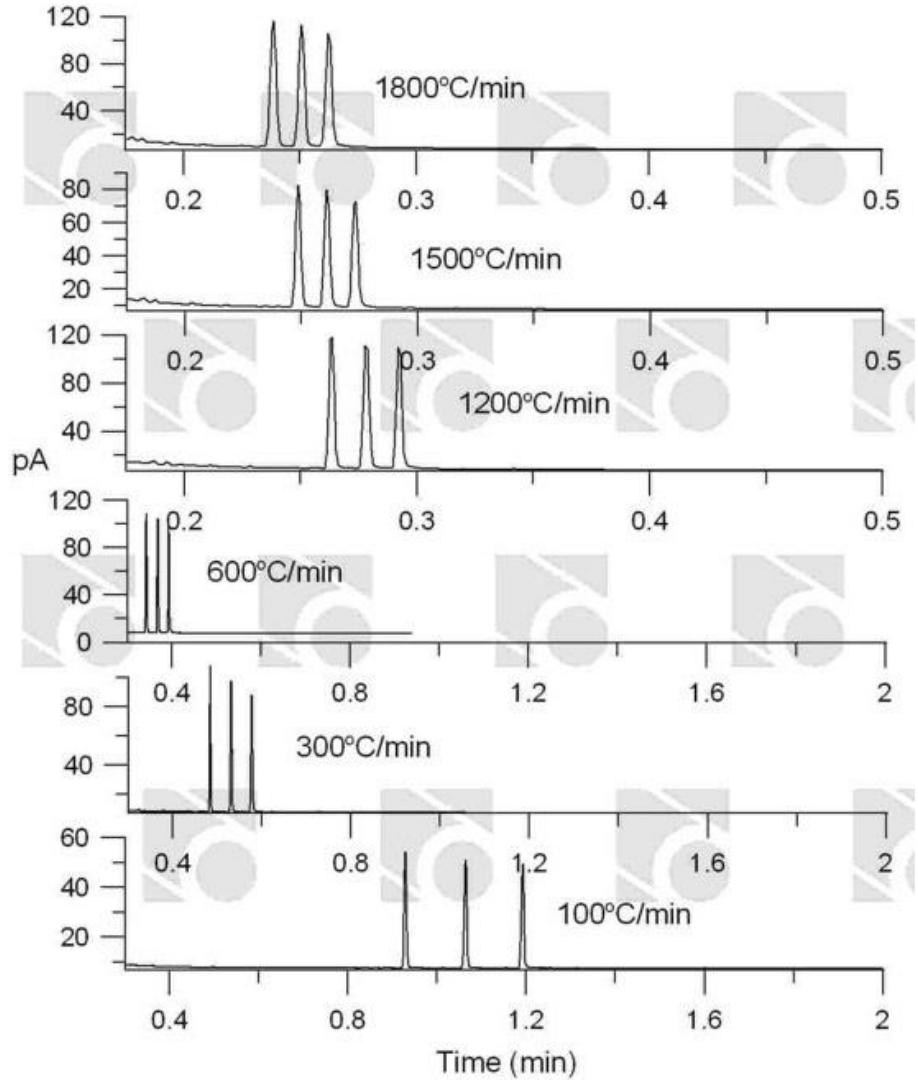
Inside View



LTM Heating Speeds

Heating speeds can be set up to 1800°C/min

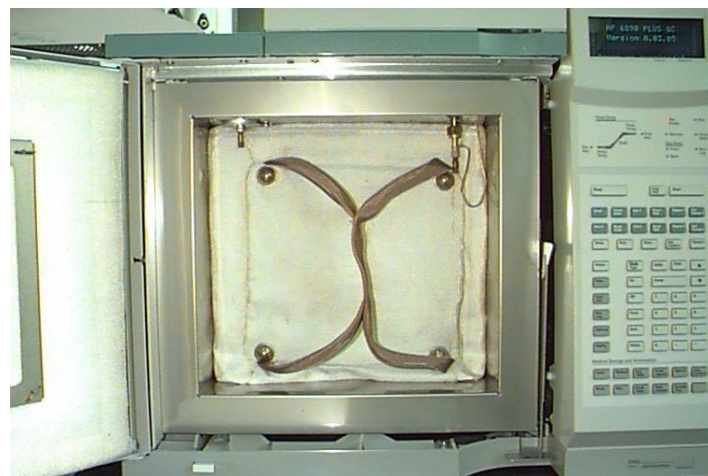
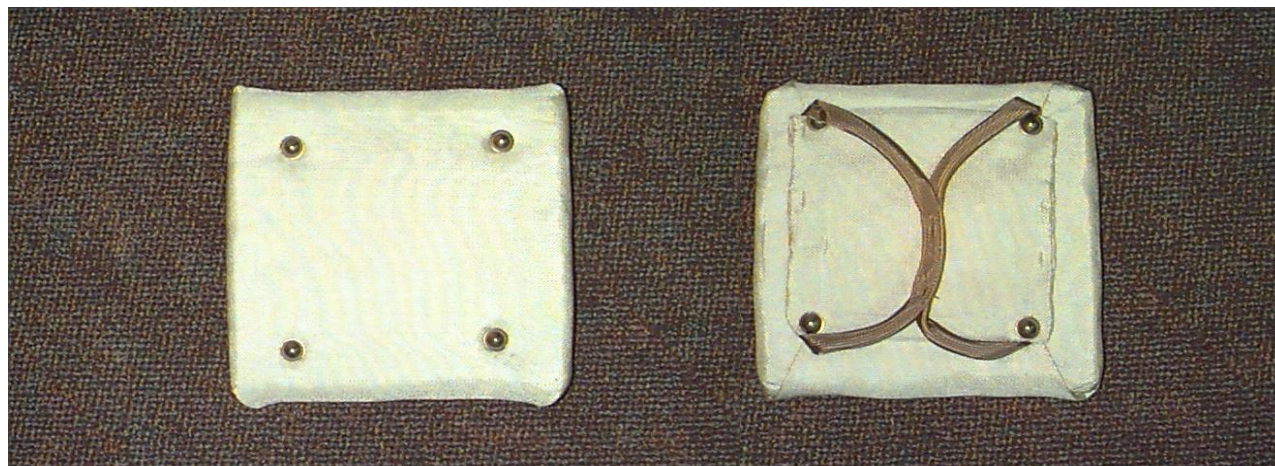
- achievable rates depend on column mass, configuration and column void times
- ... also including practical trade-offs of speed vs resolution



6890/7890 Oven Ramp Rates

	“Standard”	“Fast”		“Turbo”
Temp. Range (°C)	120V	240V	120V Insert	240V Insert
50 to 70	75	120	120	120
70 to 115	45	95	95	120
115 to 175	40	65	65	110
175 to 300	30	45	45	80
300 to 450	20	35	35	65
		(°C/min)		

Oven Insert For The 6890/7890



The insert reduces the 6890/7890 effective oven volume by 50%.

Allows 120V 6890/7890 to achieve ramp rate equal to the 240V.

Allows faster oven cool down over std 6890.

**Part No. G2646-60500
(Can't use on 6890/ 5973 MS)**

Typical Cooling Times for a LTM Column (Standard Size)

Column Length

°C	2 m	5 m	10 m	15 m	30 m
350->300	3	5	6	7	9
300->250	3	5	7	7	13
250->200	4	7	8	10	17
200->150	4	8	11	13	22
150->100	7	13	17	19	36
100->50	13	27	34	41	75

350°C → 50°C Equilibration	<u>Type</u> LTM (2m Column)	<u>Time</u> 34 seconds (= 3+3+4+4+7+13) 3 seconds
350°C → 50°C Equilibration	7890 GC	3-4 minutes 2-3 minutes

Some Practical Uses of LTM Rapid Heating/Cooling

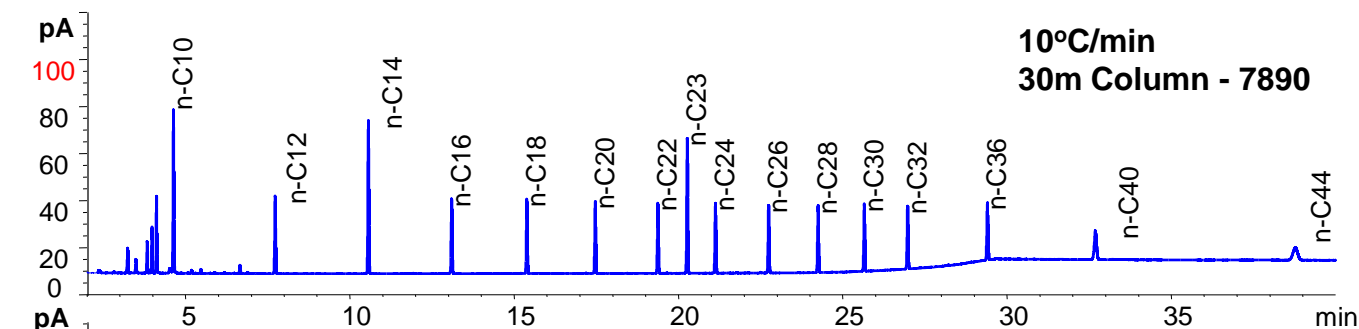
Environmental

Need for high through-put, especially for low margin samples (eg. Total Petroleum Hydrocarbon (TPH)), where analytical cycle times are a critical element in making a profit

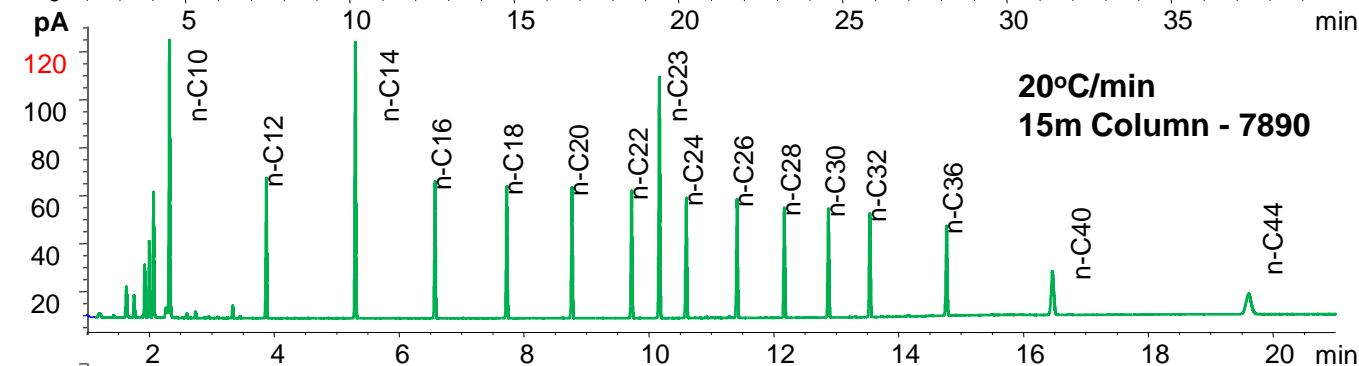
Hydrocarbon Processing

Certain process control analyses (eg. Simulated Distillation) lend themselves to batch analysis. Shorter analytical cycle times get data back to operations faster, or allow more samples/shift

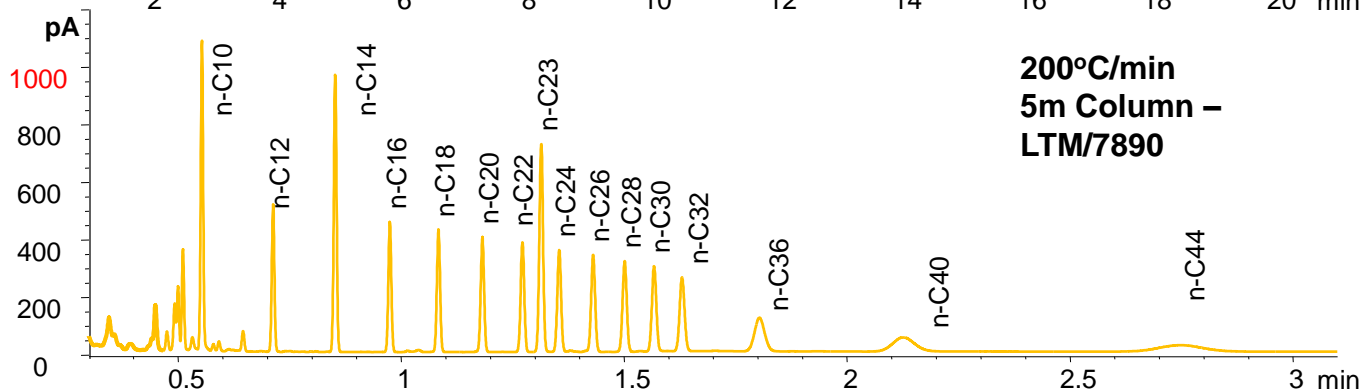
TPH Analysis with Faster GC Run Times (C₁₀→C₄₄ Standard Shown)



Standard
GC Run Time

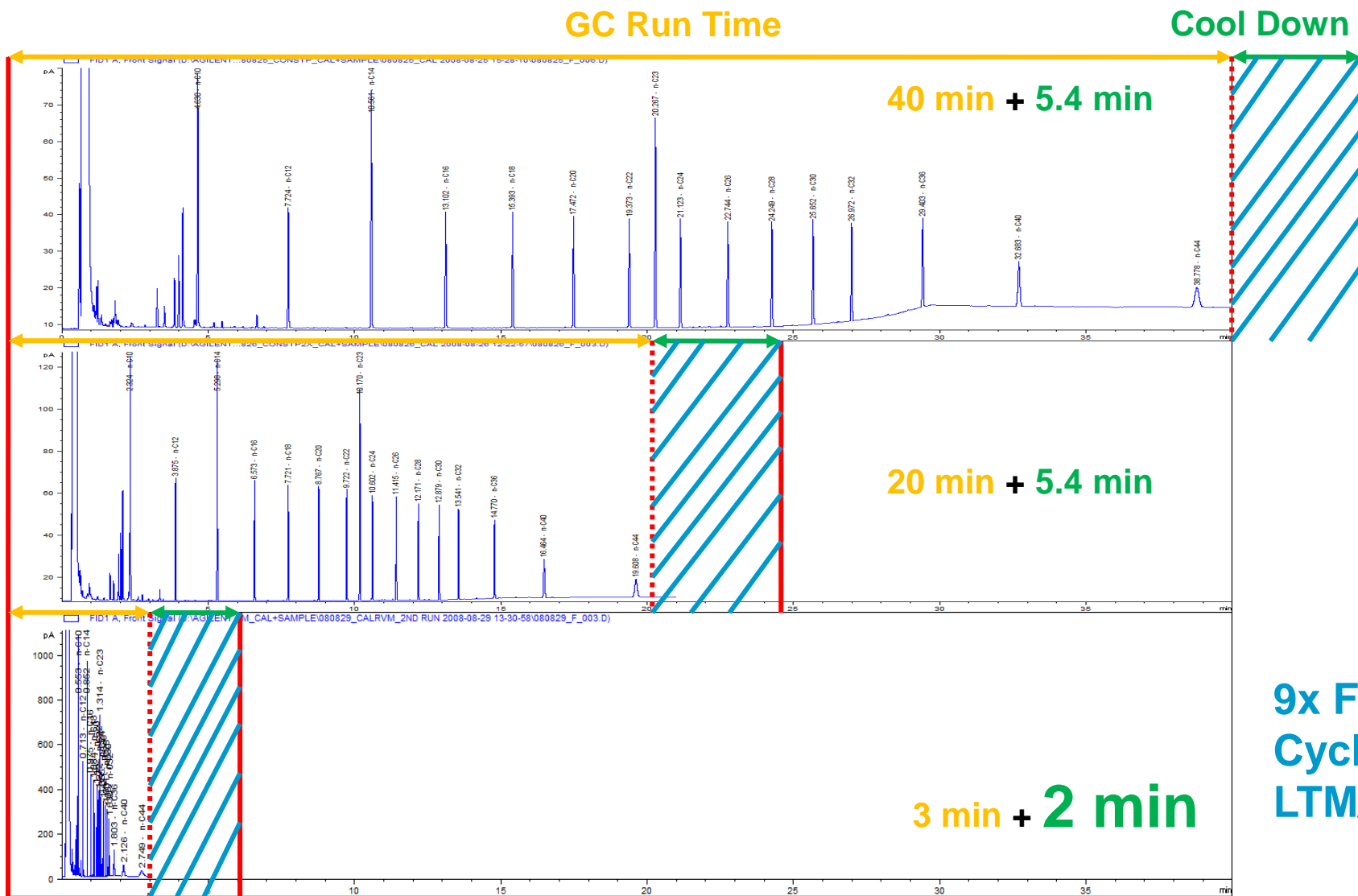


Shorter Column,
2x Faster
Ramp Rate



5m Column,
10x Faster
Ramp Rate
with LTM

LTM Also Greatly Reduces Cool-Down Times, → 9x Faster Heating/Cooling Cycle Times



CONCLUSIONS

Carrier Gas – Helium can still go fast but Hydrogen has the most advantage at high velocities, Nitrogen might work (GEC)

Diameter – Smaller allows shorter length but has less capacity

Small Change in ID Easier to Translate – Again think capacity

Length – Shorter might be possible without losing a lot of R

Temperature Program – Use MTS to scale temps properly

Method Translation Software – FREE, reliable

Capillary Flow Technology – Backflush instead of Bake Out

Low Thermal Mass GC – Fast Heating, Fast Cooling

ANY QUESTIONS?

Technical Support
1-800-227-9770, 3

E-mail: gc_column_support@Agilent.com



Daron_Decker@Agilent.com
Eric.Pavlich@Agilent.com