

Increasing sample throughput to *MACH* speed using fast oven programming

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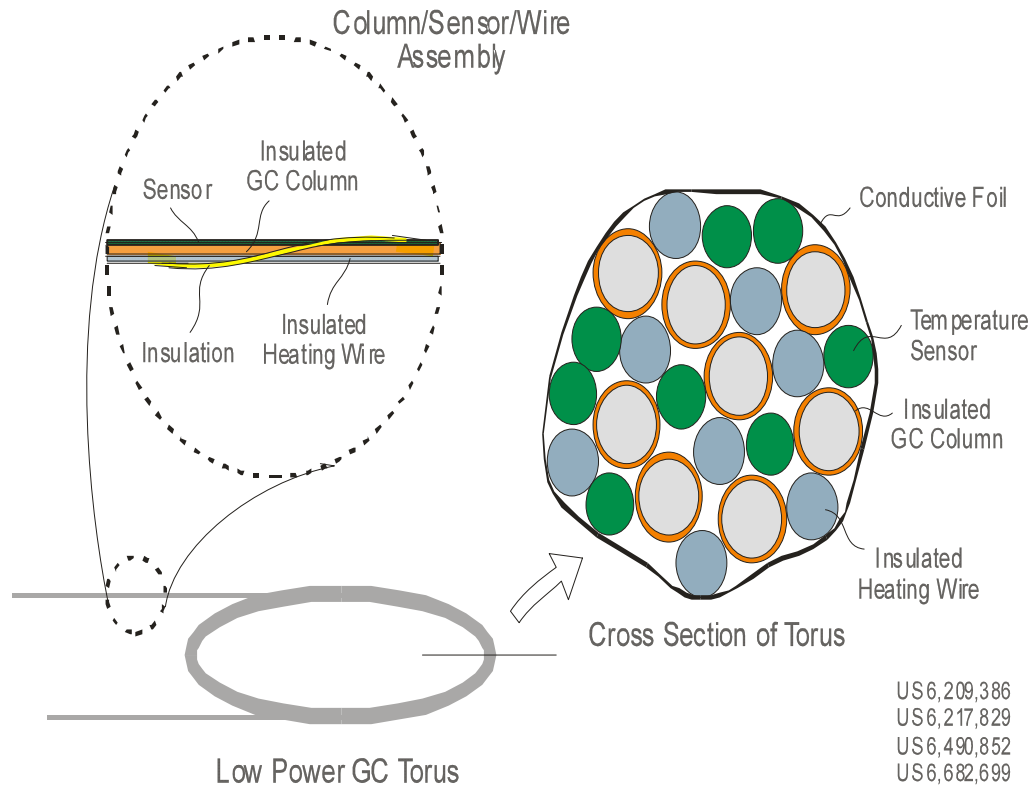
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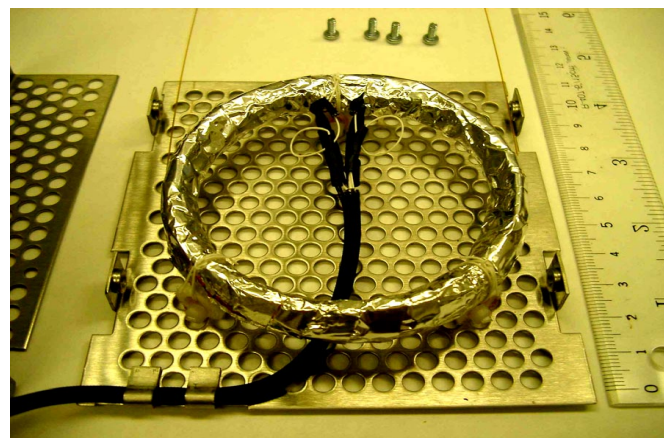
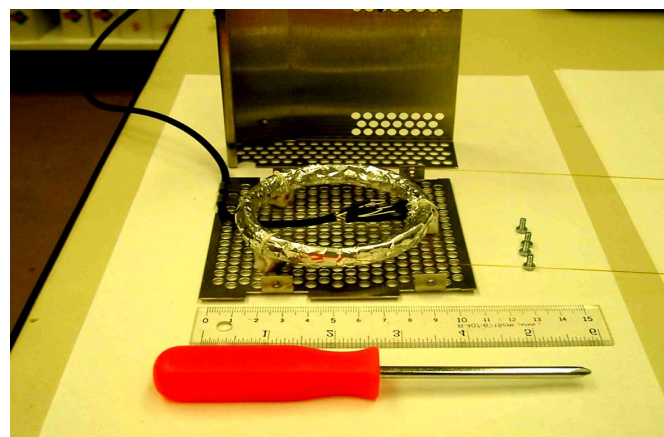


Modular Accelerated Column Heater (MACH™)

Low Thermal Mass GC Technology



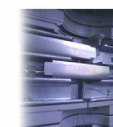
US6,209,386
 US6,217,829
 US6,490,852
 US6,682,699



Heating rates: up to 1200°C/min !!



Gerstel MACH™ Twin Module



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RVM Programming Integrated in Chemstation

The screenshot displays the MSD ChemStation software interface for an Instrument #1 - MACH4.M - EI. The main window is titled "MSD ChemStation Build 10" and contains an "Instrument Control" panel. This panel includes a status bar with "Idle", a sample name field, a "Data File: 624mix4.d" field, a green arrow button, a "Post Run Time" display showing "0.00", and buttons for "STOP", "Data", and "Help". Below this are tabs for "Sample", "Chromatography", and "Detectors". The "Chromatography" tab is active, showing icons for "Injector", "Inlets", "Columns", "Oven", "Aux", and "MS". A "GC Status Messages" box is empty, and a "GC Signal 1" box shows a value of "0.3". A "MACH FastGC - 1" window is open, displaying "MACH FastGC" and "IDLE" in a yellow bar, with a table of column temperatures:

Column	Temp (°C)
Column 1	N/A
Column 2	N/A
Column 3	N/A
Column 4	N/A

The "Oven Temperature" is displayed as "250" and "Column-1 Flow Cal." as "0.3". Below the instrument control is a "GC Signal 1" window showing a plot with a flat baseline. The x-axis is labeled from 25.0 to 29.0. A green bar at the bottom of the software window indicates "Acquisition completed". The Windows taskbar at the bottom shows the Start button and several open applications: CAG Bootp Server, Enhanced Data Anal..., Instrument #1 MSTo..., Instrument #1 - MAC..., Instrument 1 (online)..., Instrument 1 (offline)..., and Document - WordPad. The system clock shows 16:07.



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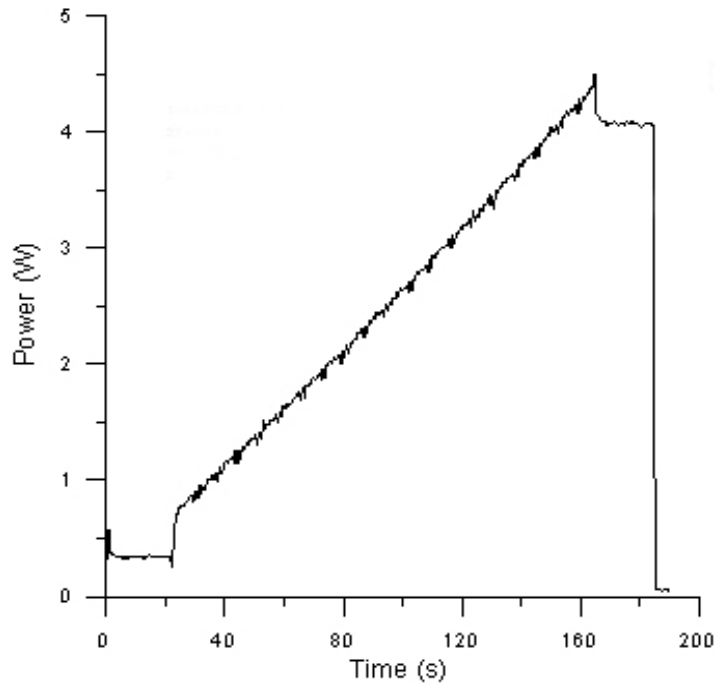


Comparison of Power Profiles between LTMGC and Conventional GC

Power Profile of LTMGC

40C – 0.35 min – 60C/min – 180C – 0.35 min

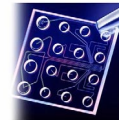
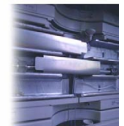
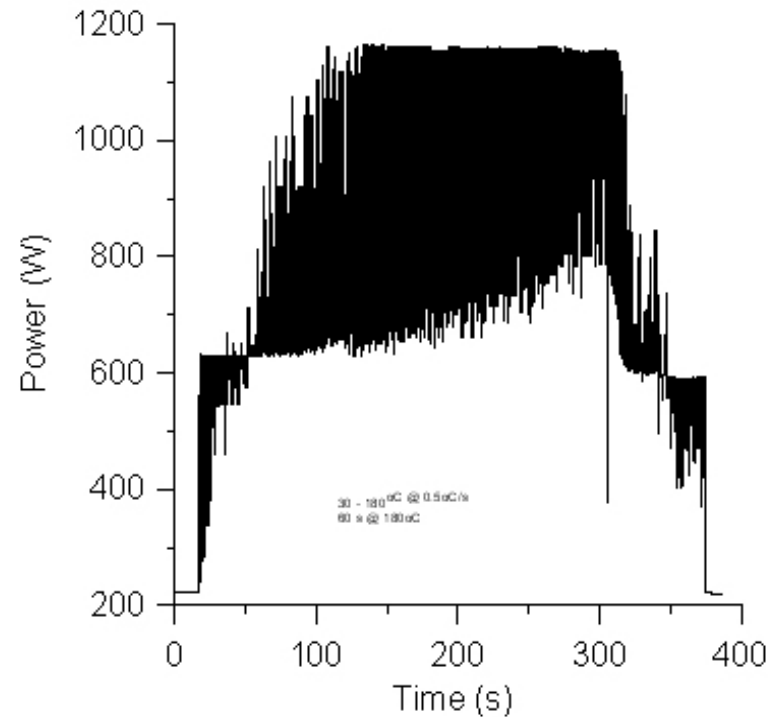
Peak power consumption: **Less than 5 watts**



Power Profile of standard GC

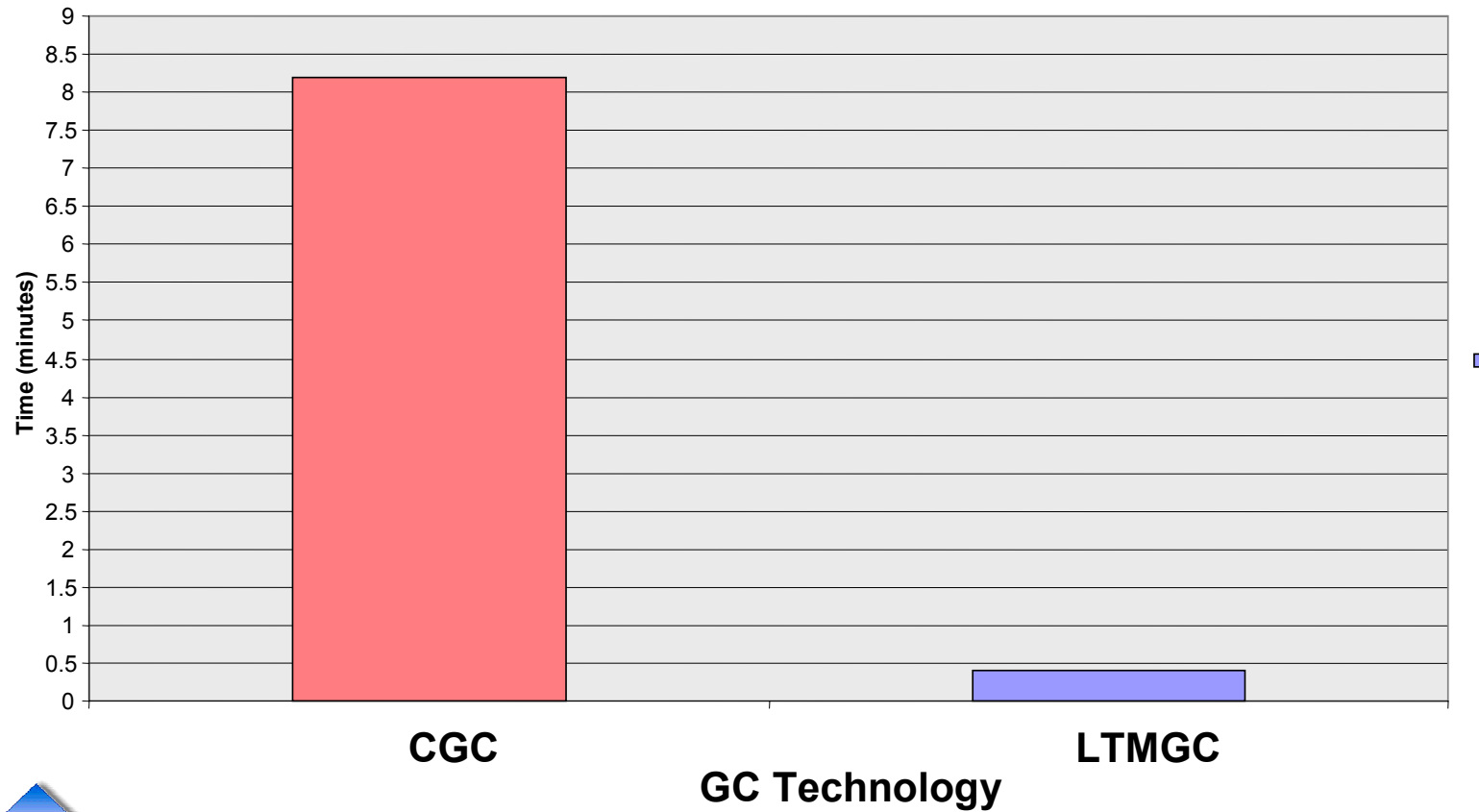
30 C – 0 min – 30C/min – 180C – 60 second

Power consumption: **Average 850 watts**

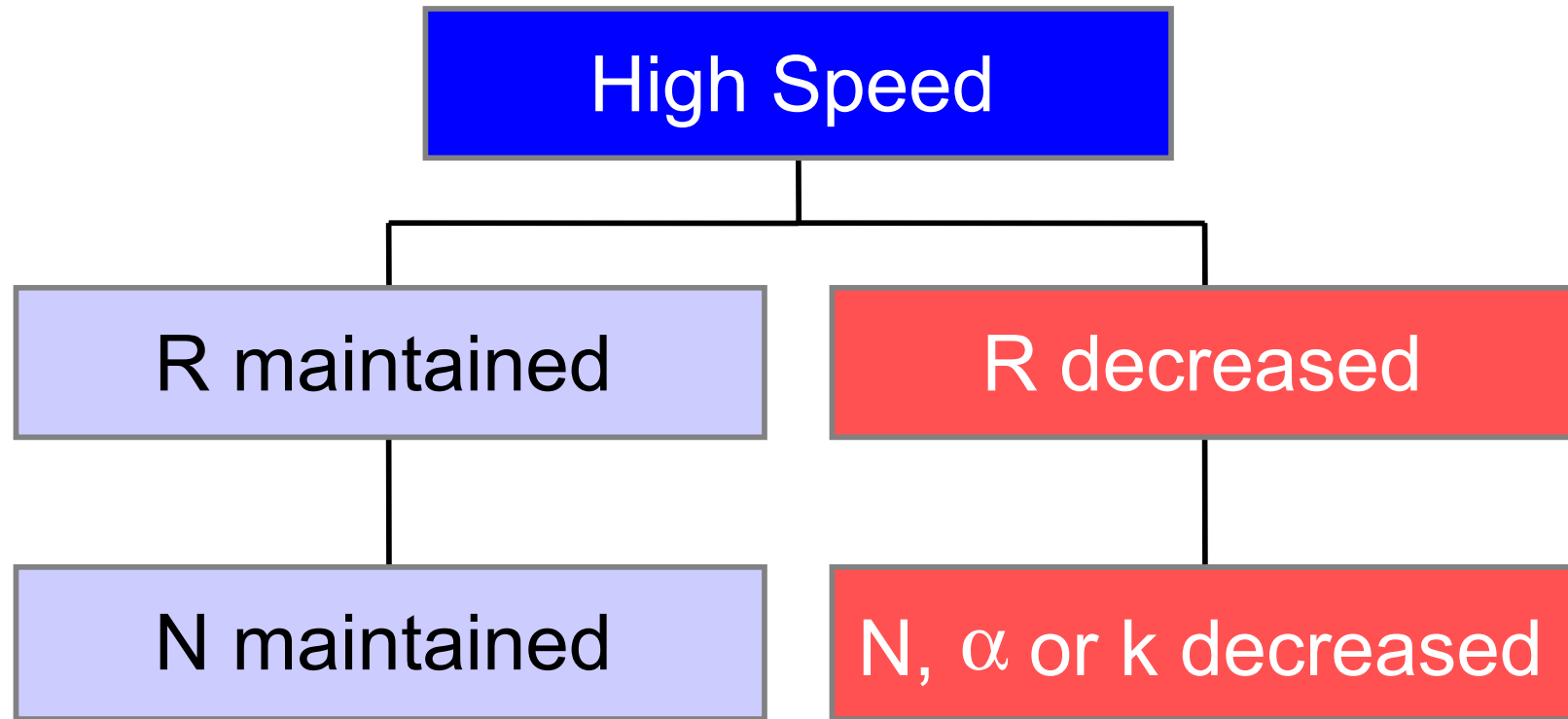


Cool Down Time Comparison

Comparison of Cool Down Time LTMGC vs. Agilent HP-6890A
Agilent HP-6890A, SN US0006181
LTMGC: 5 metre, 0.1 mm id, 0.12 micron DB-1



Increasing Speed of Analysis Strategies



Increasing Speed of Analysis Strategies

- Fast GC = short column ($L \downarrow$)
- Fast GC = increase u ($H_2 > He > N_2$)
 - Vacuum outlet conditions

- Narrow bore column
- Optimized phase ratio
- **Optimized temperature program**

- Fast GC = low capacity factor (k)
- High β ratio: wide bore - thin film
- High temperature
- **Fast temperature program**



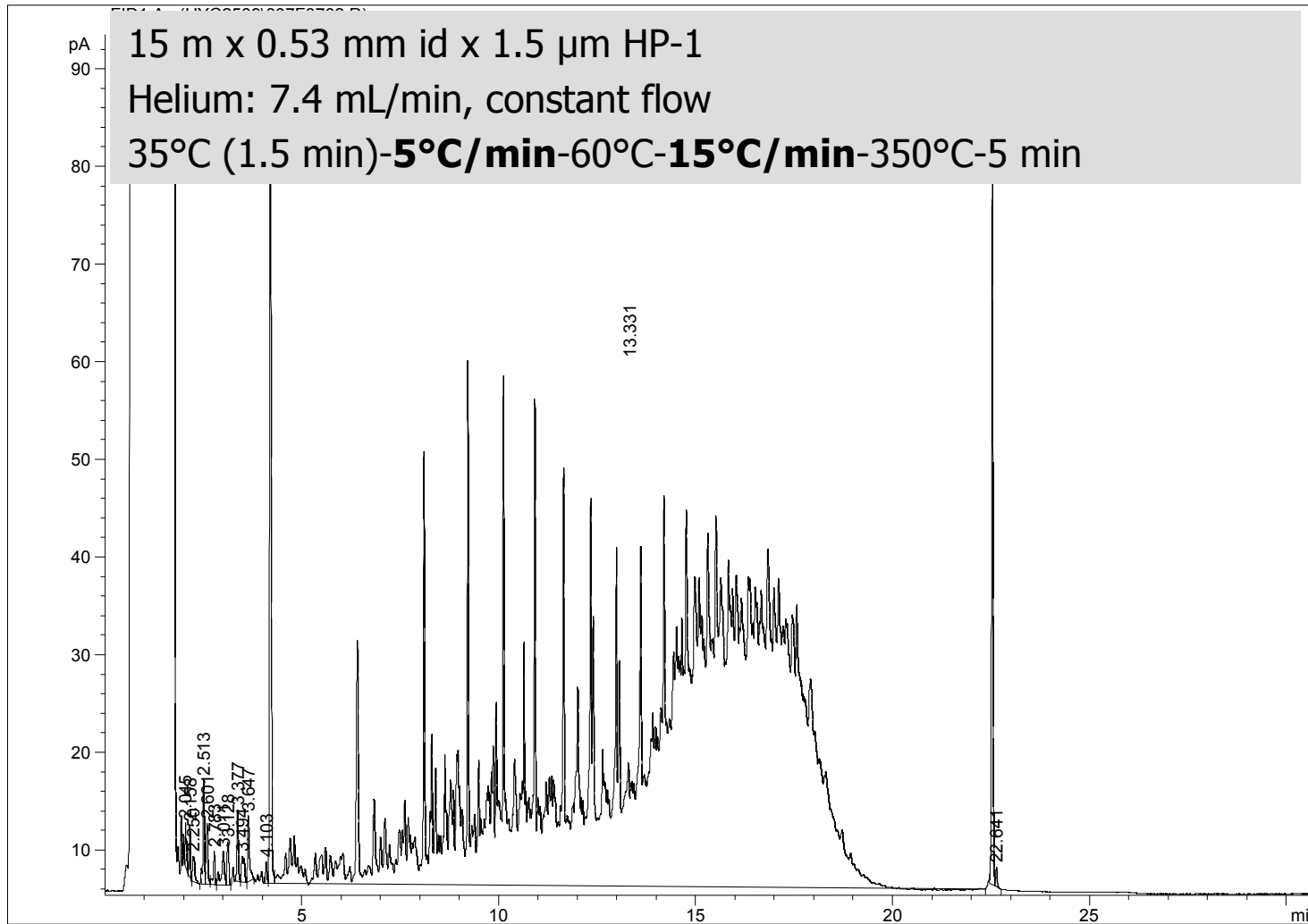
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Hydrocarbon Index (ISO 9377-2 and ISO 9377-4)



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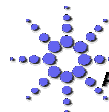
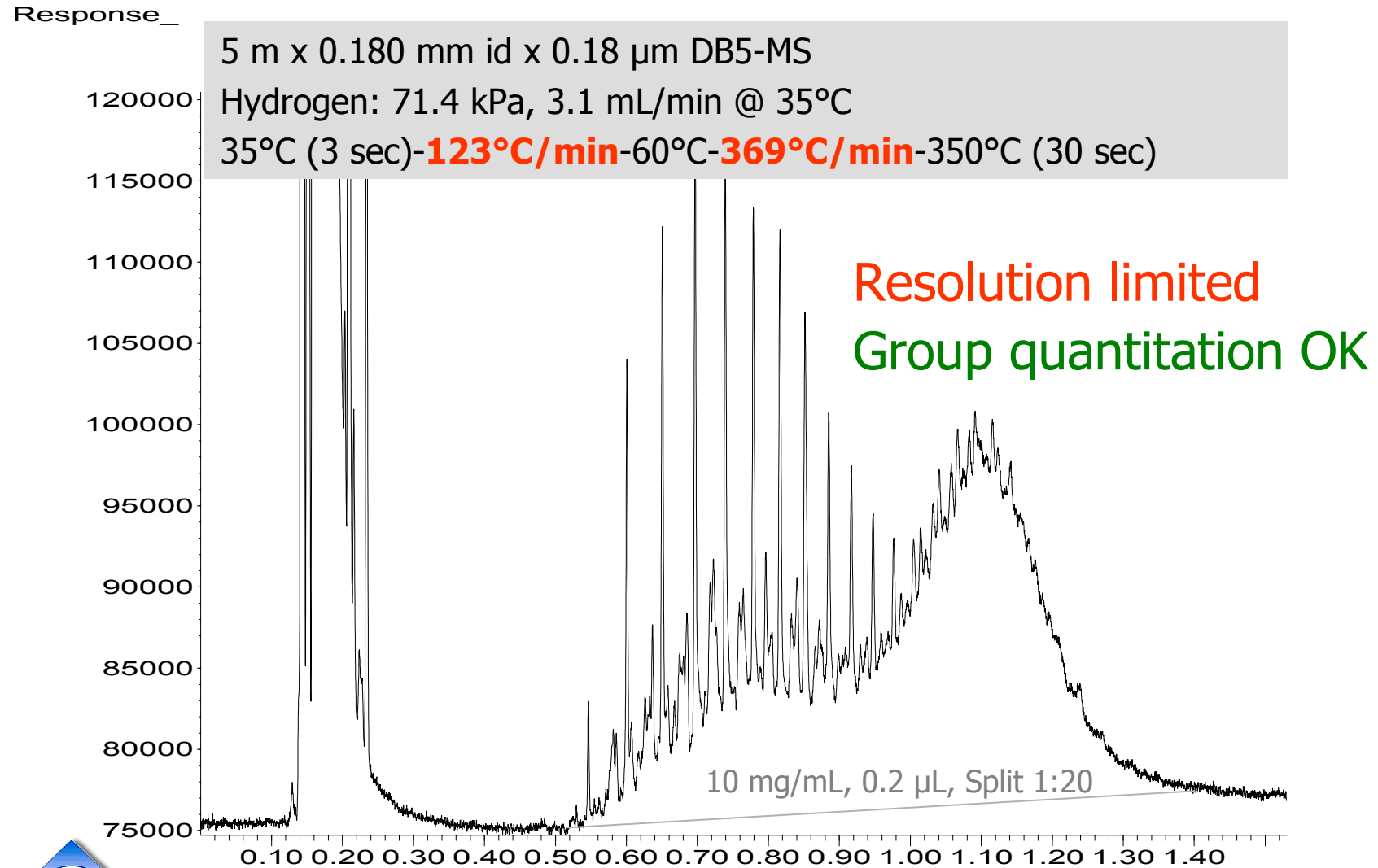


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Hydrocarbon Index (*MACH* method 1)

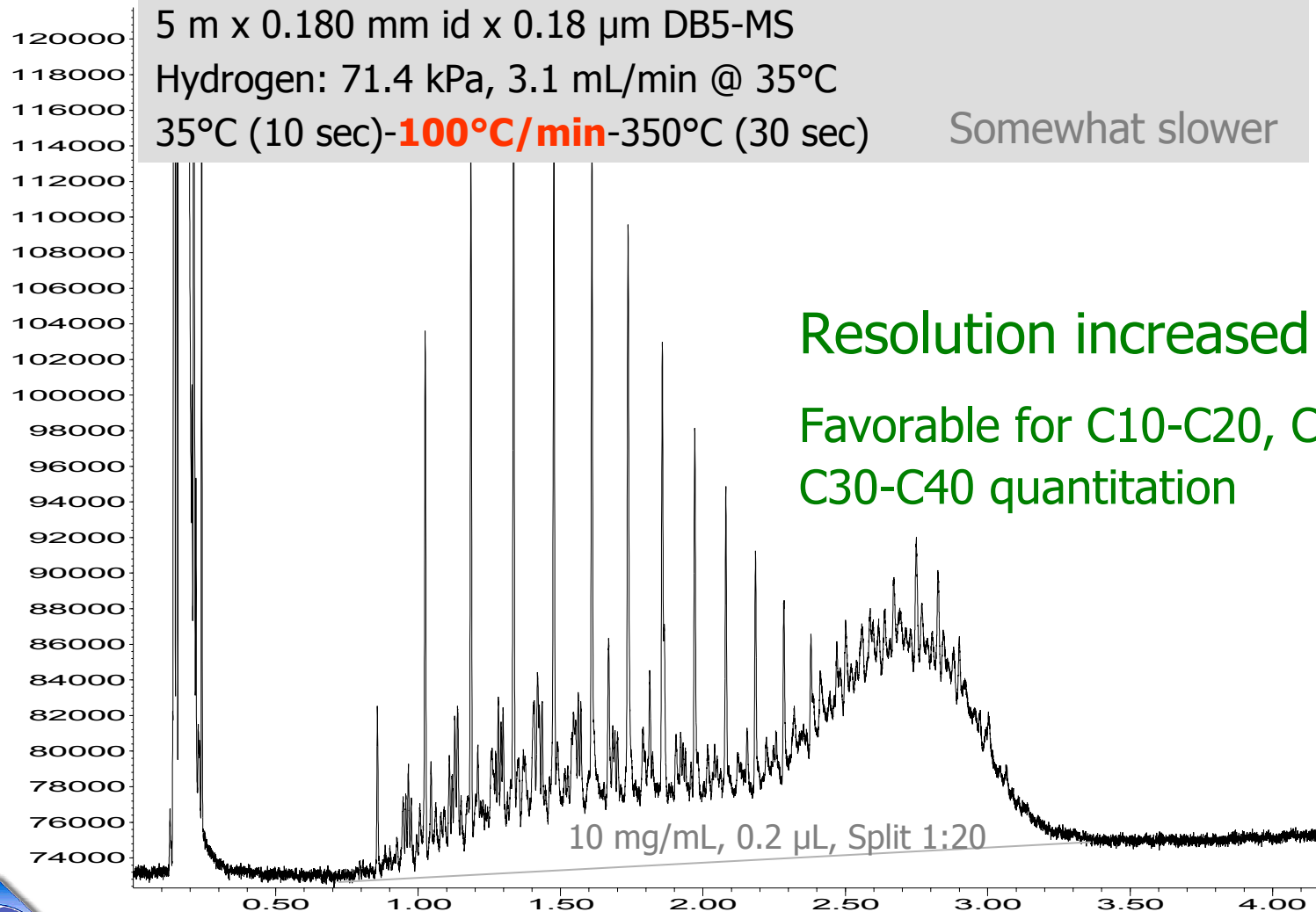


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Hydrocarbon Index (*MACH* method 2)

Response_

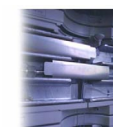
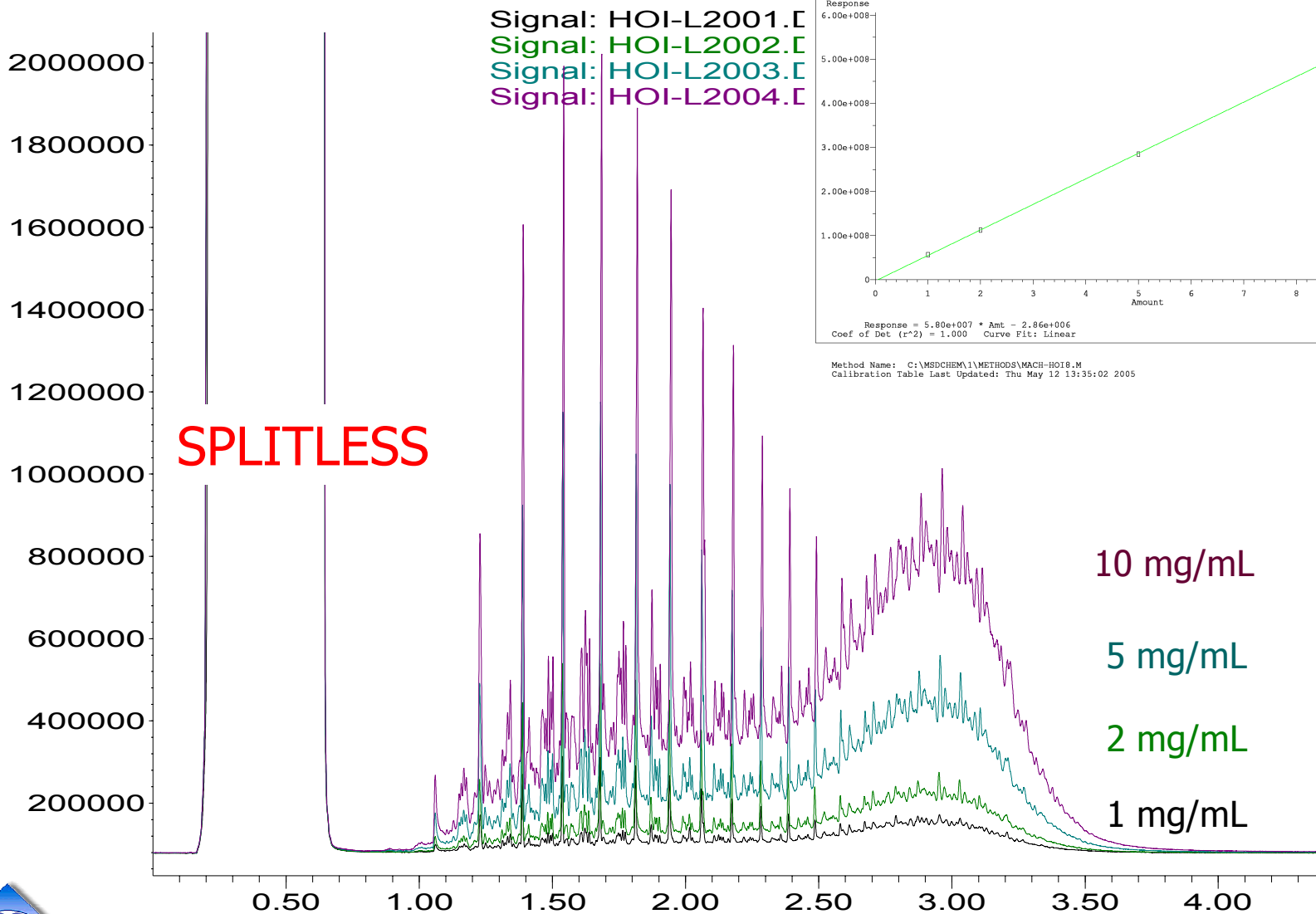


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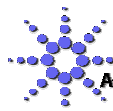
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Linearity

Response_



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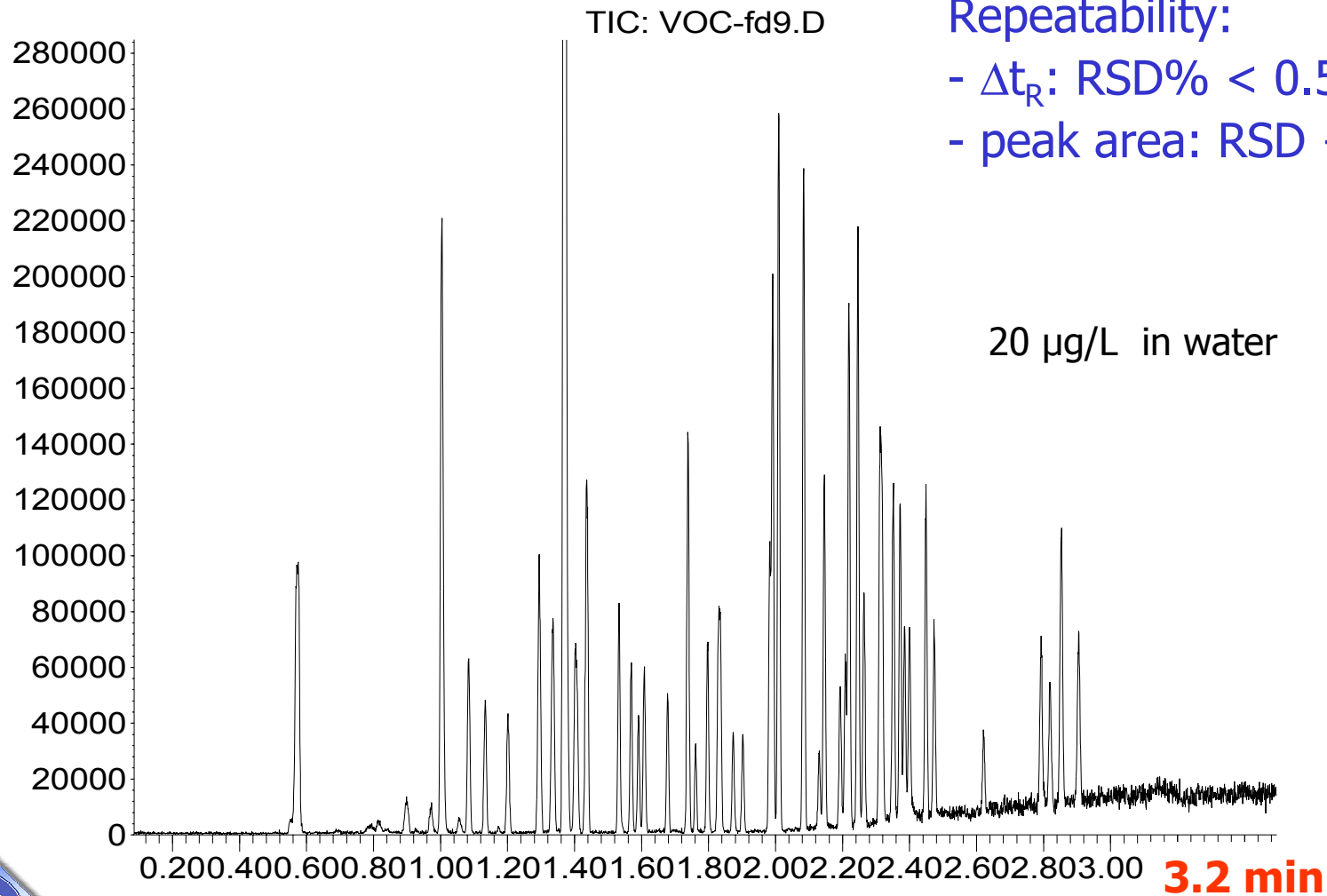
Ultra fast SHS-GC-MSD of VOCs in water

- SHS:
 - 10 mL water sample in 20 mL vial
 - 80°C – 10min equilibration (MPS2 sample prep-ahead !!)
 - 1 mL injection, Split 1:20
- 20 m x 0.18 mm id x 1 µm DB-624
- Helium: 279 kPa (70 cm/s, 2.5 mL/min@40°C)
- 40°C – 30 s – 100°C/min – 260°C – 30 s
- MSD (5973 Inert): fast scan, m/z 40-350 (19.5 scans/sec)



Ultra fast SHS-GC-MSD: EPA Volatiles

Abundance



Repeatability:

- Δt_R : RSD% < 0.5%
- peak area: RSD < 7%



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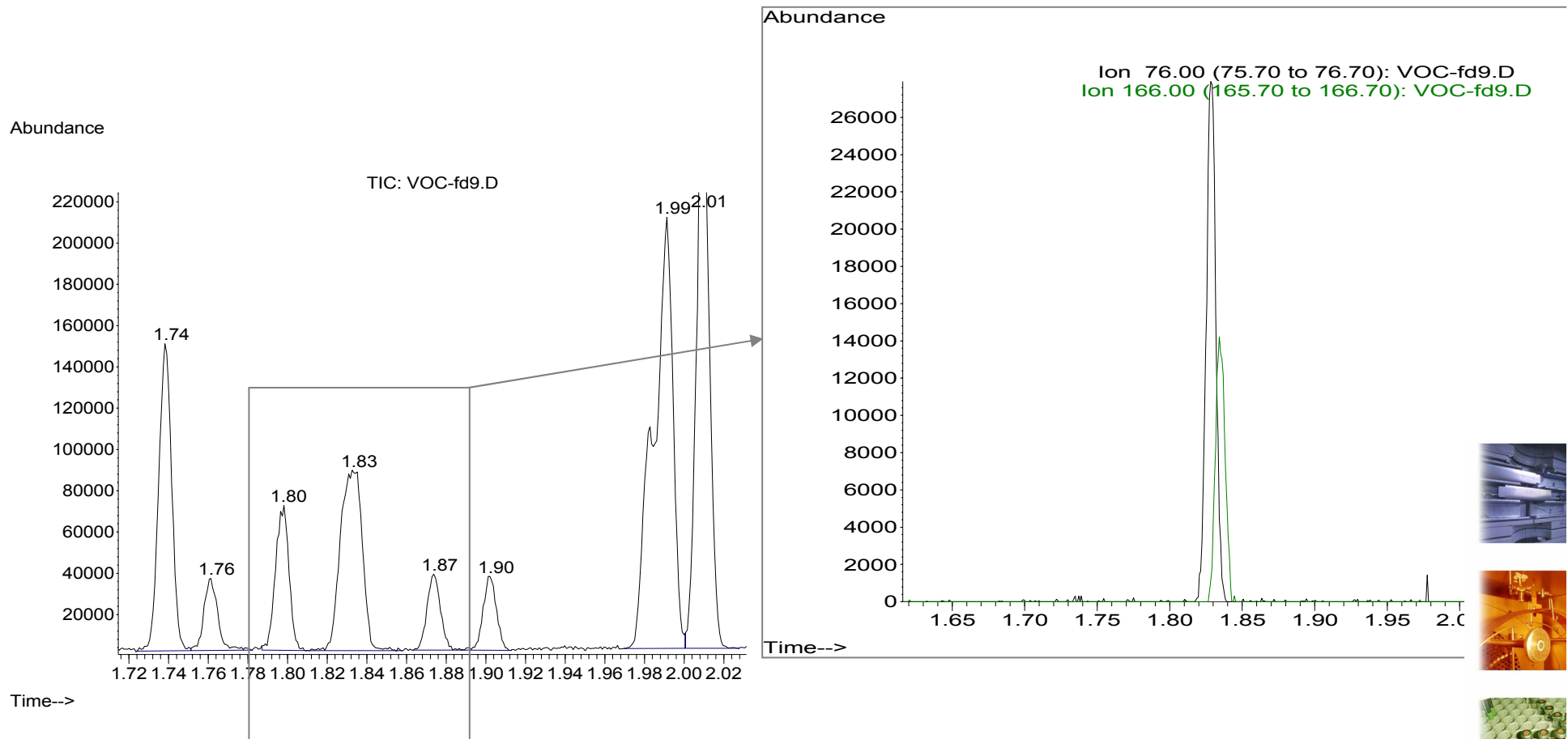


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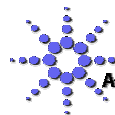
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'Deconvolution' of critical pairs



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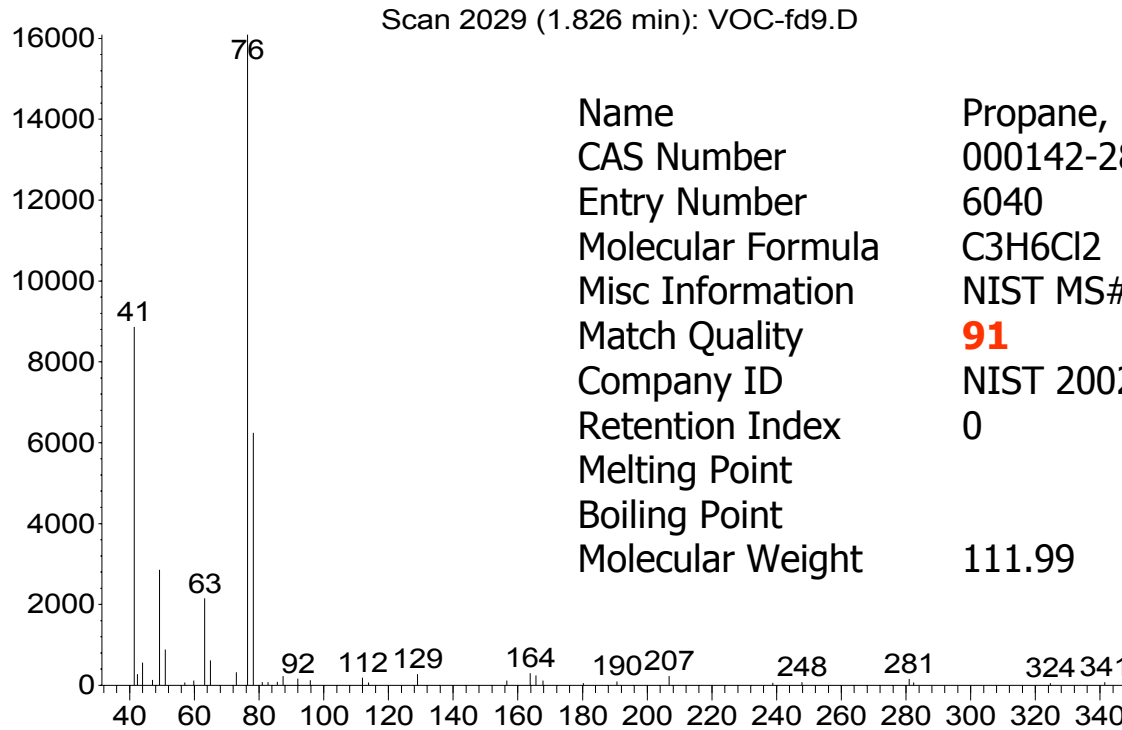


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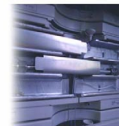
Identification

Abundance

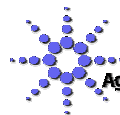


Name	Propane, 1,3-dichloro-
CAS Number	000142-28-9
Entry Number	6040
Molecular Formula	C3H6Cl2
Misc Information	NIST MS# 230388, Seq# M34731
Match Quality	91
Company ID	NIST 2002
Retention Index	0
Melting Point	
Boiling Point	
Molecular Weight	111.99

m/z-->



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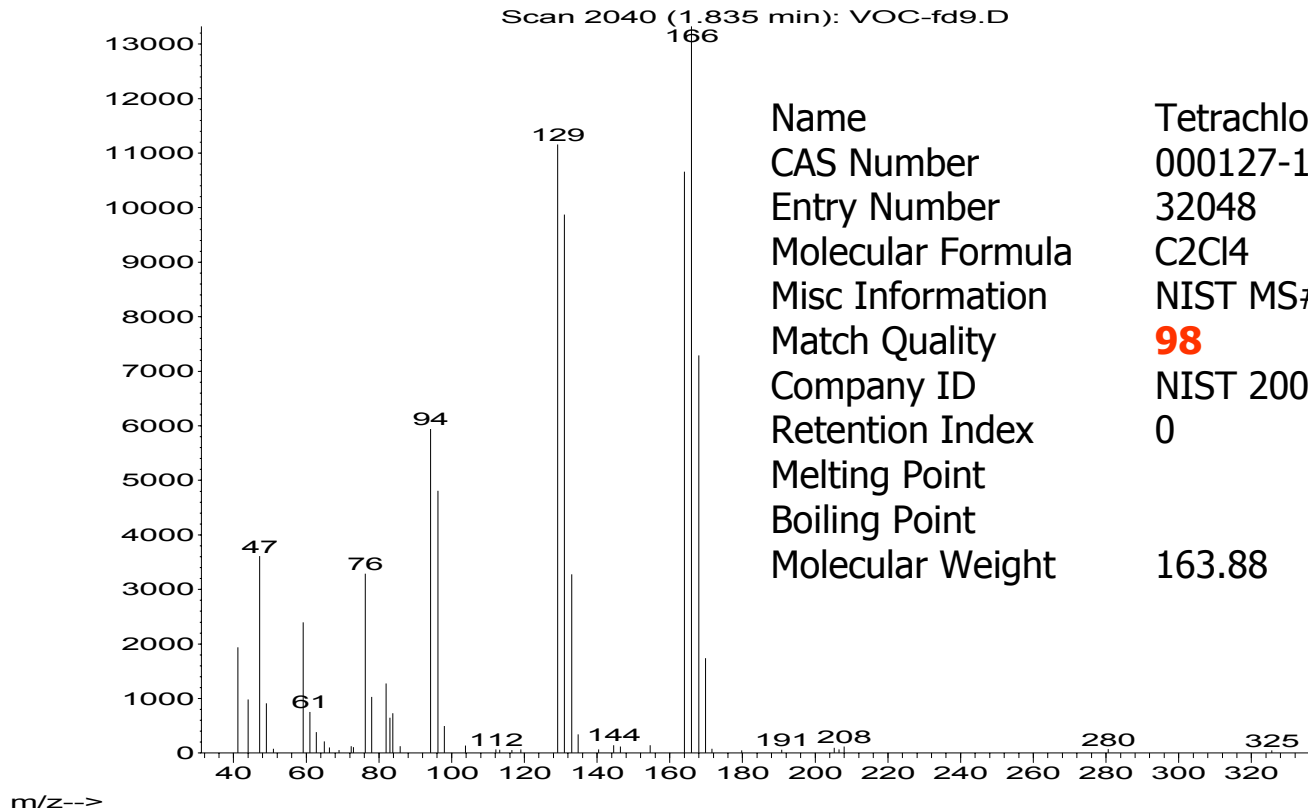


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Identification

Abundance



Name	Tetrachloroethylene
CAS Number	000127-18-4
Entry Number	32048
Molecular Formula	C2Cl4
Misc Information	NIST MS# 107150, Seq# R21538
Match Quality	98
Company ID	NIST 2002
Retention Index	0
Melting Point	
Boiling Point	
Molecular Weight	163.88



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Polyaromatic Hydrocarbons

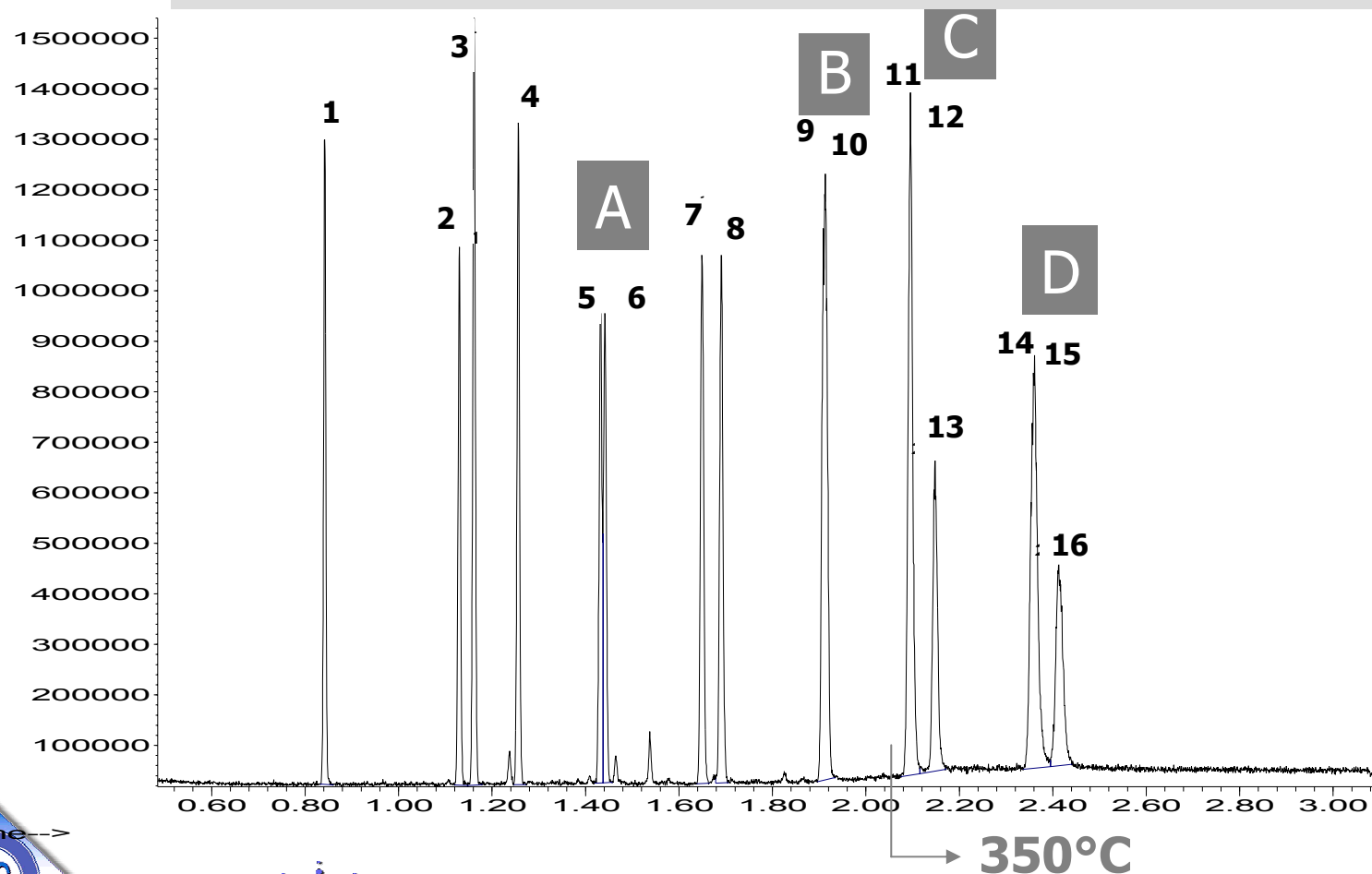
5 m x **0.180 mm id** x 0.18 μm DB5-MS

Helium: 45.5 kPa, 1.4 mL/min @ 50°C

50°C (3 sec)-**150°C/min**-350°C (60 sec)

MSD: **fast scan**, m/z 100-300 (**25 Scans/sec**)

Abundance



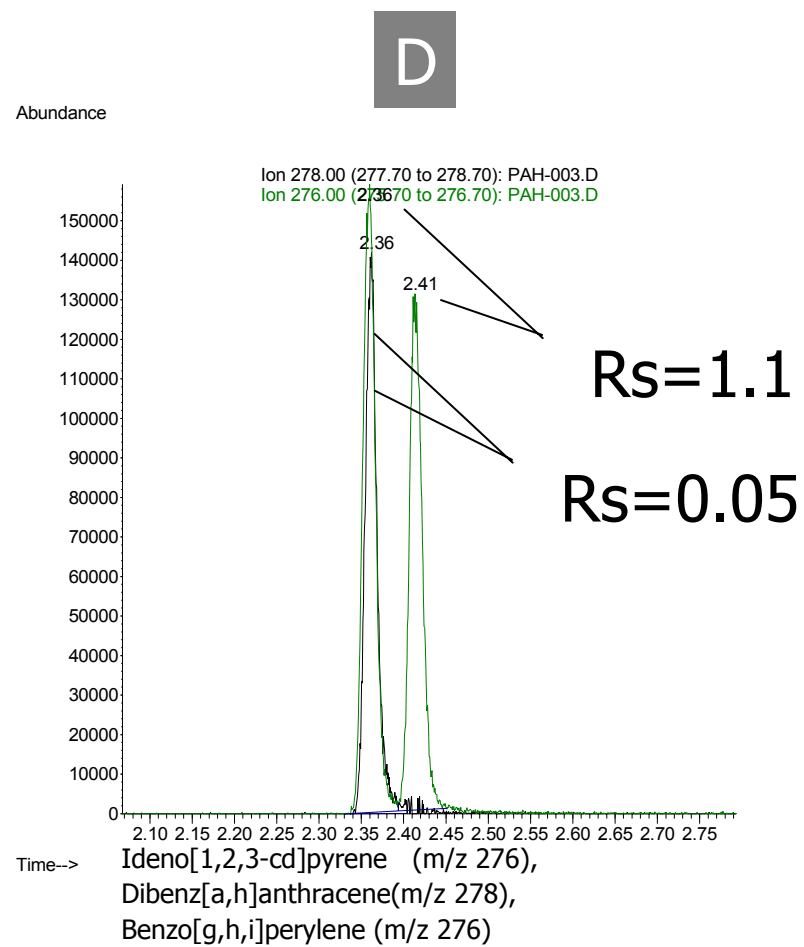
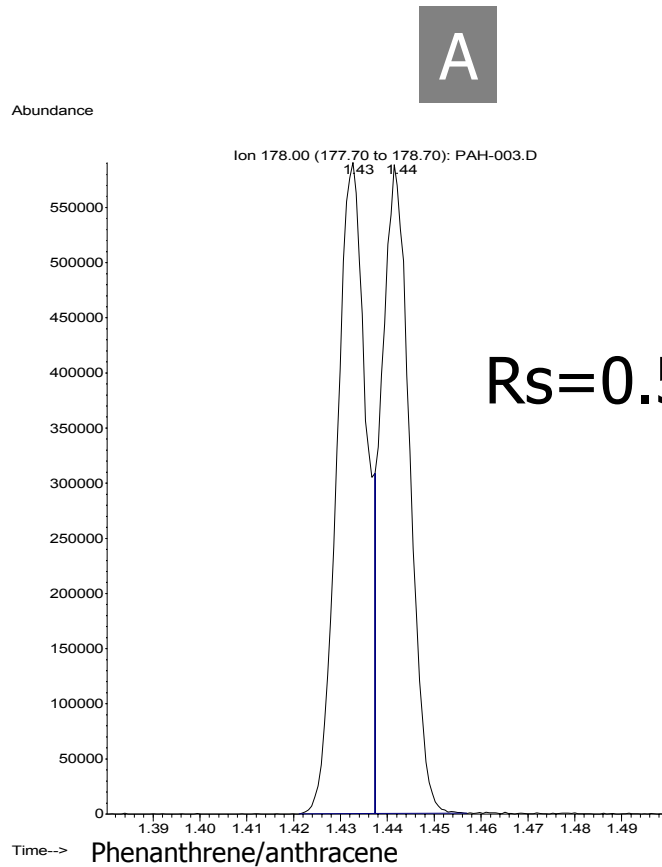
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1. naphthalene, 2. acenaphthylene, 3. acenaphthene, 4. fluorene, 5. phenanthrene, 6. anthracene, 7. fluoranthene, 8. pyrene, 9. benzo(a)anthracene, 10. chrysene, 11. benzo(b)fluoranthene, 12. benzo(k)fluoranthene, 13. benzo(a)pyrene, 14. indeno(1,2,3-cd-pyrene), 15. dibenzo(a,h)anthracene, 16. benzo(g,h,i)perylene



Critical pairs



→ Narrow bore column / slower t° programming/ optimal u...
at the cost of time !!



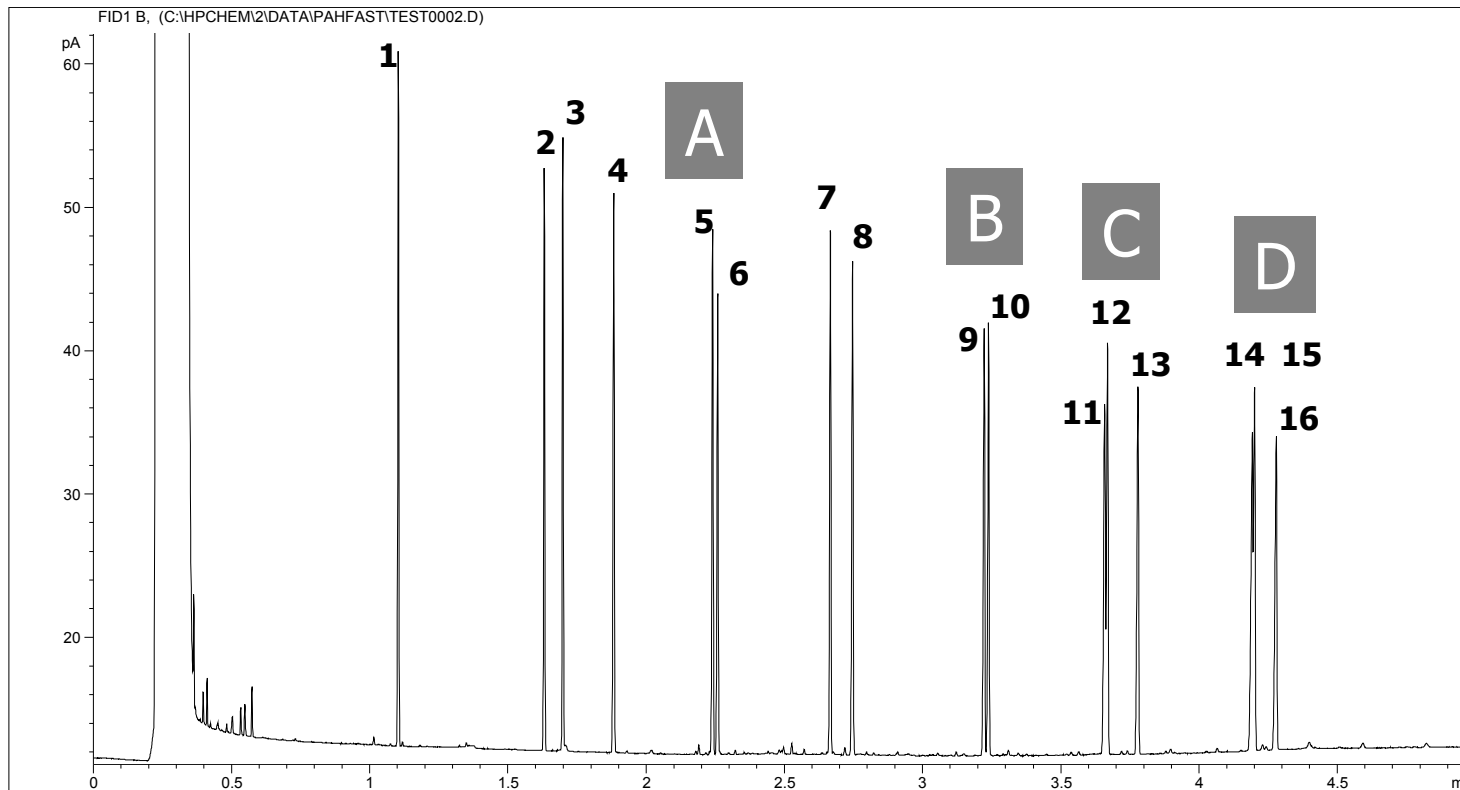
Polyaromatic Hydrocarbons

10 m x **0.100 mm id** x 0.1 μm HP5

Hydrogen: 437.9 kPa, 2 mL/min @ 50°C

50°C (0)-**75°C/min**-325°C (120 sec)

FID (100 Hz)

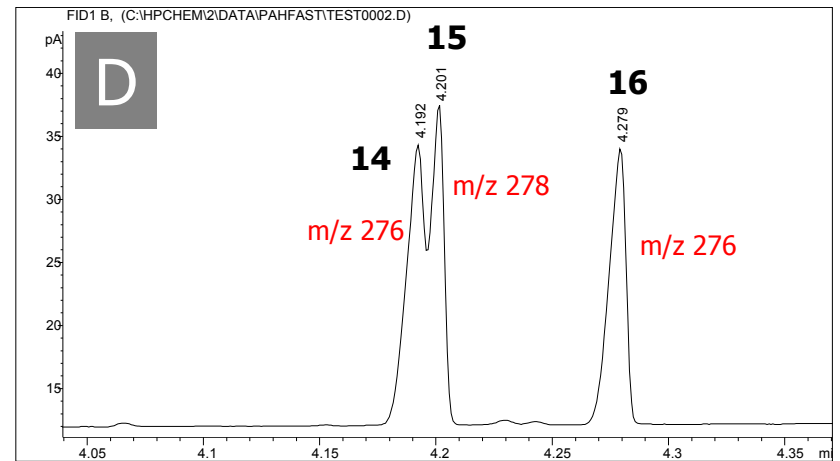
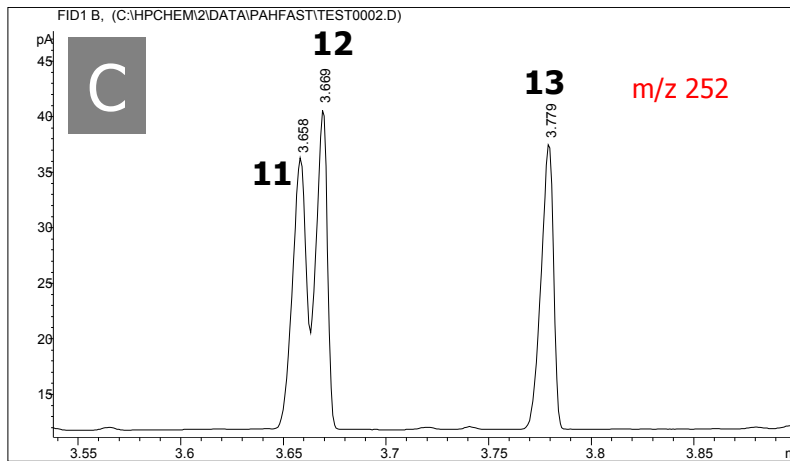
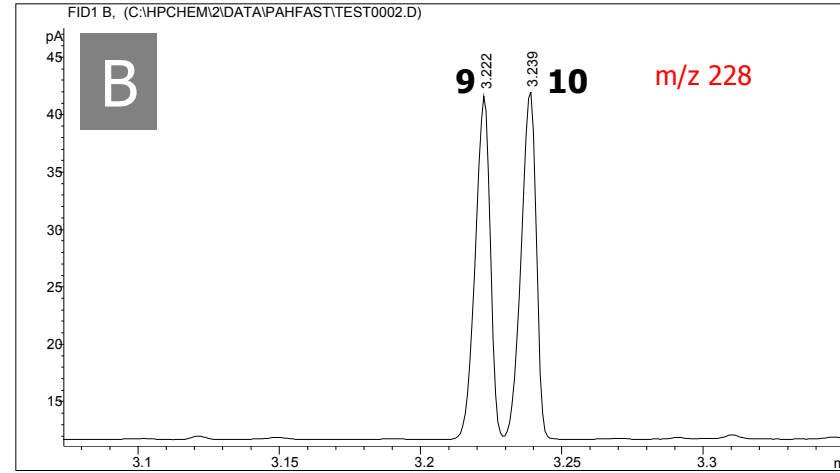
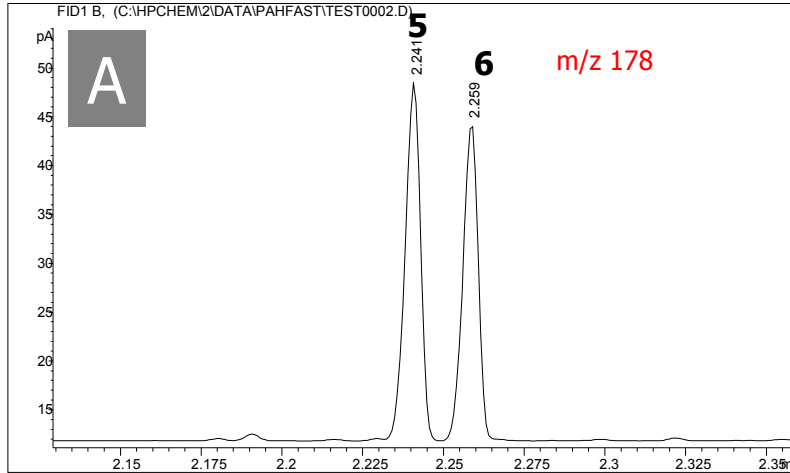


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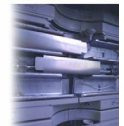


1. naphthalene, 2. acenaphthylene, 3. acenaphthene, 4. fluorene, 5. phenanthrene, 6. anthracene, 7. fluoranthene, 8. pyrene, 9. benzo(a)anthracene, 10. chrysene, 11. benzo(b)fluoranthene, 12. benzo(k)fluoranthene, 13. benzo(a)pyrene, 14. indeno(1,2,3-cd)pyrene, 15. dibenzo(a,h)anthracene, 16. benzo(g,h,i)perylene

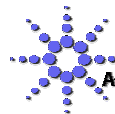
Critical pairs



1. naphthalene, 2. acenaphthylene, 3. acenaphthene, 4. fluorene, **5. phenanthrene**, **6. anthracene**, 7. fluoranthene, 8. pyrene, **9. benzo(a)anthracene**, **10. chrysene**, **11. benzo(b)fluoranthene**, **12. benzo(k)fluoranthene**, 13. benzo(a)pyrene, **14. indeno(1,2,3-cd)pyrene**, **15. dibenzo(a,h)anthracene**, 16. benzo(g,h,i)perylene



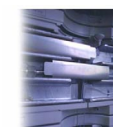
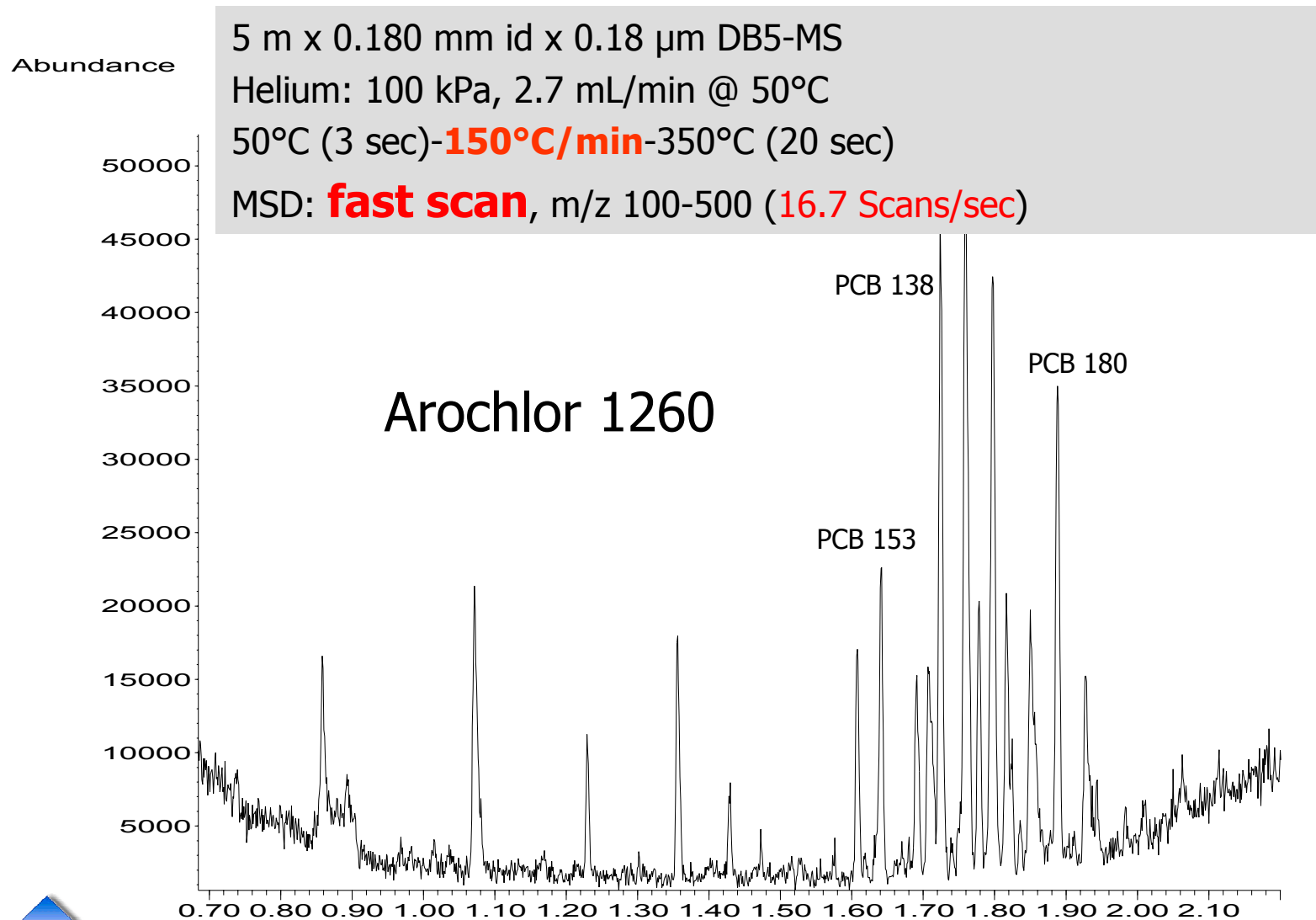
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Polychlorinated Biphenyls



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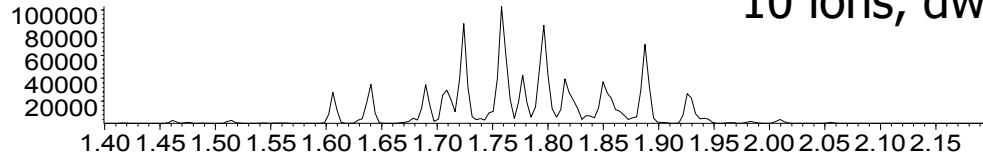
5 m x 0.180 mm id x 0.18 μm DB5-MS

Helium: 100 kPa, 2.7 mL/min @ 50°C

50°C (3 sec)-**150°C/min**-350°C (20 sec)

MSD: **SIM**, m/z 256, 258, 290, 292, 324, 326, 360, 362, 394, 396

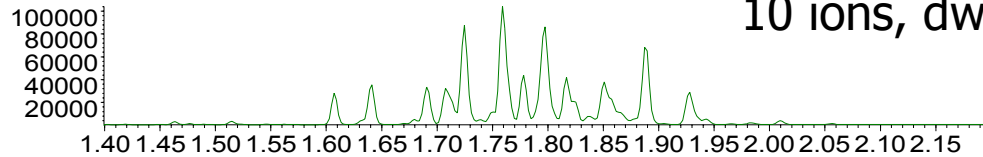
Abundance



10 ions, dwell 20 msec = **4.0** cycles/sec

Time-->
Abundance

TIC: PCB-6.D

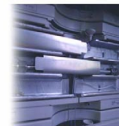
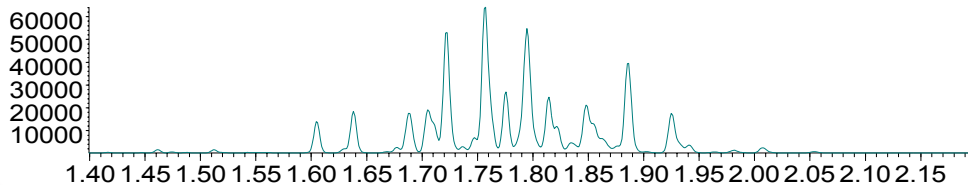


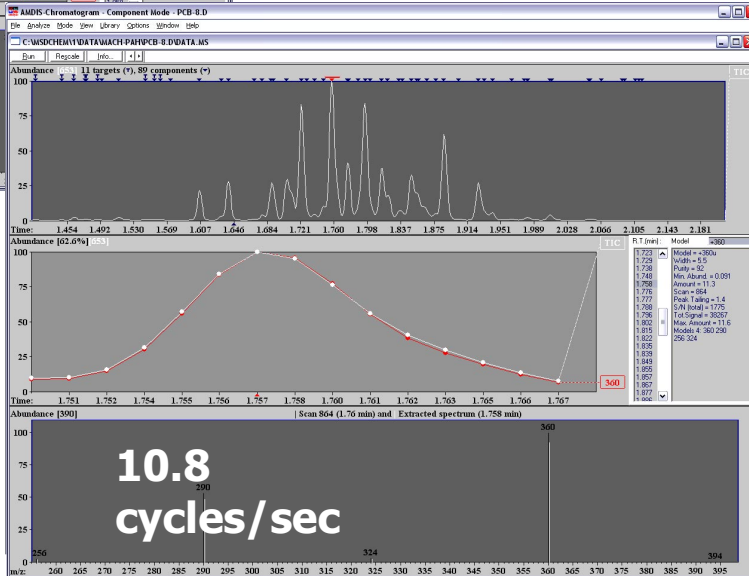
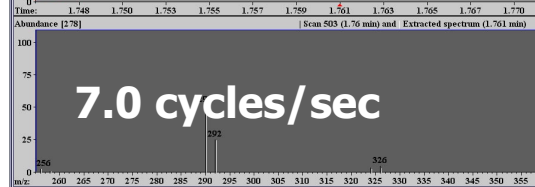
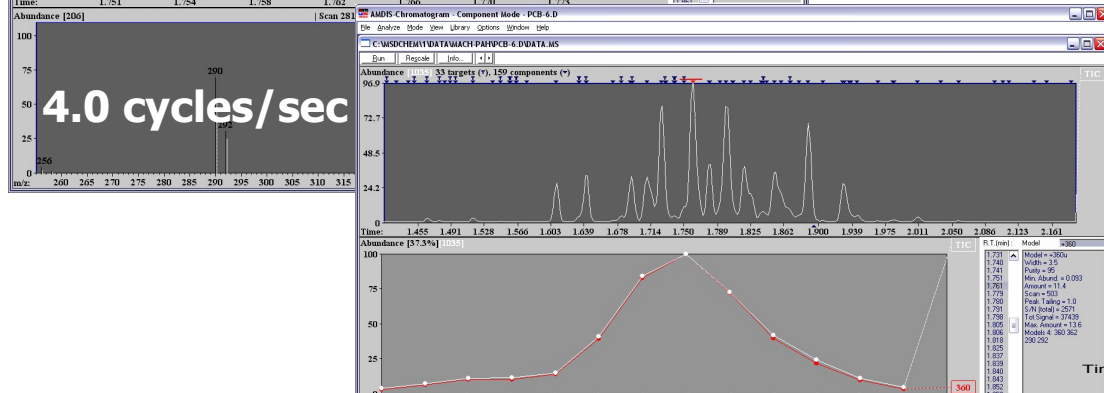
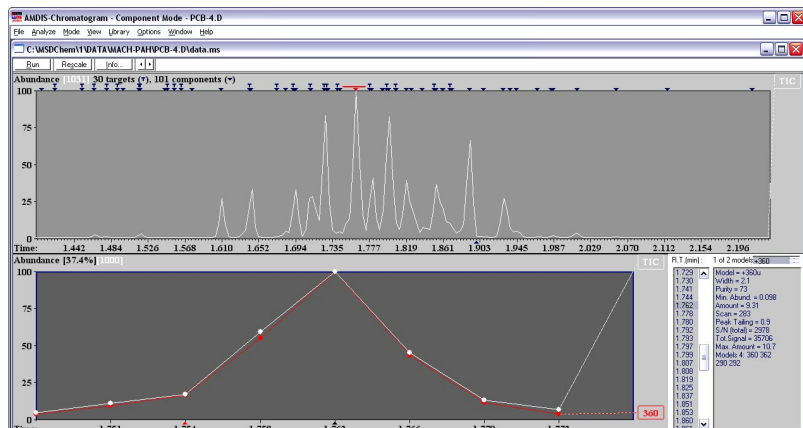
10 ions, dwell 10 msec = **7.0** cycles/sec

Time-->
Abundance

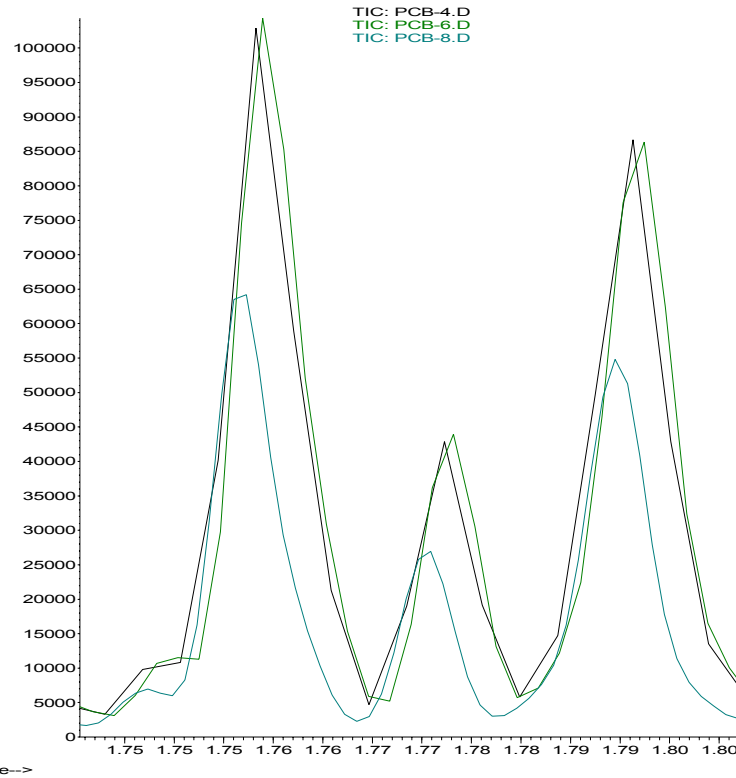
TIC: PCB-8.D

5 ions, dwell 10 msec = **10.8** cycles/sec





Abundance



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Conclusions

- Hardware: *MACHTM*
- Speed versus resolution?
 - Increase speed of analysis with or without loss of resolution ?
- Chromatographic resolution or mass spectroscopic resolution ?
- Required sample capacity?

