

# High Throughput GC and 2D-GC solutions using LTM Technology in combination with the Agilent 5975 MSD and 7000 Triple Quadrupole MS.

Frank David



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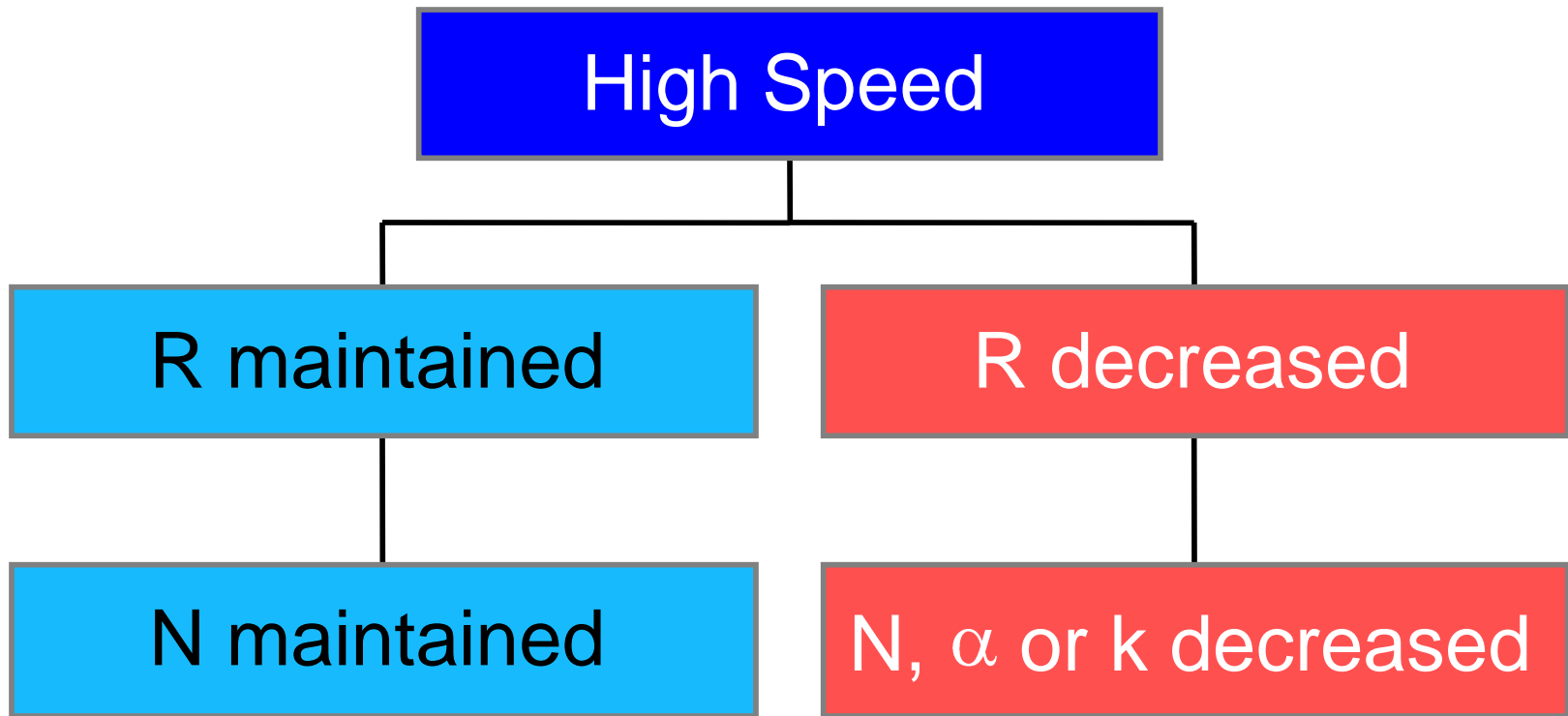
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# Agilent Low Thermal Mass (LTM) module

- Fast GC analysis  
(1200 C/min)
- Independent column temperature control
- of up to 4 different columns



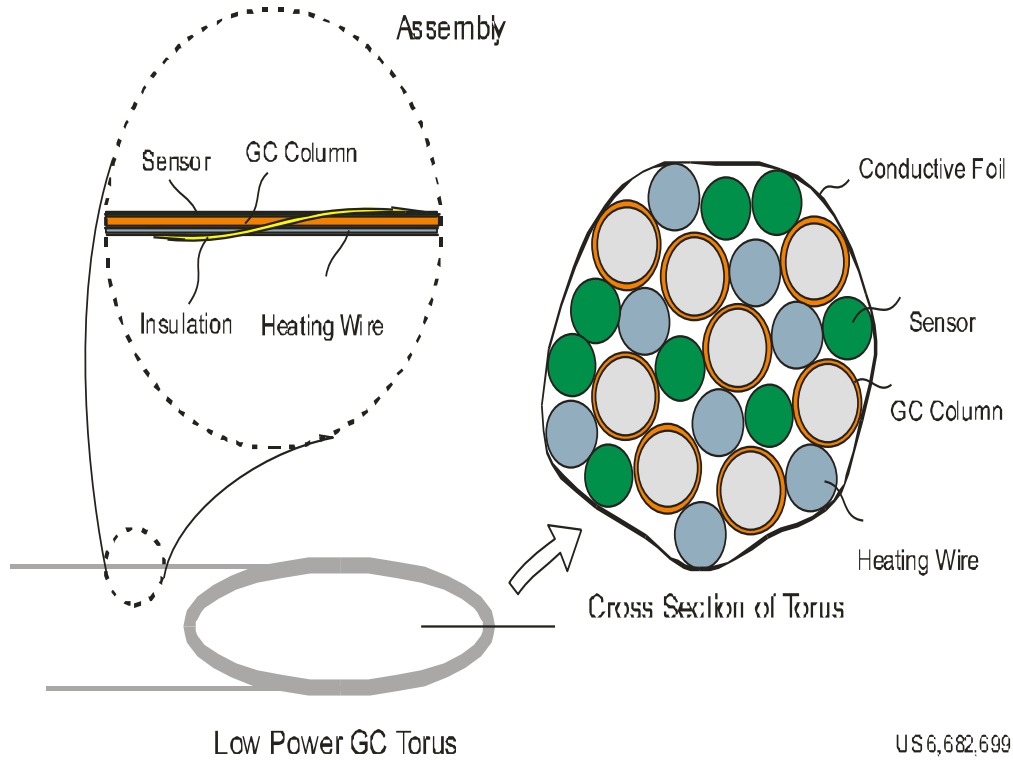
# High Speed CGC



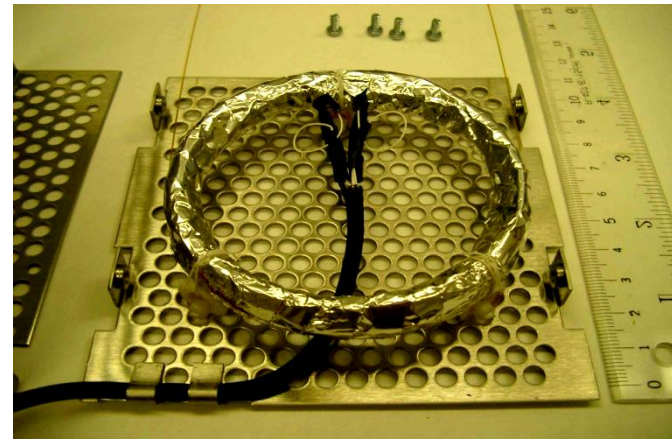
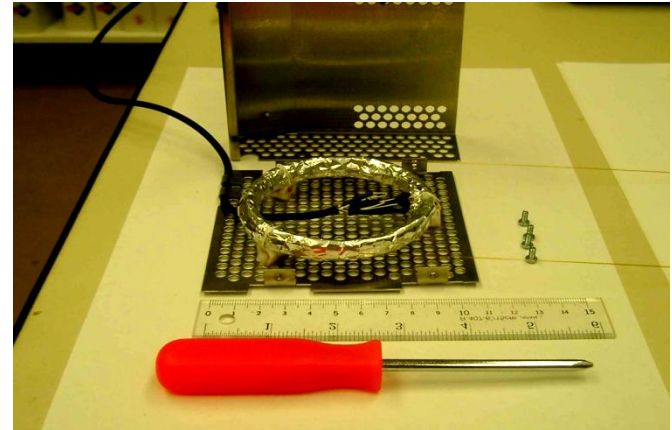
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# Low Thermal Mass GC Technology



US 6,682,699



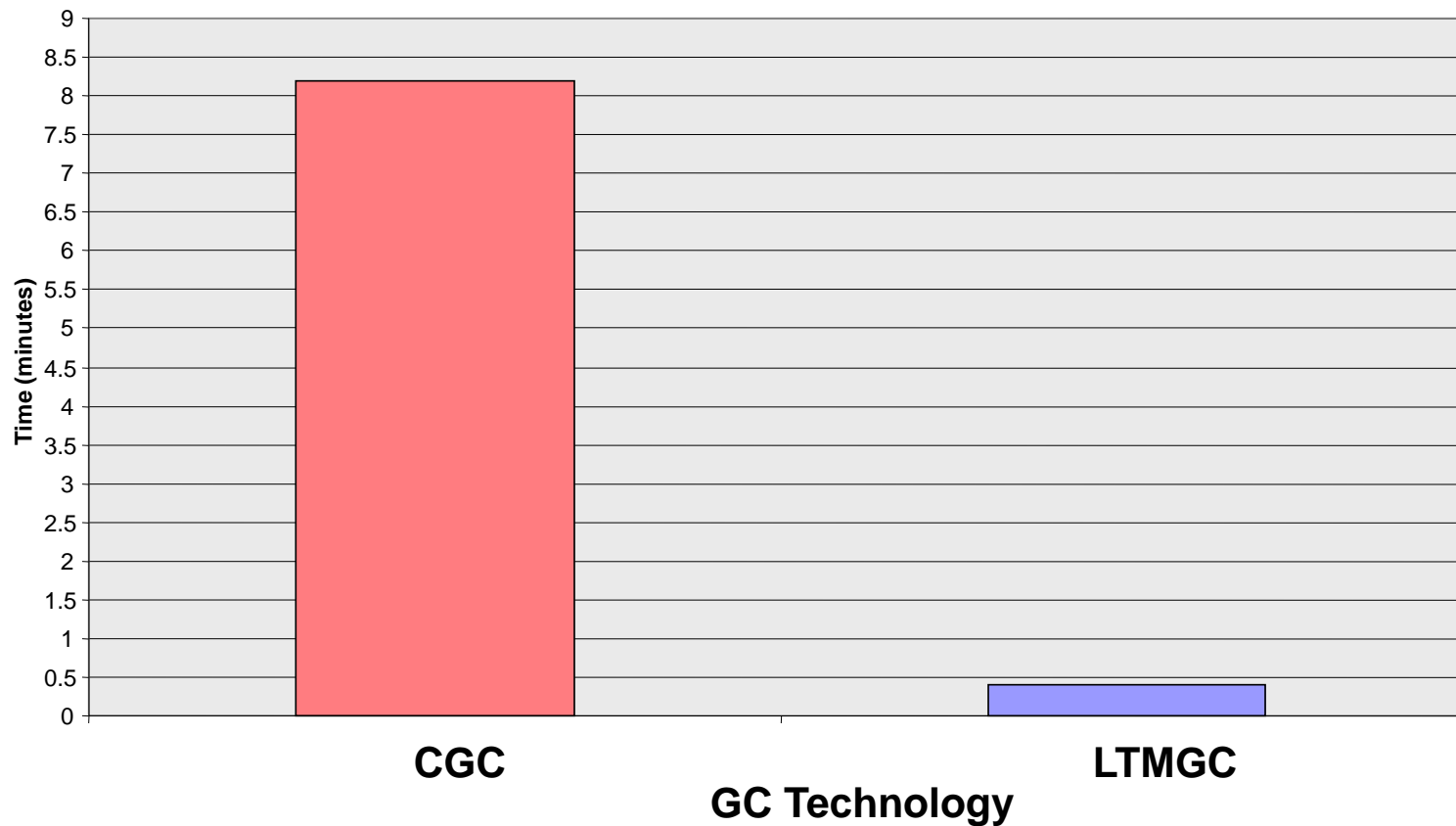
Heating rates: up to 1000 C/min !!



# Cool Down Time Comparison

Comparison of Cool Down Time LTMGC vs. Agilent HP-6890A

LTMGC: 5 meter, 0.1 mm id, 0.12 micron DB-1

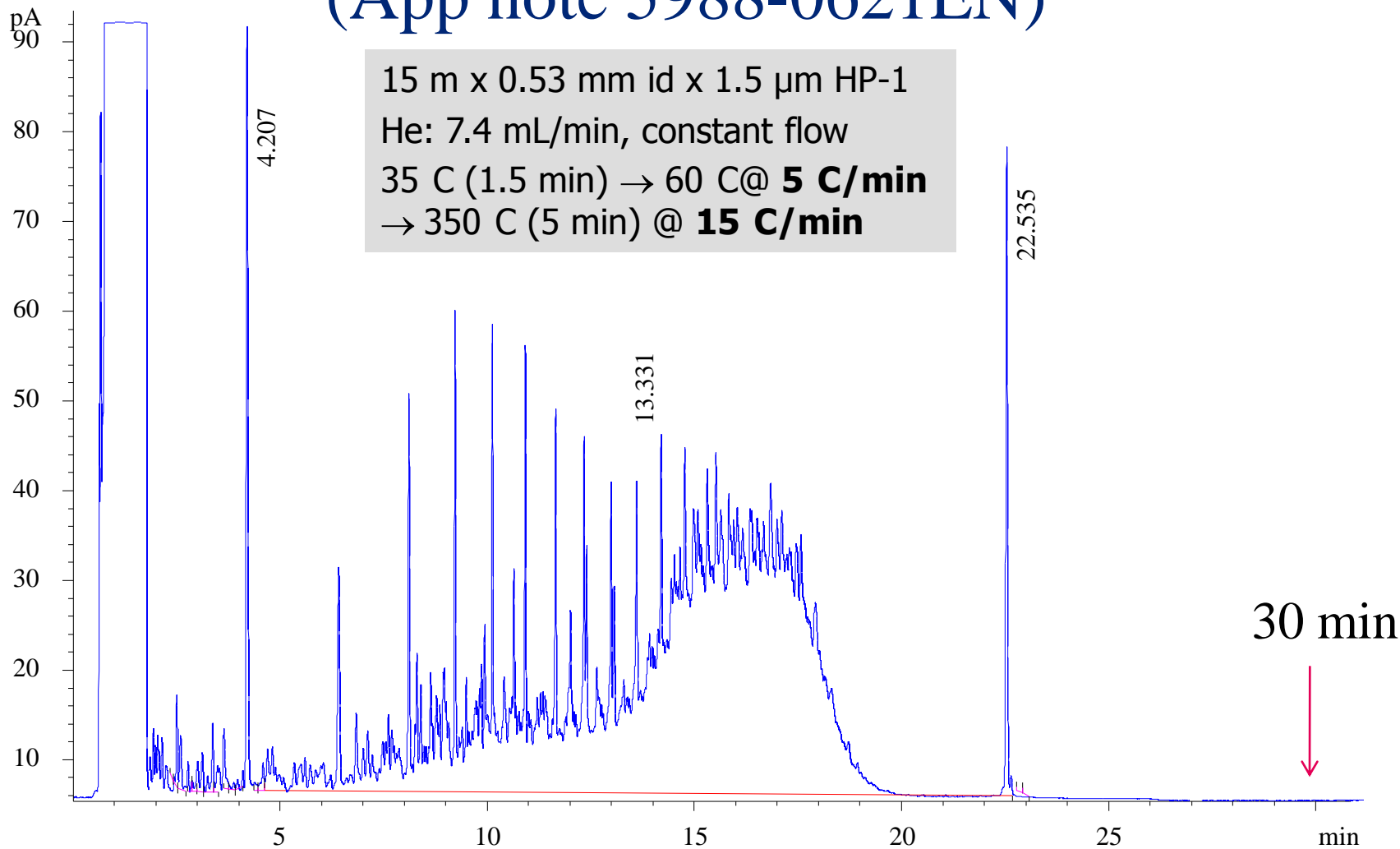


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# Typical Hydrocarbon Index Analysis by GC (App note 5988-0621EN)

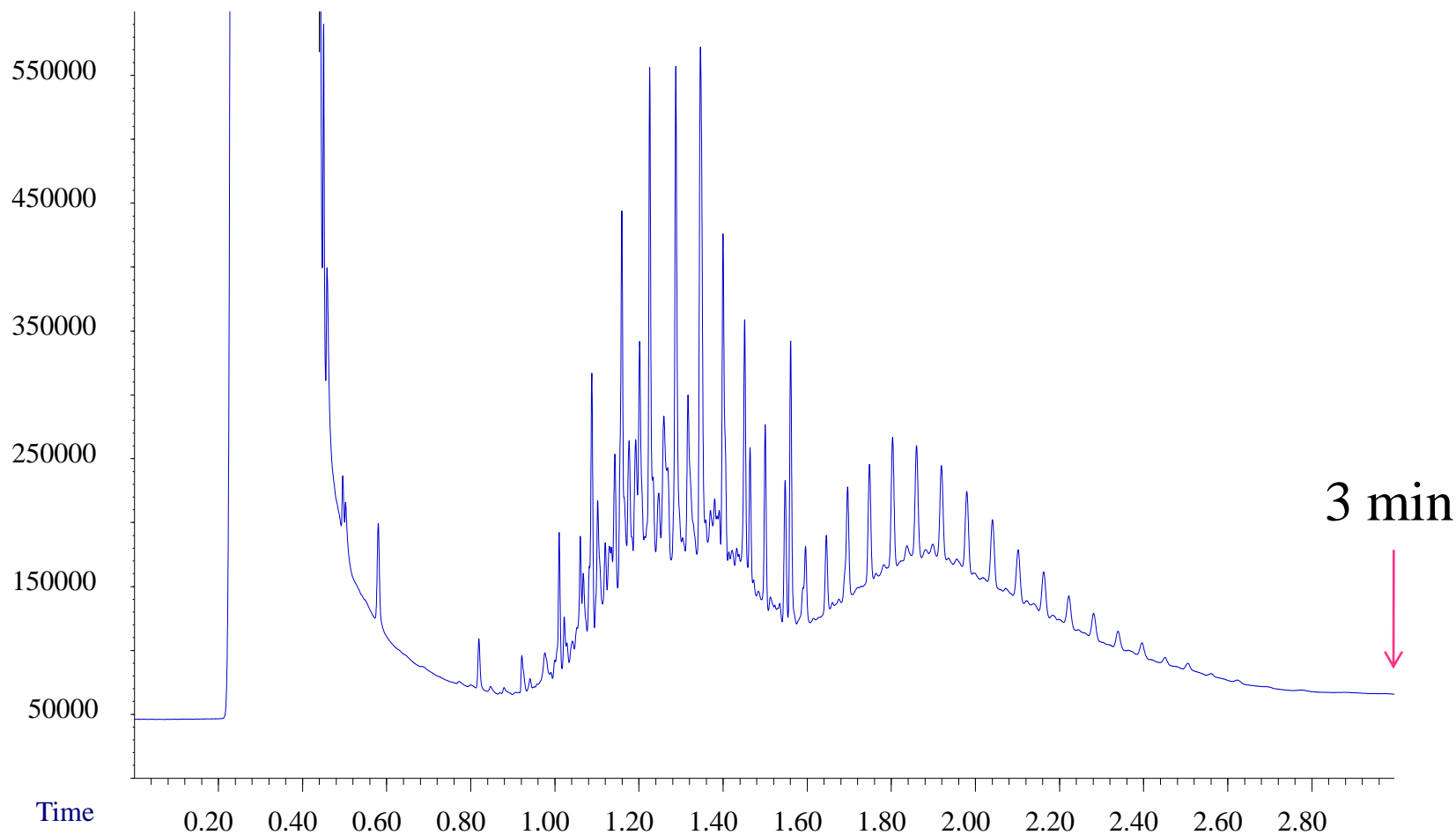


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# Mineral Oil Calibration 100 mg/L

Response



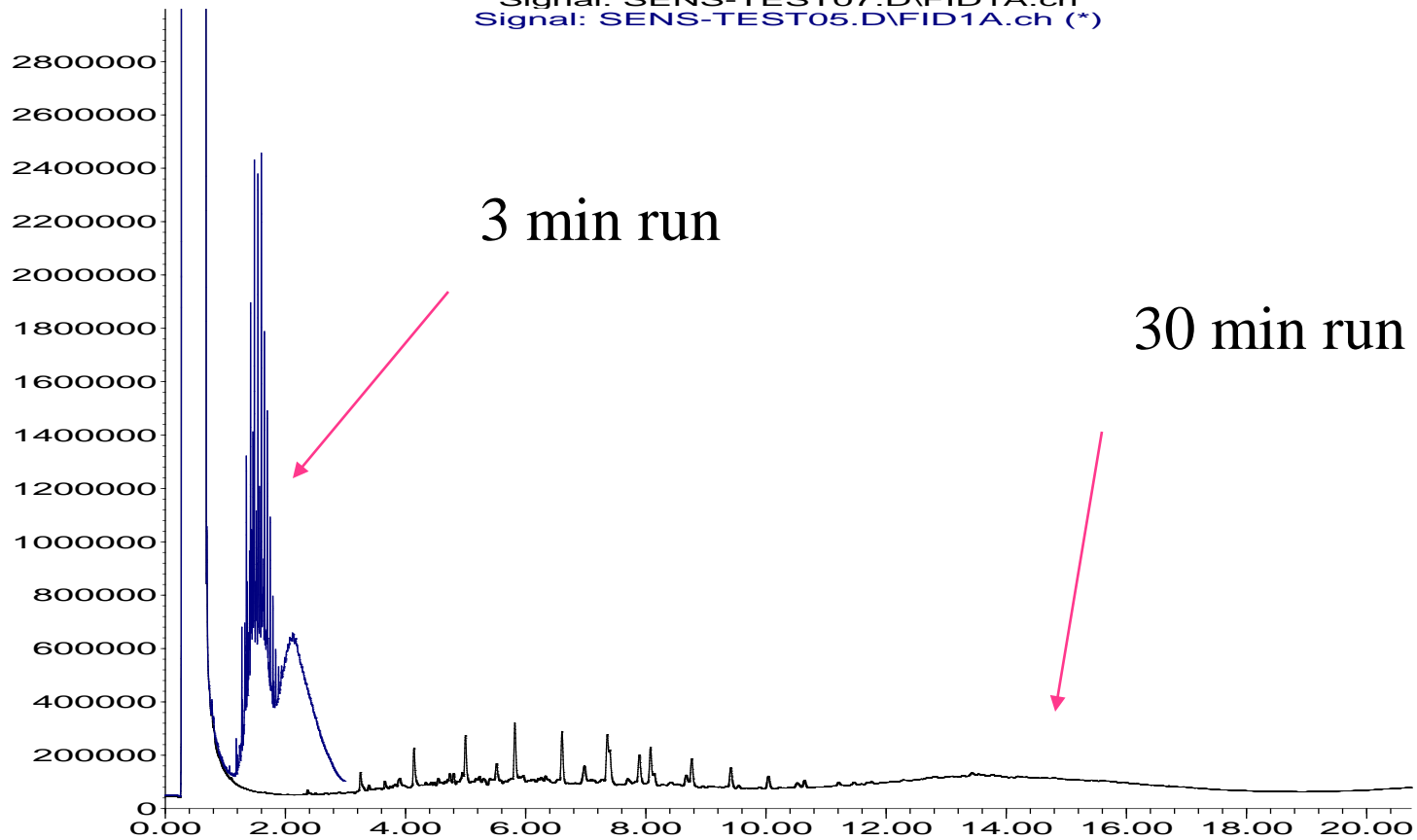
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# Extra: sensitivity gain

Response\_

Signal: SENS-TEST07.D\FID1A.ch  
Signal: SENS-TEST05.D\FID1A.ch (\*)



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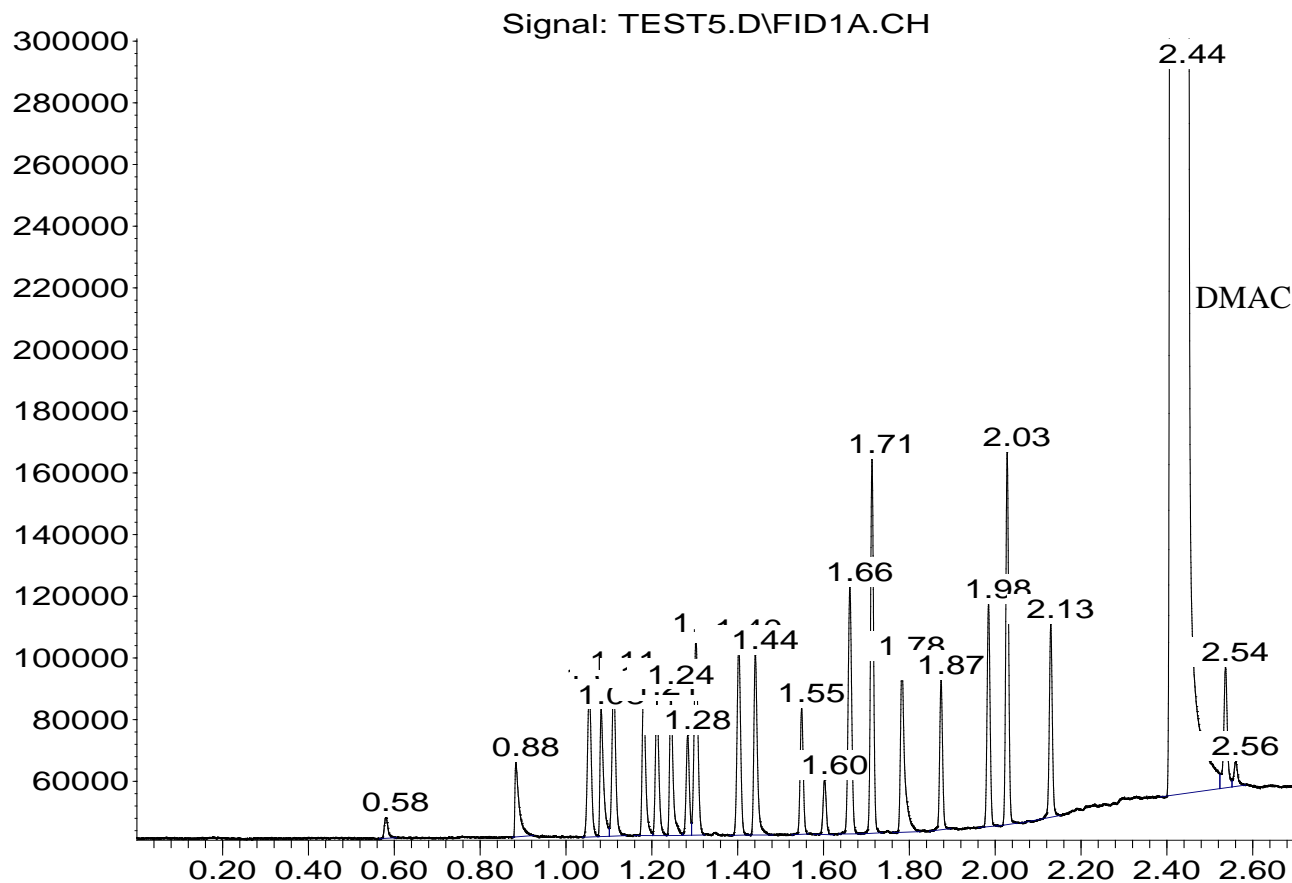
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# Residual solvent analysis

## *LTM* method (2.6 min)

Response\_



Time

**2.6 min**



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# Repeatability: Retention Times and Area RSD

Compound	tR (min)		Area
	Mean	s	RSD (%)
Methanol	0,888	0,0005	2,85
Pentane	1,054	0,0005	0,77
Ethanol	1,085	0,0000	3,90
Ether	1,110	0,0000	1,25
Aceton	1,180	0,0004	2,28
2-Propanol	1,213	0,0004	4,93
Acetonitrile	1,246	0,0004	3,30
Dichloormethane	1,284	0,0005	3,50
t-Butanol	1,303	0,0000	5,18
Hexane	1,402	0,0005	1,47
Propanol	1,444	0,0004	4,68
Ethyl acetate	1,549	0,0000	2,93
Chloroform	1,603	0,0004	3,81
Cyclohexane	1,661	0,0005	1,71
Benzene	1,713	0,0000	2,73
Butanol	1,785	0,0007	5,41
1,4-Dioxane	1,874	0,0005	7,70
4-Methyl-2-pentanone	1,985	0,0004	8,21
Toluene	2,028	0,0000	3,10
Butyl acetate	2,130	0,0004	7,03



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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  $\mu$ m 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: **fast scan**, m/z 40-350 (**19.5 scans/sec**)

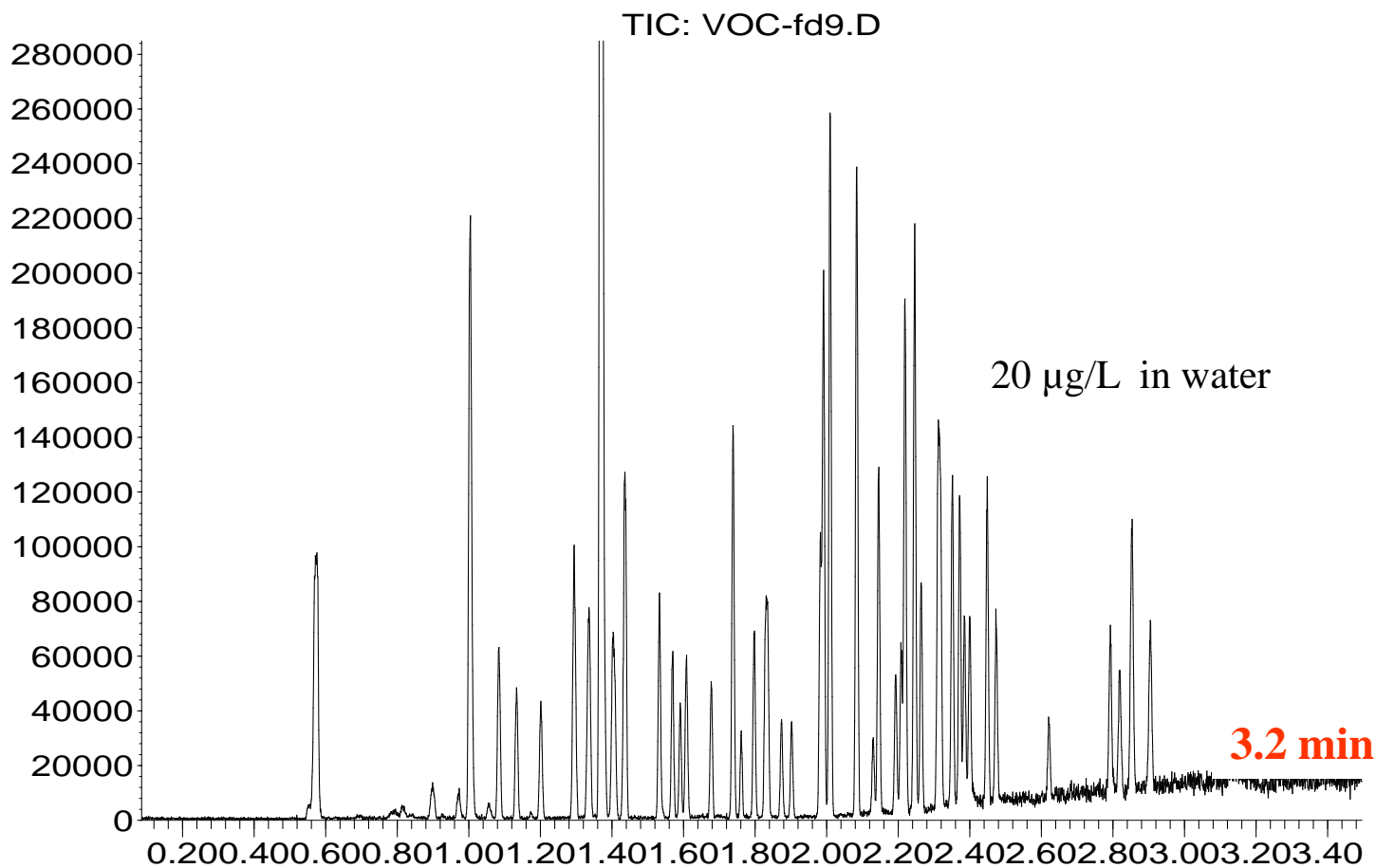


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# Ultra fast SHS-GC-MSD: EPA Volatiles

Abundance



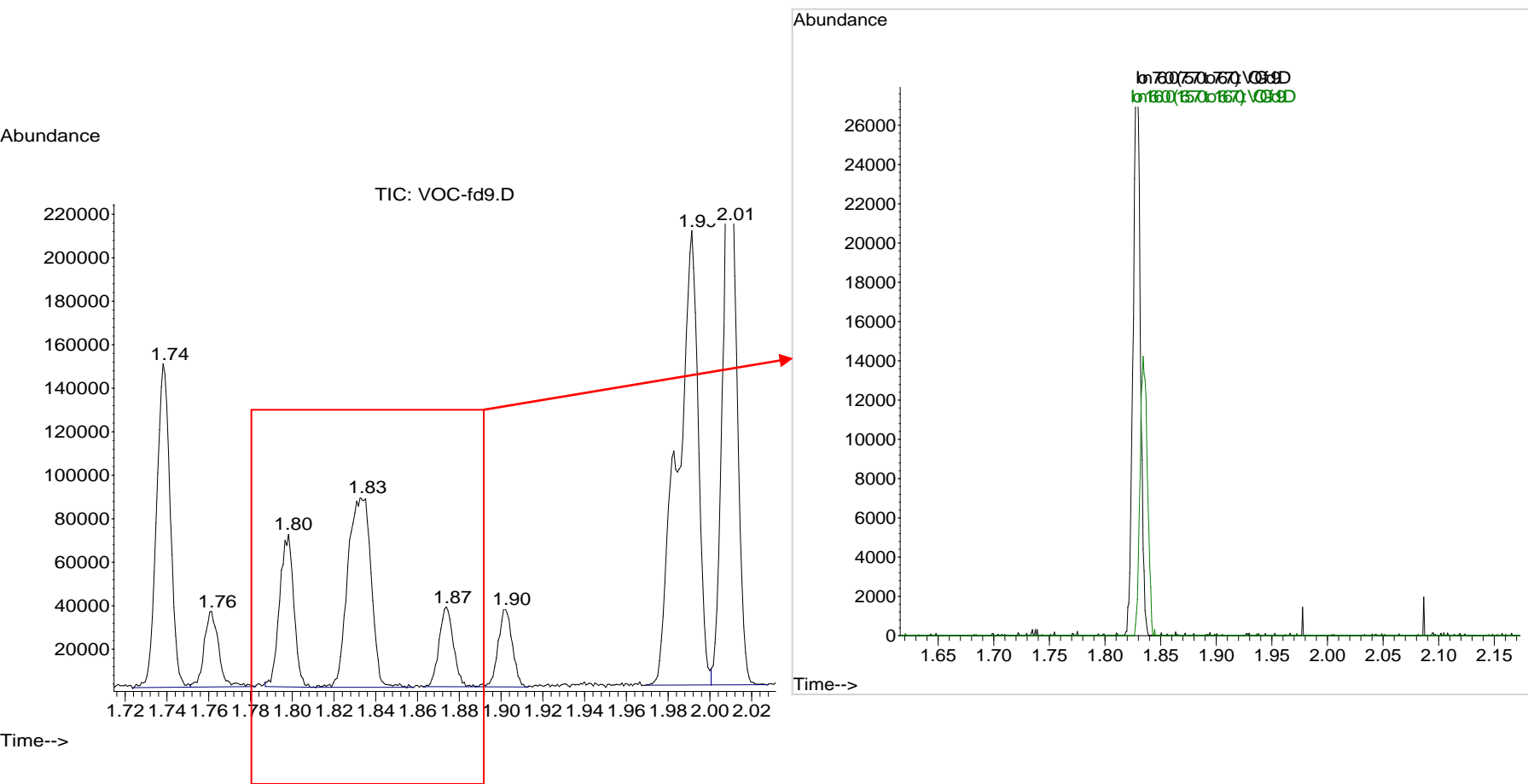
Time-->



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# 'Deconvolution' of critical pairs



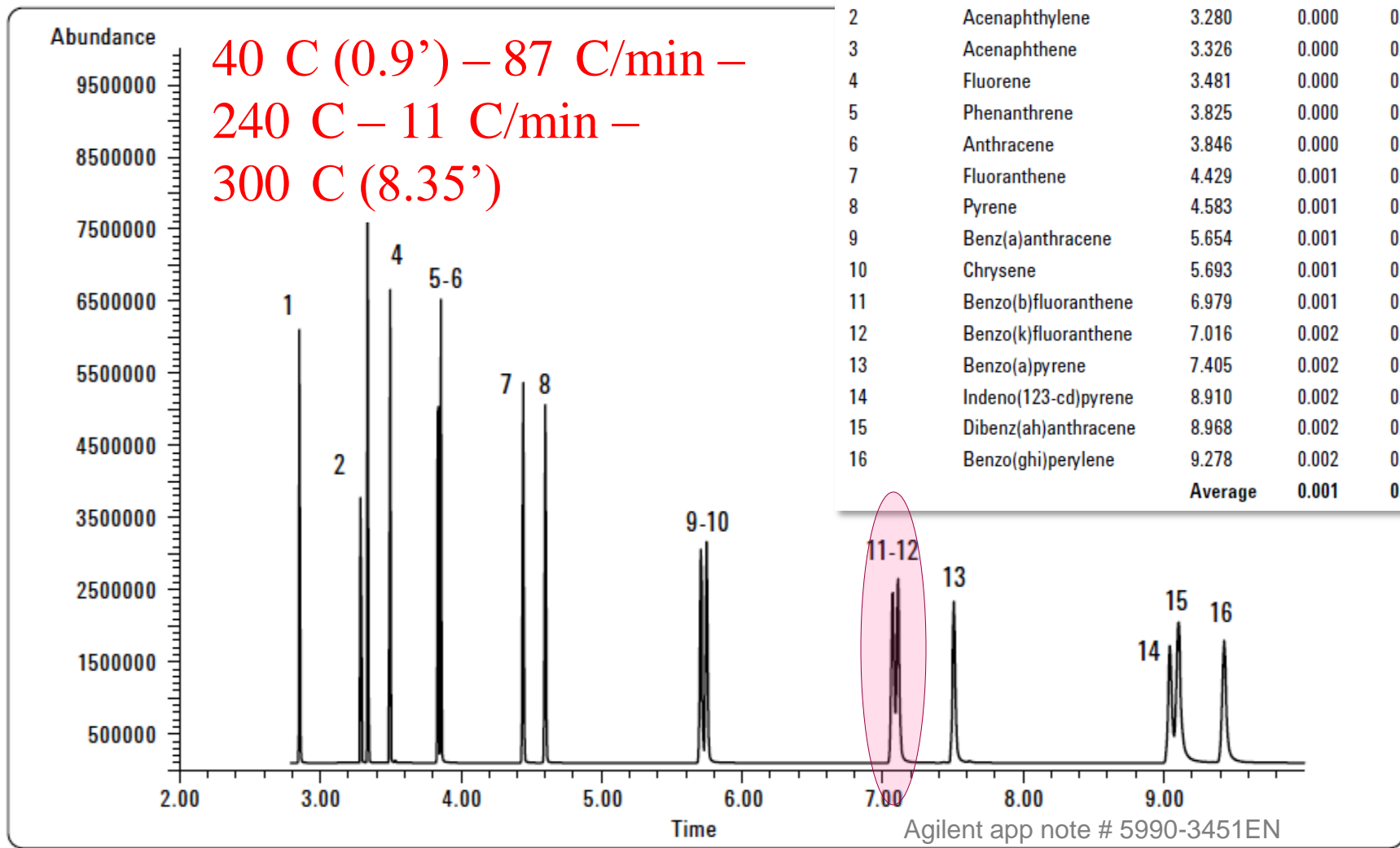
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# Fast LTM analysis of PAH

## 20 m x 180 $\mu$ m ID x 0.18 $\mu$ m DB-5MS

Order of elution	Compound name	Mean $t_R$ (min)	$\sigma$	% RSD
1	Naphthalene	2.850	0.000	0.00
2	Acenaphthylene	3.280	0.000	0.00
3	Acenaphthene	3.326	0.000	0.00
4	Fluorene	3.481	0.000	0.00
5	Phenanthrene	3.825	0.000	0.01
6	Anthracene	3.846	0.000	0.00
7	Fluoranthene	4.429	0.001	0.01
8	Pyrene	4.583	0.001	0.01
9	Benz(a)anthracene	5.654	0.001	0.02
10	Chrysene	5.693	0.001	0.01
11	Benzo(b)fluoranthene	6.979	0.001	0.02
12	Benzo(k)fluoranthene	7.016	0.002	0.02
13	Benzo(a)pyrene	7.405	0.002	0.02
14	Indeno(123-cd)pyrene	8.910	0.002	0.02
15	Dibenz(ah)anthracene	8.968	0.002	0.02
16	Benzo(ghi)perylene	9.278	0.002	0.02
	<b>Average</b>	<b>0.001</b>	<b>0.01</b>	<b>0.01</b>





# Soil extract - GC-MS in scan/SIM – scan trace

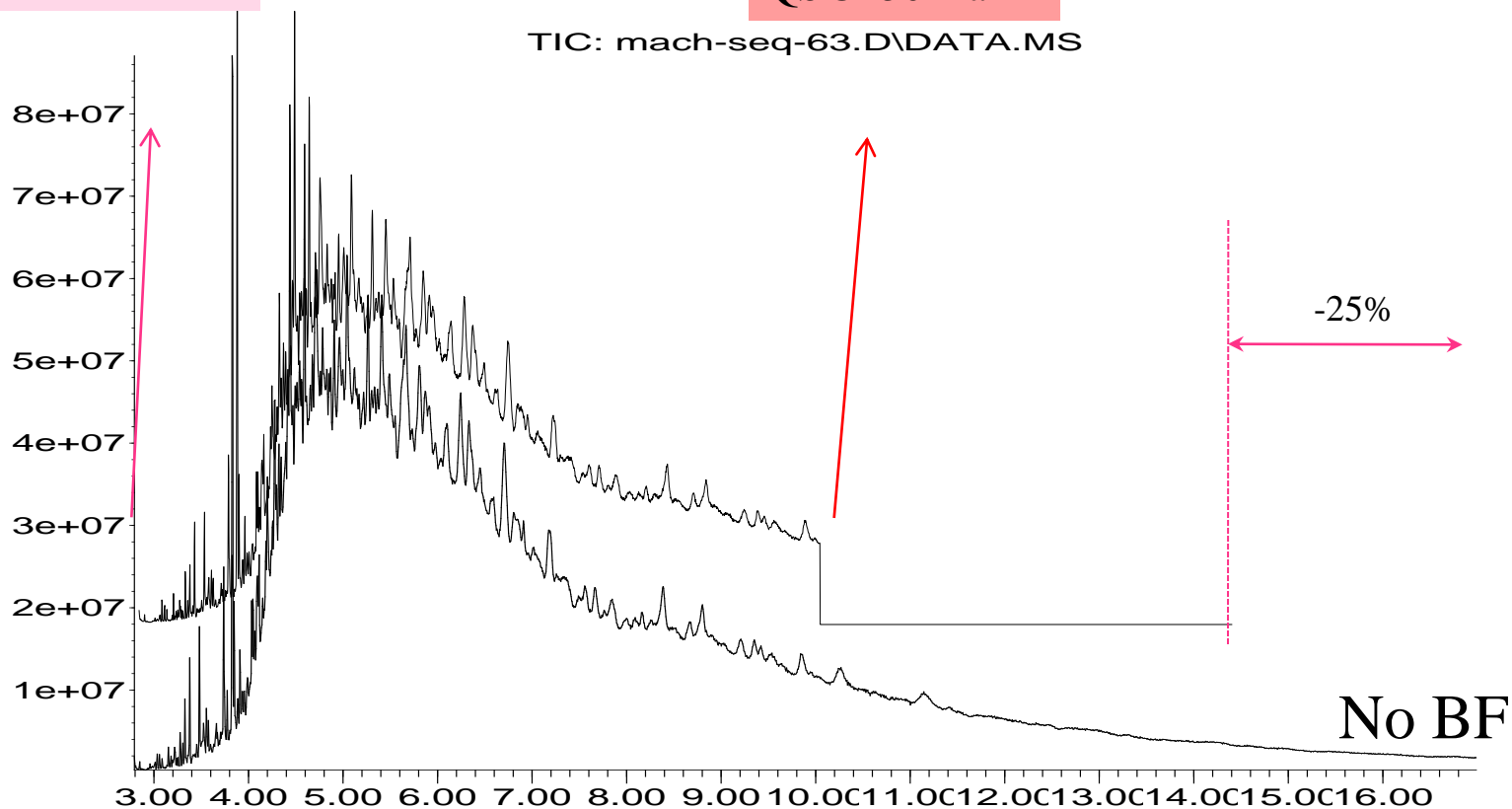
INJ @ 213-392kPa

QS@28 kPa

INJ @ 10kPa

QS@250kPa

TIC: mach-seq-63.D\DATA.MS

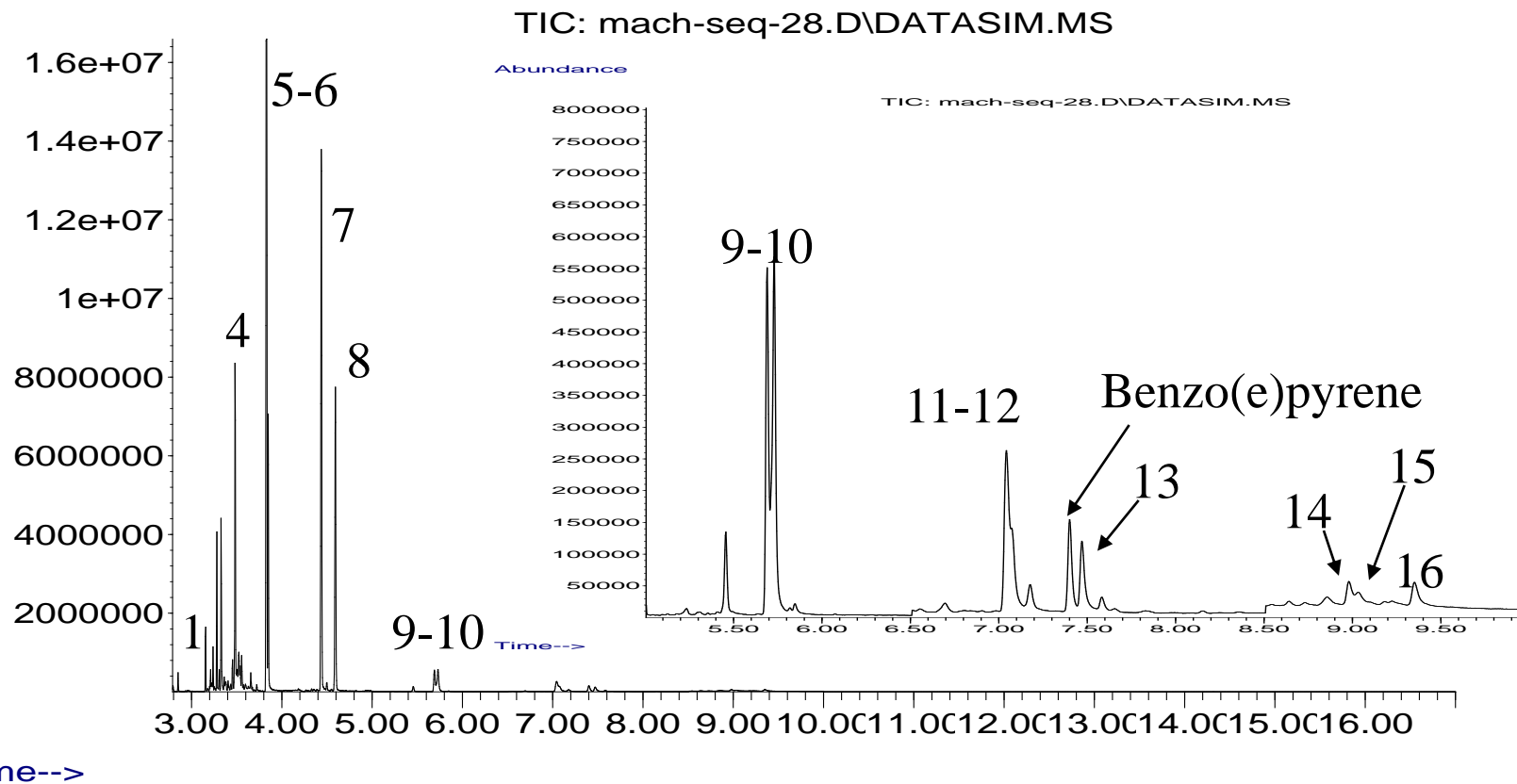


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# Soil extract - GC-MS in scan/SIM – SIM trace

Abundance



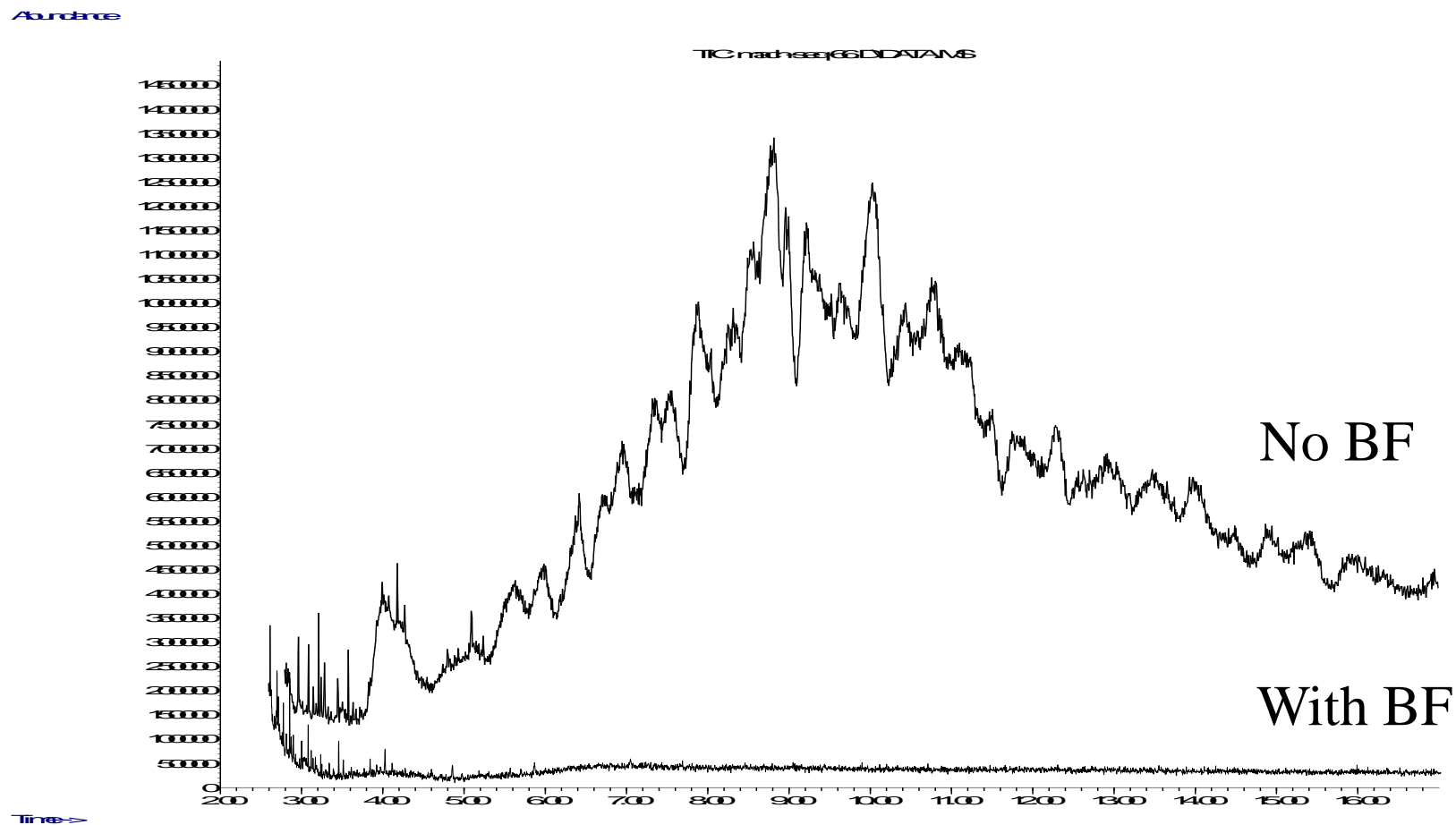
Time-->



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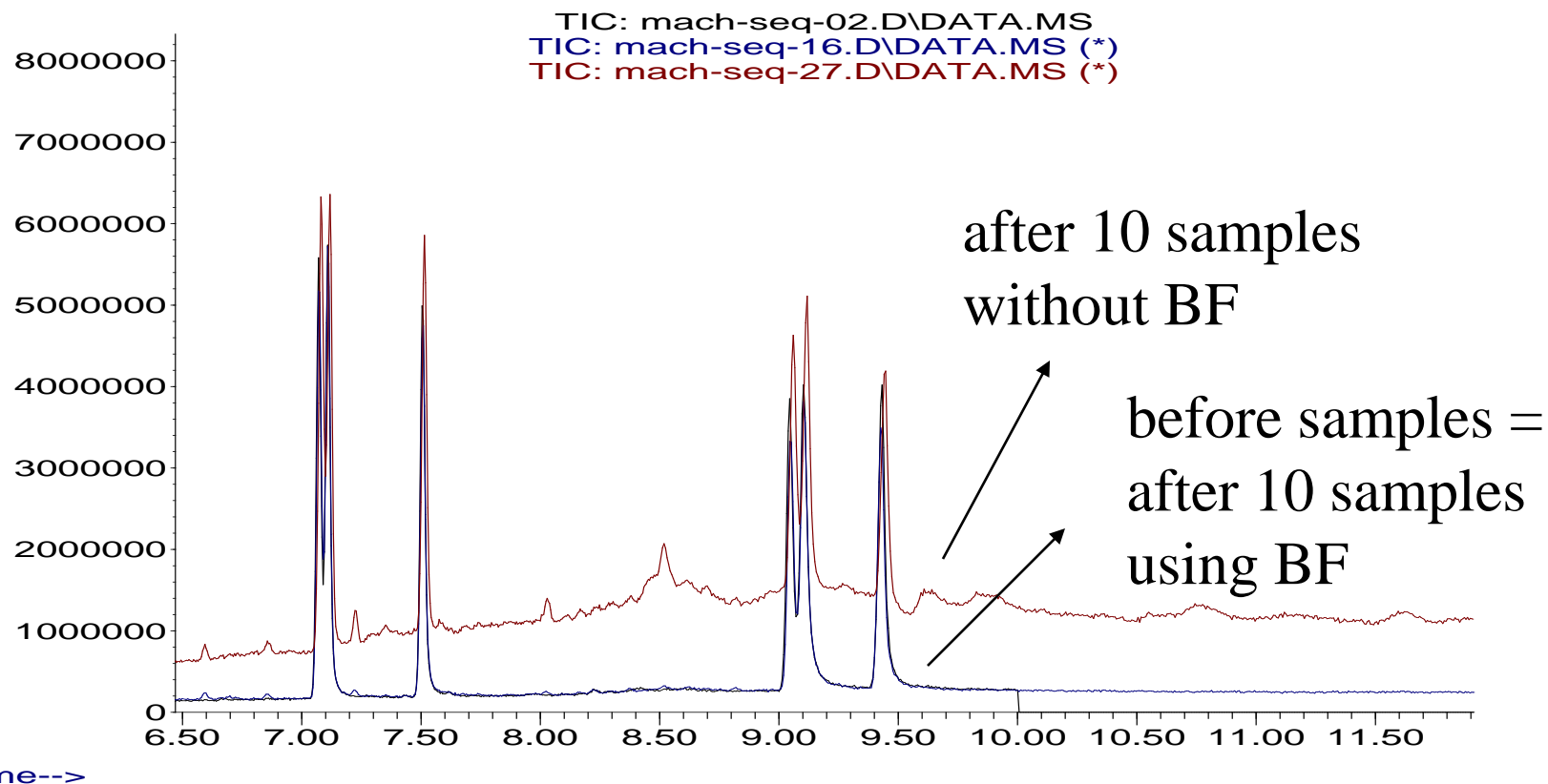
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# Blank run after soil extract - scan trace



# Check-out sample after soil extract sequence scan trace

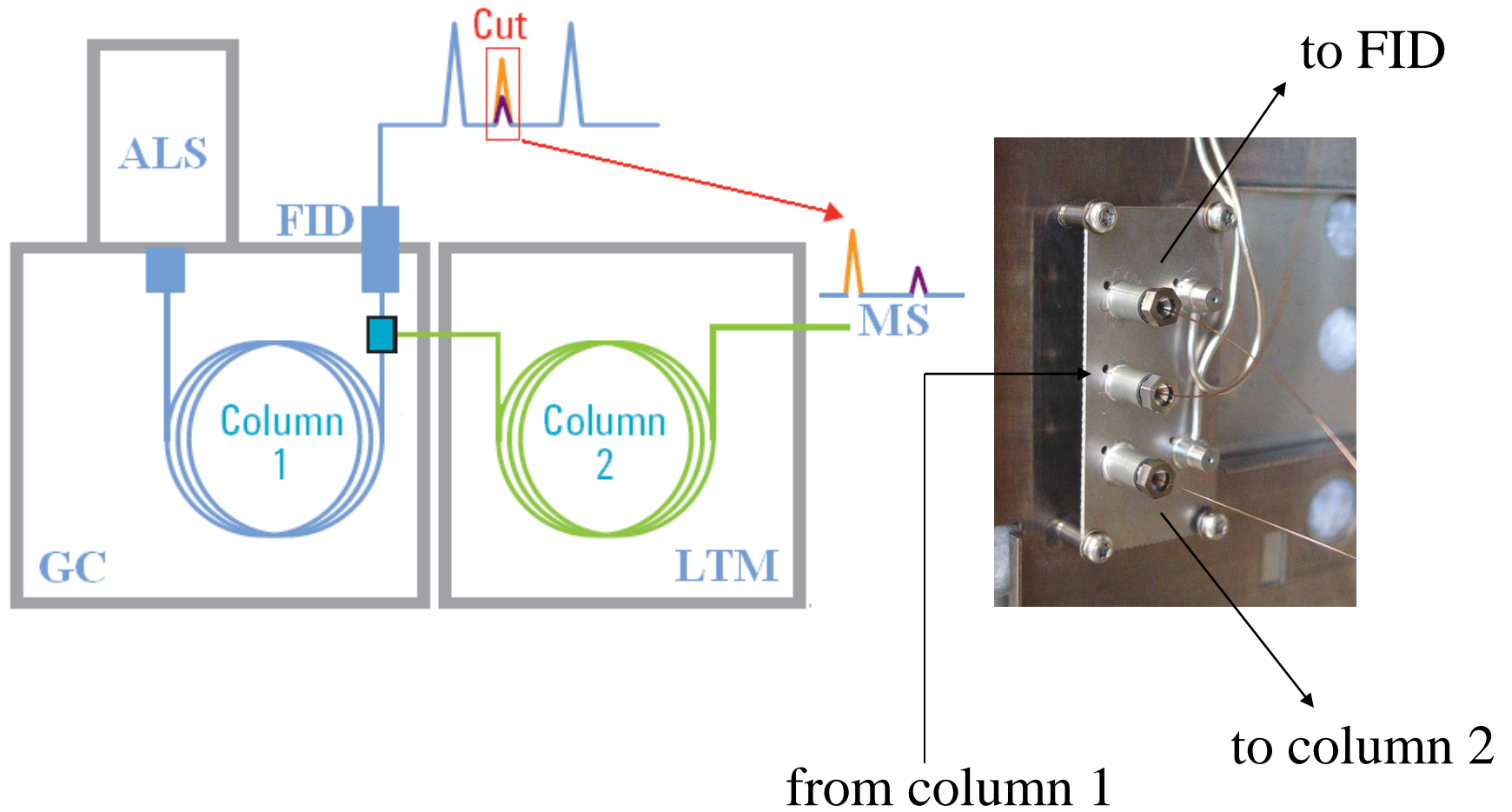
Abundance



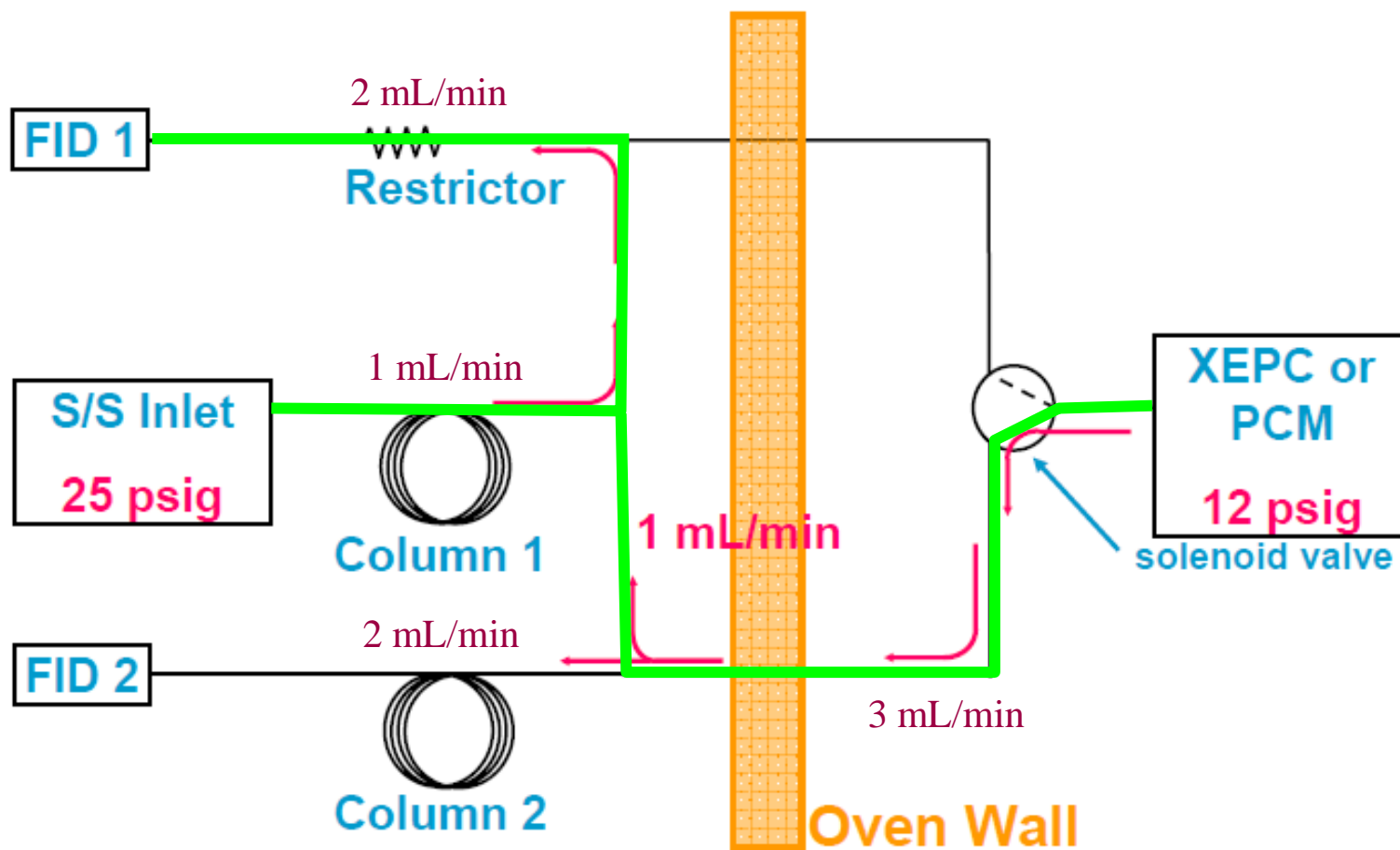
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# CFT based 2D-GC



# Deans Switch principle – 1D analysis (FID1)

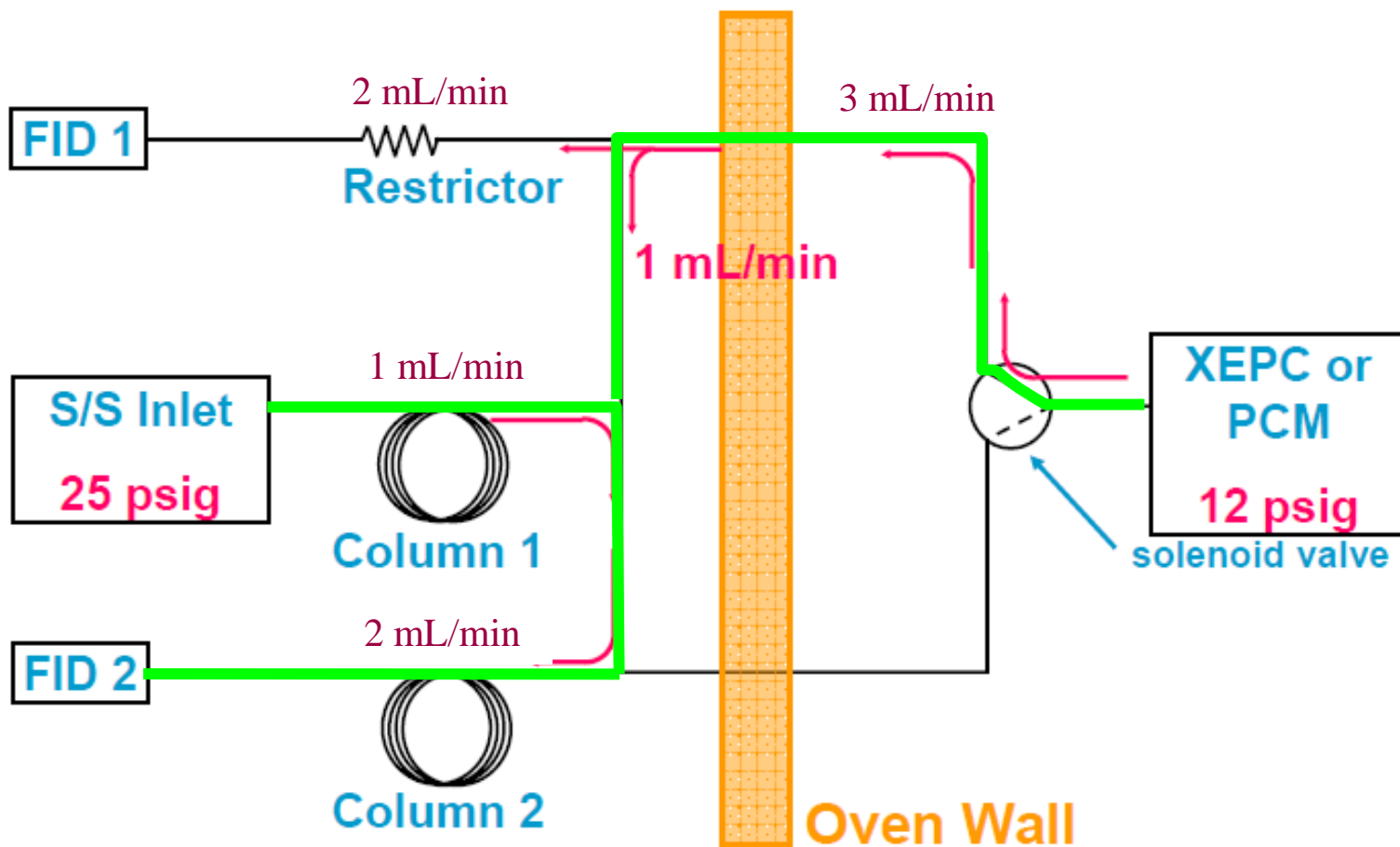


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# Deans Switch principle – ‘heart cut’ to 2D GC column

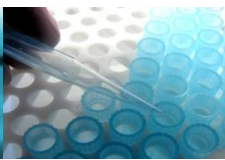
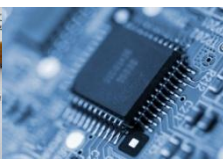
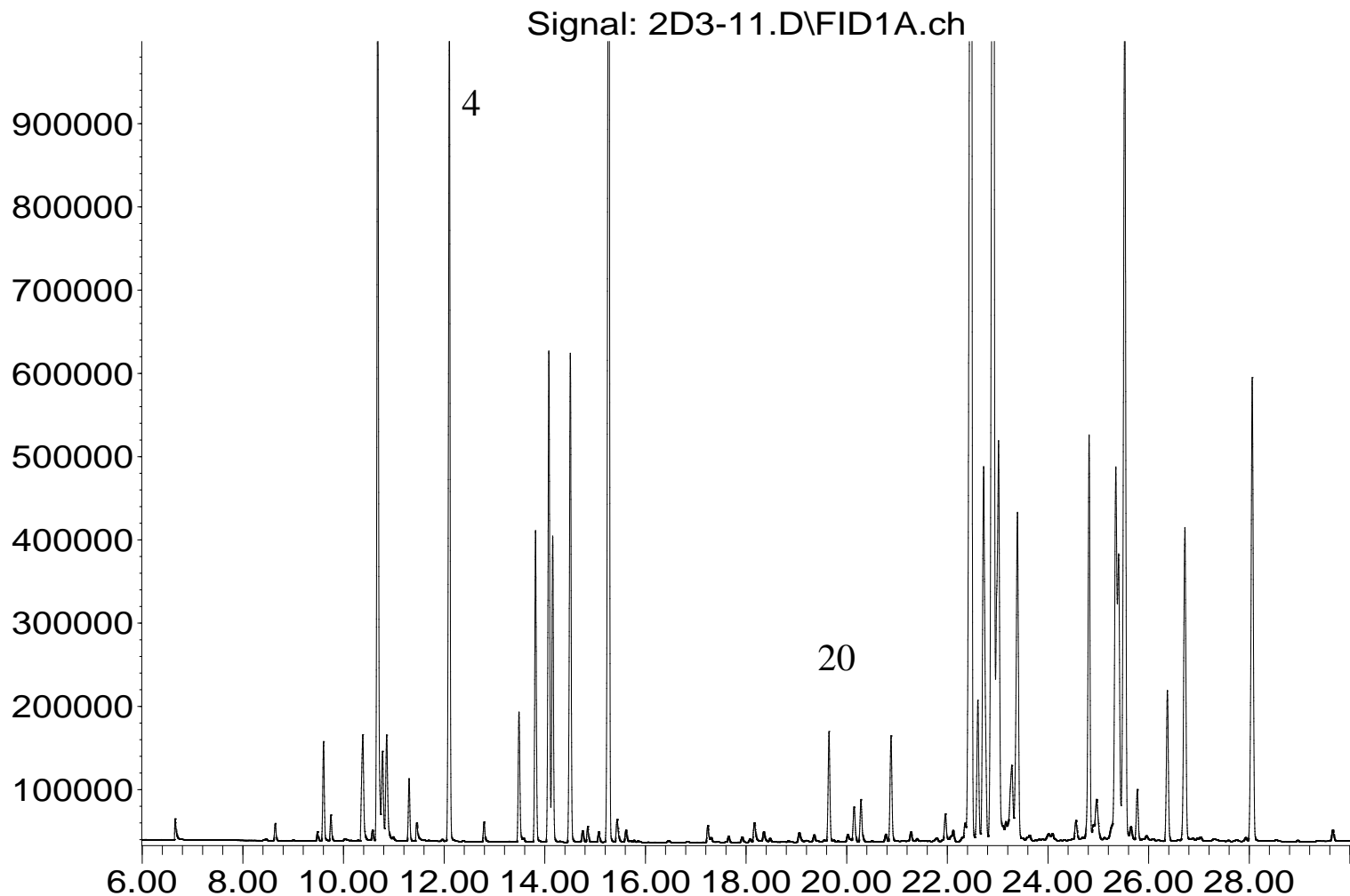


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# Suspected Flavor and Fragrance Allergens in Perfumes

Abundance

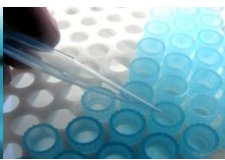
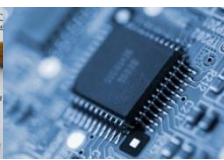
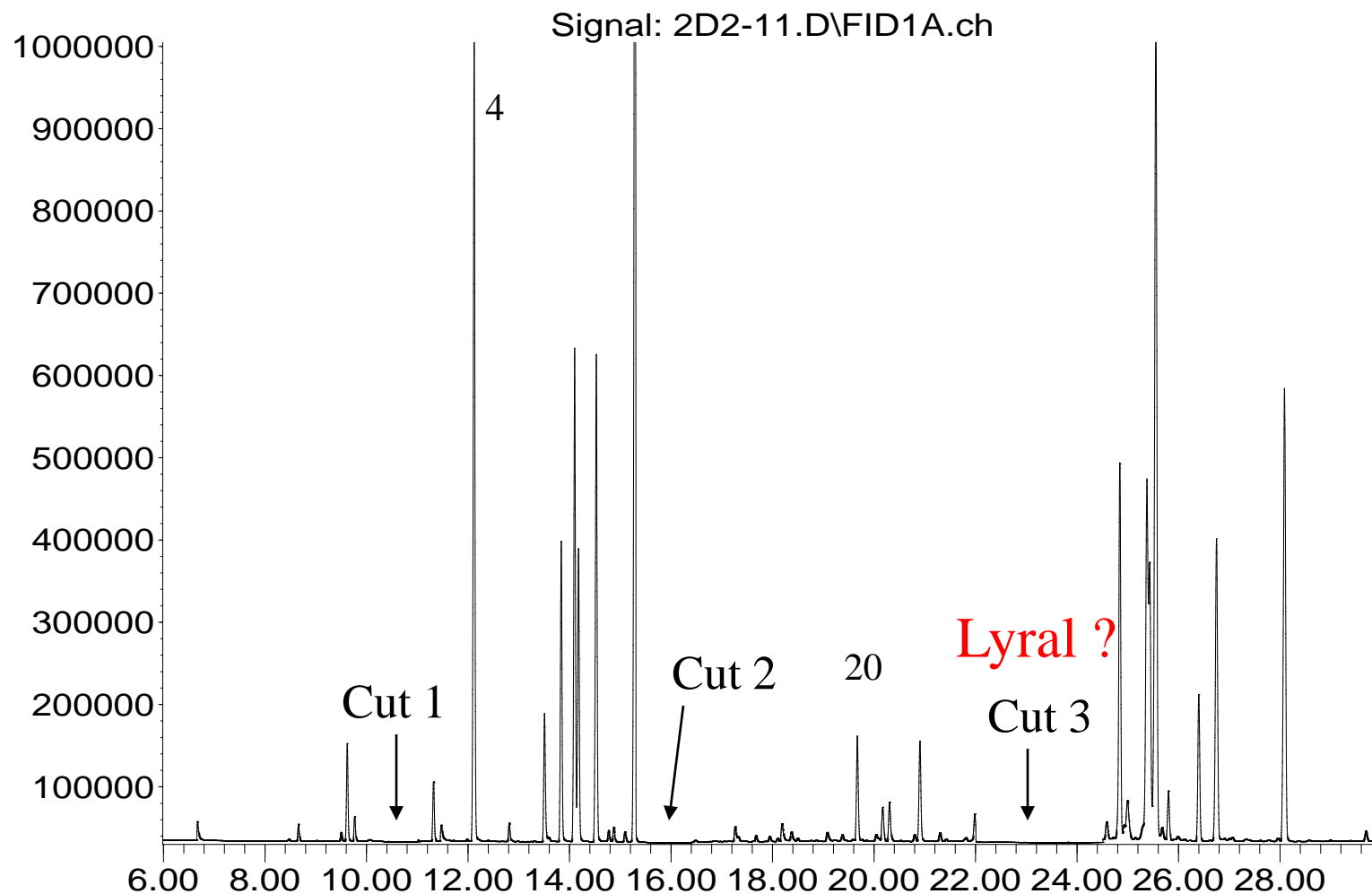


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# Suspected Flavor and Fragrance Allergens in Perfumes

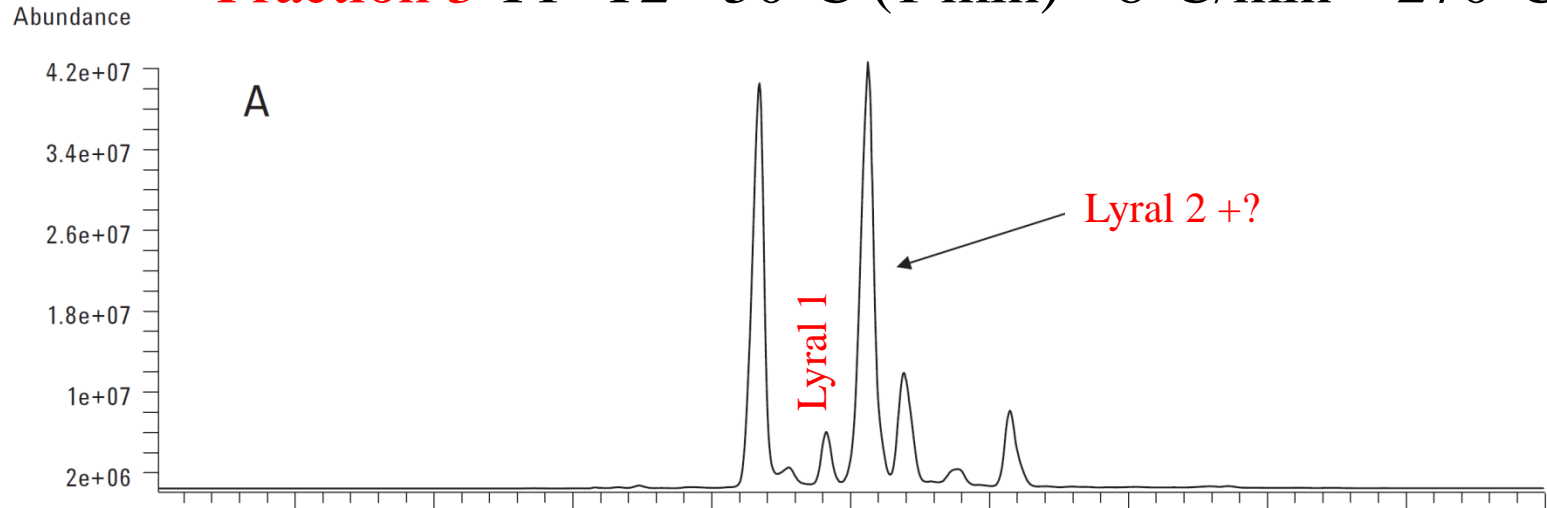
Abundance



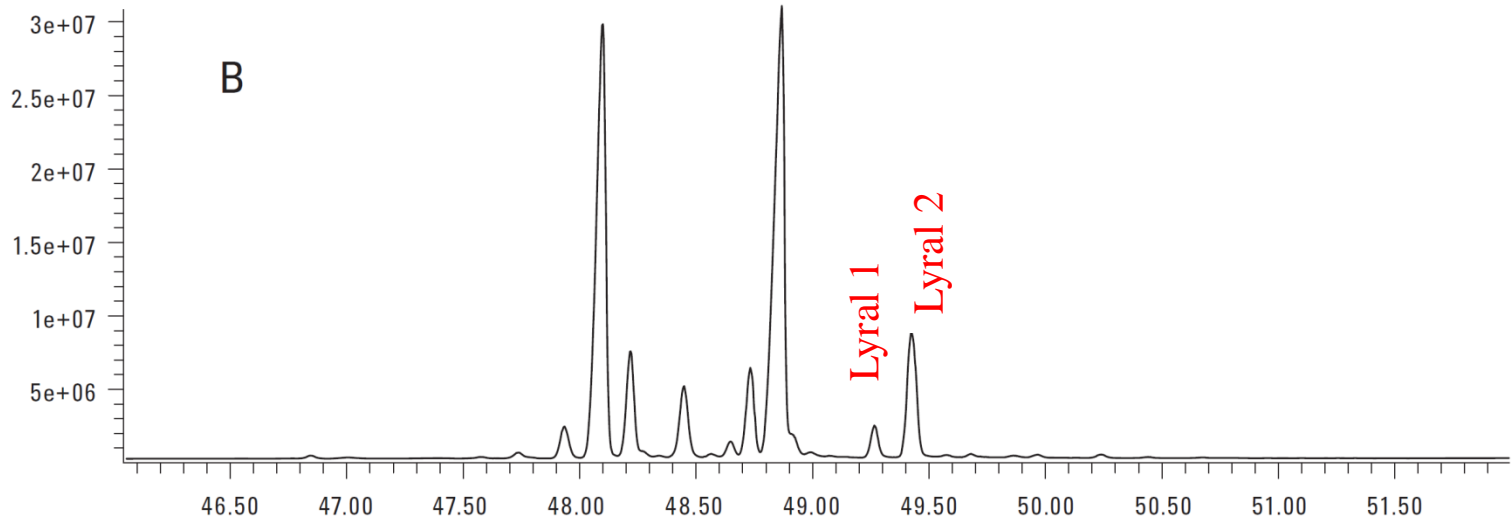
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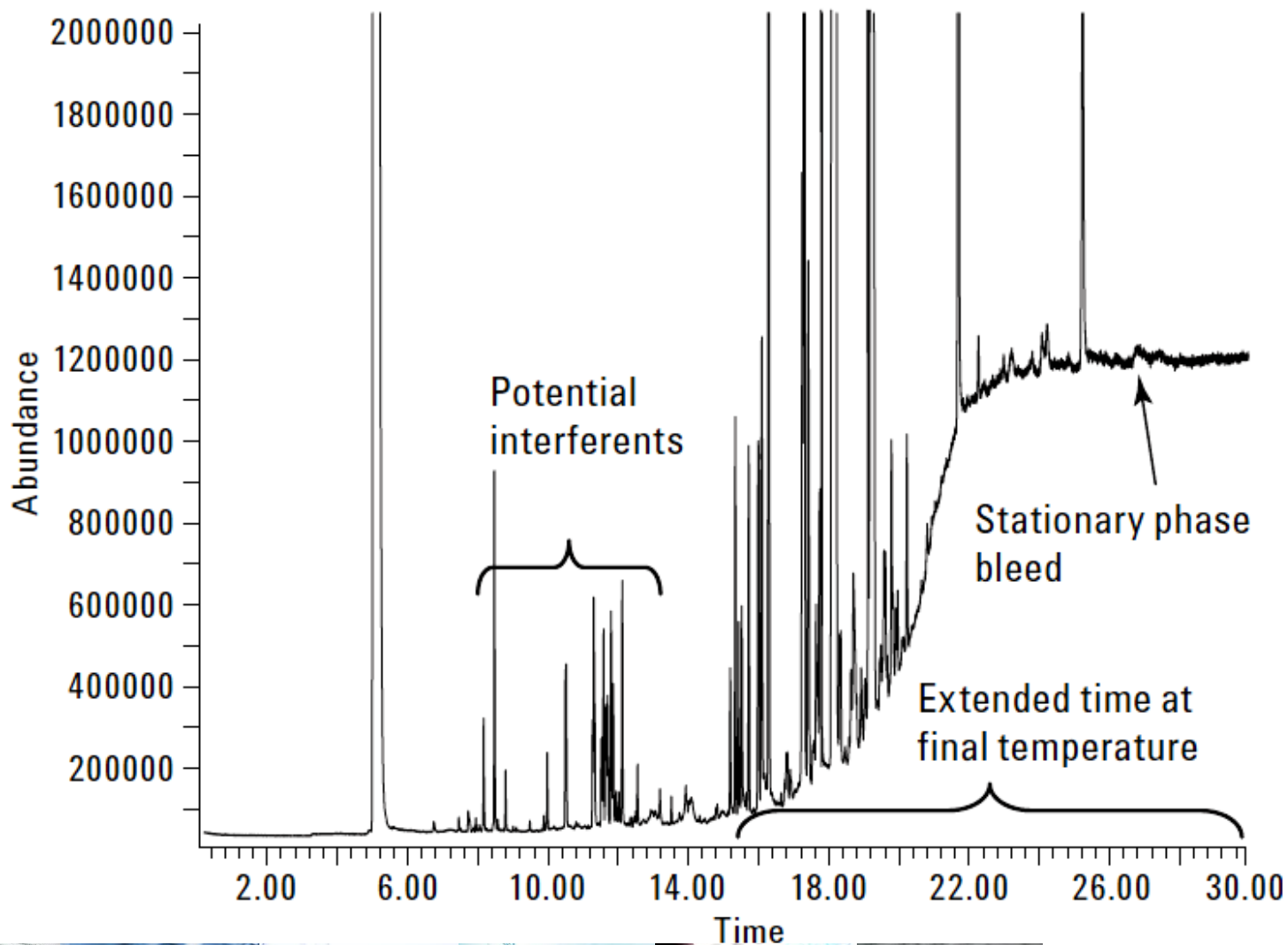
# Fraction 3 T1=T2= 50 C (1 min) - 8 C/min – 270 C



# Fraction 3 T1≠T2; T2= 50 C (25 min) - 6 C/min - 250 C



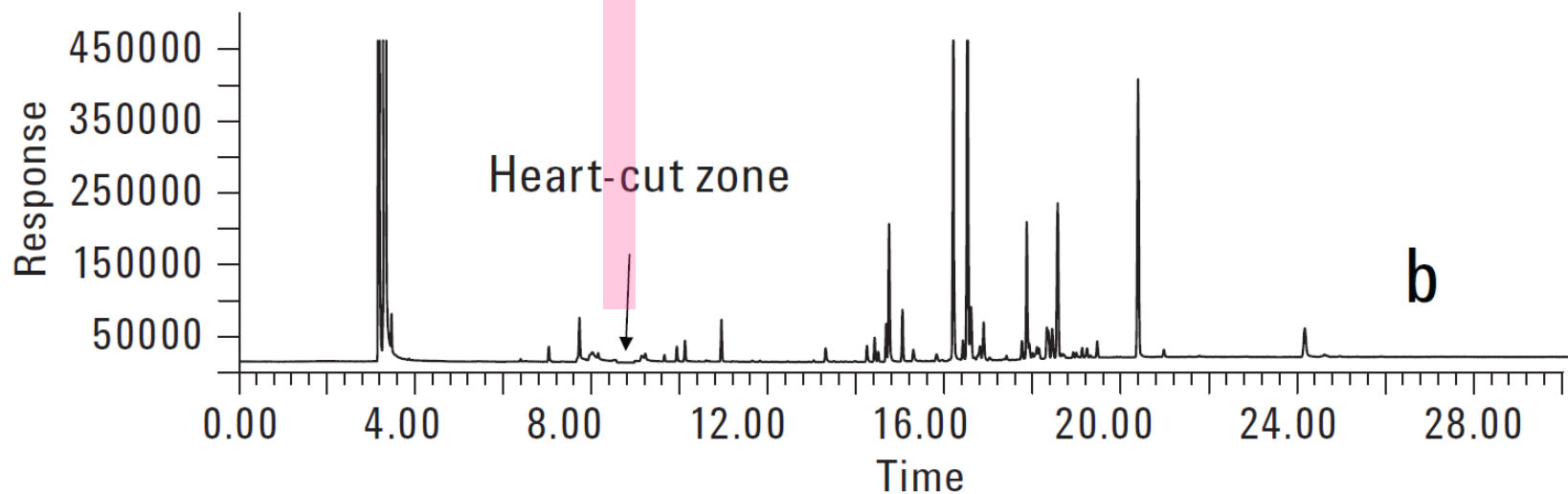
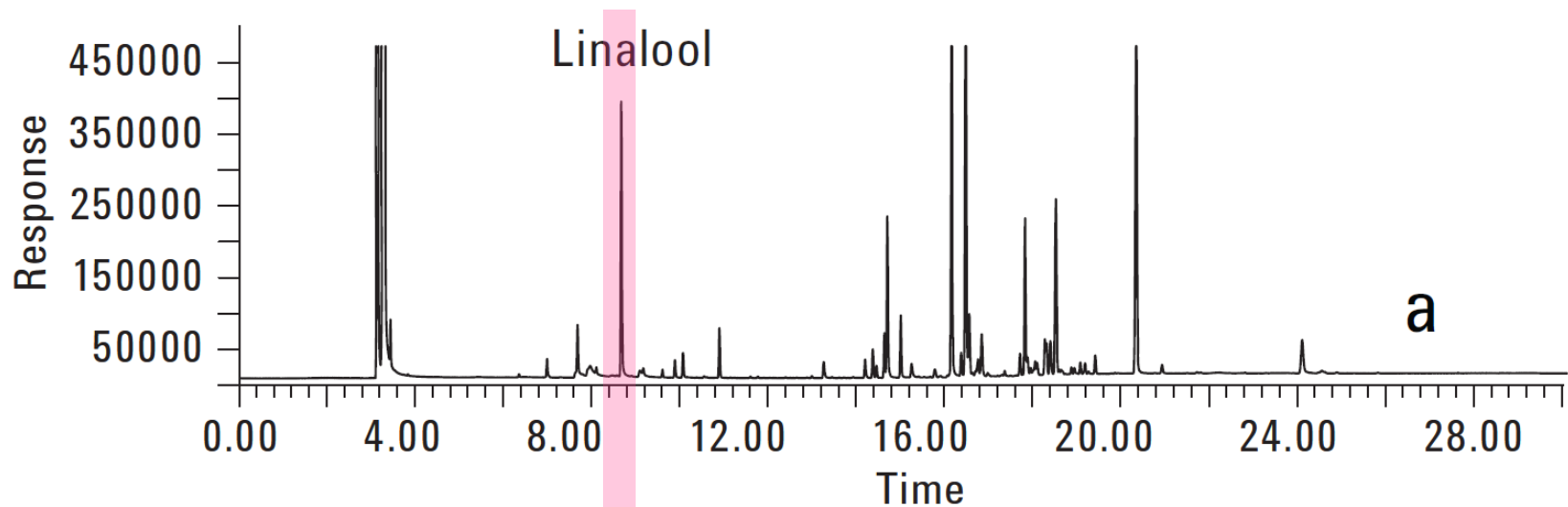
# Linalool analysis in essential oil on chiral GC column (CycloDex-B)



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# Linalool analysis in essential oil on <sup>1</sup>GC column (HP-1MS)



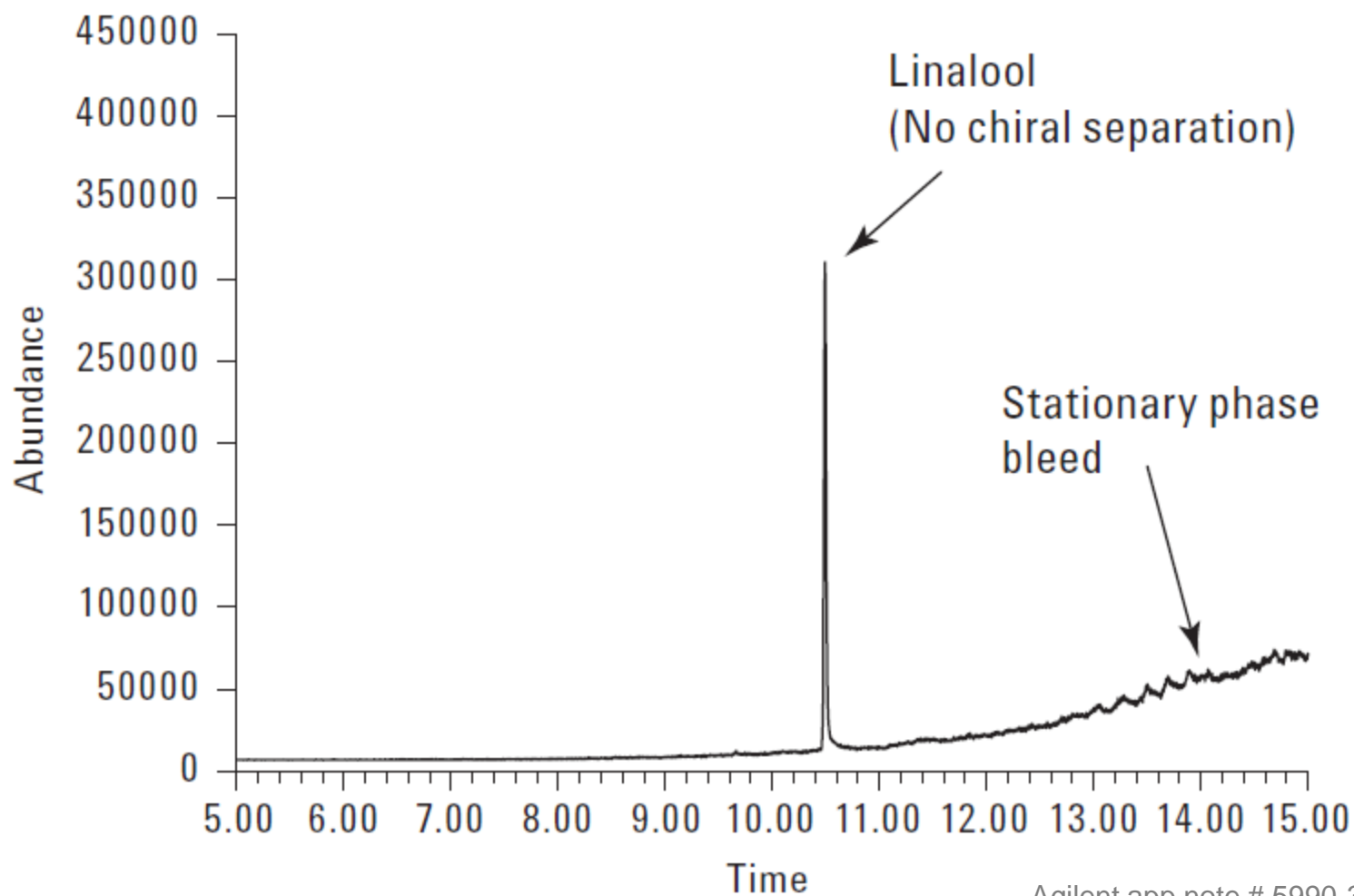
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## 2DGC analysis of fraction 8.5-8.9 min

**T1=T2= 70 C (1 min) - 10 C/min – 200 C**



Agilent app note # 5990-3428EN

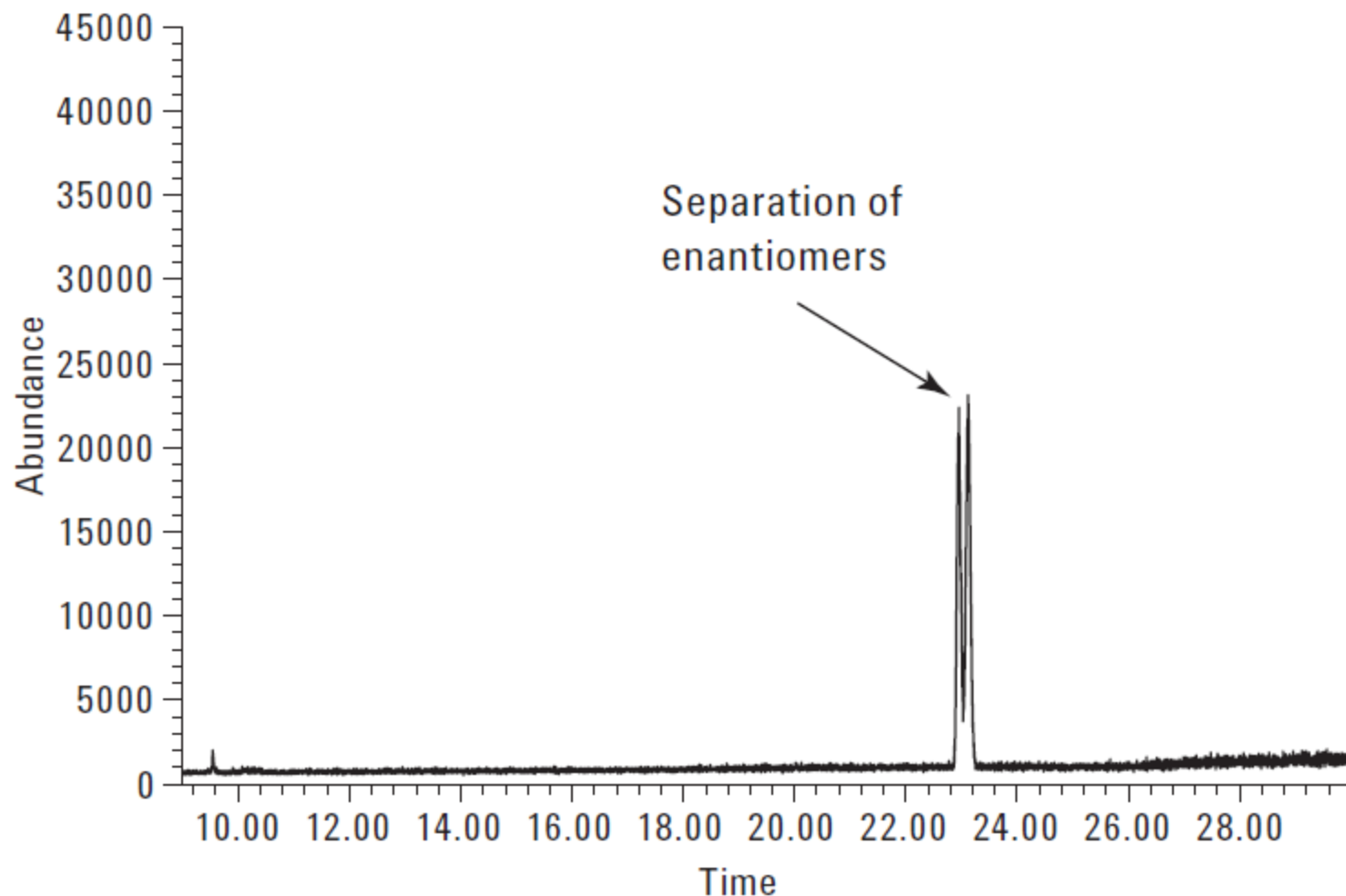


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## 2DGC analysis of fraction 8.5-8.9 min

**T1≠T2**; T2= 80 C (9 min) - 1 C/min - 100 C



Agilent app note # 5990-3428EN



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# Determination of PAHs in Mineral Oil (Food Grade Oil)

## Dim 1: HP-5MS – Heart-cut of B(b)F, B(k)F and B(a)P fraction

Abundance

Signal: Find\_Learn\_10g.D\FIDAch

1800000  
1600000  
1400000  
1200000  
1000000  
800000  
600000  
400000  
200000

OIL: 100 mg/mL  
1  $\mu$ L inject  
1/10 split  
= 10  $\mu$ g on-column

10.00 15.00 20.00 25.00 30.00 35.00

Time-->

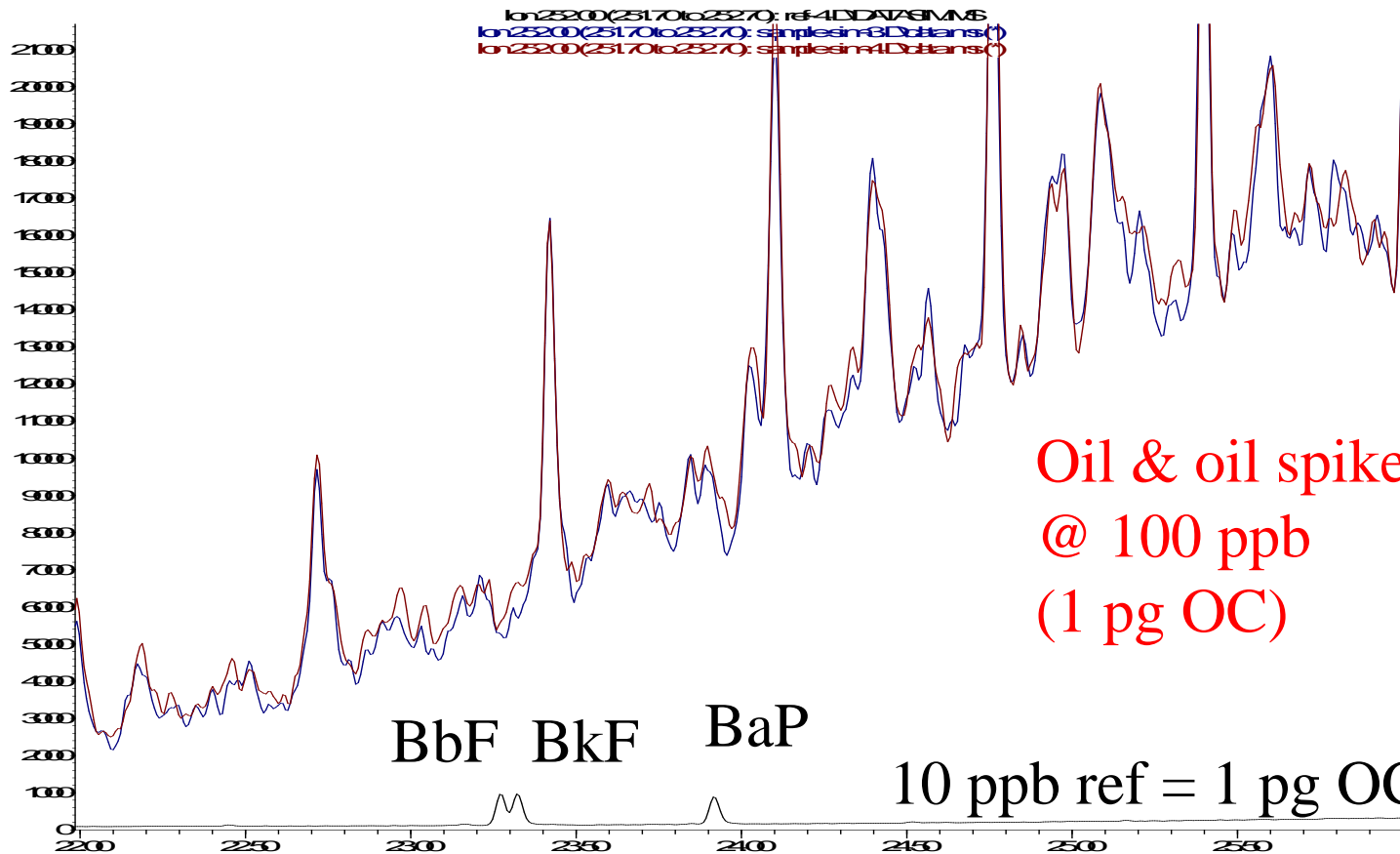


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# Determination of PAHs in Mineral Oil direct analysis by GC-MSD

Abundance



Oil & oil spiked  
@ 100 ppb  
(1 pg OC)

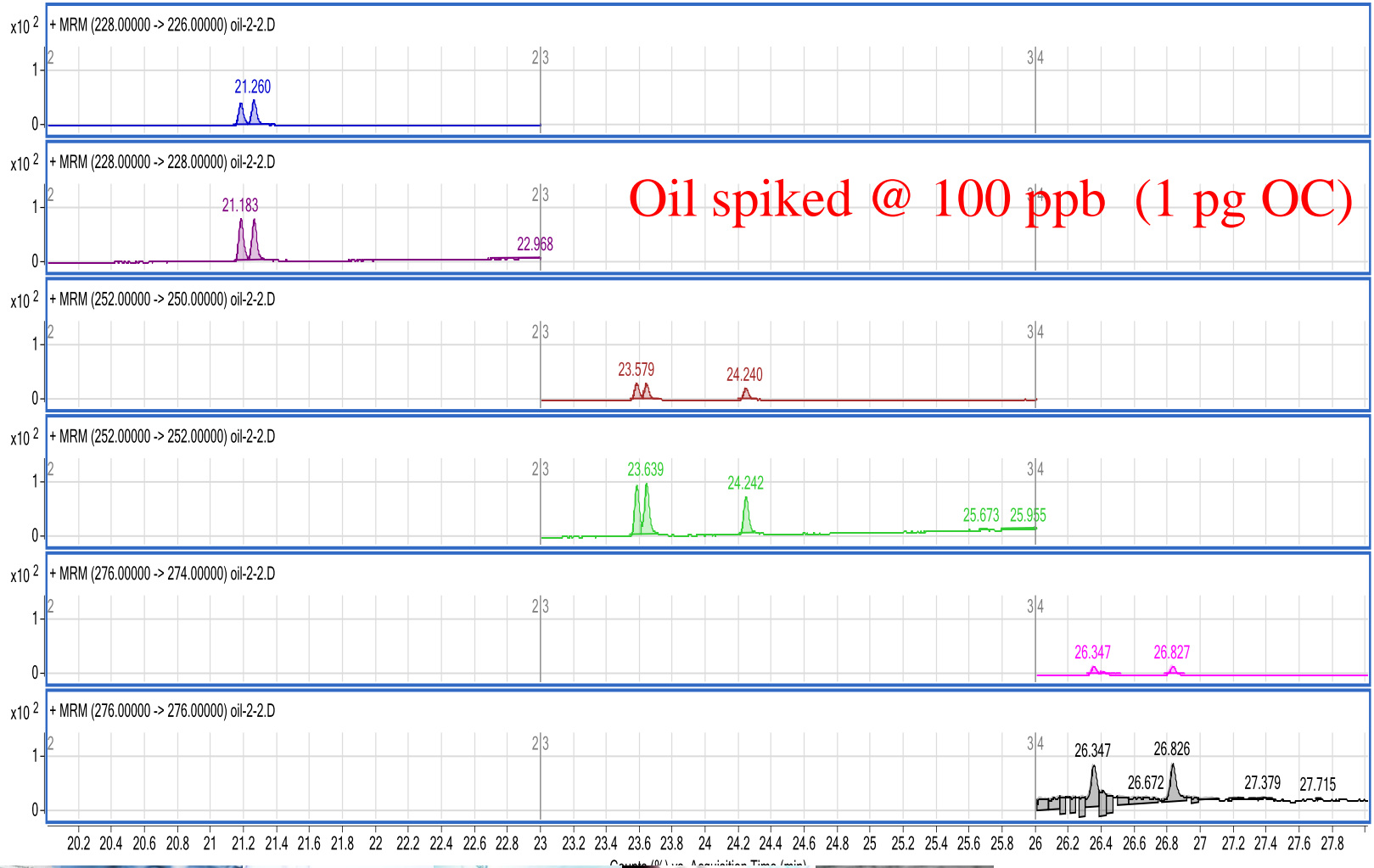
Time



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# Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



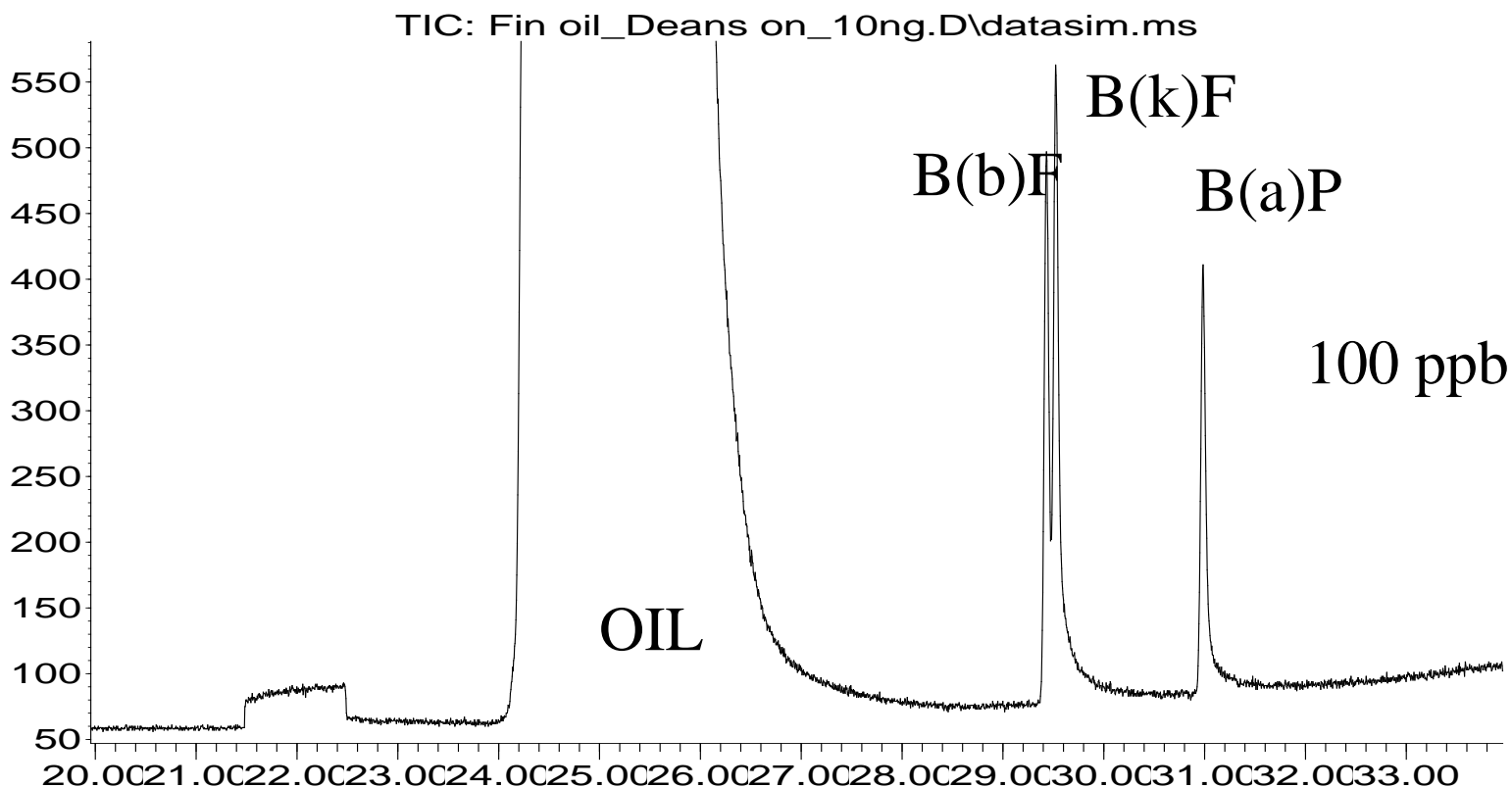
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# Determination of PAHs in Mineral Oil (Food Grade Oil) analysis by 2DGC-MSD

## Dim 2: DB-17 – Analysis of B(b)F, B(k)F and B(a)P fraction

Abundance



Time-->

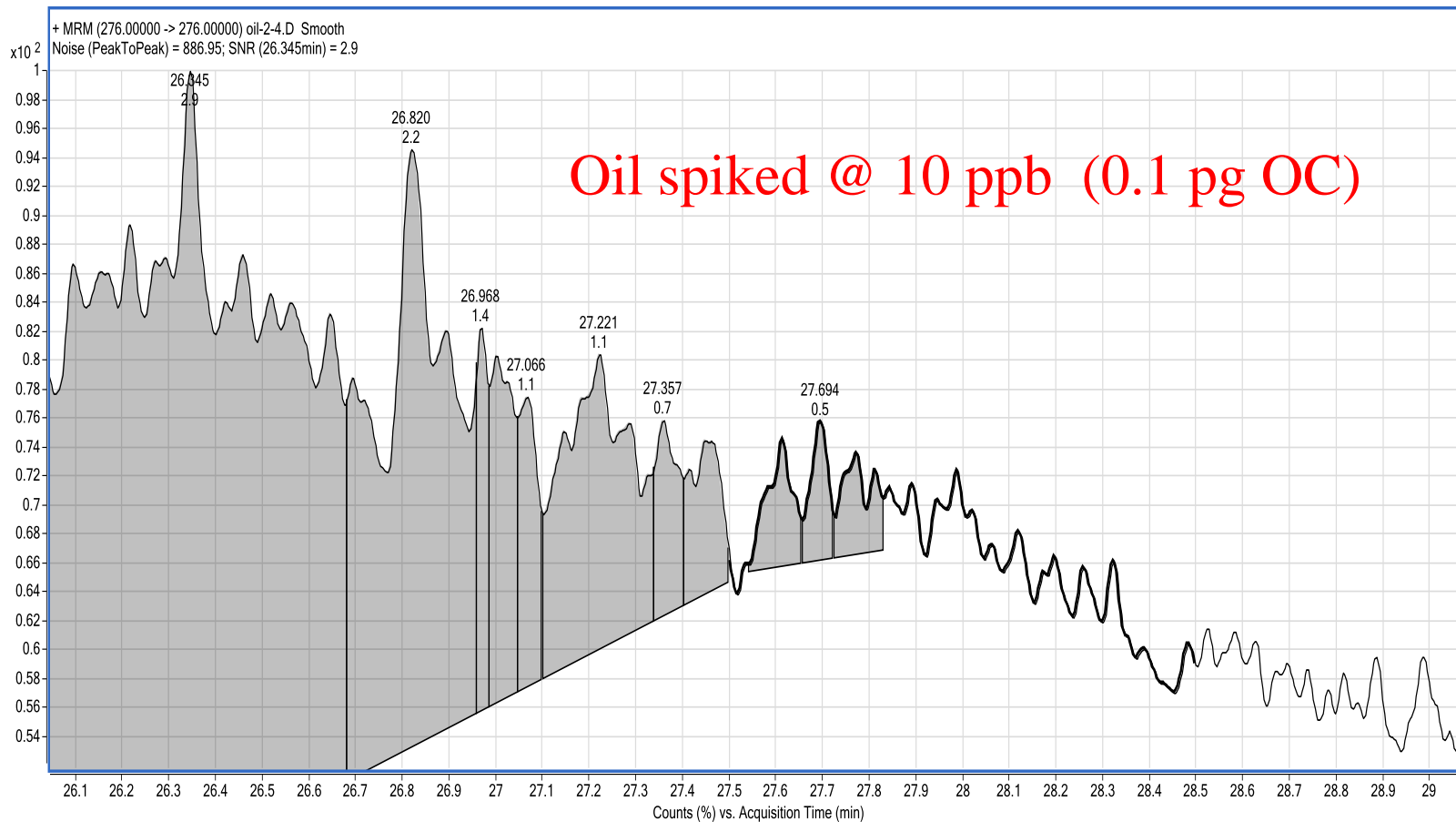


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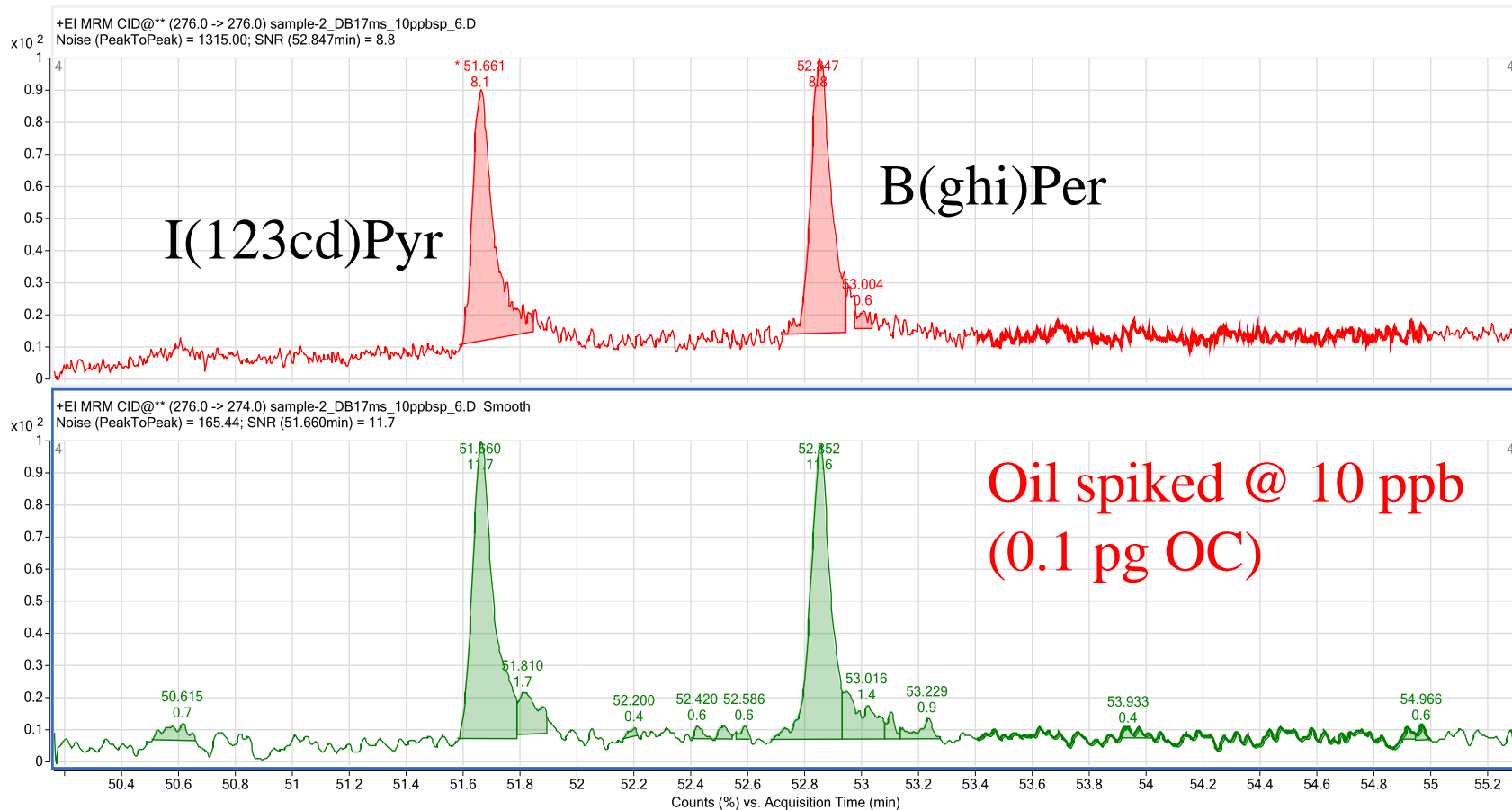
# Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



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# Determination of PAHs in Mineral Oil analysis by 2DGC-QQQ



# Determination of Genotoxics in API

- Matrix
  - Non-volatile (semi-volatile)
  - Wide polarity range (log P from  $< 0$  to  $> 6$ )
  - Stability ?
- Concentration:
  - Target LOD: 1 ppm = 1  $\mu\text{g/g}$  drug substance
  - This is about 3 orders of magnitude lower than classical QC (purity determination)



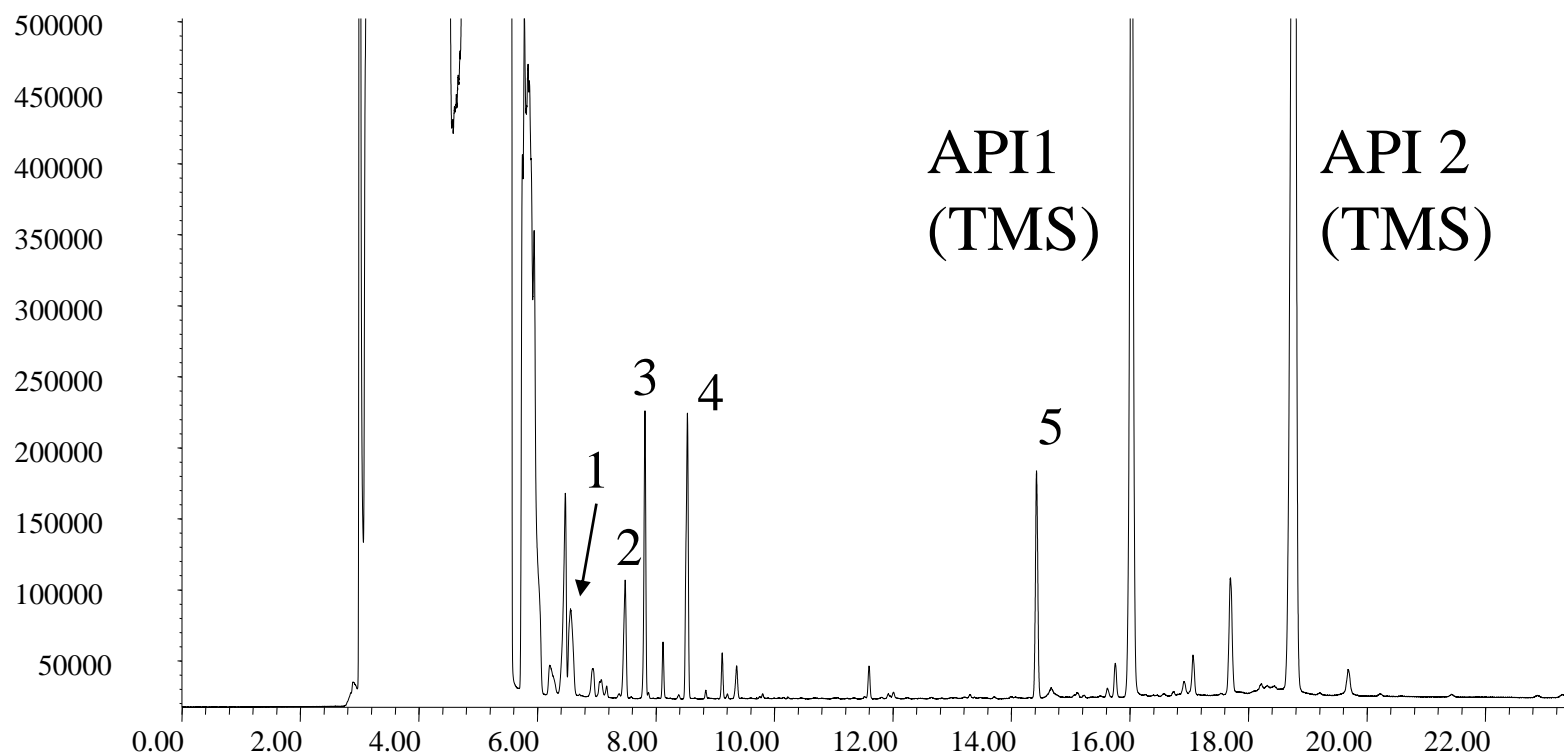
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# Halo-alcohols (TMS der) in Carbamazepine on Dim 1 - scouting run

TMS-ethers of 2-bromoethanol (1), 2-iodoethanol (2), 4-chloro-1-butanol (3), 2-(2-chloroethoxy)ethanol (4), 11-bromo-undecanol (5)

Response\_



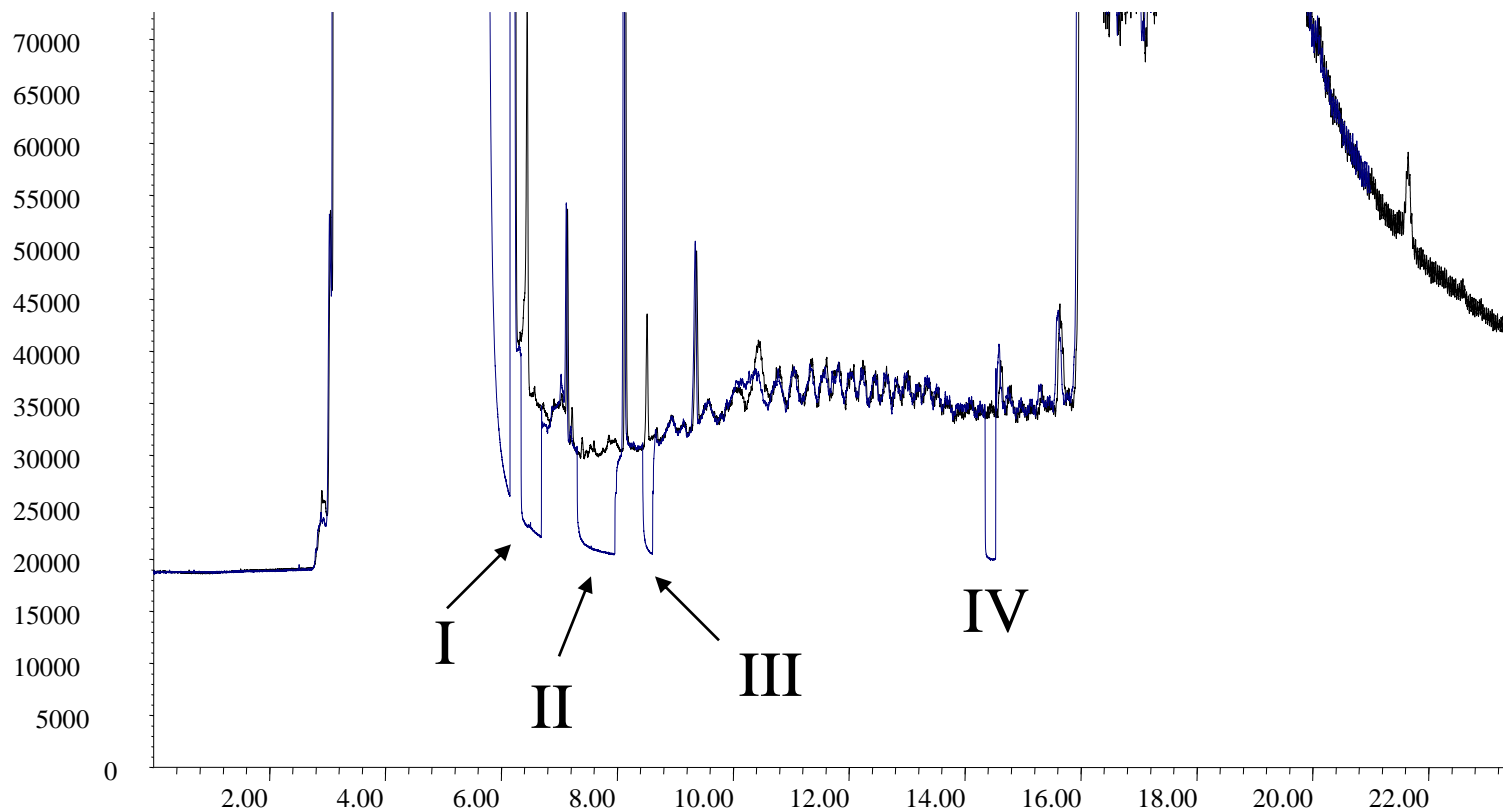
Time



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# Halo-alcohols (TMS der) in Carbamazepine (1 ppm) on Dim 1 with and without heart-cut

Response\_



Time



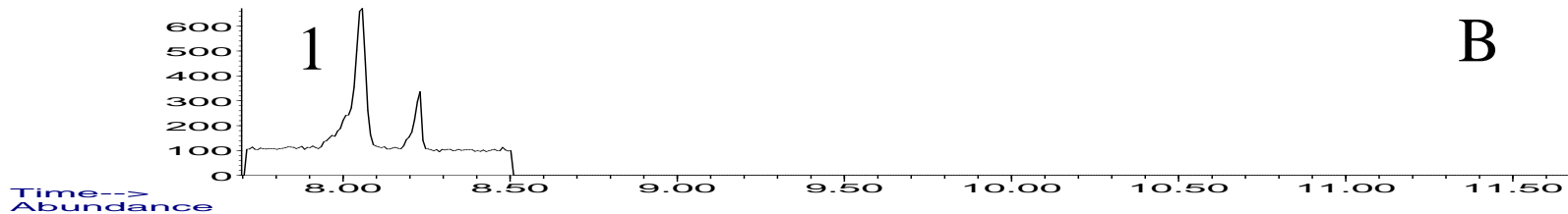
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# Dim 2

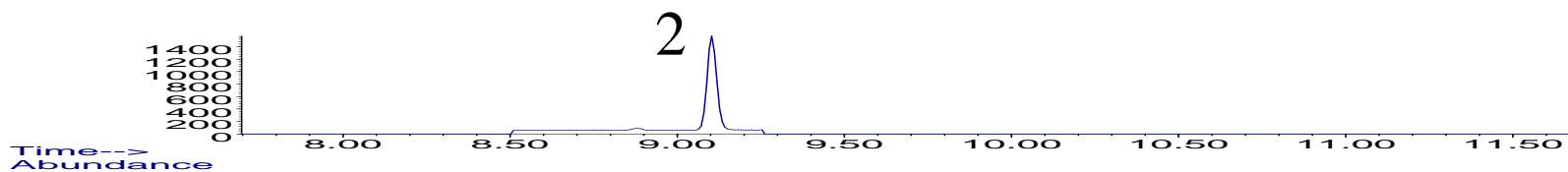
Abundance

Ion 1300 (137.0 to 137.0): 5.g/AP\_Velconesthompis\_4.D\\DATA\\SIMS

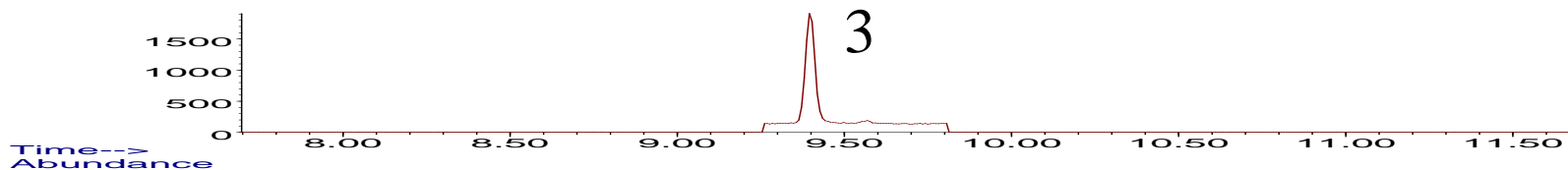


B

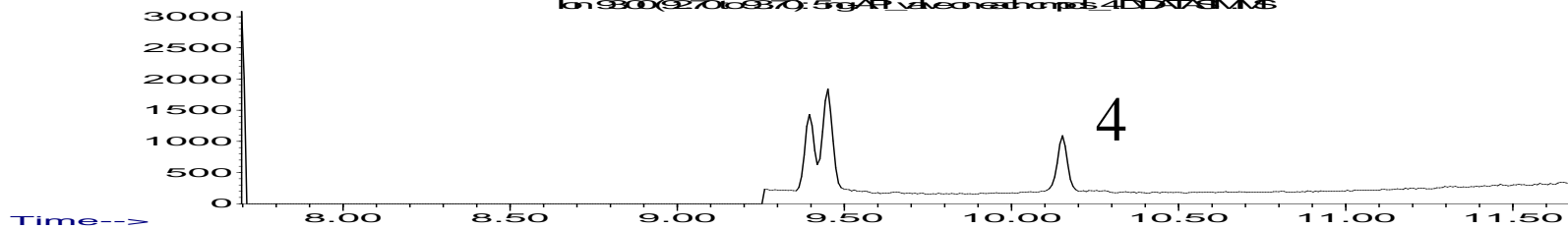
Ion 1350 (134.0 to 135.0): 5.g/AP\_Velconesthompis\_4.D\\DATA\\SIMS



Ion 1200 (122.0 to 123.0): 5.g/AP\_Velconesthompis\_4.D\\DATA\\SIMS



Ion 930 (92.0 to 93.0): 5.g/AP\_Velconesthompis\_4.D\\DATA\\SIMS



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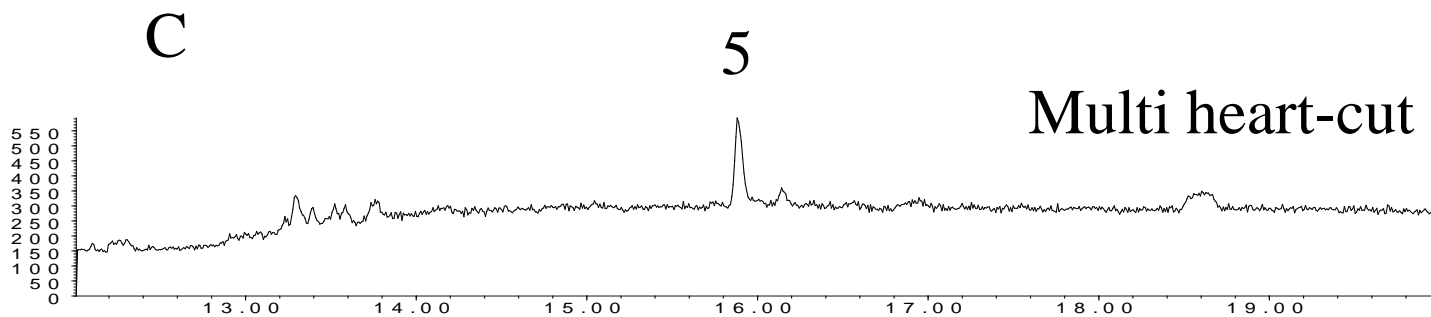


# Dim 2

## TMS-ether of 11-bromo-undecanol (5)

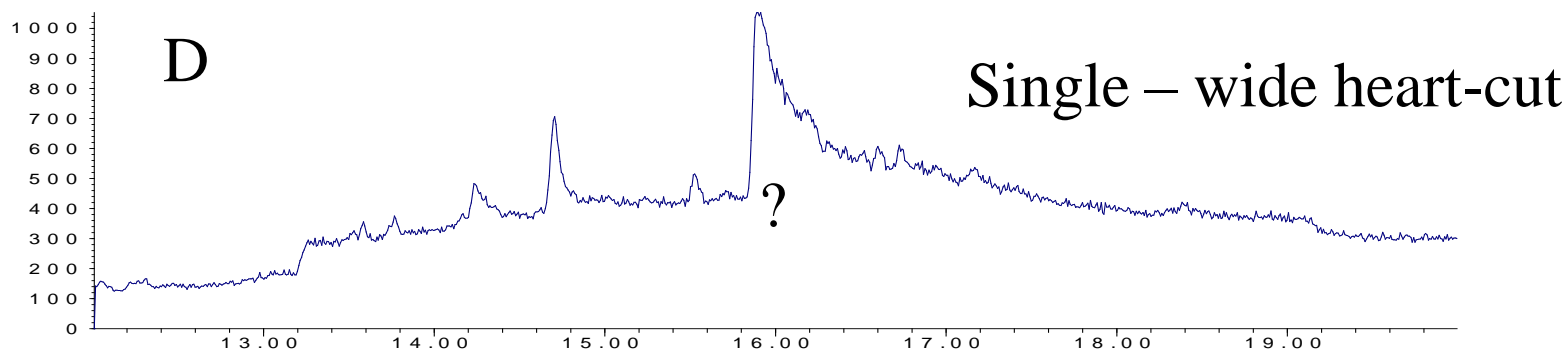
Abundance

Ion 169.00 (168.70 to 169.70): 5ng-API\_valve on each cmpds\_4.D\DATASIM.MS



Time-->  
Abundance

Ion 169.00 (168.70 to 169.70): 5ng-API\_valve on all cmpds\_4.D\DATASIM.MS (\*)



Time-->

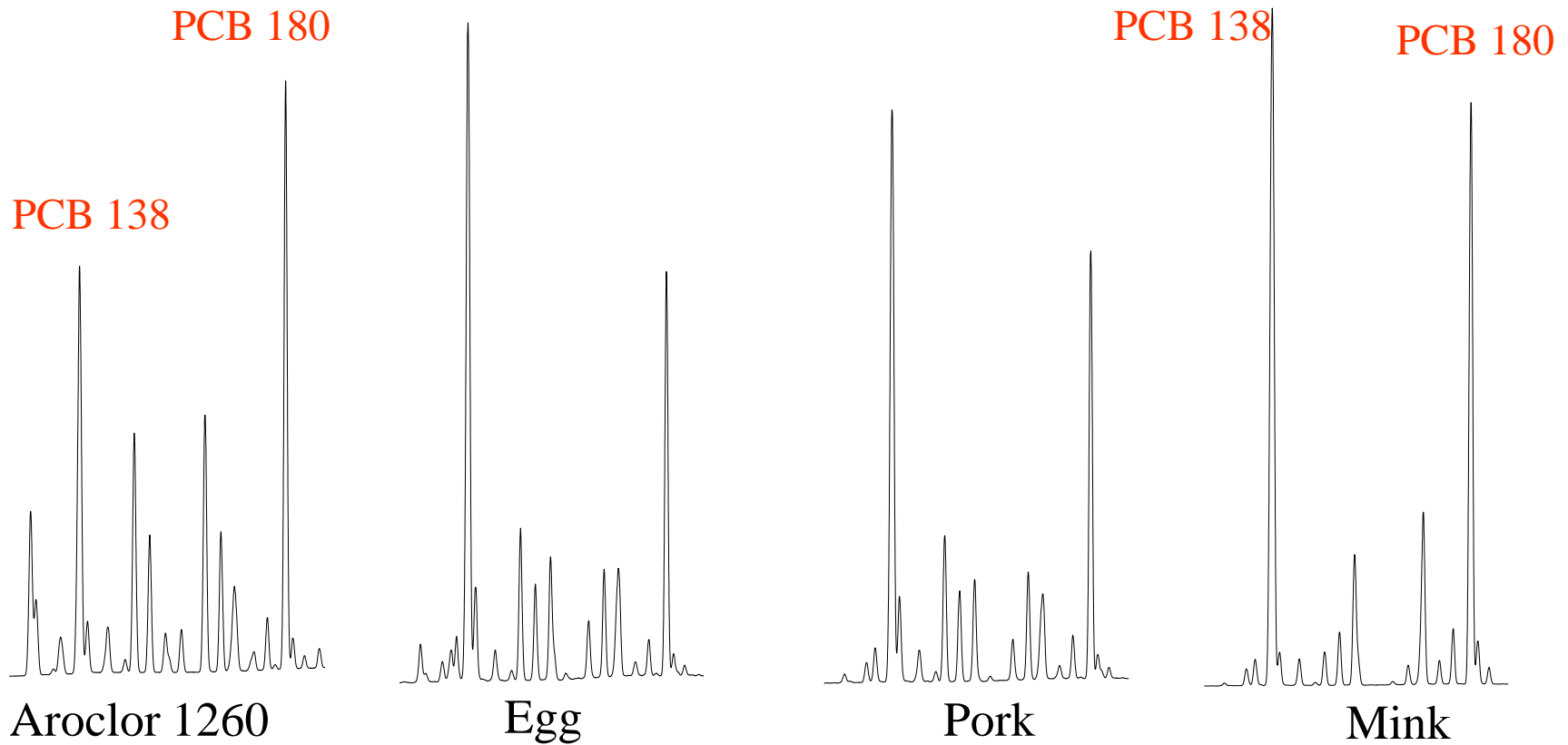


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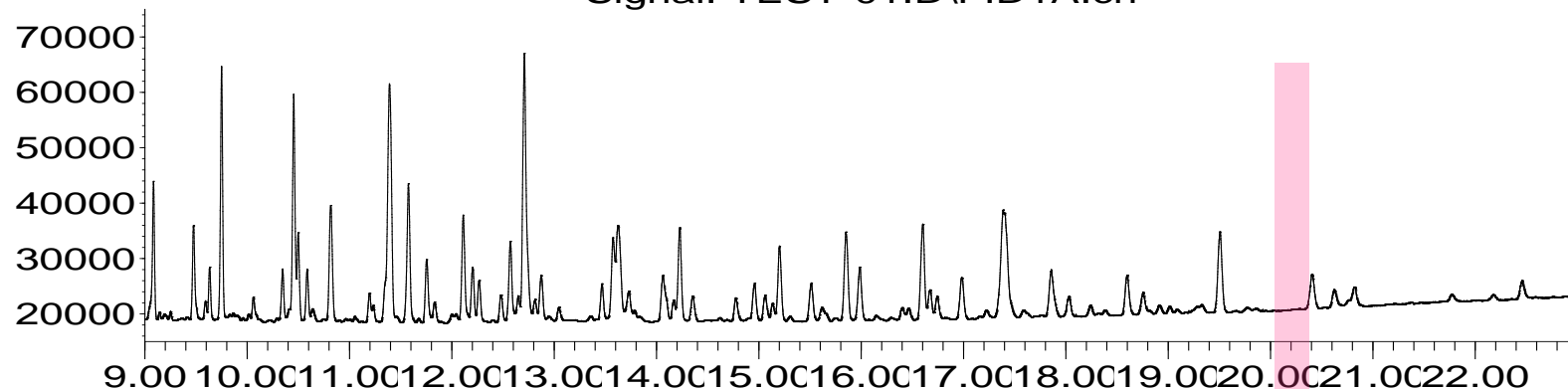
# PCBs in Food Products

*GC- $\mu$ ECD profiles of PCB extracts:  
Watch metabolism  $\Rightarrow$  co-planar PCBs?*

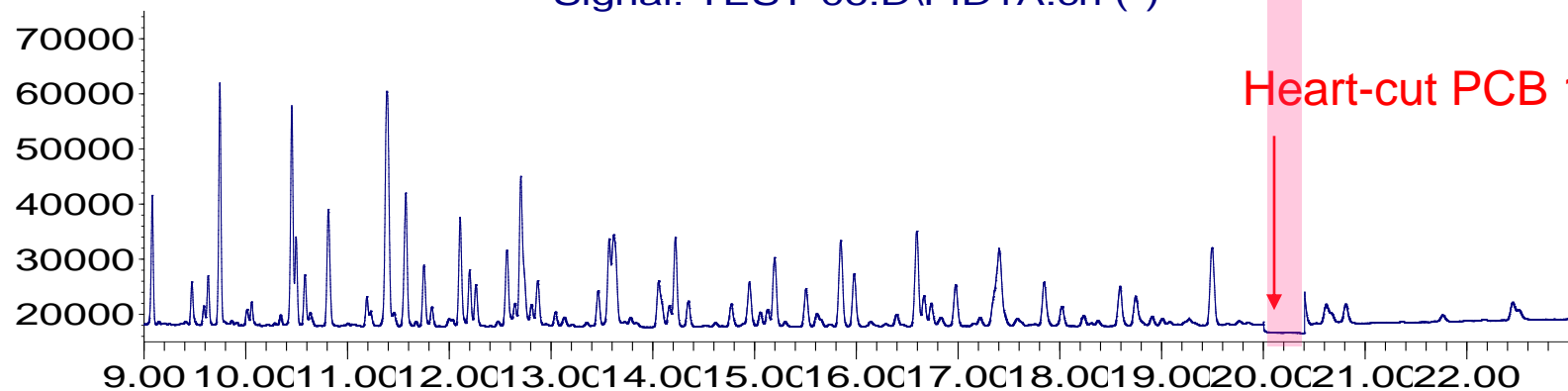


# PCB analysis by GC-GC-MS: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)

Signal: TEST-01.D\FID1A.ch



Signal: TEST-06.D\FID1A.ch (\*)



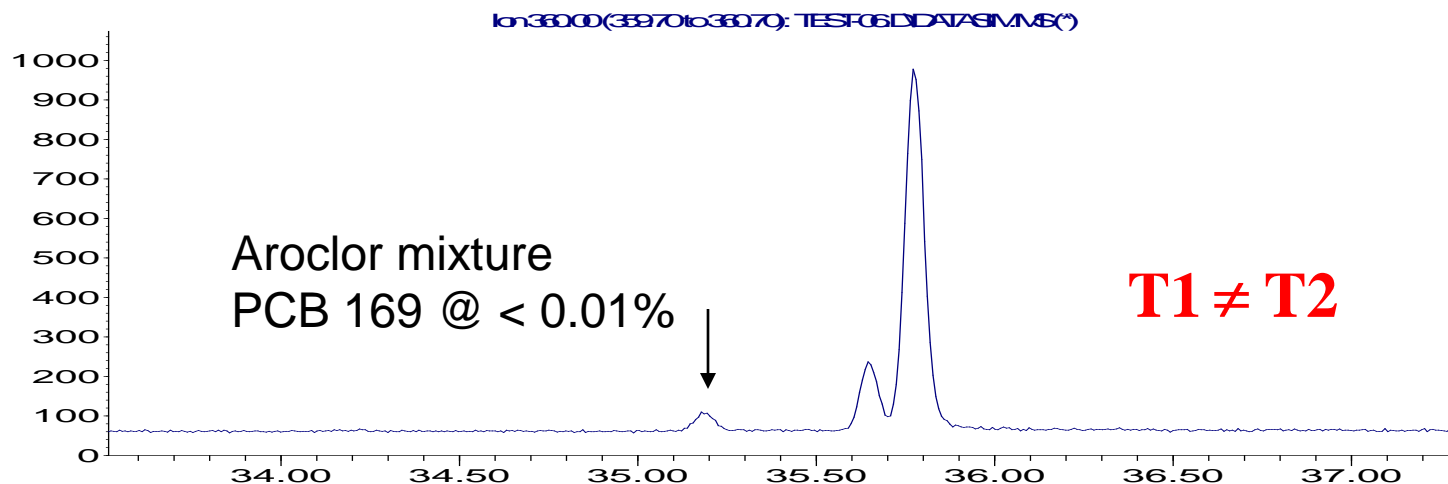
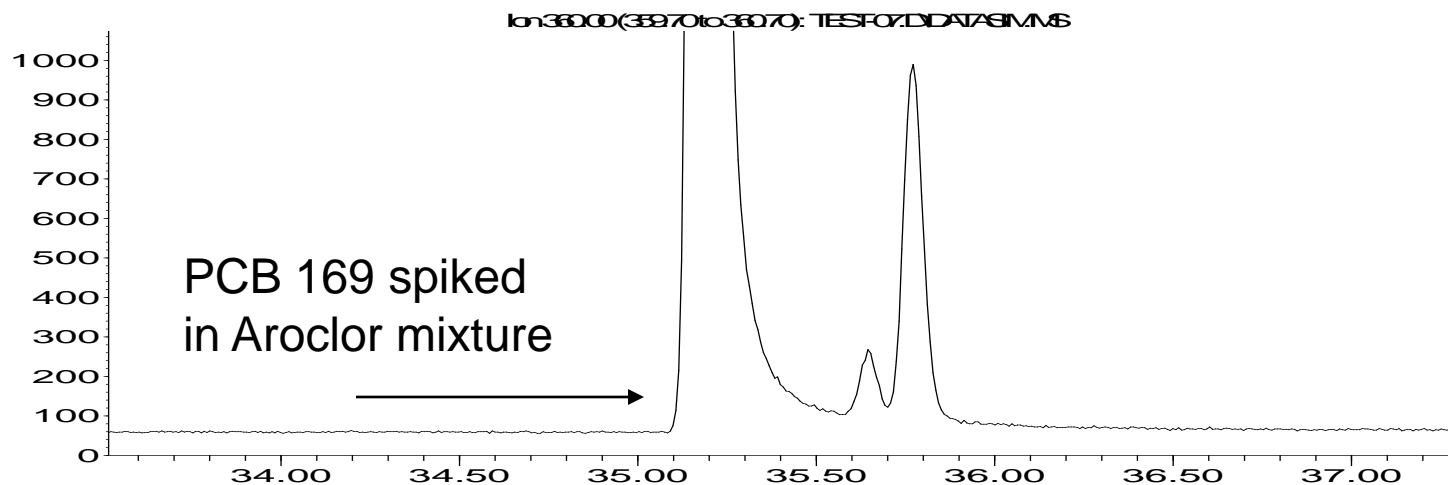
Time



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# PCB analysis by 2DGC-MSD: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



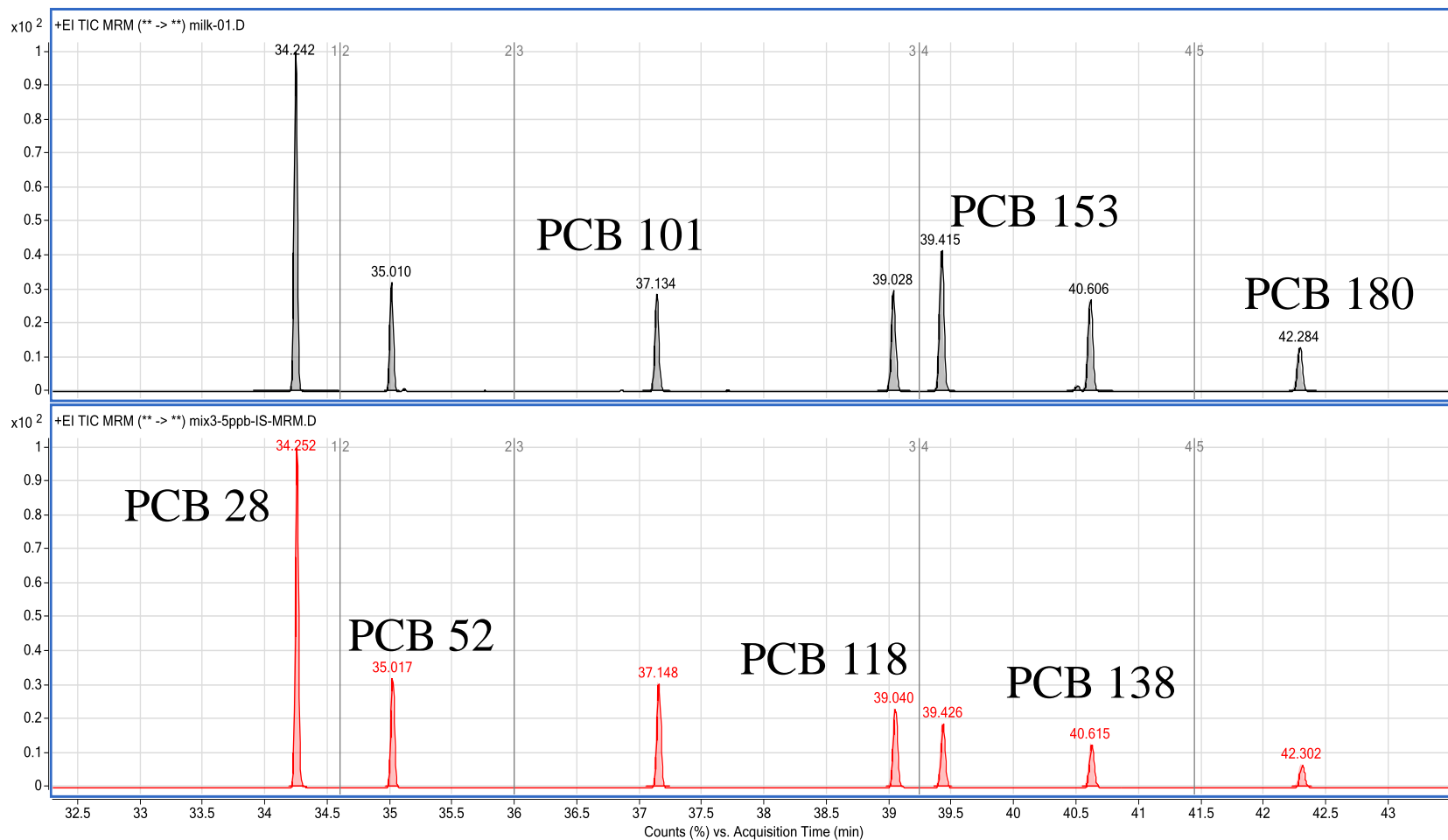
Time-->



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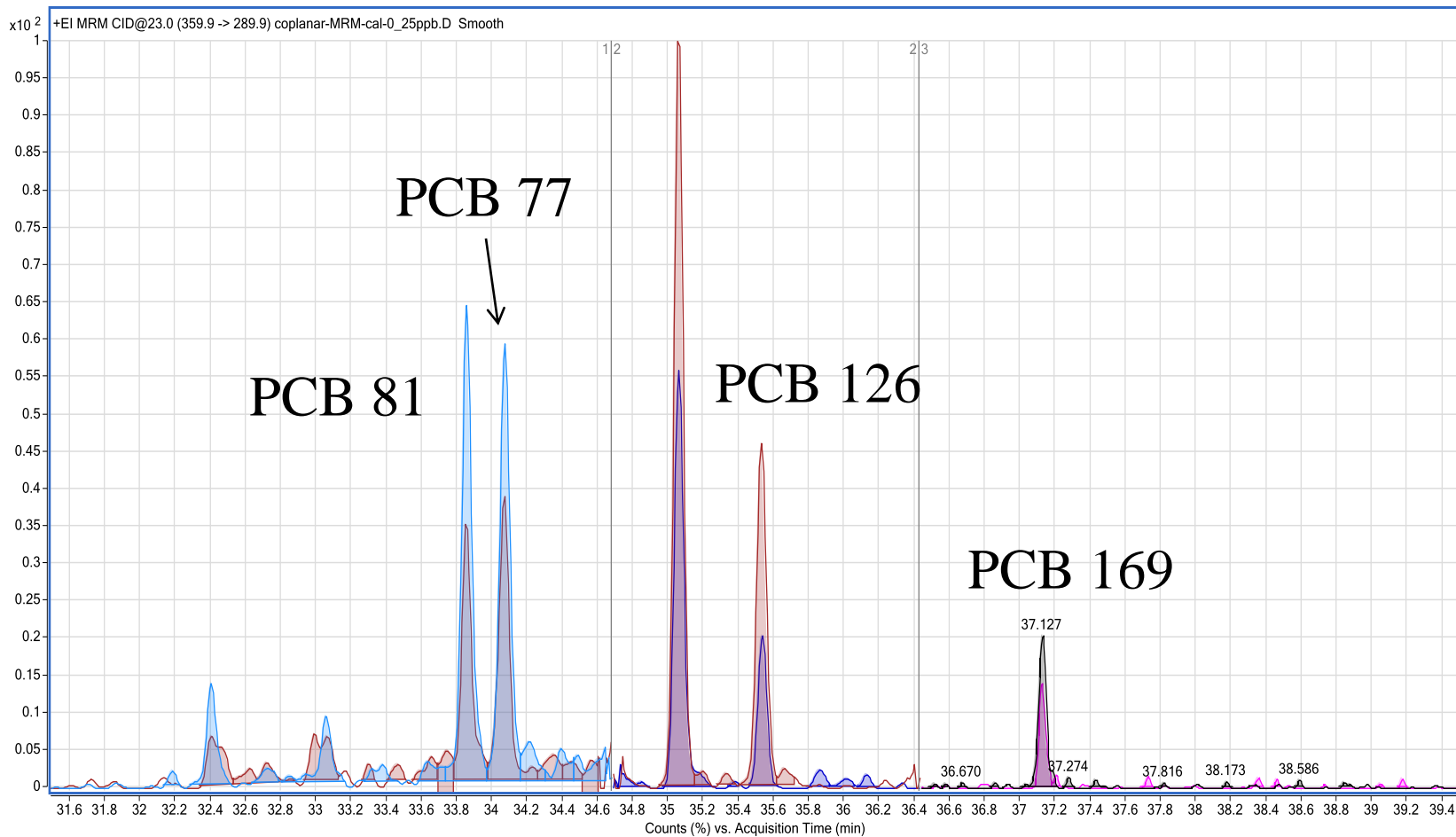
# PCB analysis in milk powder by 2DGC-QQQ: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



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# Coplanar PCBs 0.25 $\mu\text{g}/\mu\text{L}$

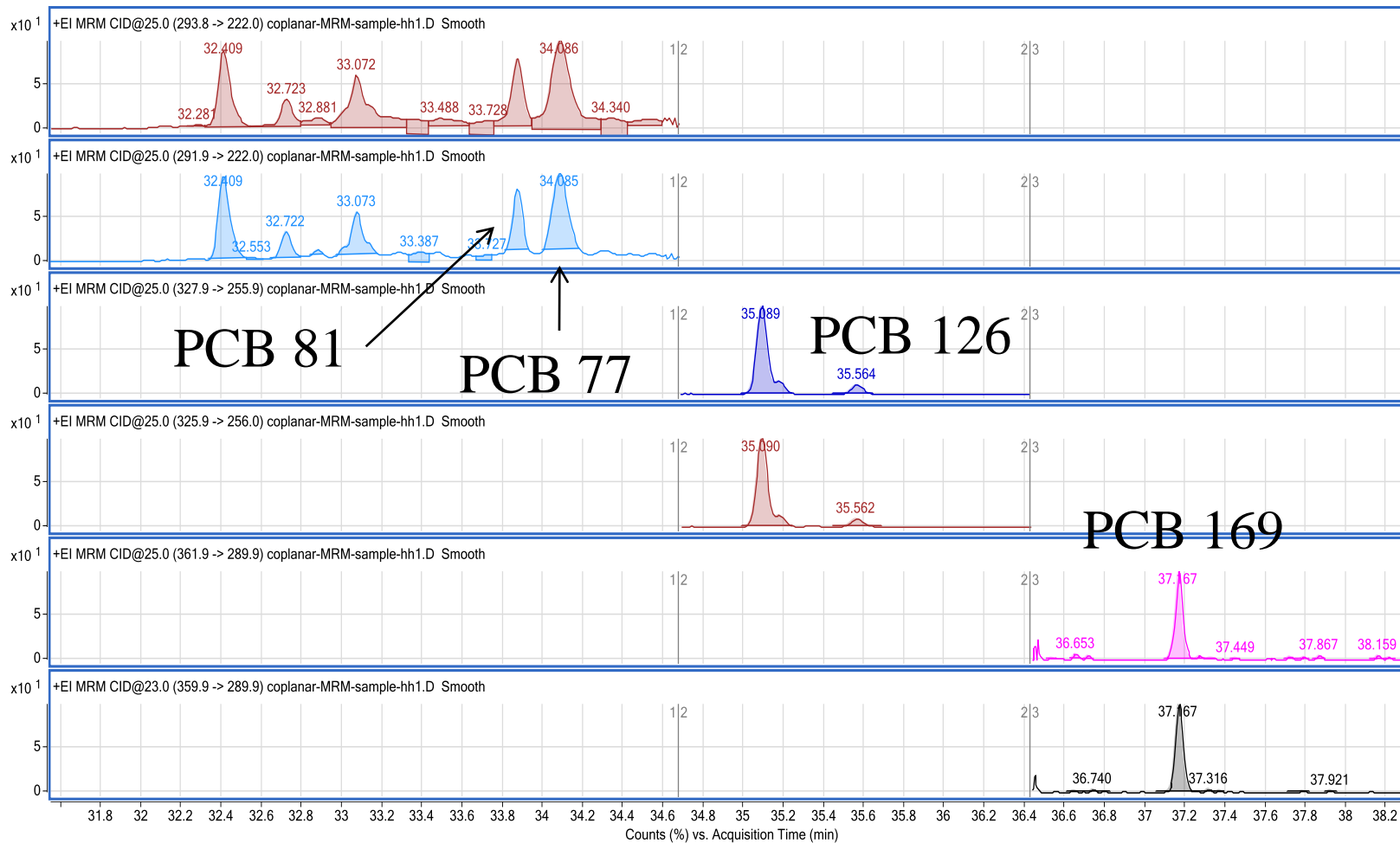


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# Fish (Chub) extract



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# Agilent's new 5975T Transportable GCMS

## Laboratory quality performance GCMS for out-of-laboratory applications



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