

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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Agilent Low Thermal Mass (LTM) module

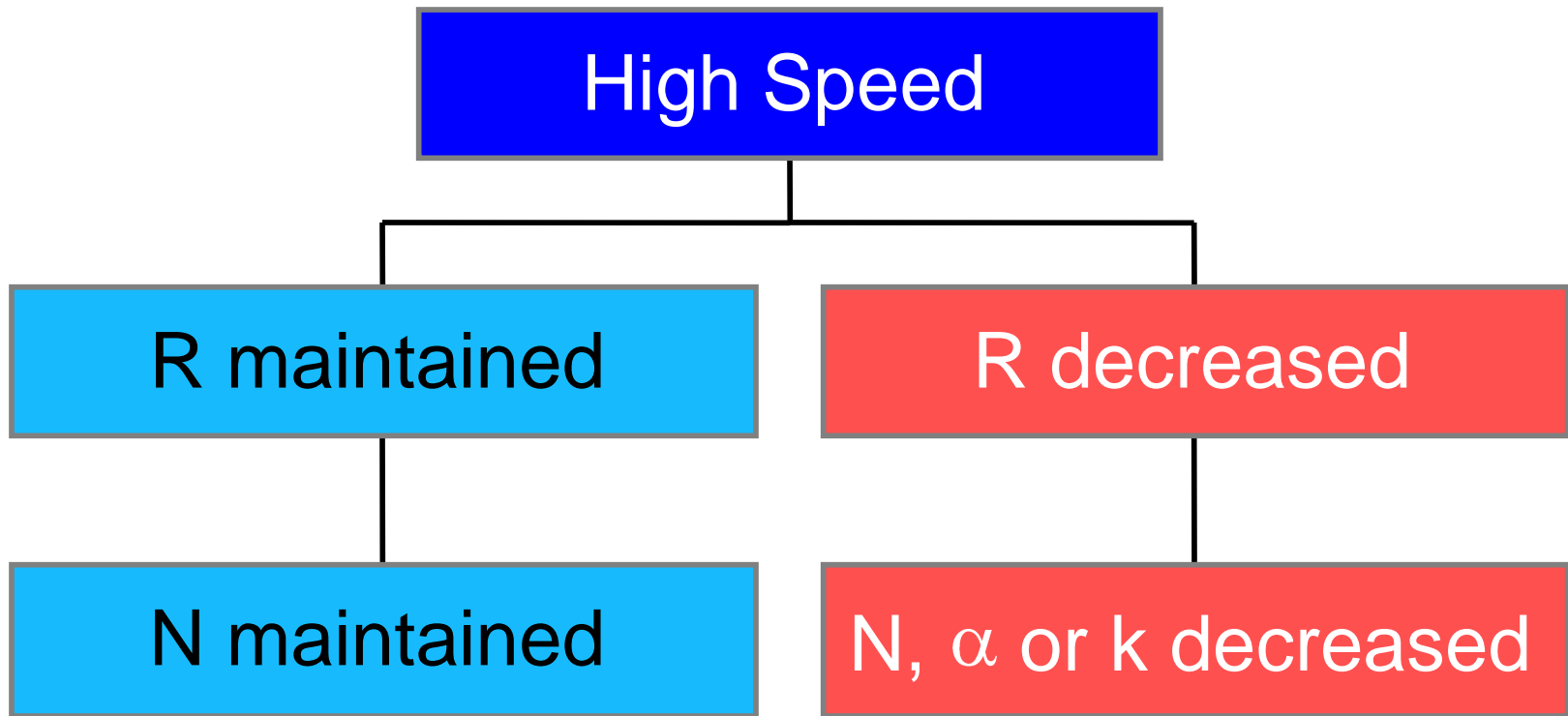
- Fast GC analysis
(1200 C/min)
- Independent column temperature control
- of up to 4 different columns



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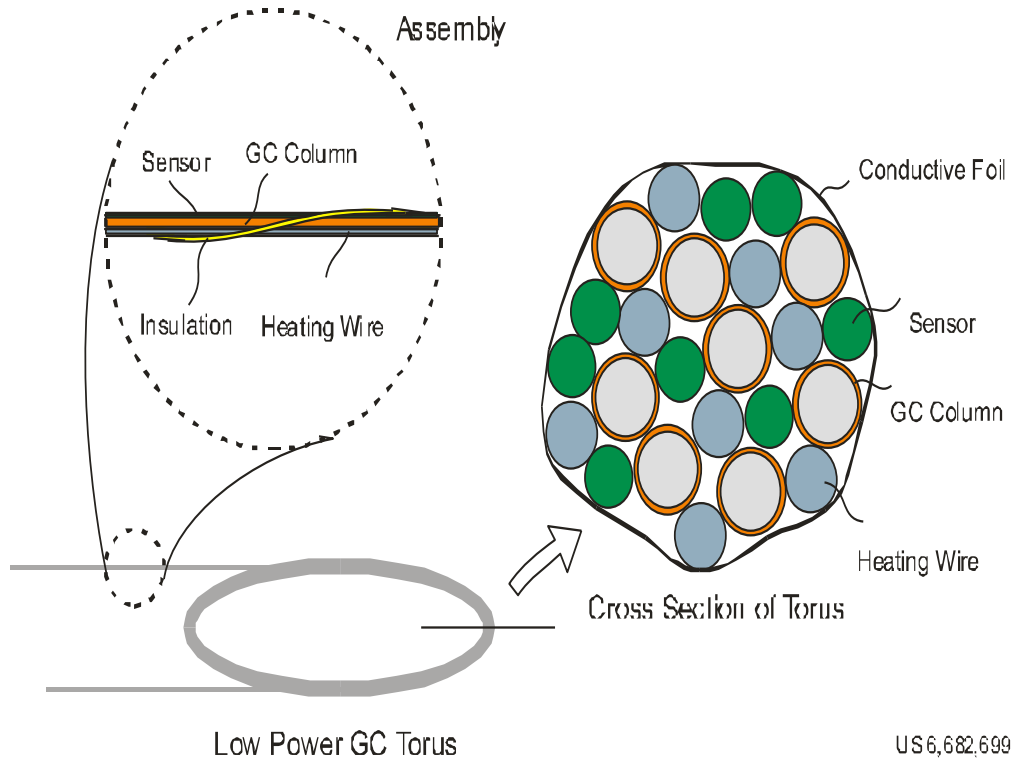
High Speed CGC



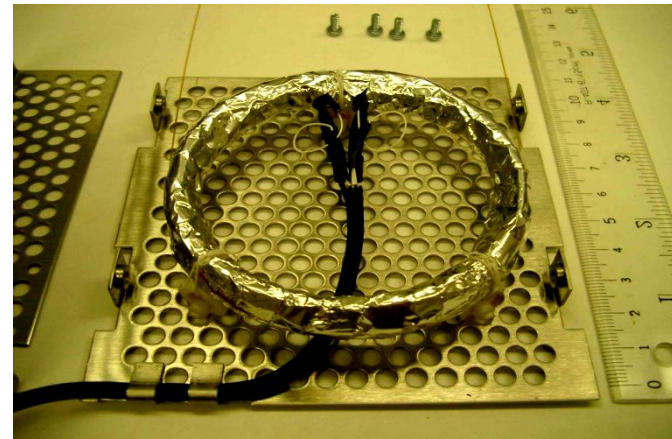
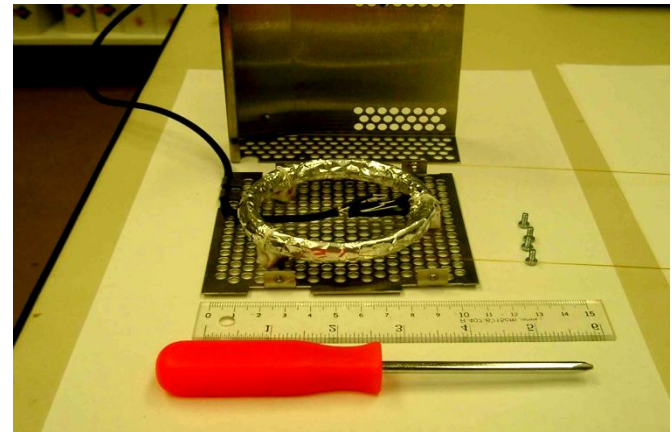
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Low Thermal Mass GC Technology



US 6,682,699



Heating rates: up to 1000 C/min !!



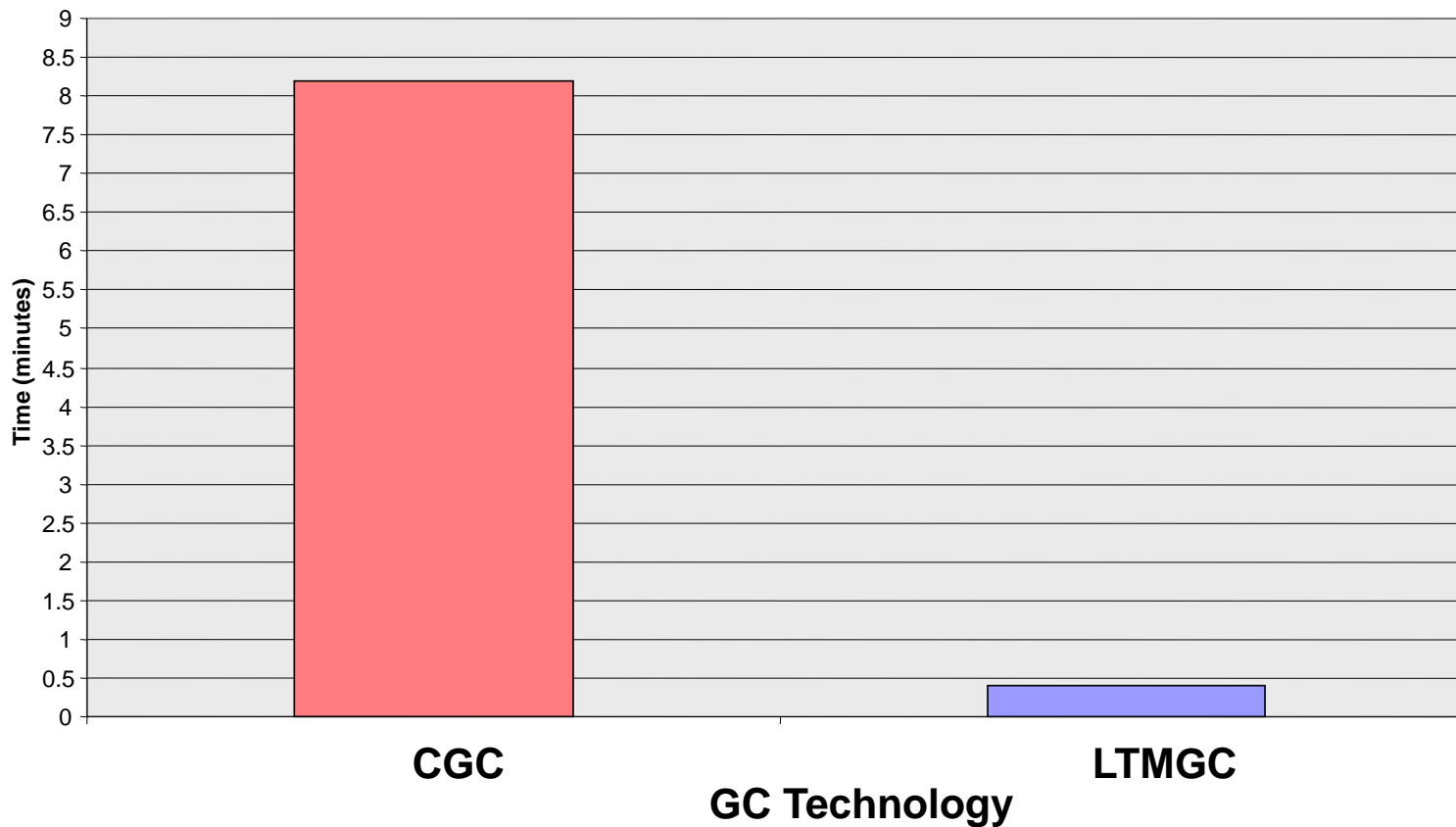
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Cool Down Time Comparison

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

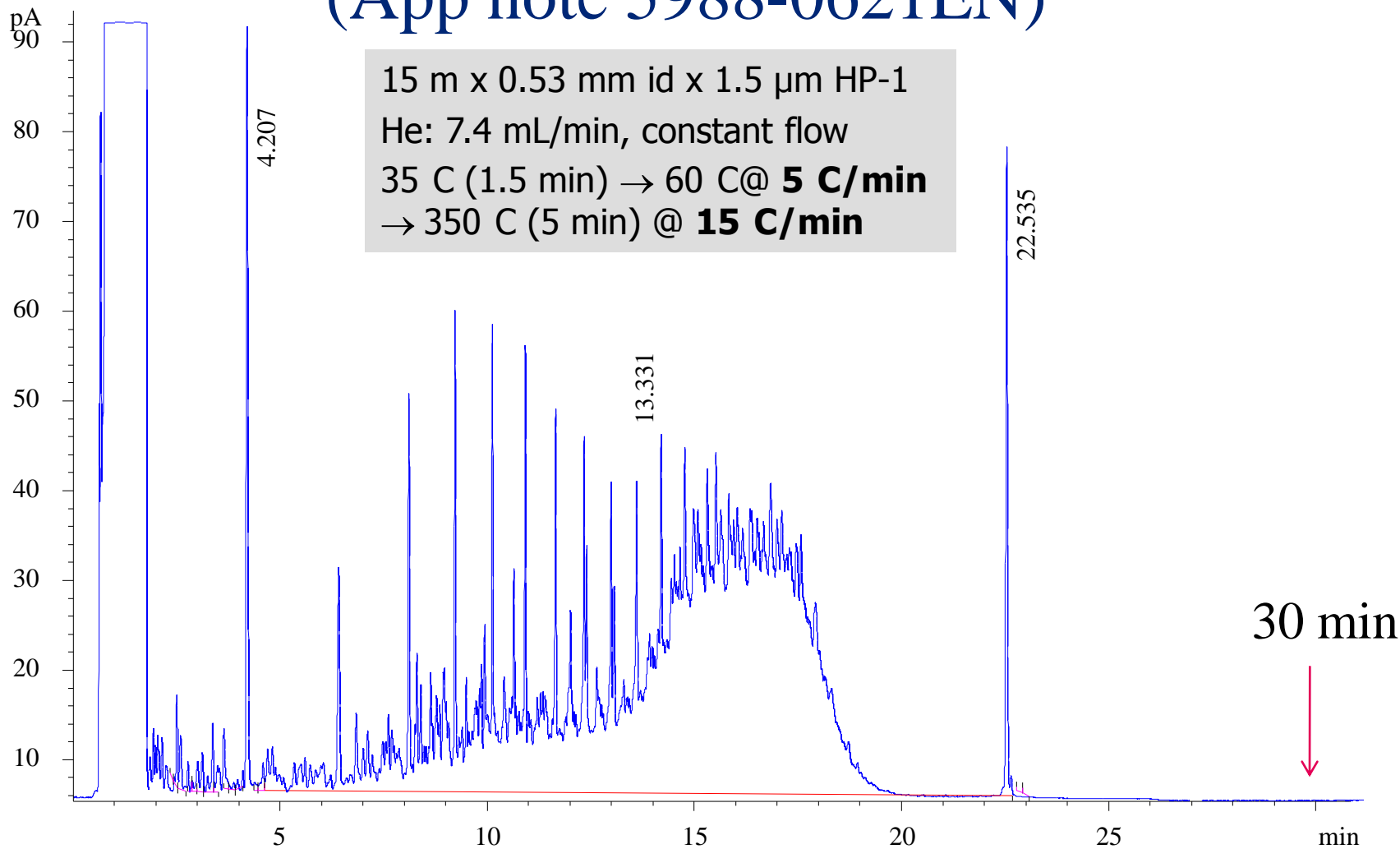
LTMGC: 5 meter, 0.1 mm id, 0.12 micron DB-1



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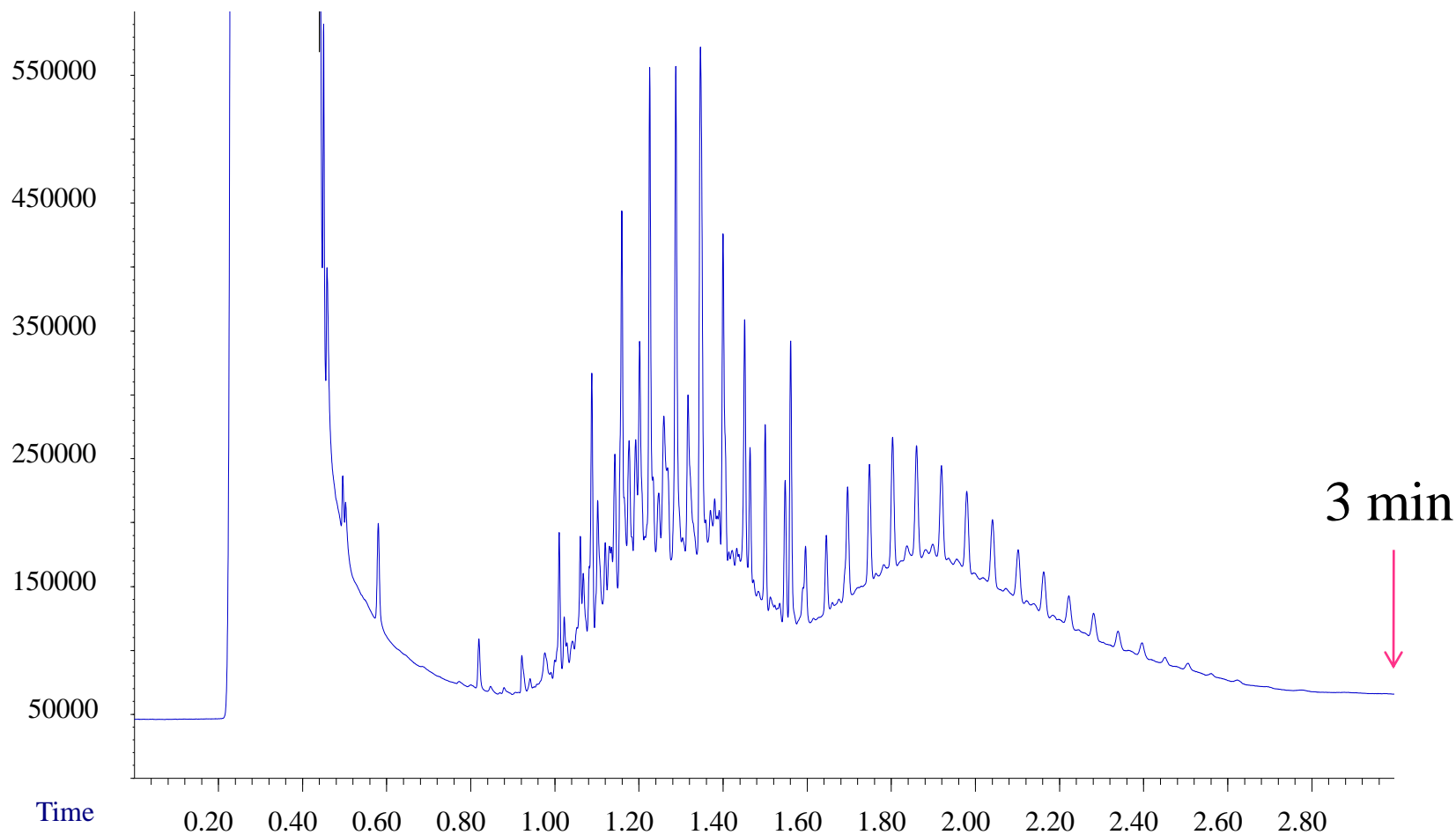
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Typical Hydrocarbon Index Analysis by GC (App note 5988-0621EN)



Mineral Oil Calibration 100 mg/L

Response



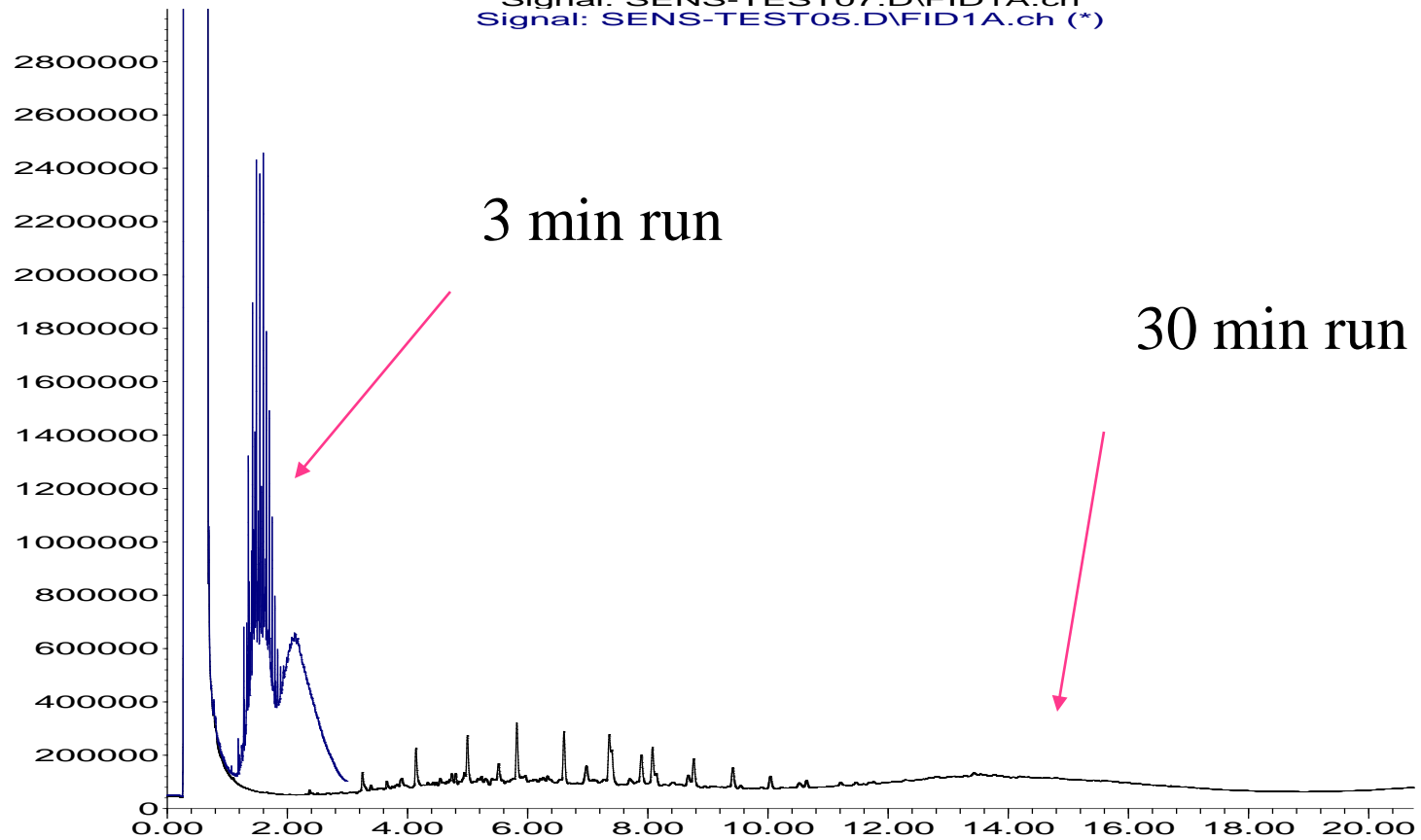
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Extra: sensitivity gain

Response_

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



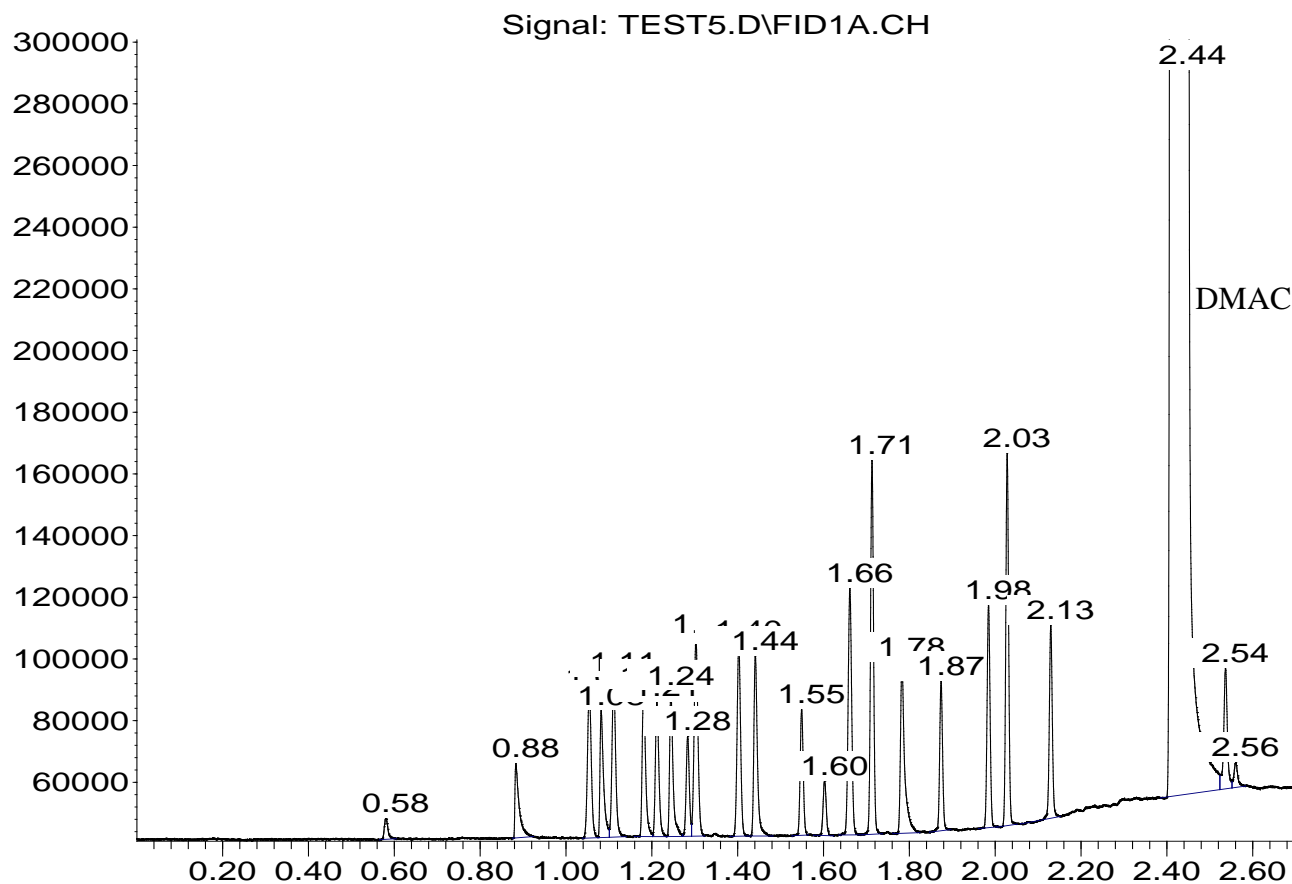
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Residual solvent analysis

LTM method (2.6 min)

Response_



Time

2.6 min

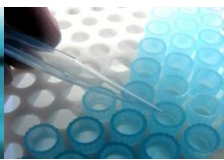
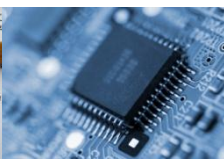


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Repeatability: Retention Times and Area RSD

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 μ 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**)

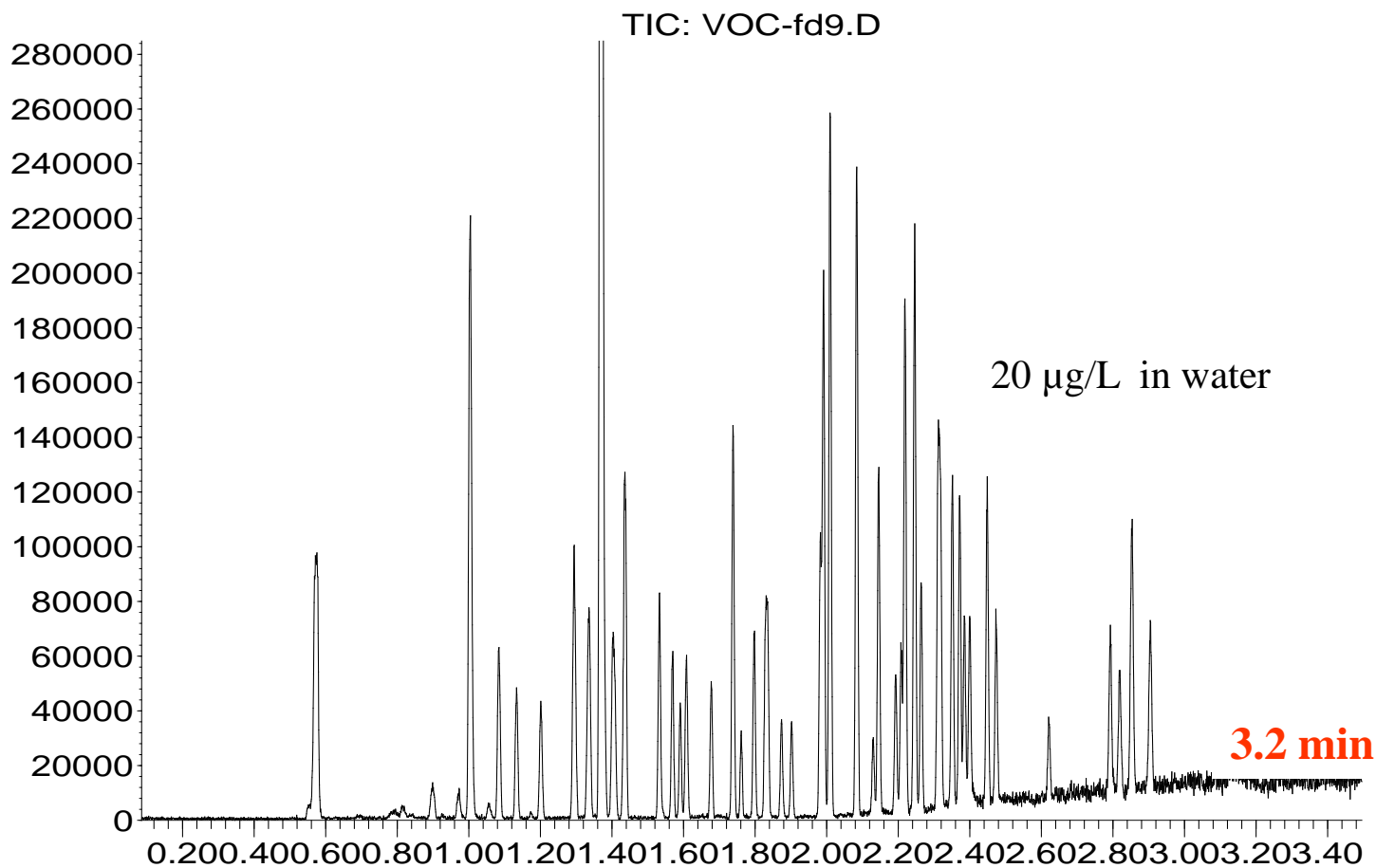


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Ultra fast SHS-GC-MSD: EPA Volatiles

Abundance



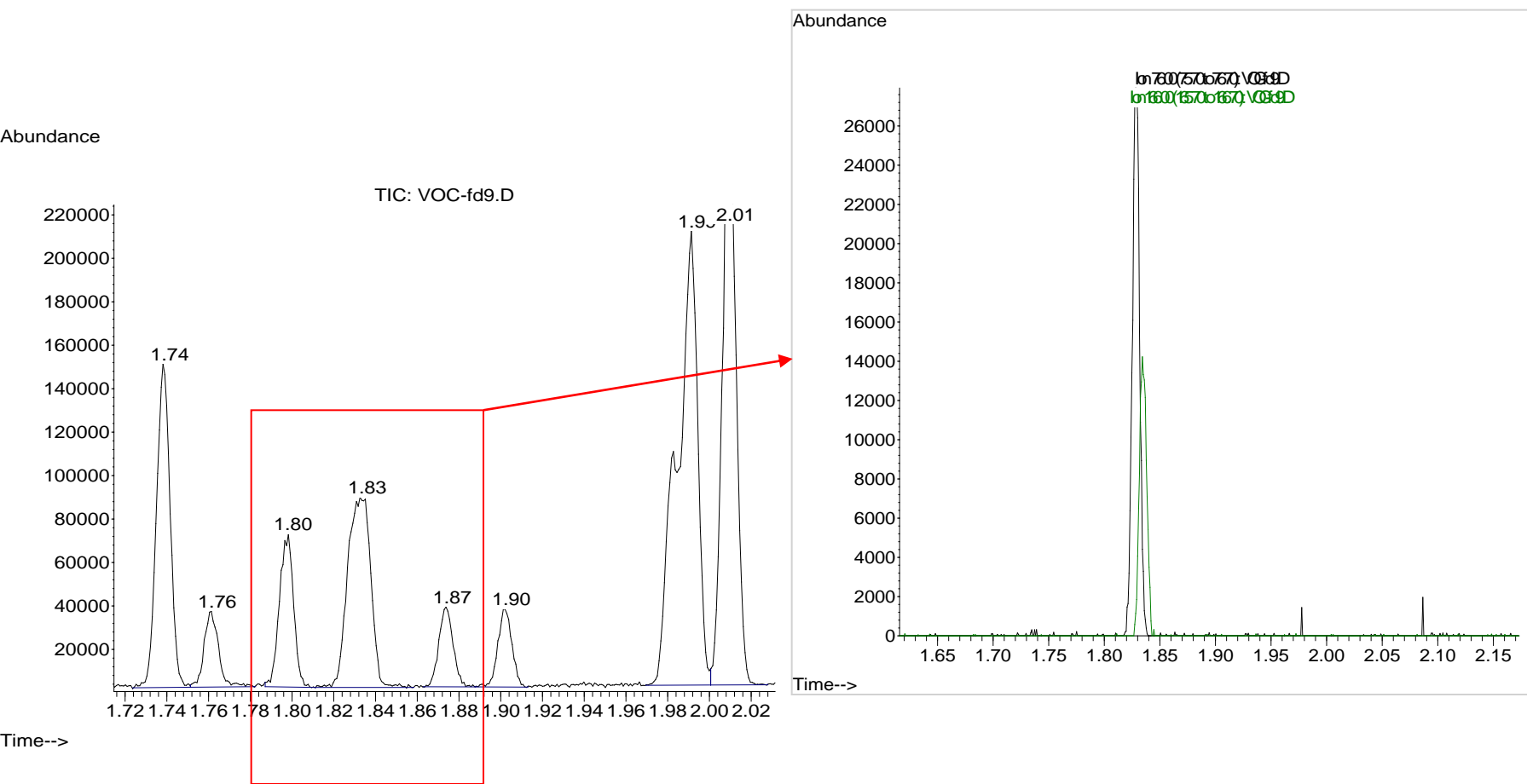
Time-->



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'Deconvolution' of critical pairs



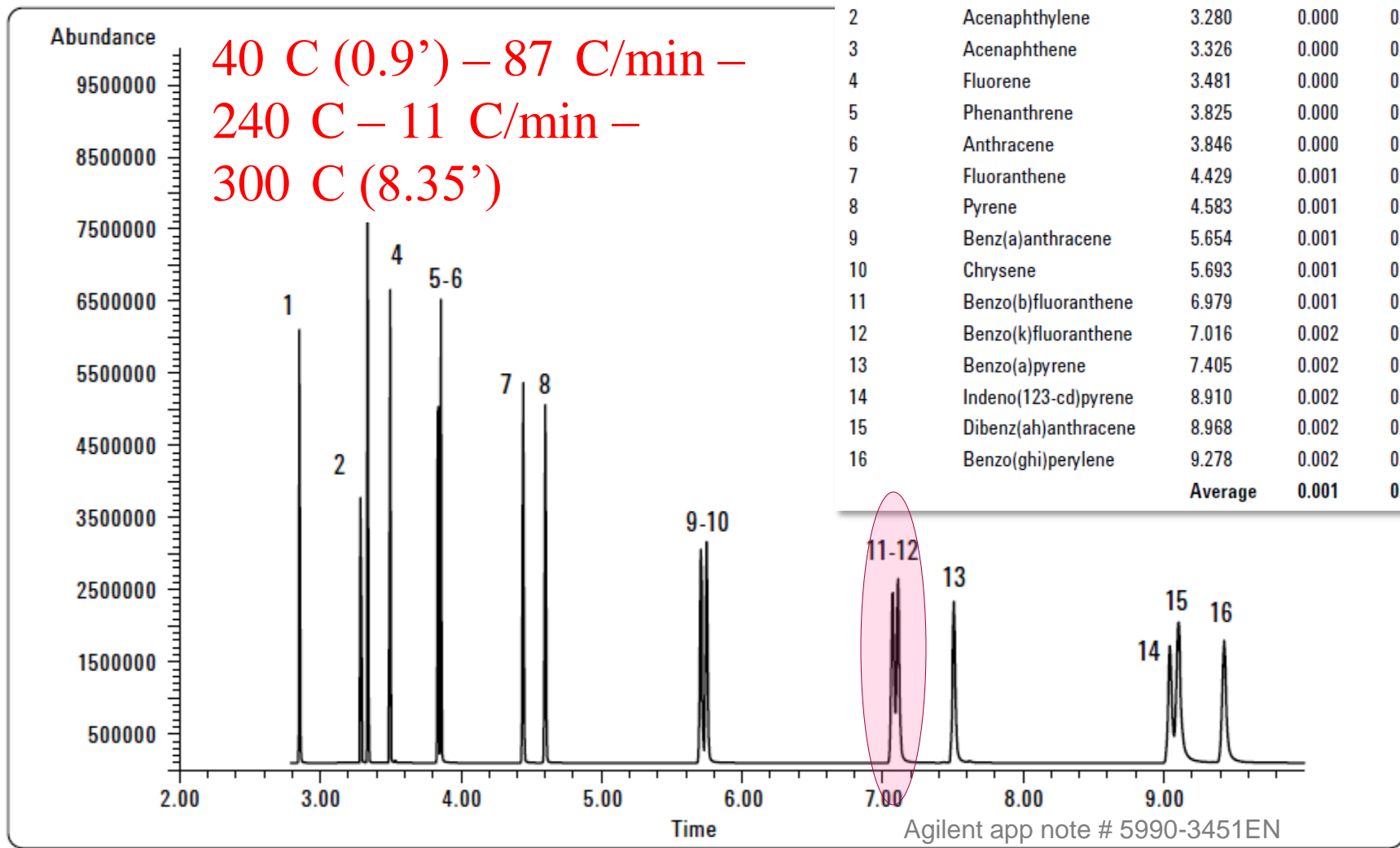
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Fast LTM analysis of PAH

20 m x 180 μm ID x 0.18 μm DB-5MS

Order of elution	Compound name	Mean t_R (min)	σ	% 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
	Average	0.001	0.01	0.01



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

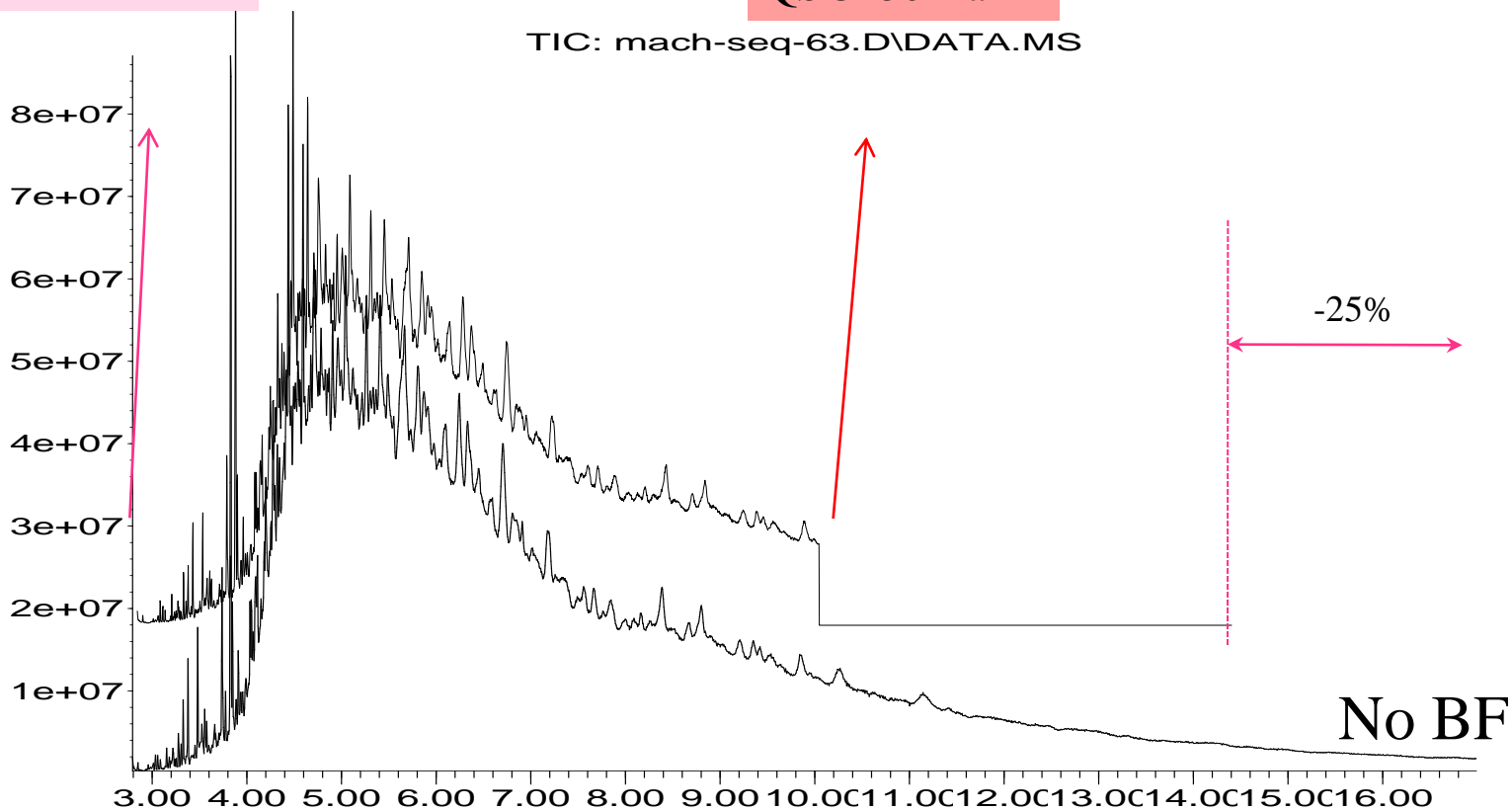
INJ @ 213-392kPa

QS@28 kPa

INJ @ 10kPa

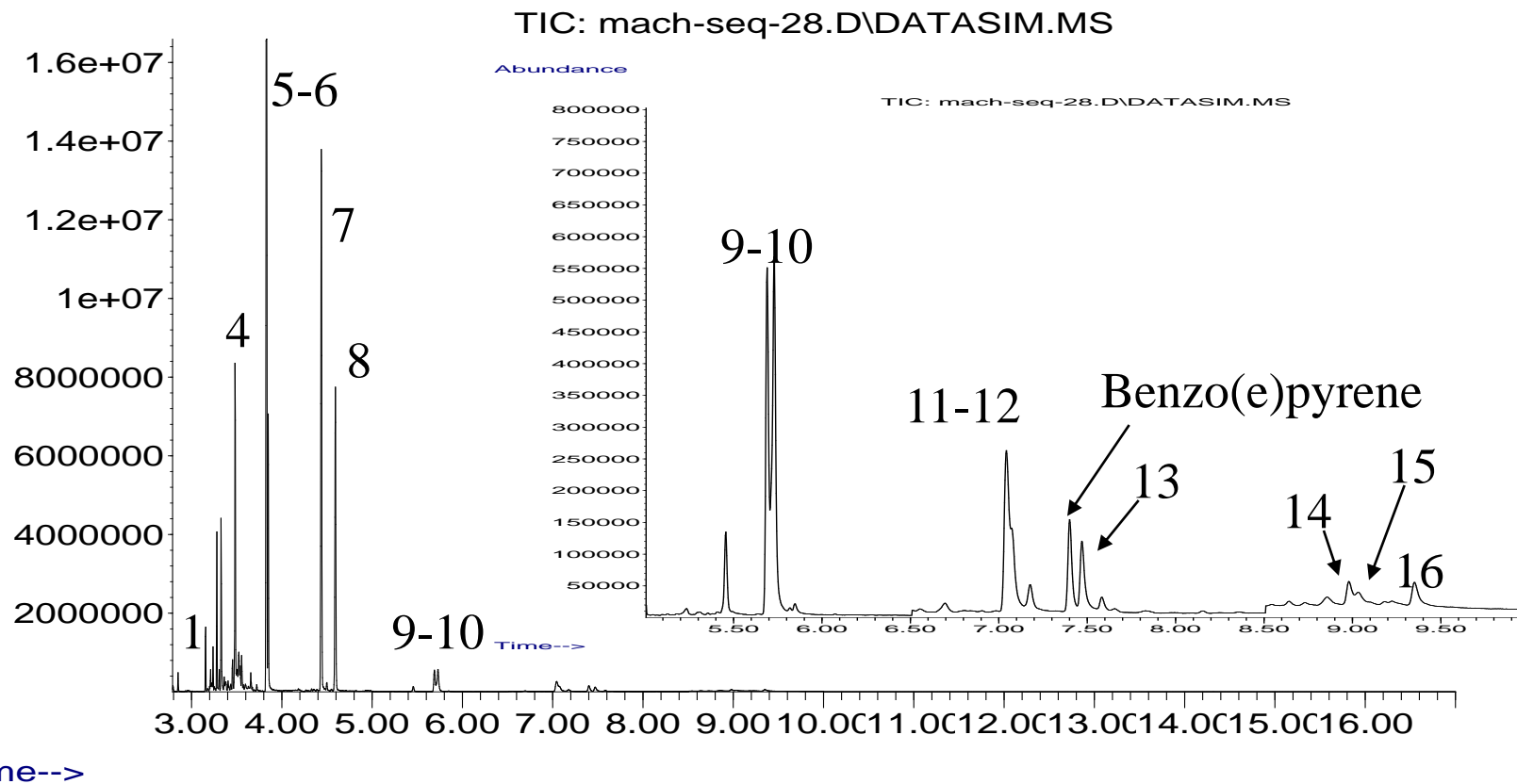
QS@250kPa

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



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

Abundance



Time-->

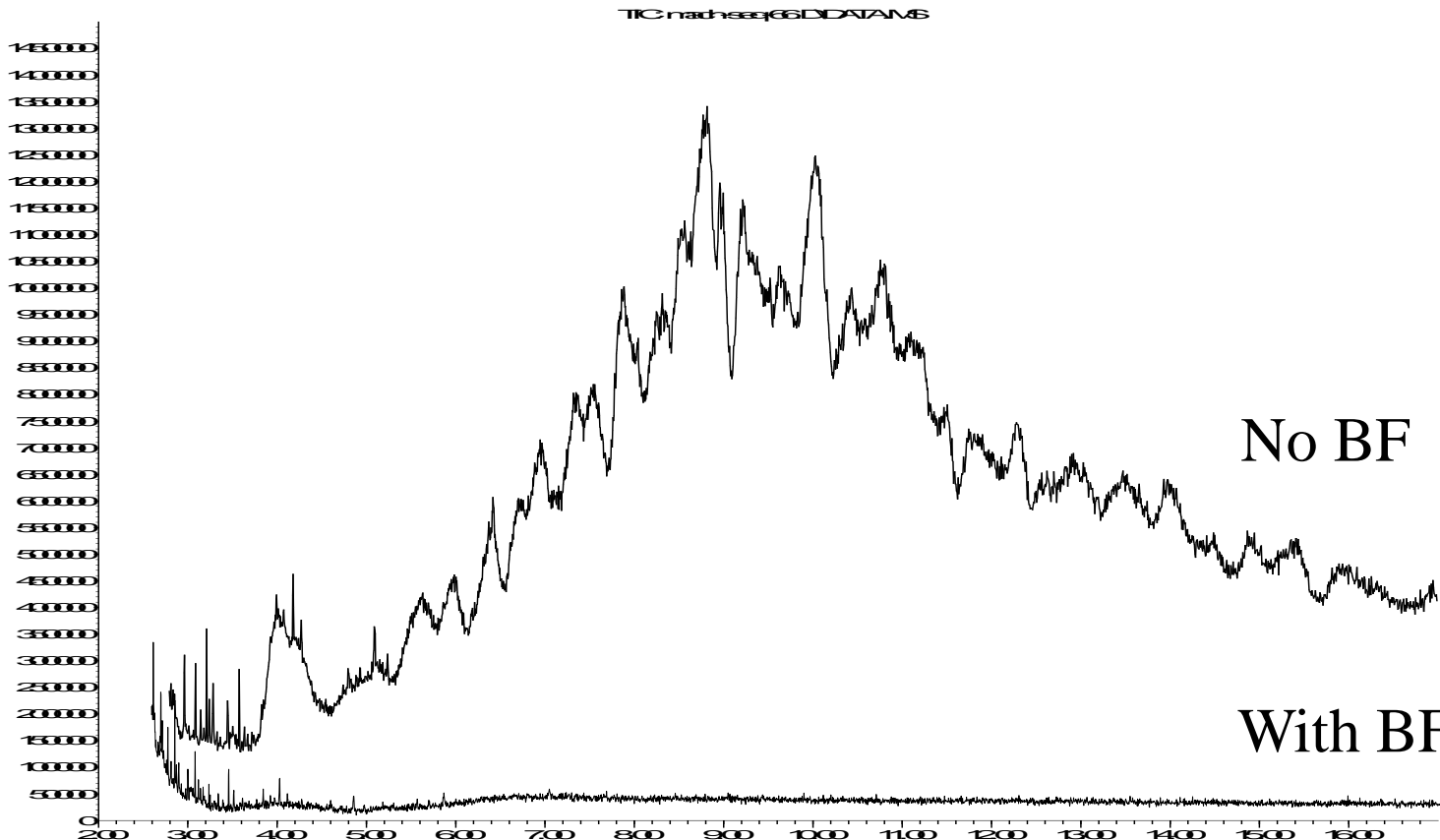


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Blank run after soil extract - scan trace

Abundance



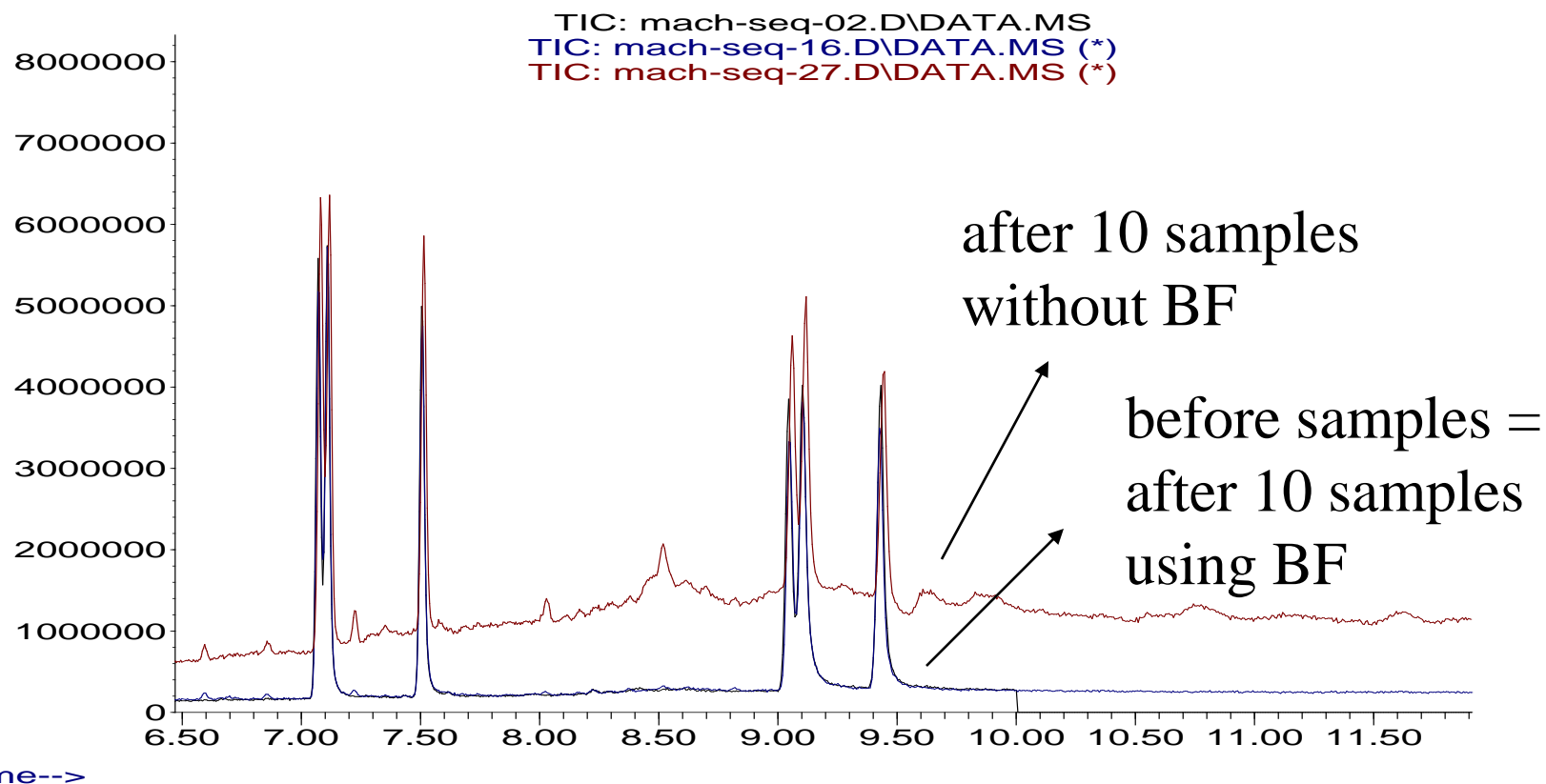
With BF

Time



Check-out sample after soil extract sequence scan trace

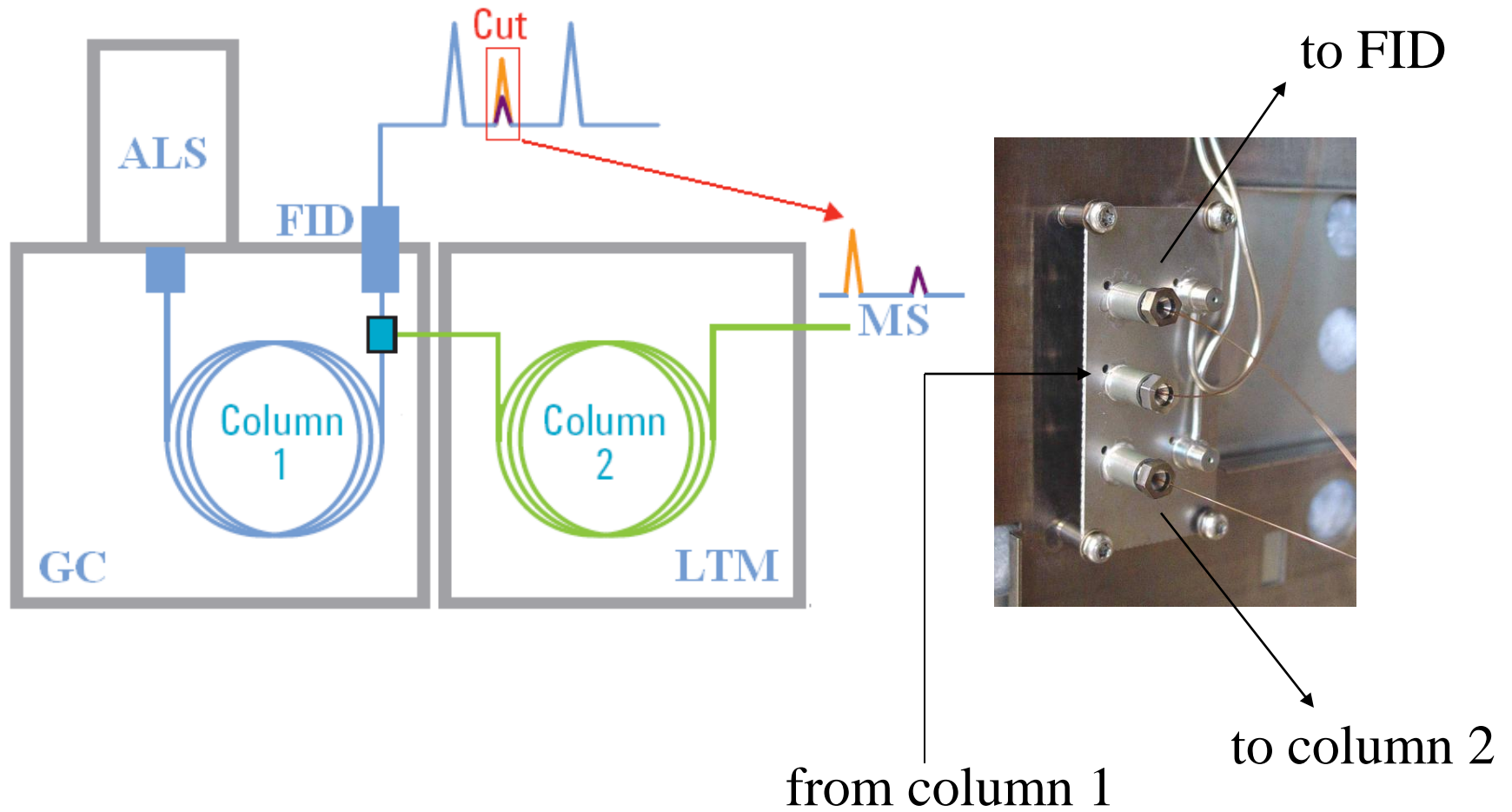
Abundance



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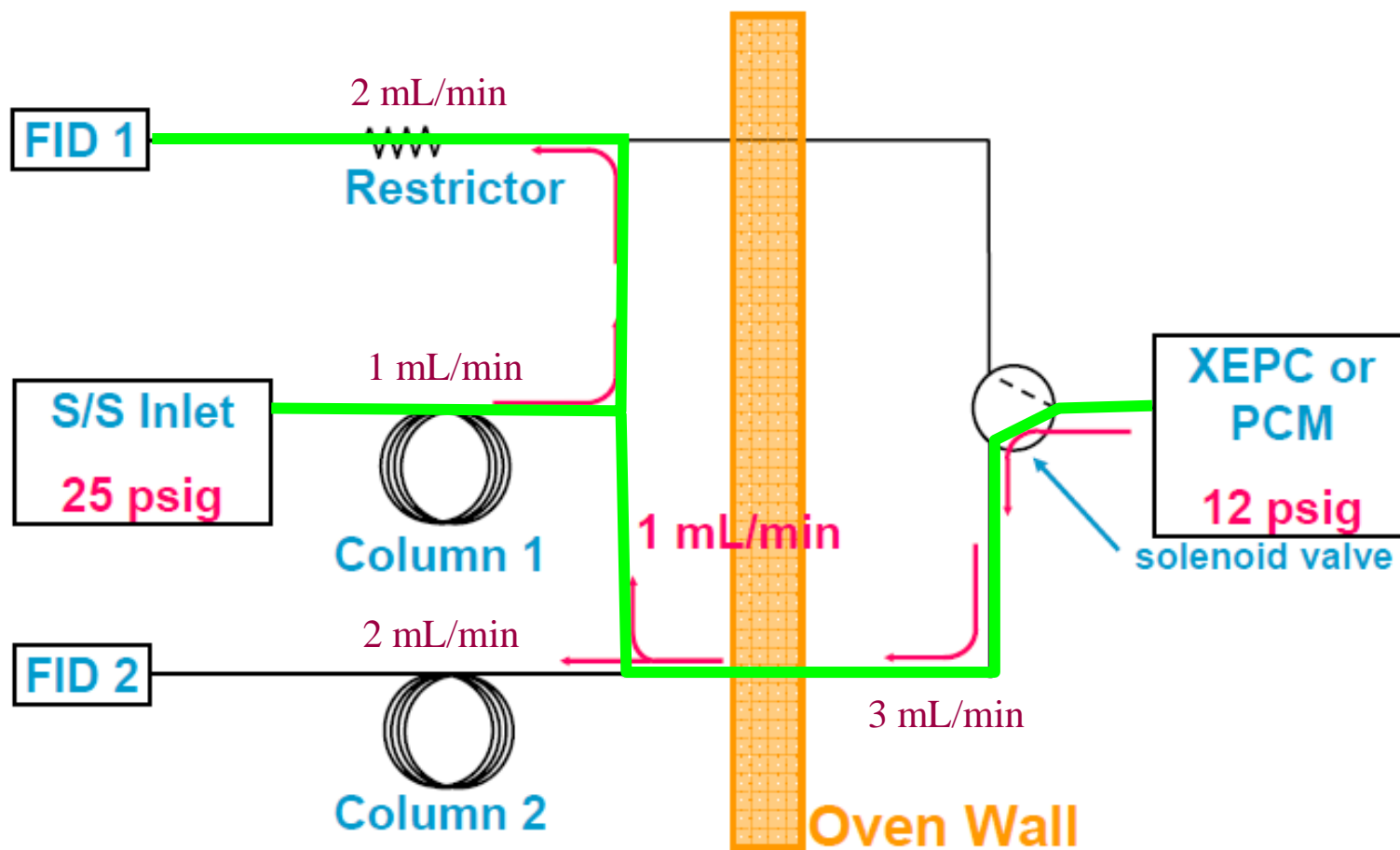
CFT based 2D-GC



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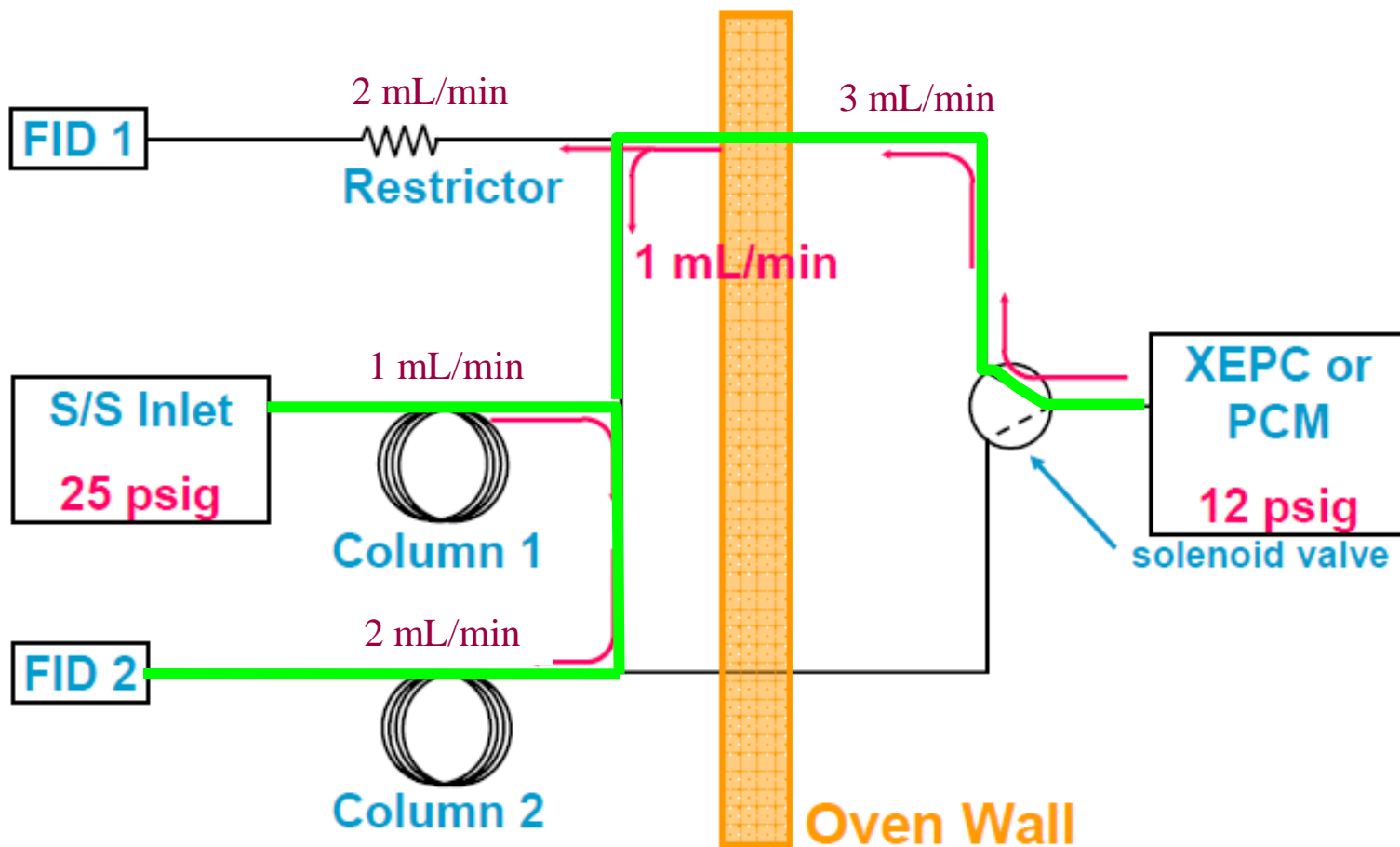
Deans Switch principle – 1D analysis (FID1)



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

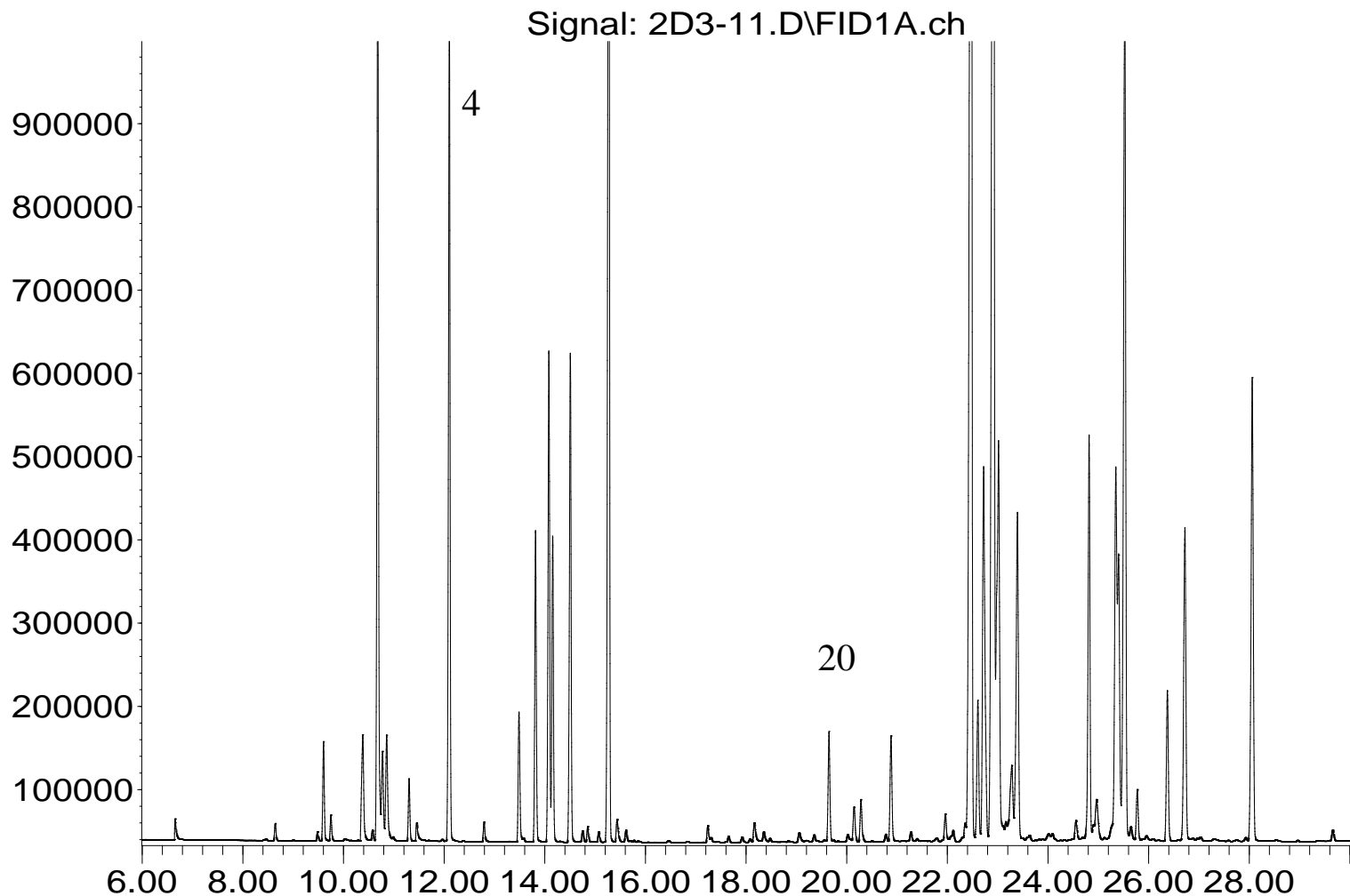


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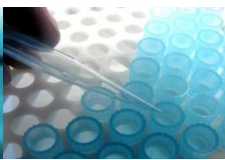
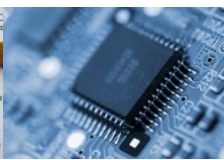
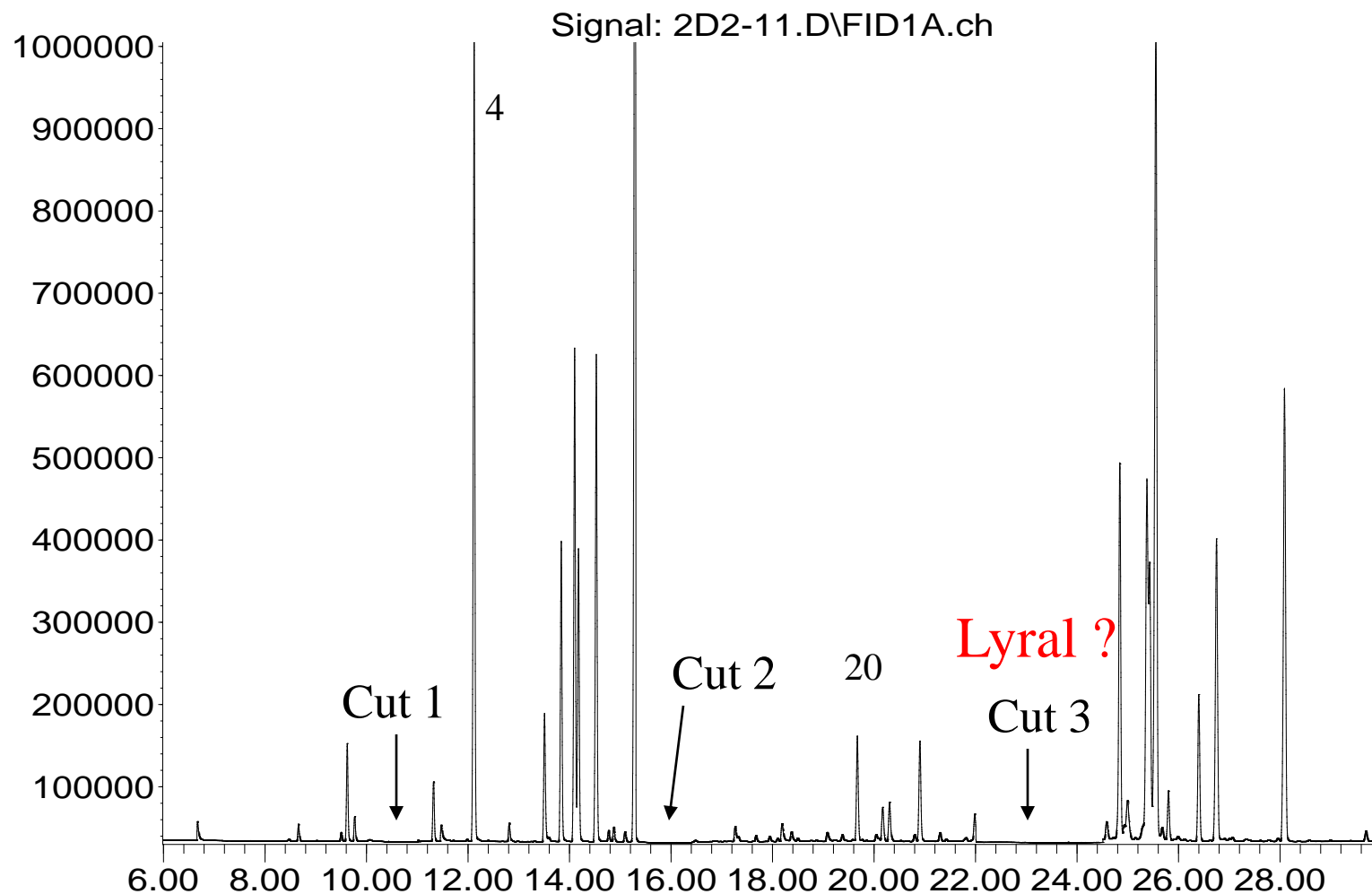
Suspected Flavor and Fragrance Allergens in Perfumes

Abundance



Suspected Flavor and Fragrance Allergens in Perfumes

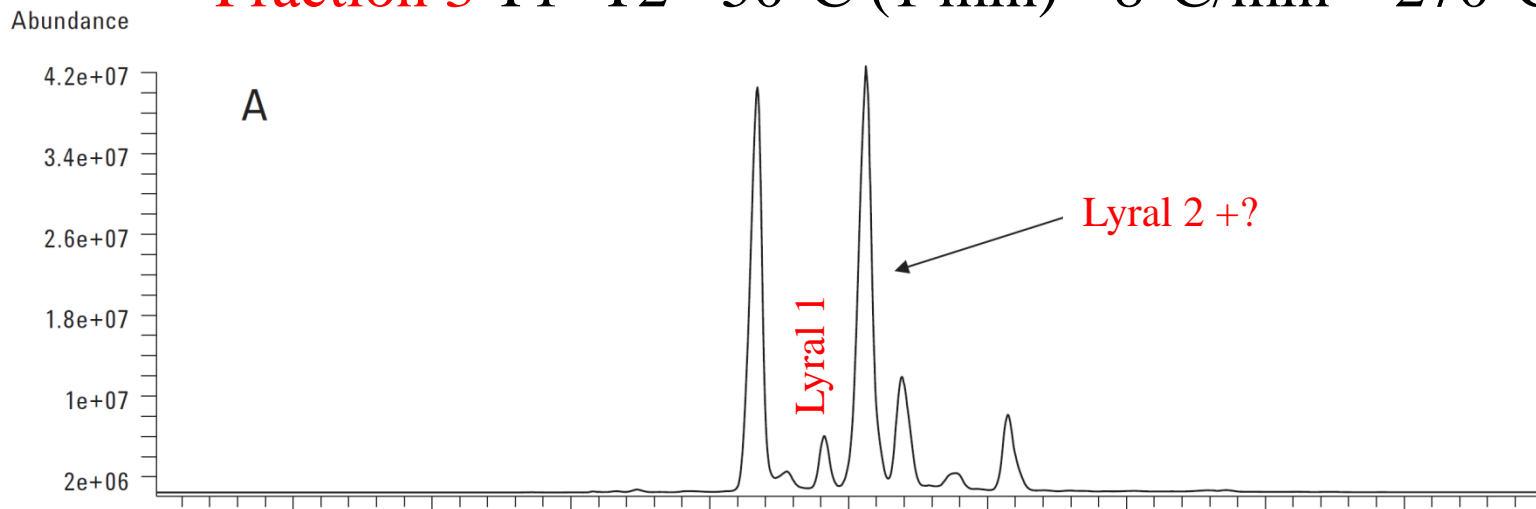
Abundance



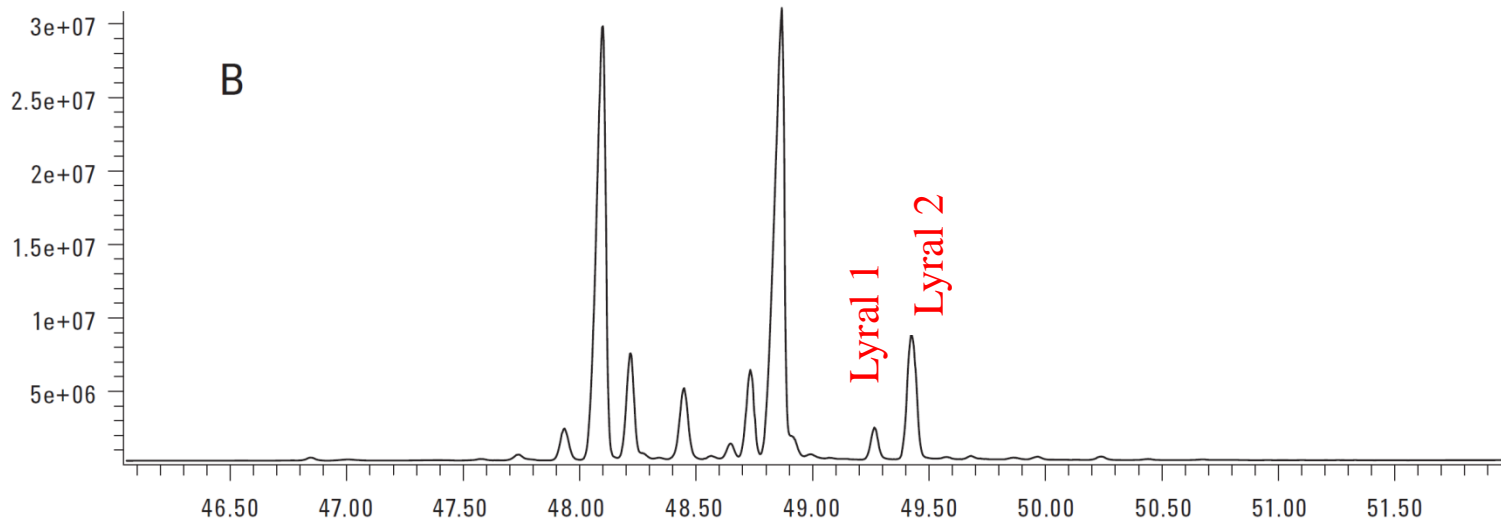
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Fraction 3 T1=T2= 50 C (1 min) - 8 C/min – 270 C



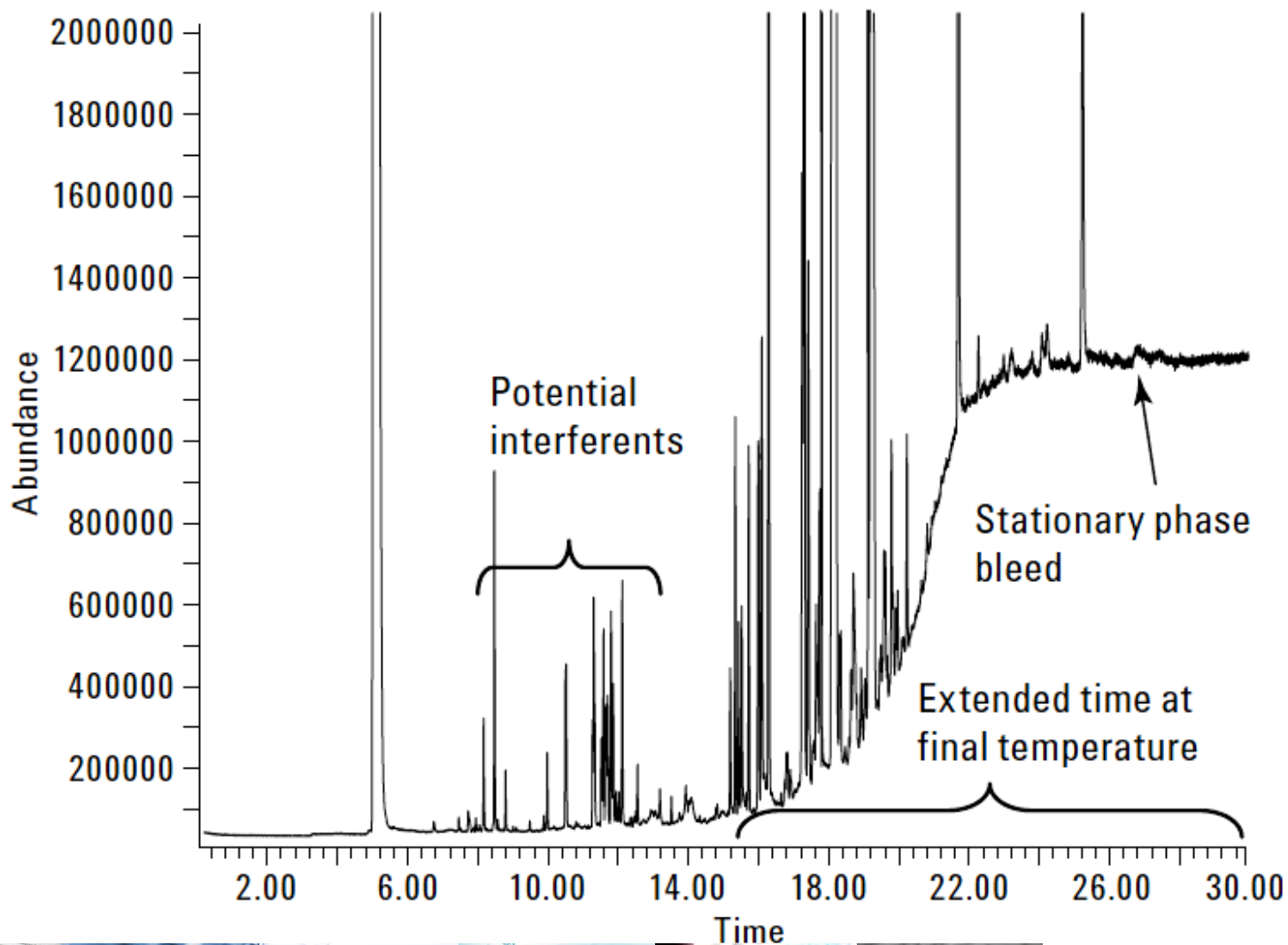
Fraction 3 T1≠T2; T2= 50 C (25 min) - 6 C/min - 250 C



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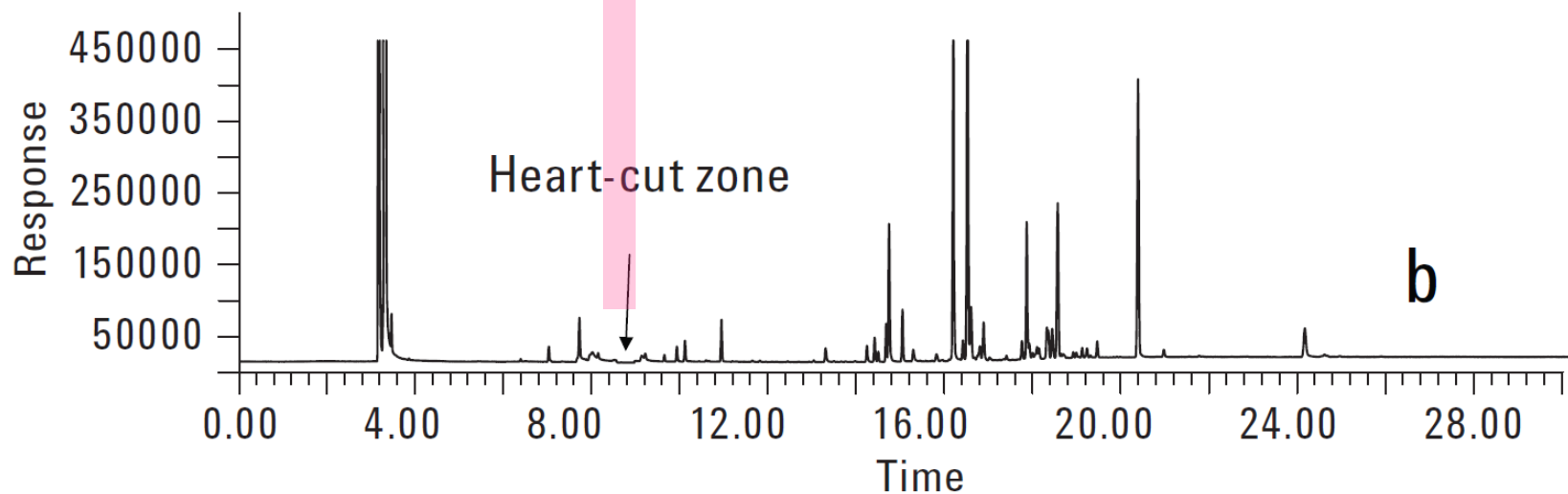
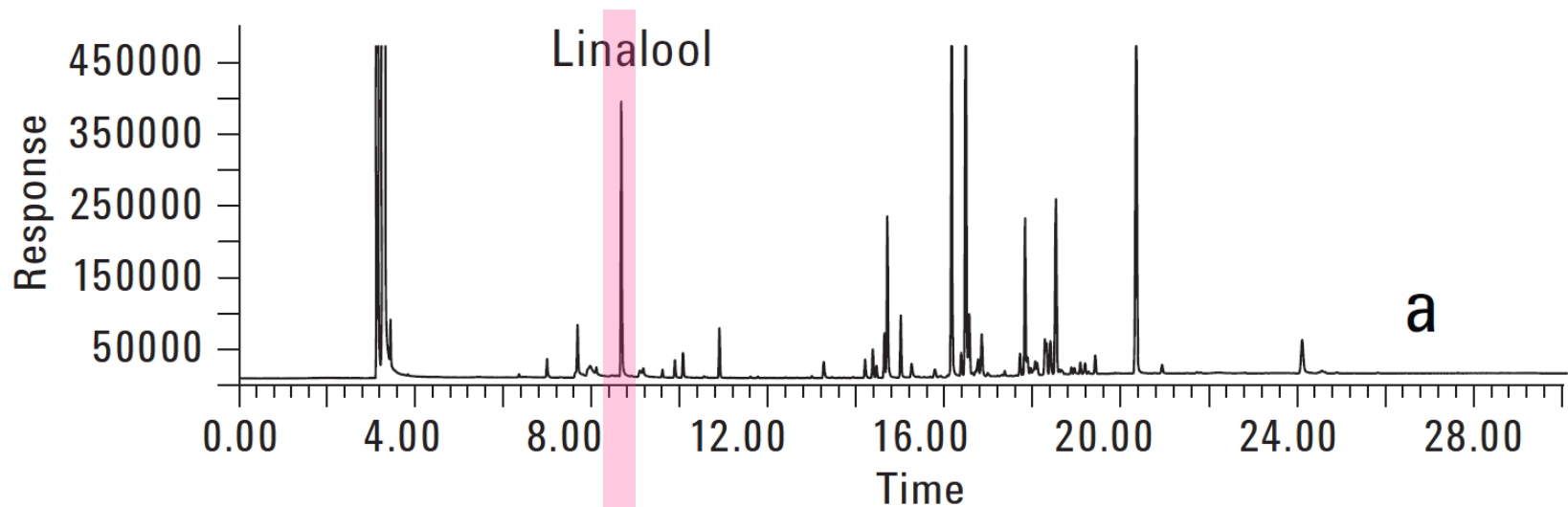
Linalool analysis in essential oil on chiral GC column (CycloDex-B)



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Linalool analysis in essential oil on ¹GC column (HP-1MS)

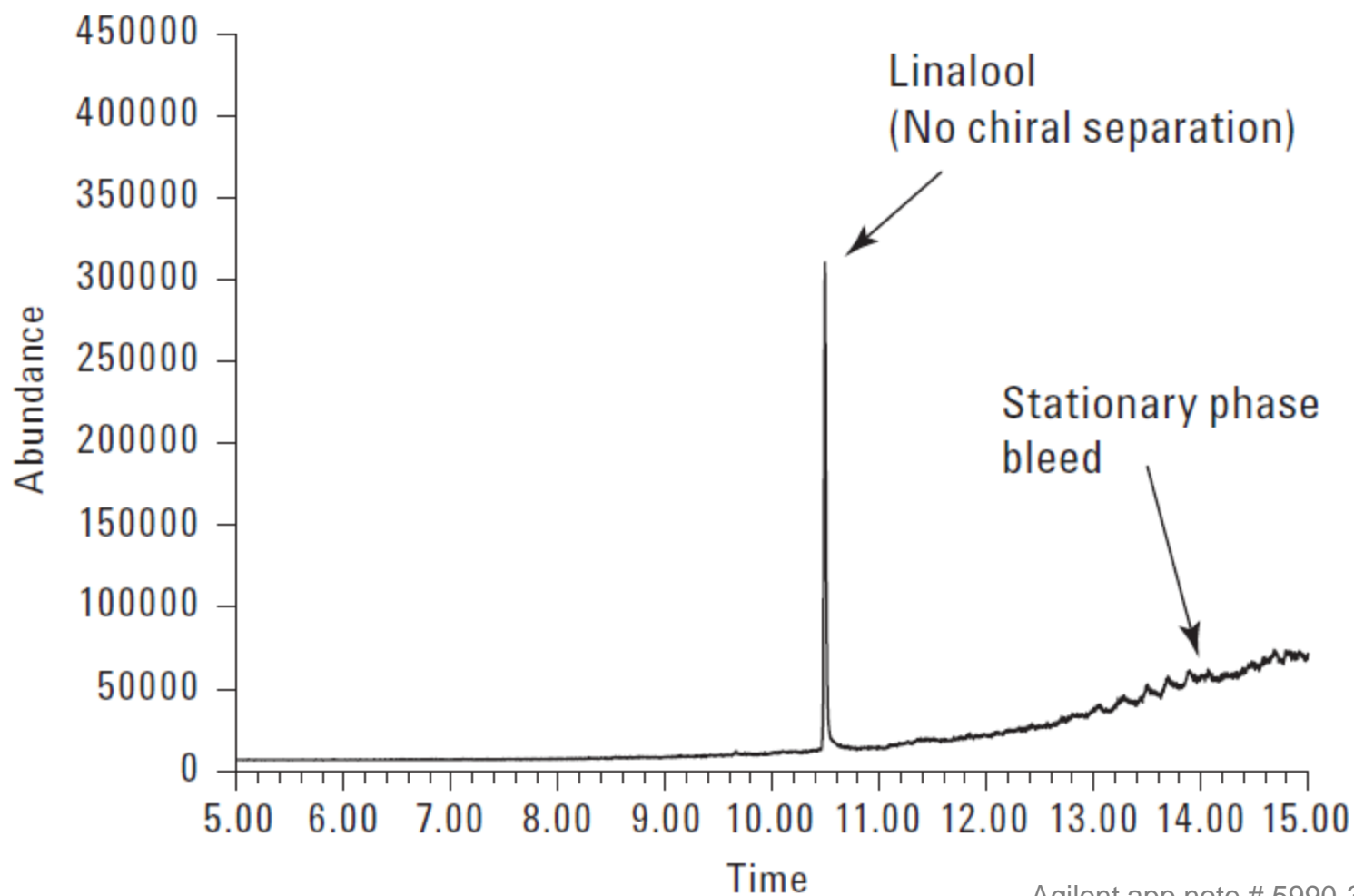


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2DGC analysis of fraction 8.5-8.9 min

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



Agilent app note # 5990-3428EN

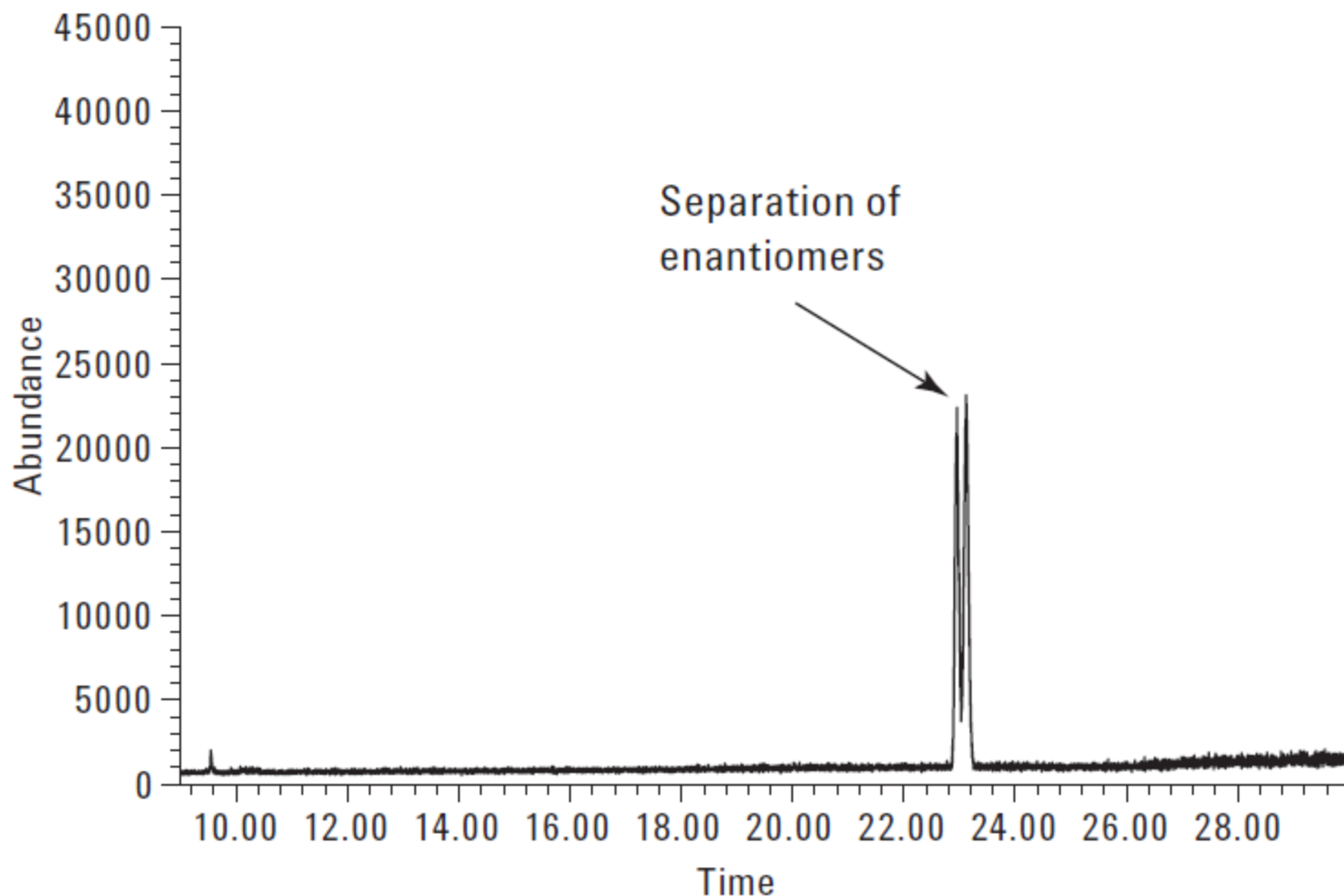


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2DGC analysis of fraction 8.5-8.9 min

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



Agilent app note # 5990-3428EN



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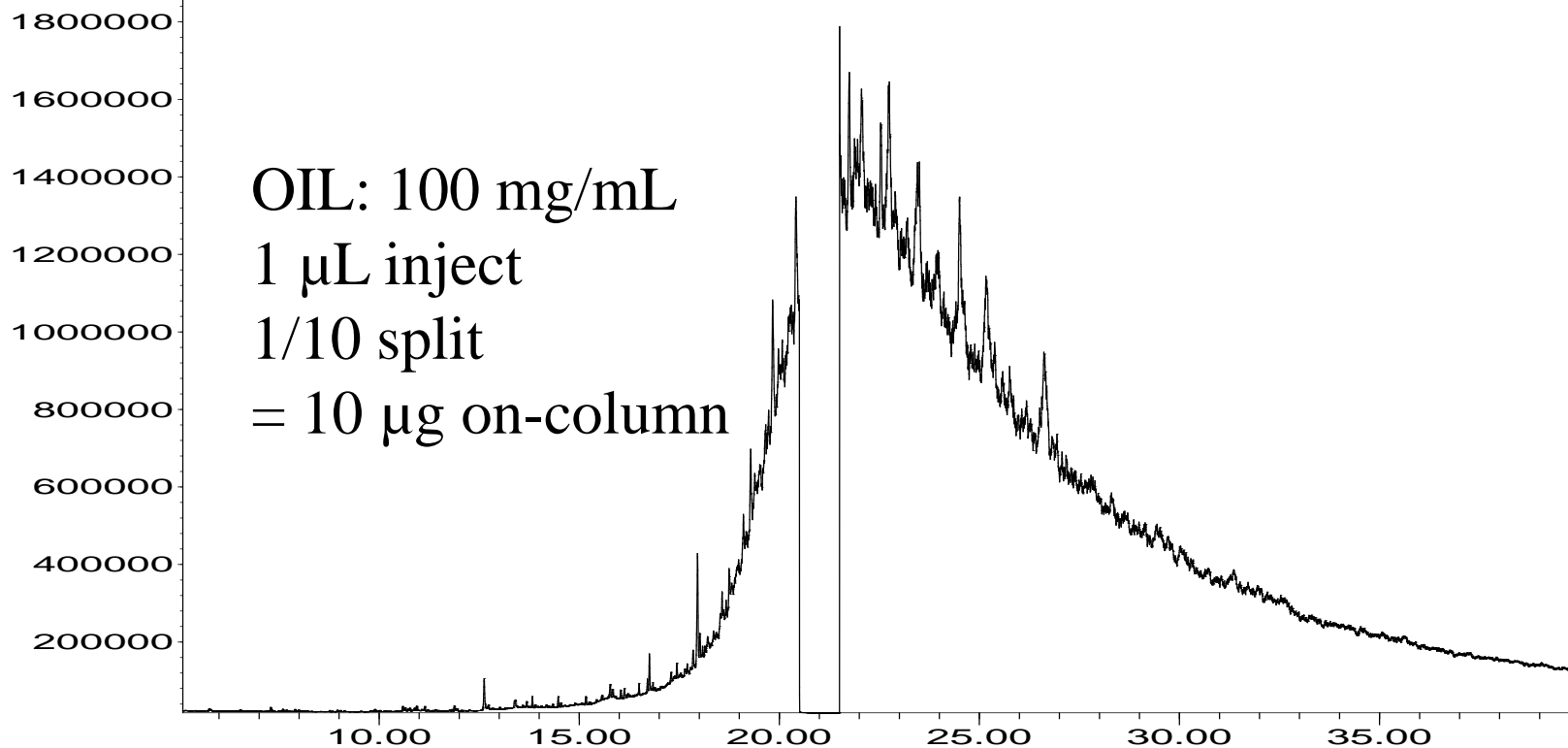
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Determination of PAHs in Mineral Oil (Food Grade Oil)

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



Time-->

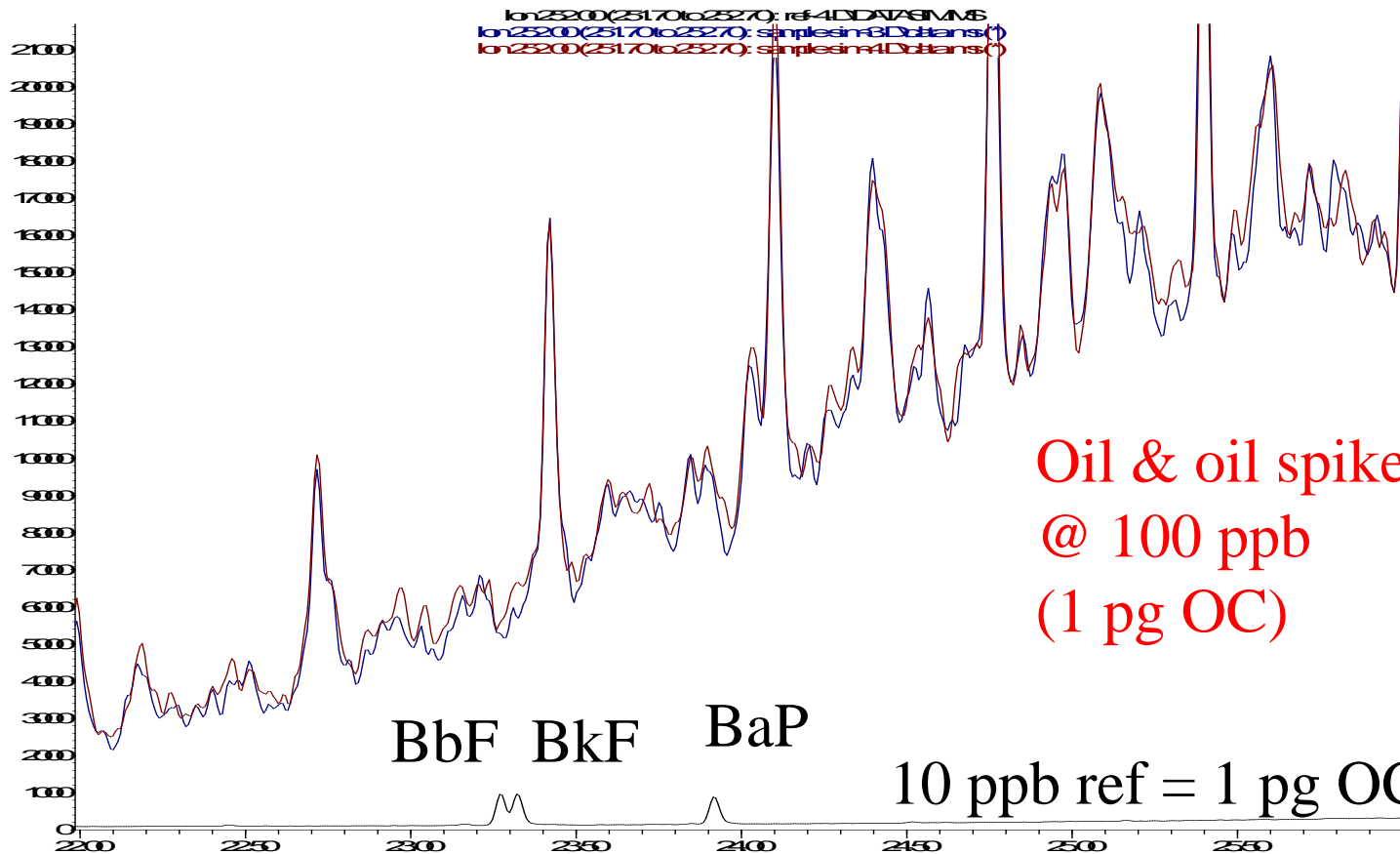


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Determination of PAHs in Mineral Oil direct analysis by GC-MSD

Abundance



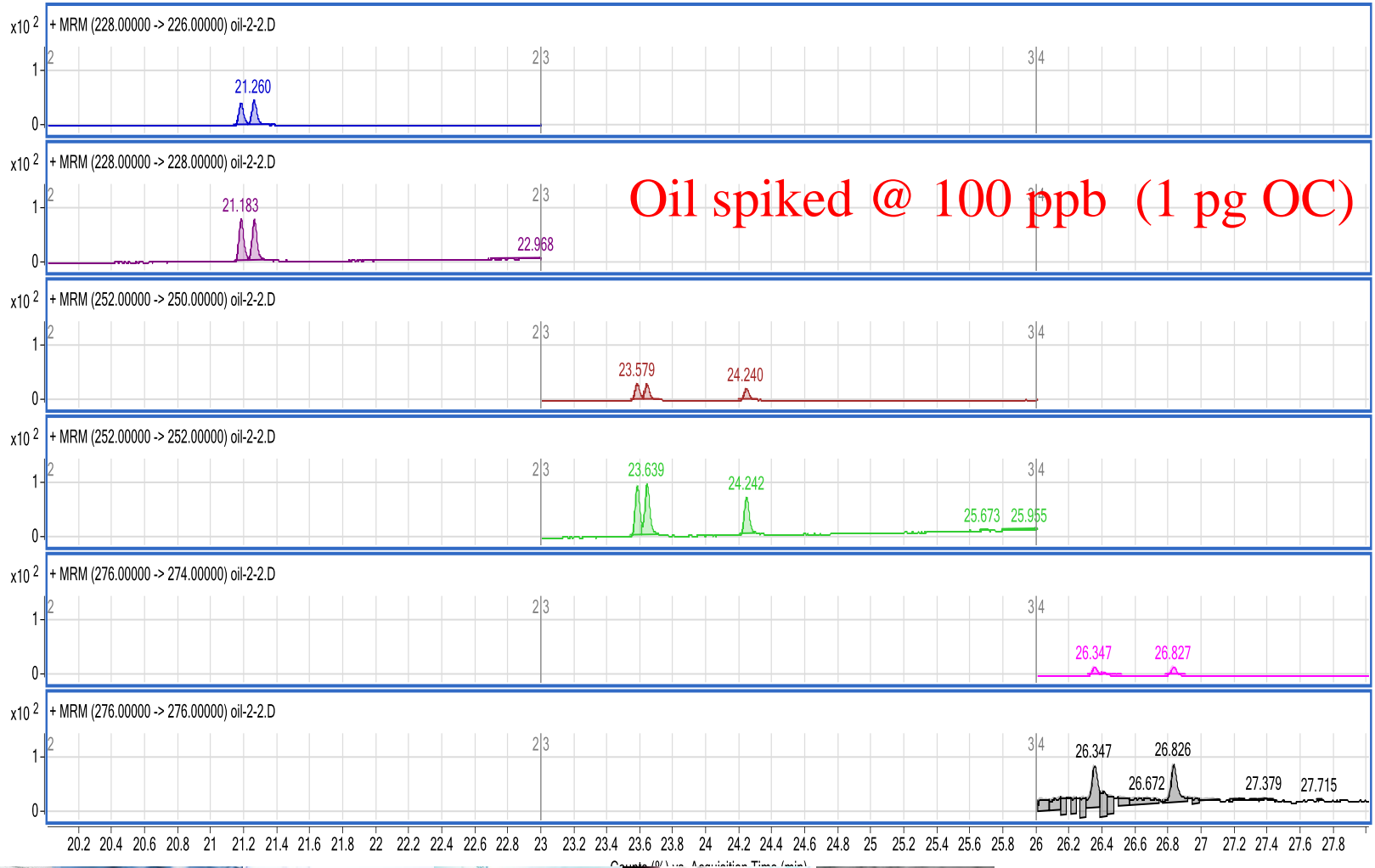
Time



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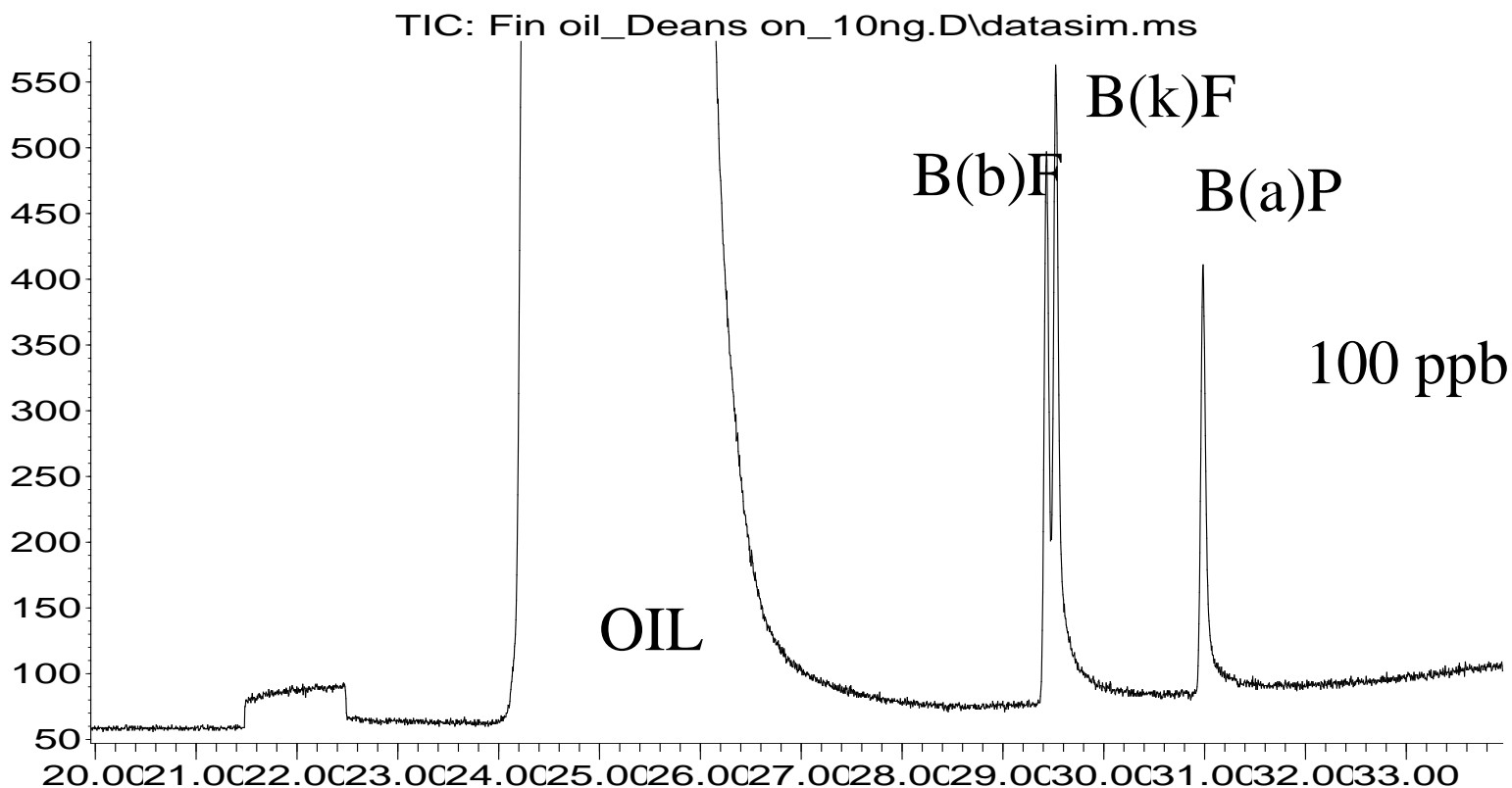
Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



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



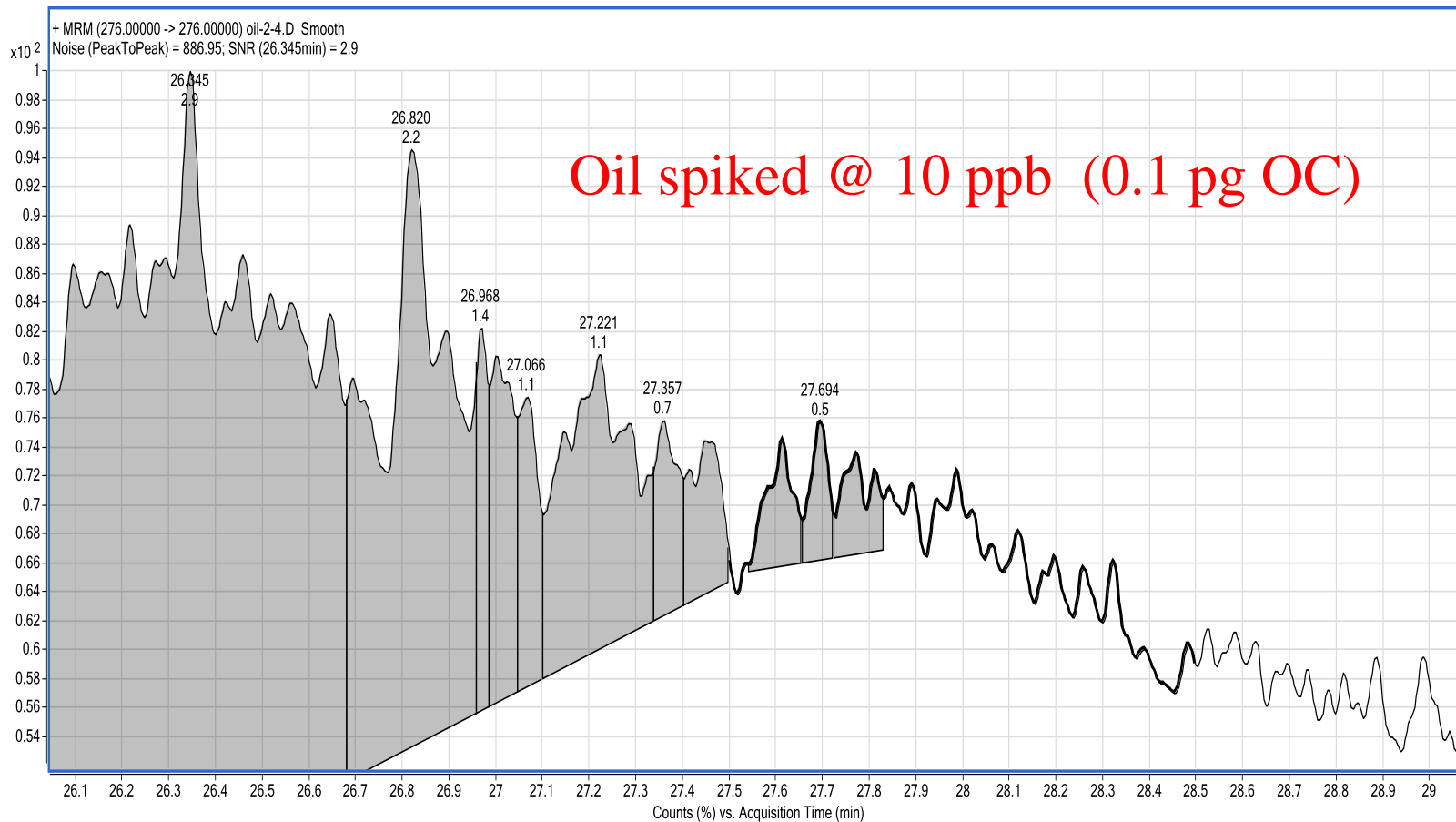
Time-->



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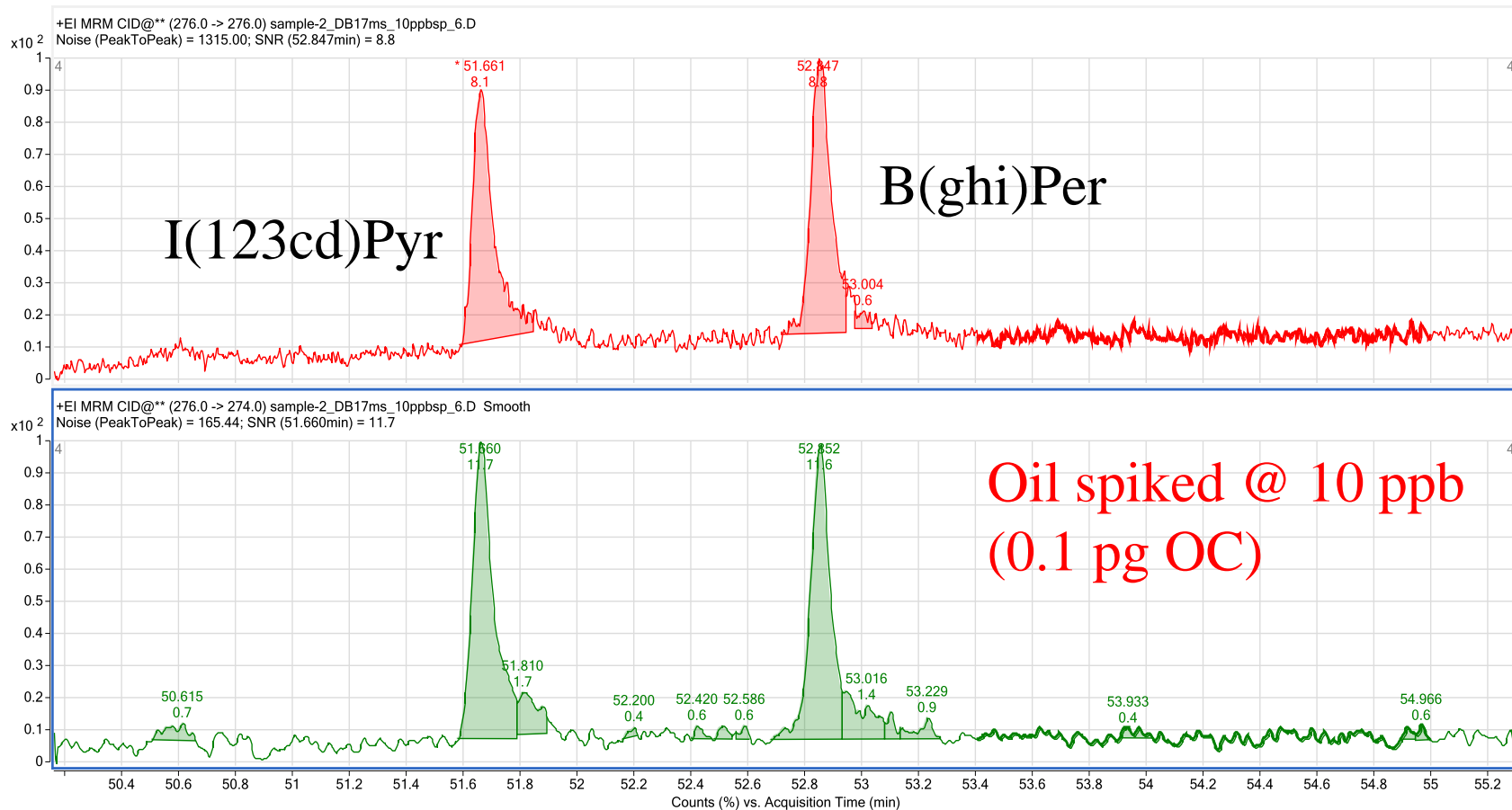
Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



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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)



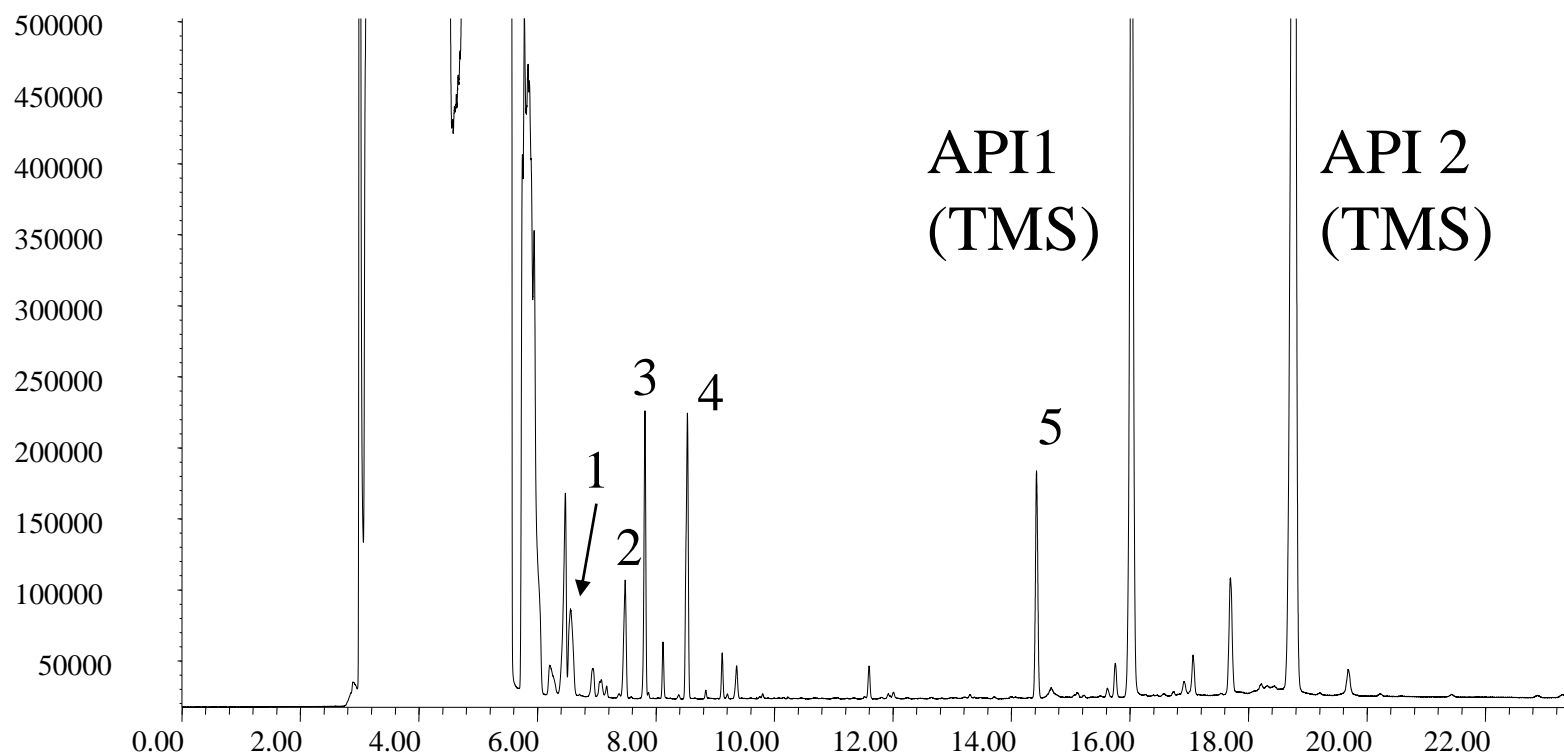
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Halo-alcohols (TMS der) in Carbamazepine on Dim 1 - scouting run

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

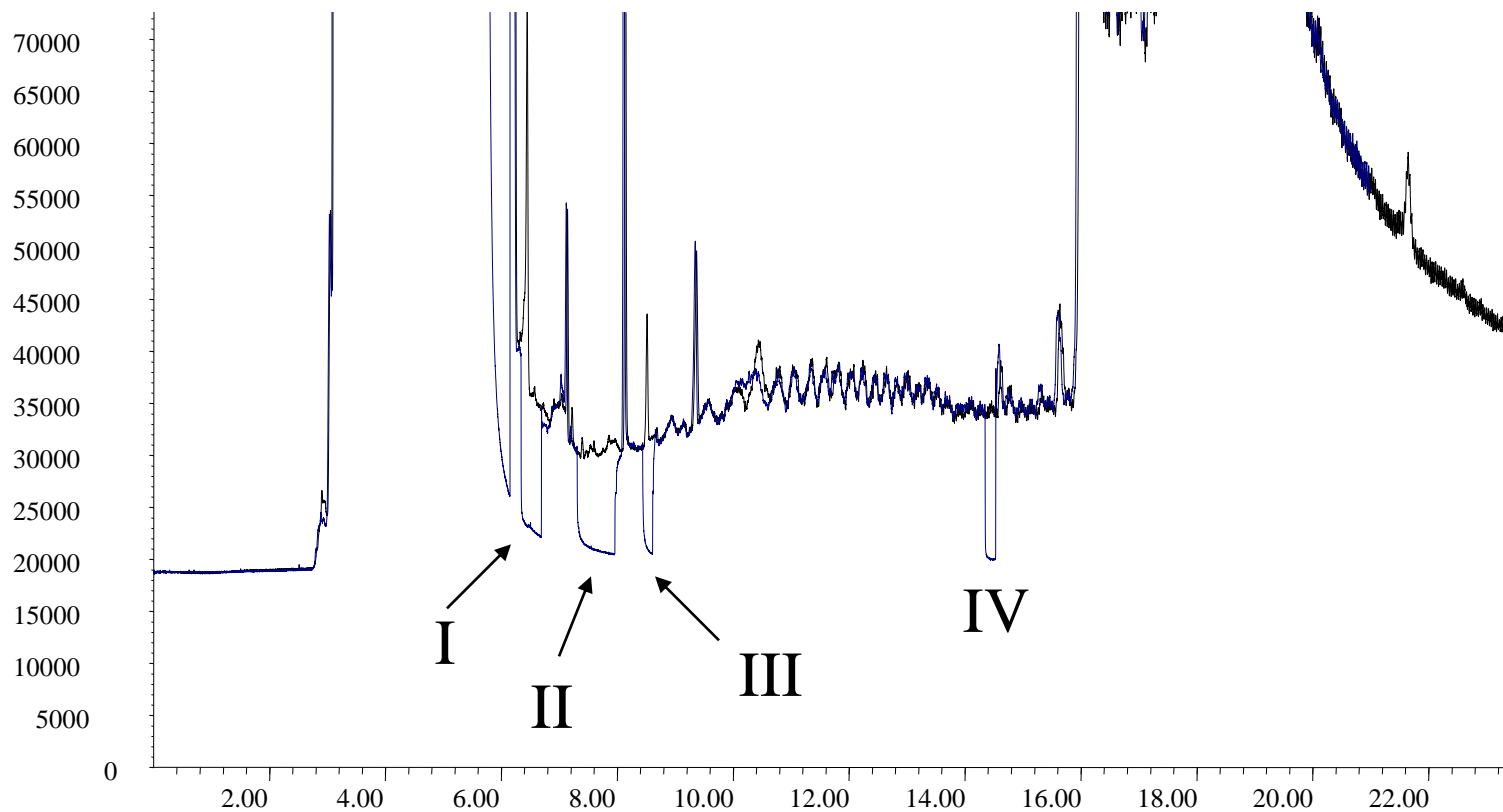


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Halo-alcohols (TMS der) in Carbamazepine (1 ppm) on Dim 1 with and without heart-cut

Response_



Time



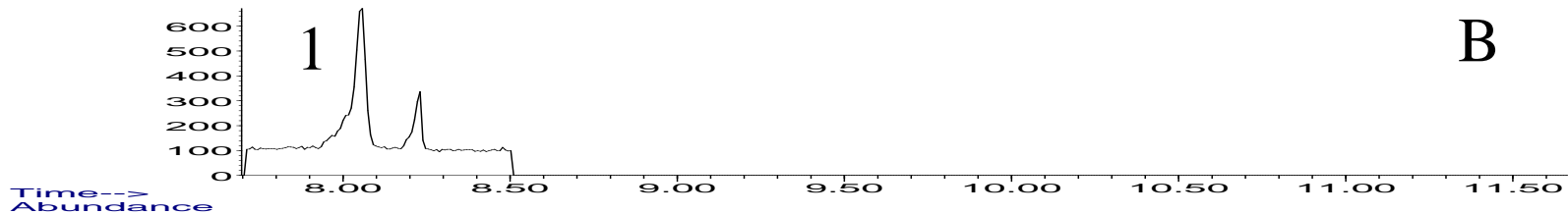
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Dim 2

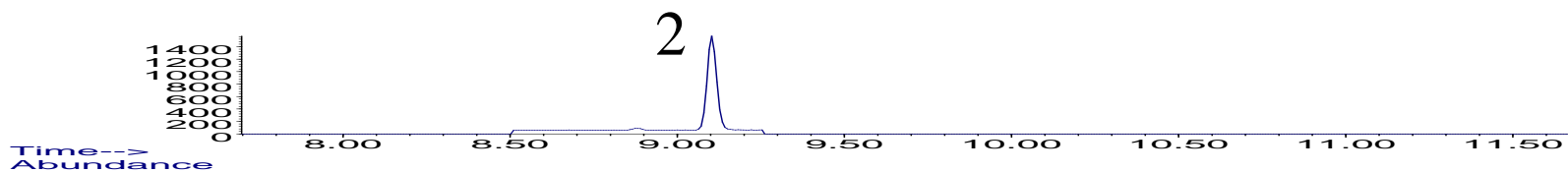
Abundance

Ion 1300 (137.0 to 137.0): 5.g/AP_Velconesthompis_4.D.DAT/MS

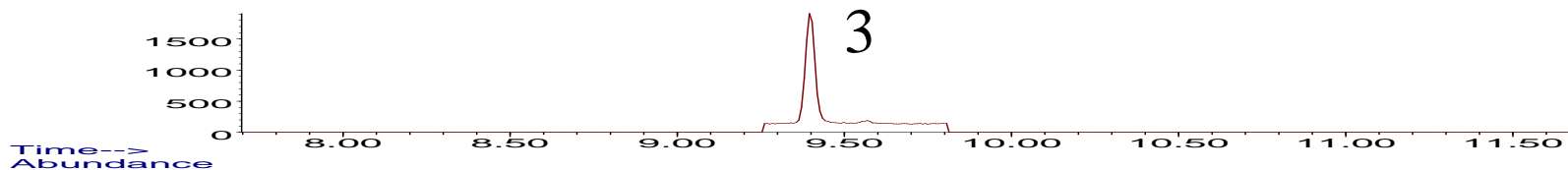


B

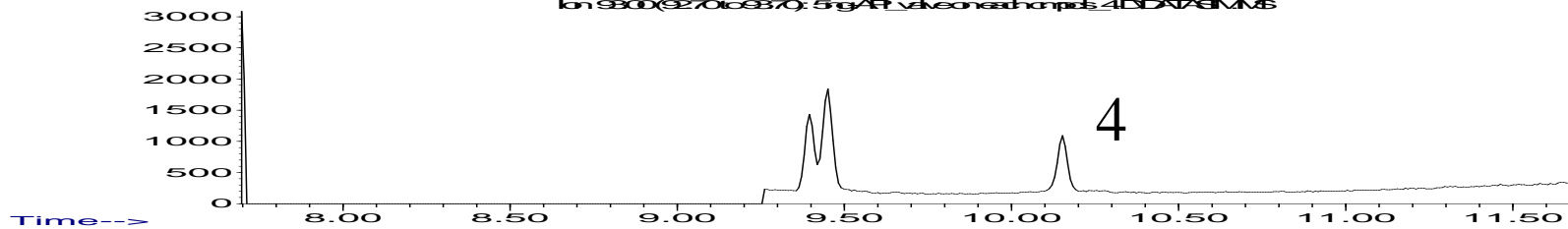
Ion 1350 (137.0 to 137.0): 5.g/AP_Velconesthompis_4.D.DAT/MS



Ion 1200 (127.0 to 127.0): 5.g/AP_Velconesthompis_4.D.DAT/MS



Ion 930 (927.0 to 927.0): 5.g/AP_Velconesthompis_4.D.DAT/MS



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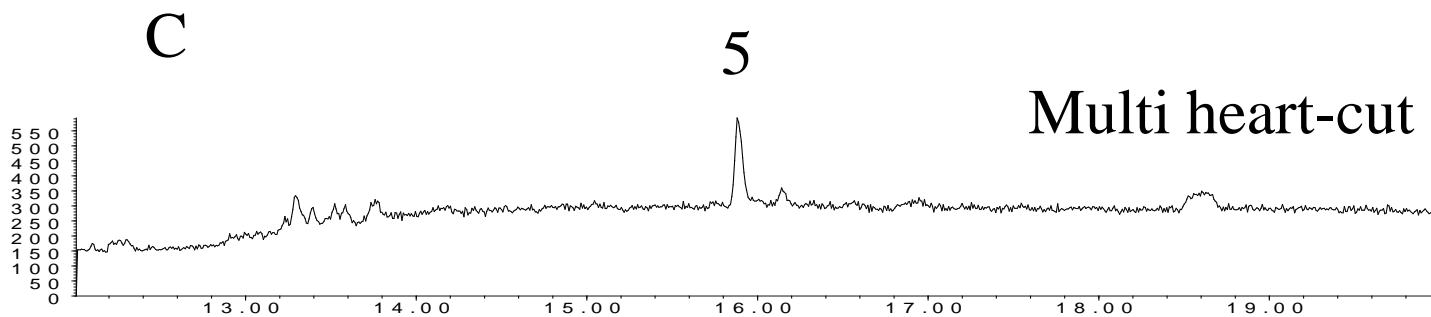
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Dim 2

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

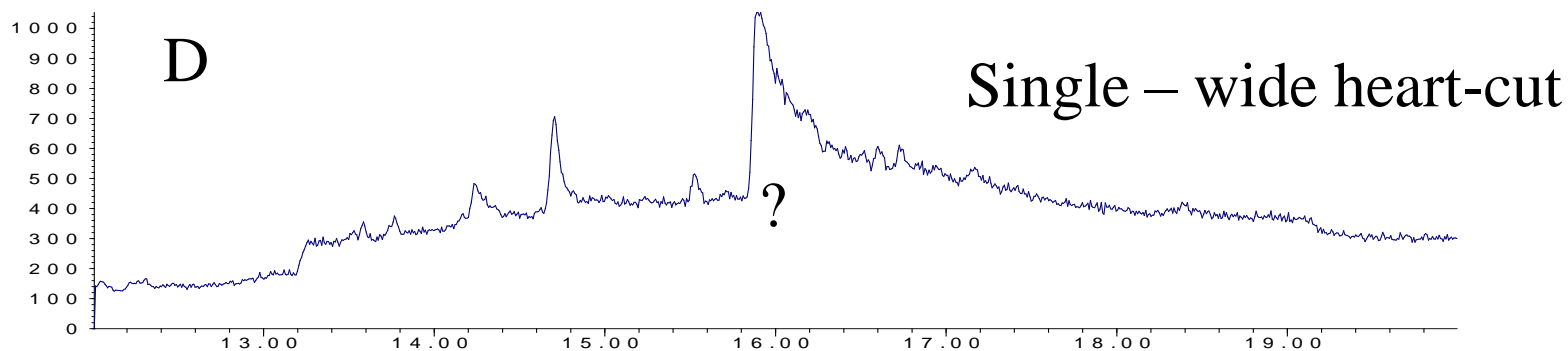
Abundance

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-->

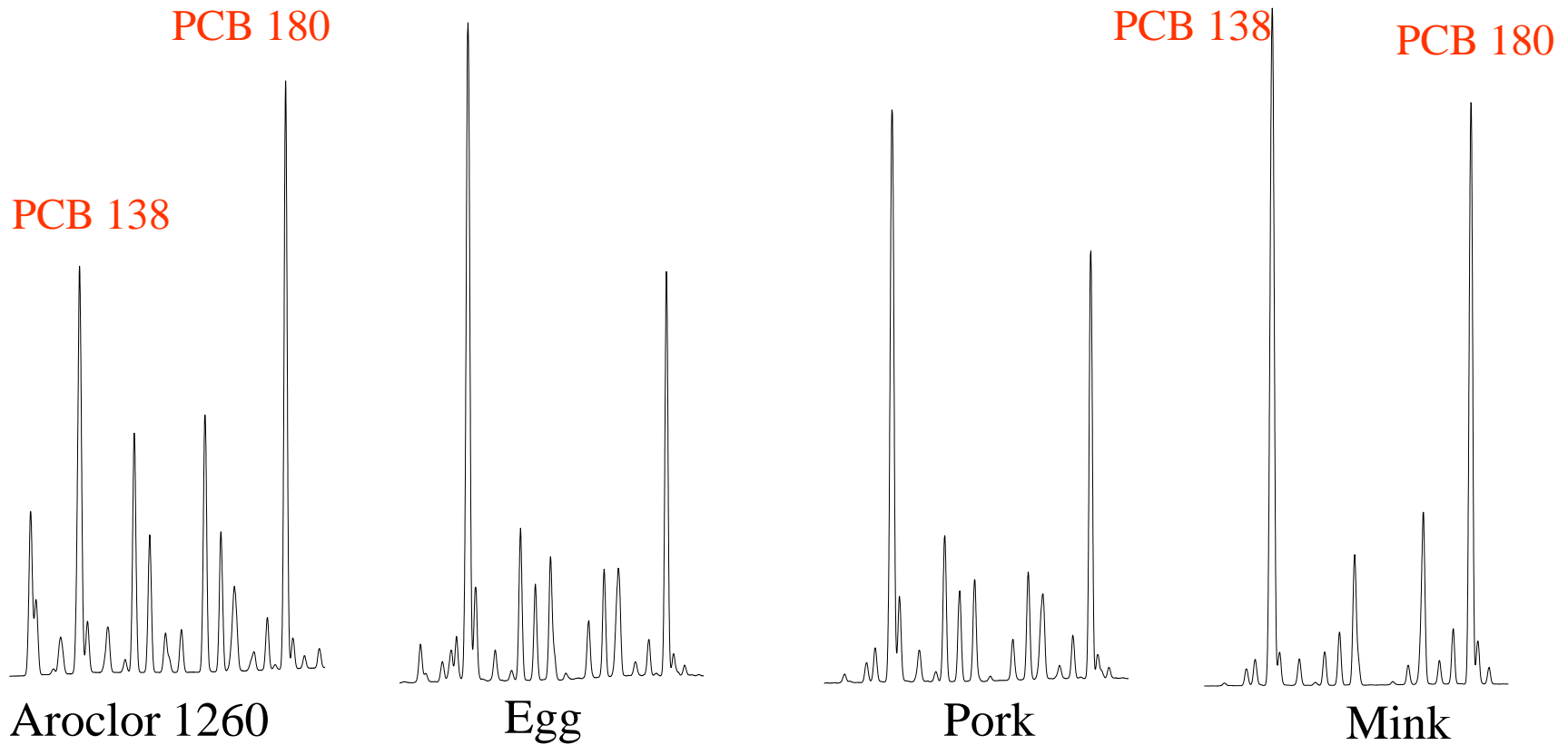


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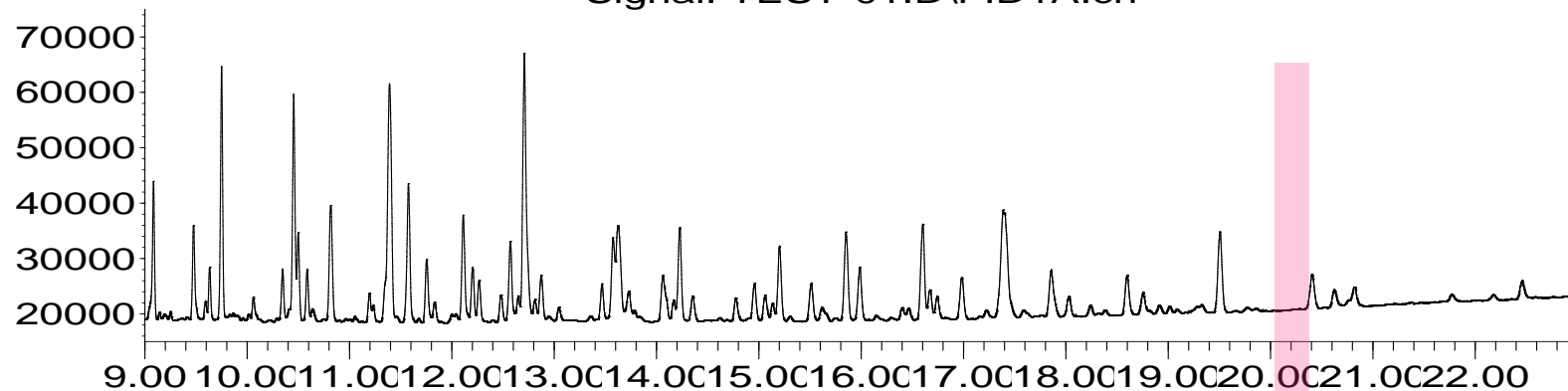
PCBs in Food Products

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

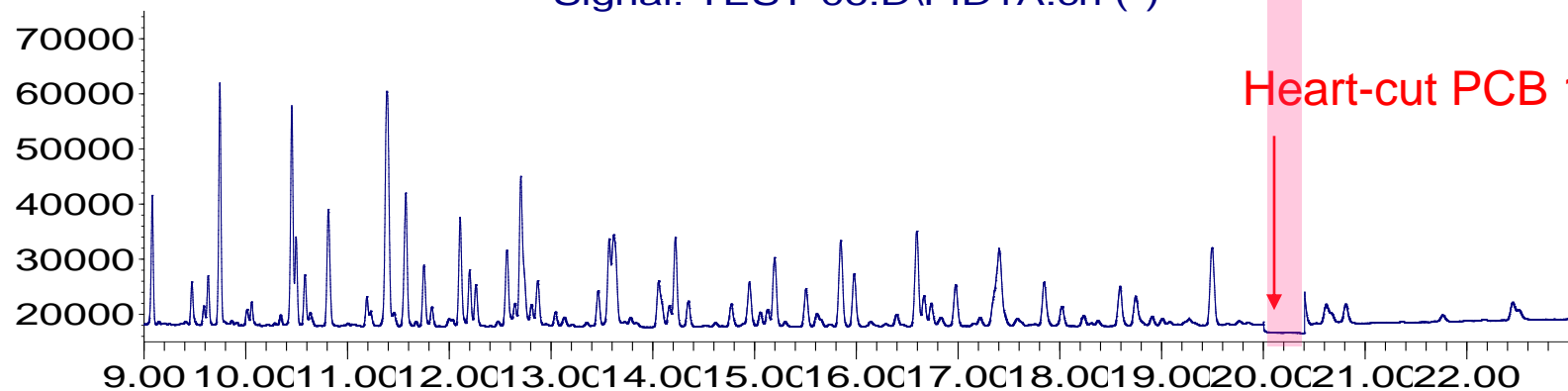


PCB analysis by GC-GC-MS: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)

Signal: TEST-01.D\FID1A.ch



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

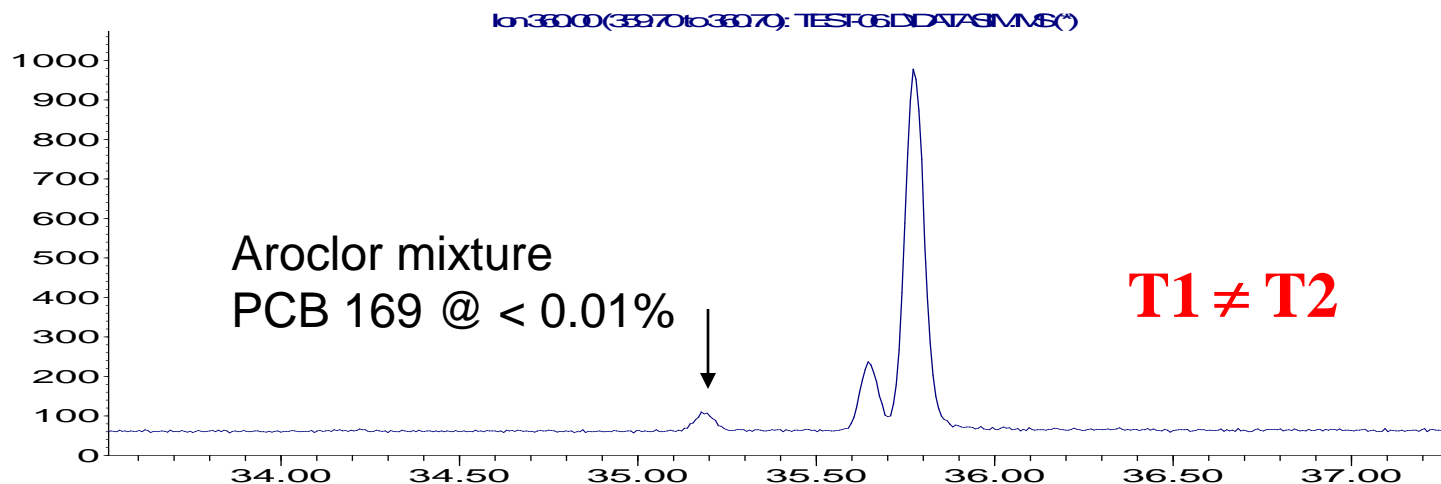
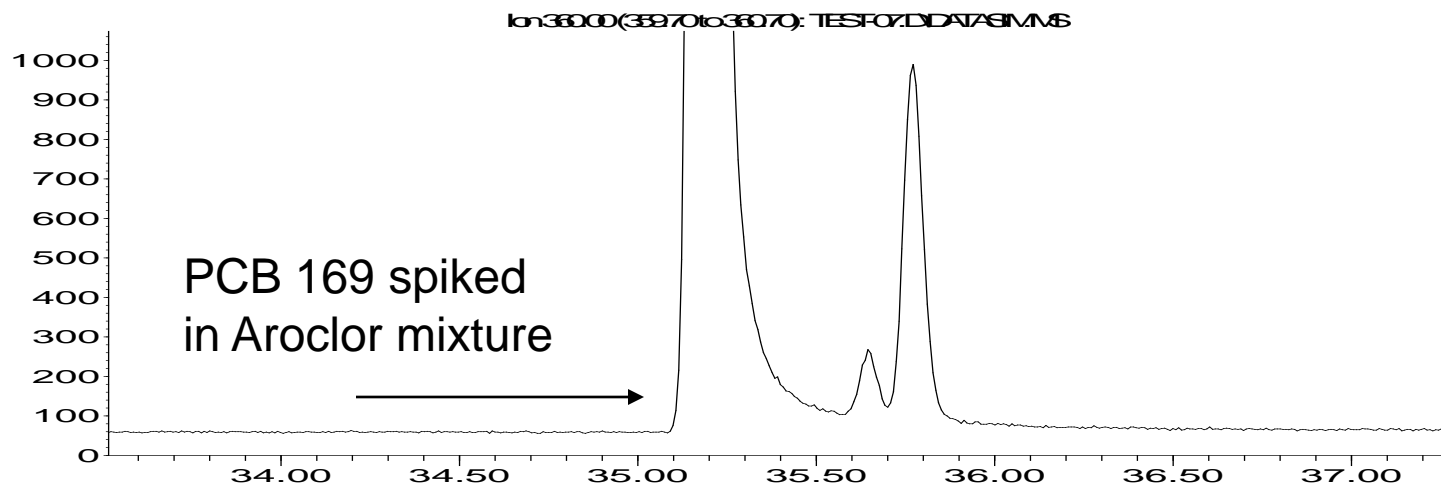


Time



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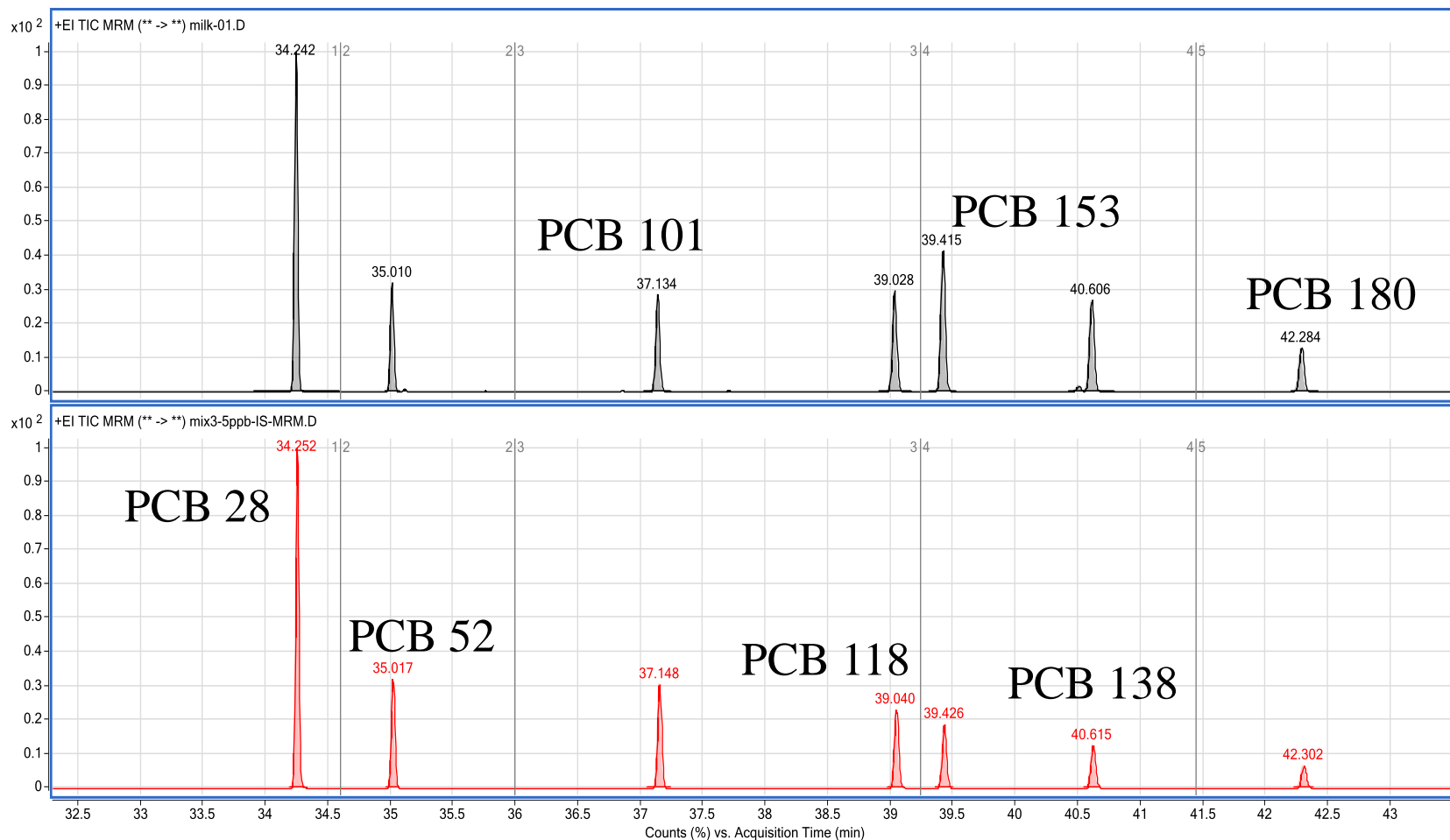
PCB analysis by 2DGC-MSD: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



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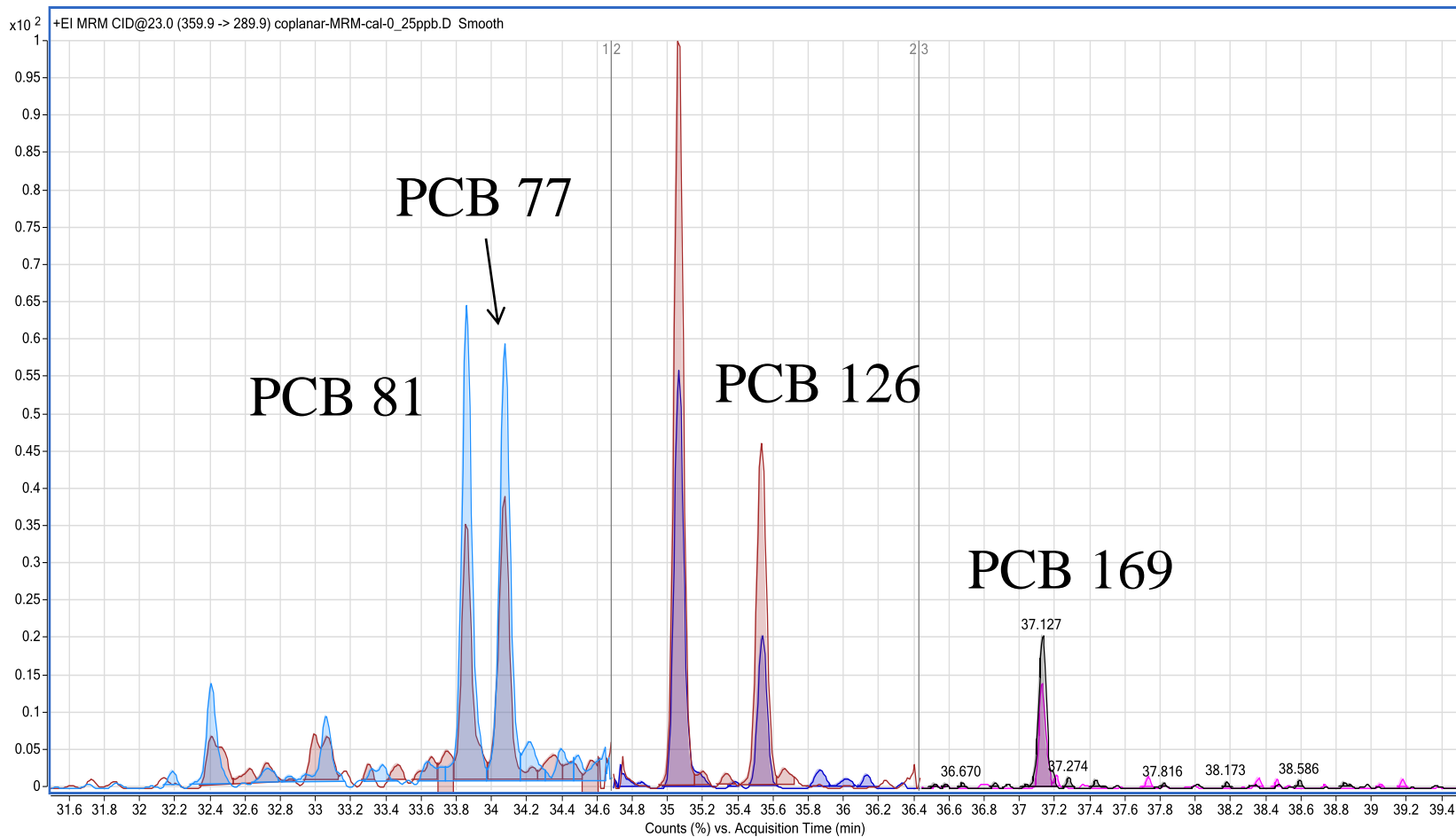
PCB analysis in milk powder by 2DGC-QQQ: 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



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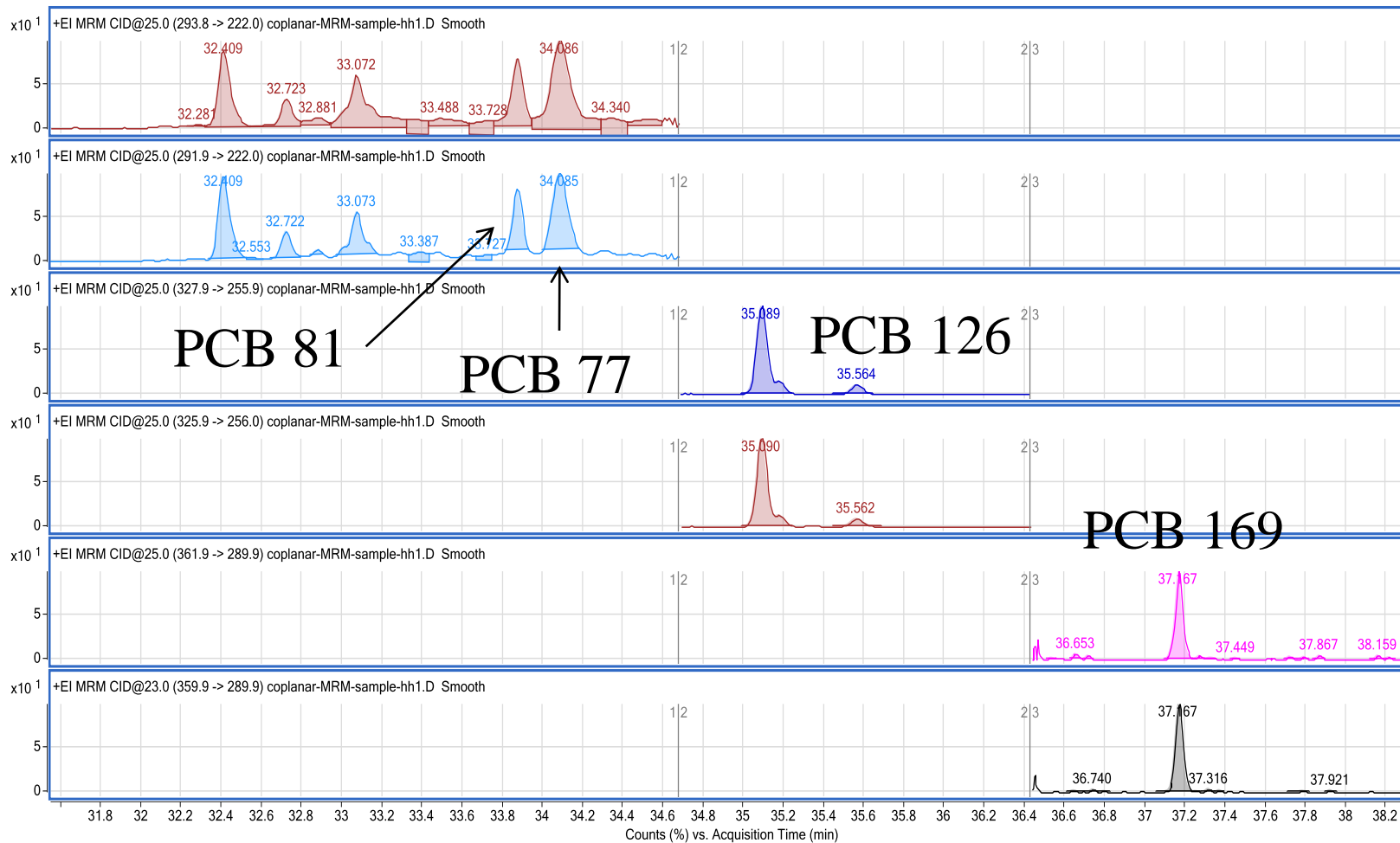
Coplanar PCBs 0.25 $\mu\text{g}/\mu\text{L}$



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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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