

# 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

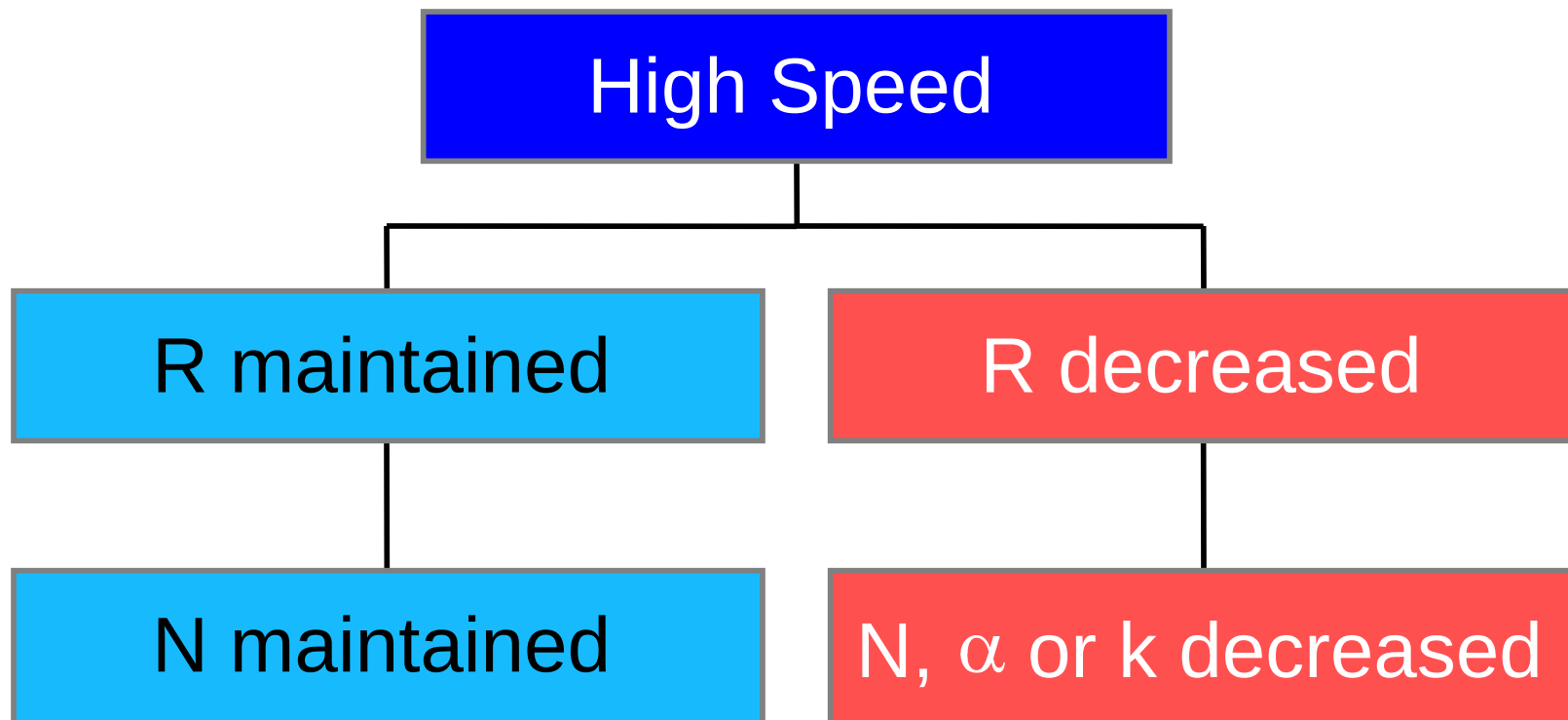
- Fast GC analysis  
(1200 C/min)
- Independant column  
temperature control
- of up to 4 different columns



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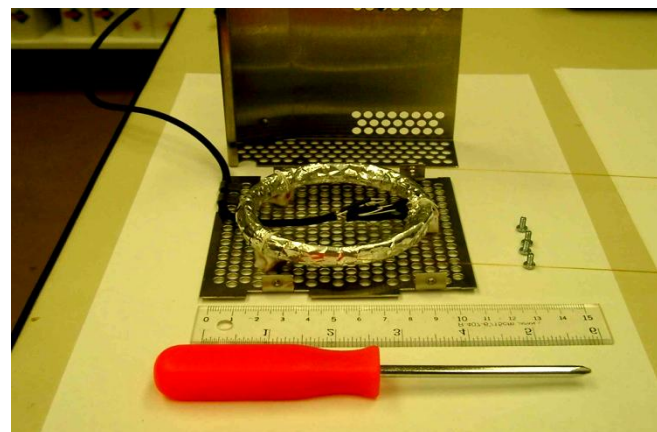
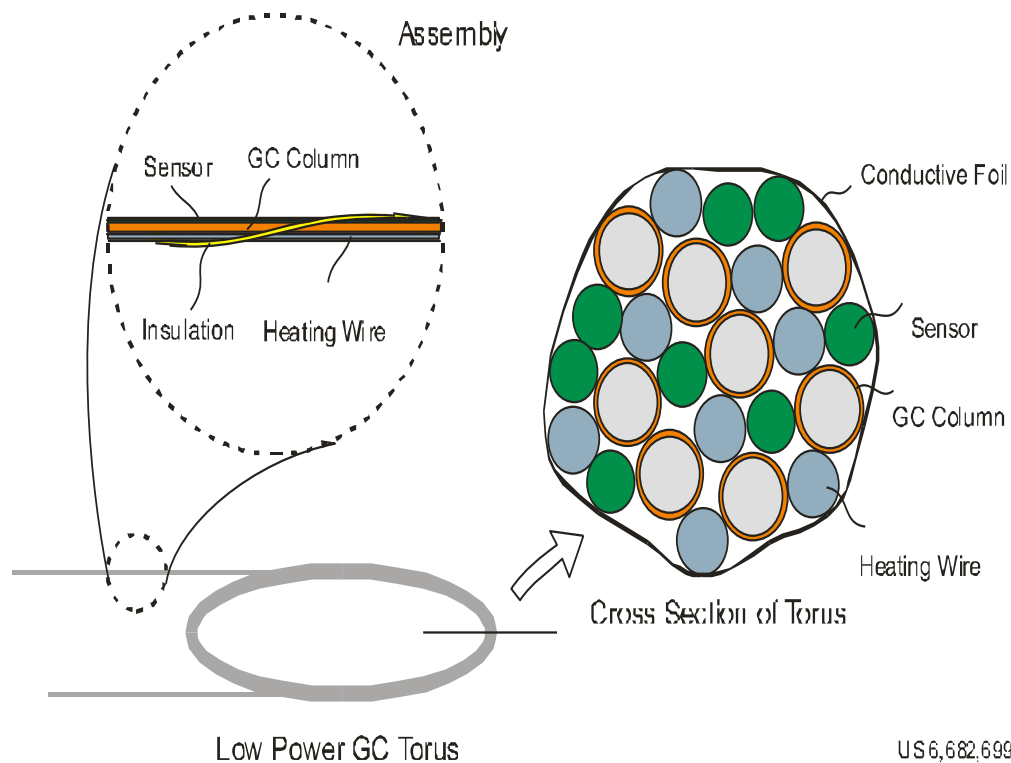
# High Speed CGC



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# Low Thermal Mass GC Technology



Heating rates: up to 1000 C/min !!



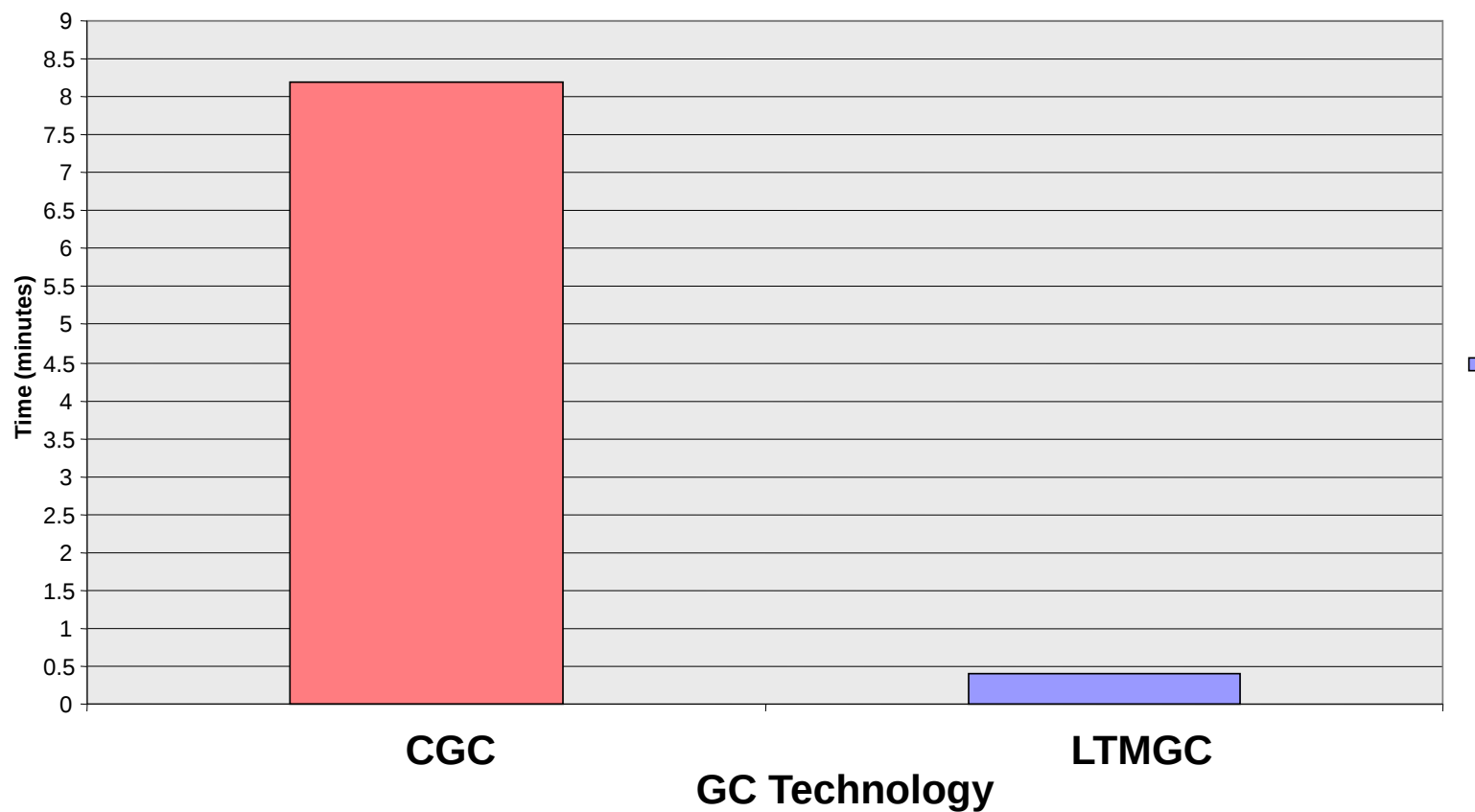
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# Cool Down Time Comparison

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

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

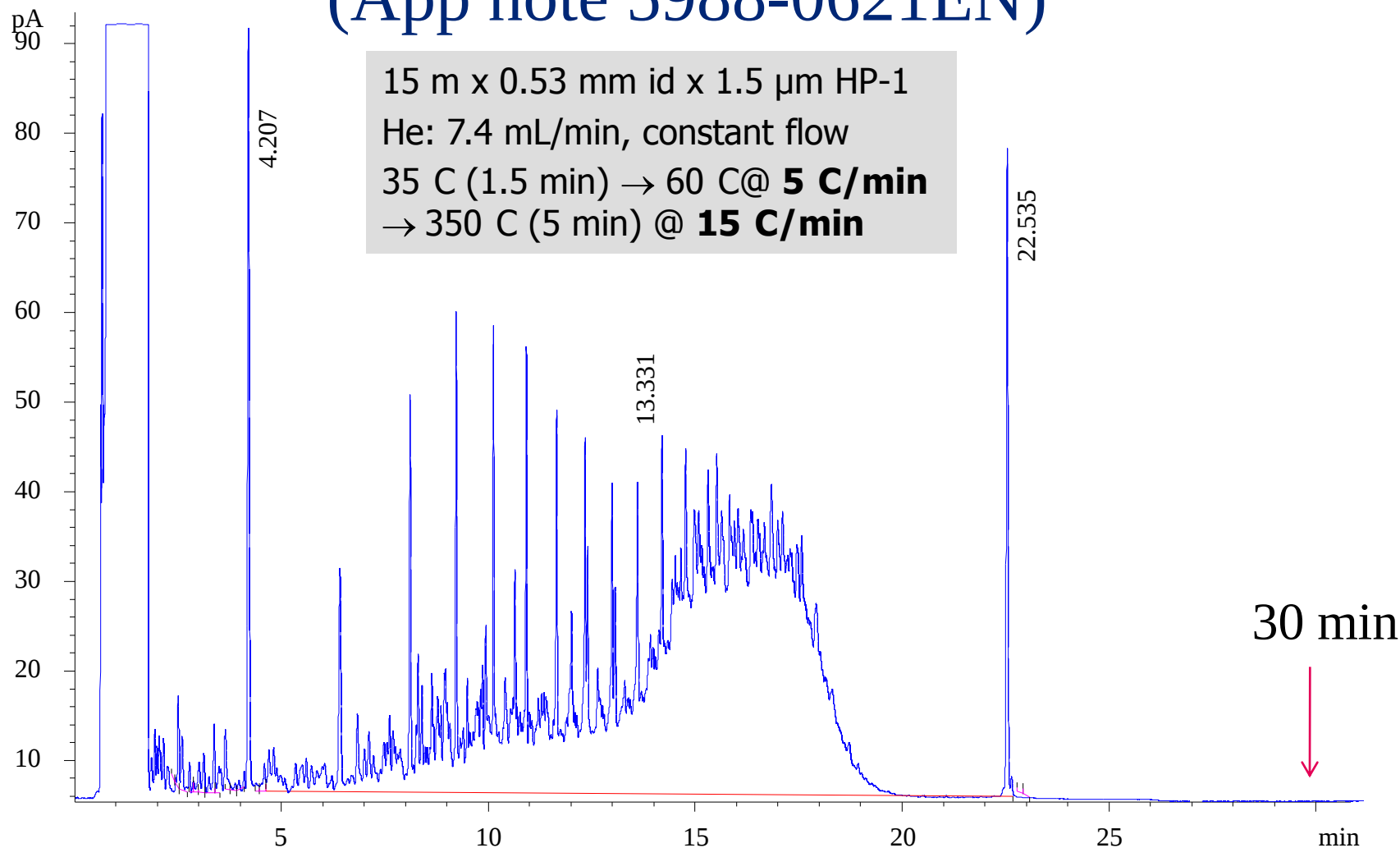


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# Typical Hydrocarbon Index Analysis by GC

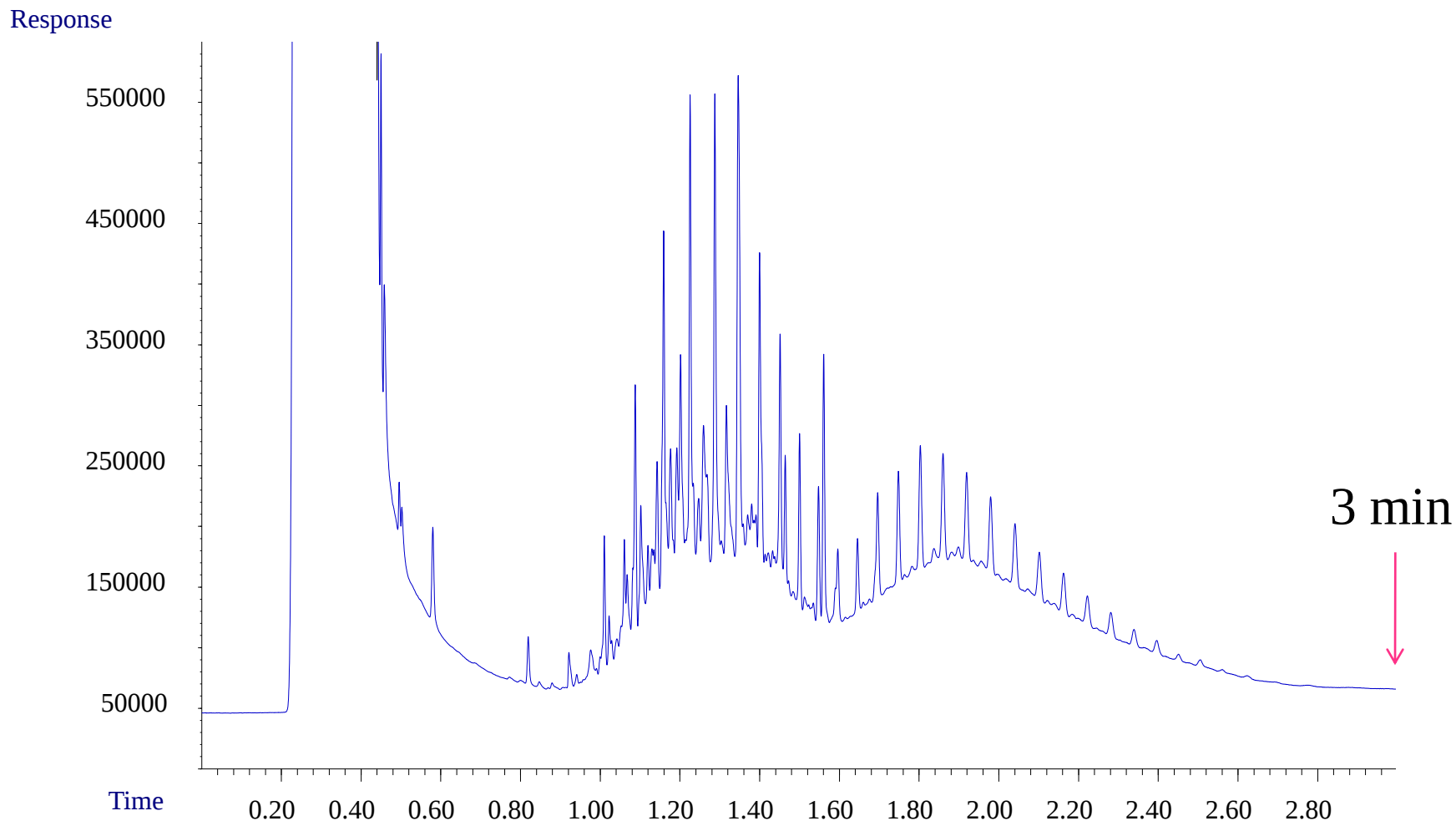
## (App note 5988-0621EN)



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# Mineral Oil Calibration 100 mg/L



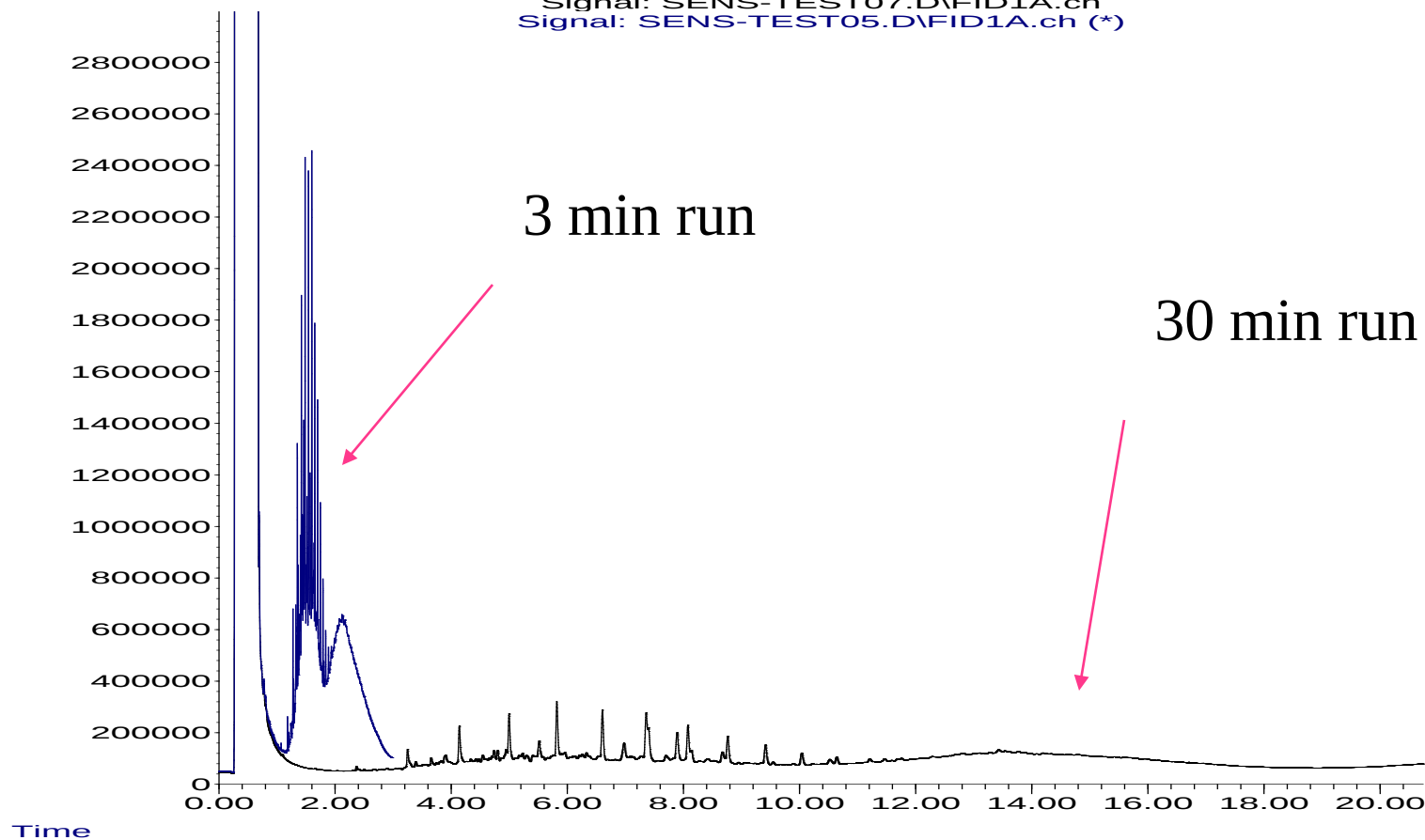
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# Extra: sensitivity gain

Response\_

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



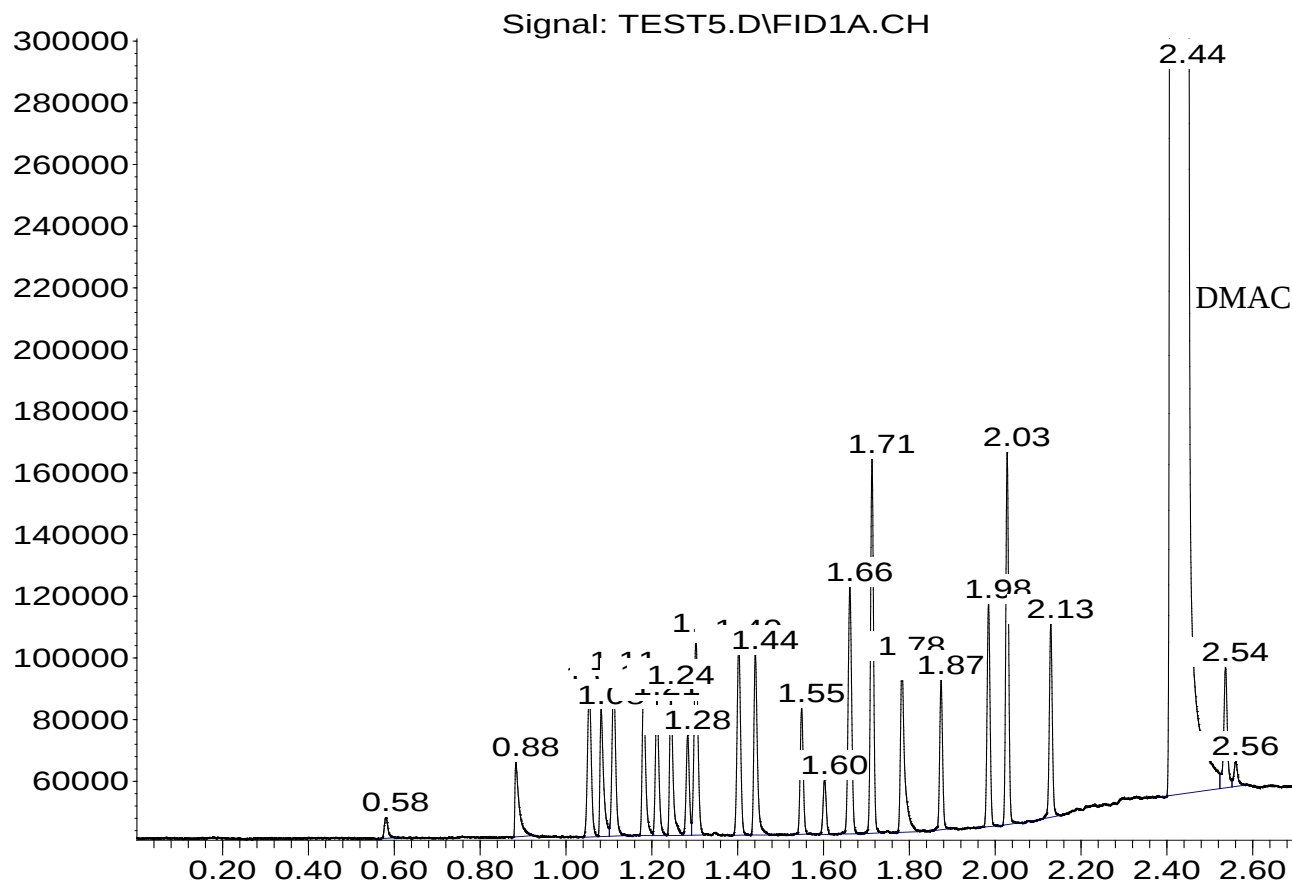
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# Residual solvent analysis

## *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  $\mu$ 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**)

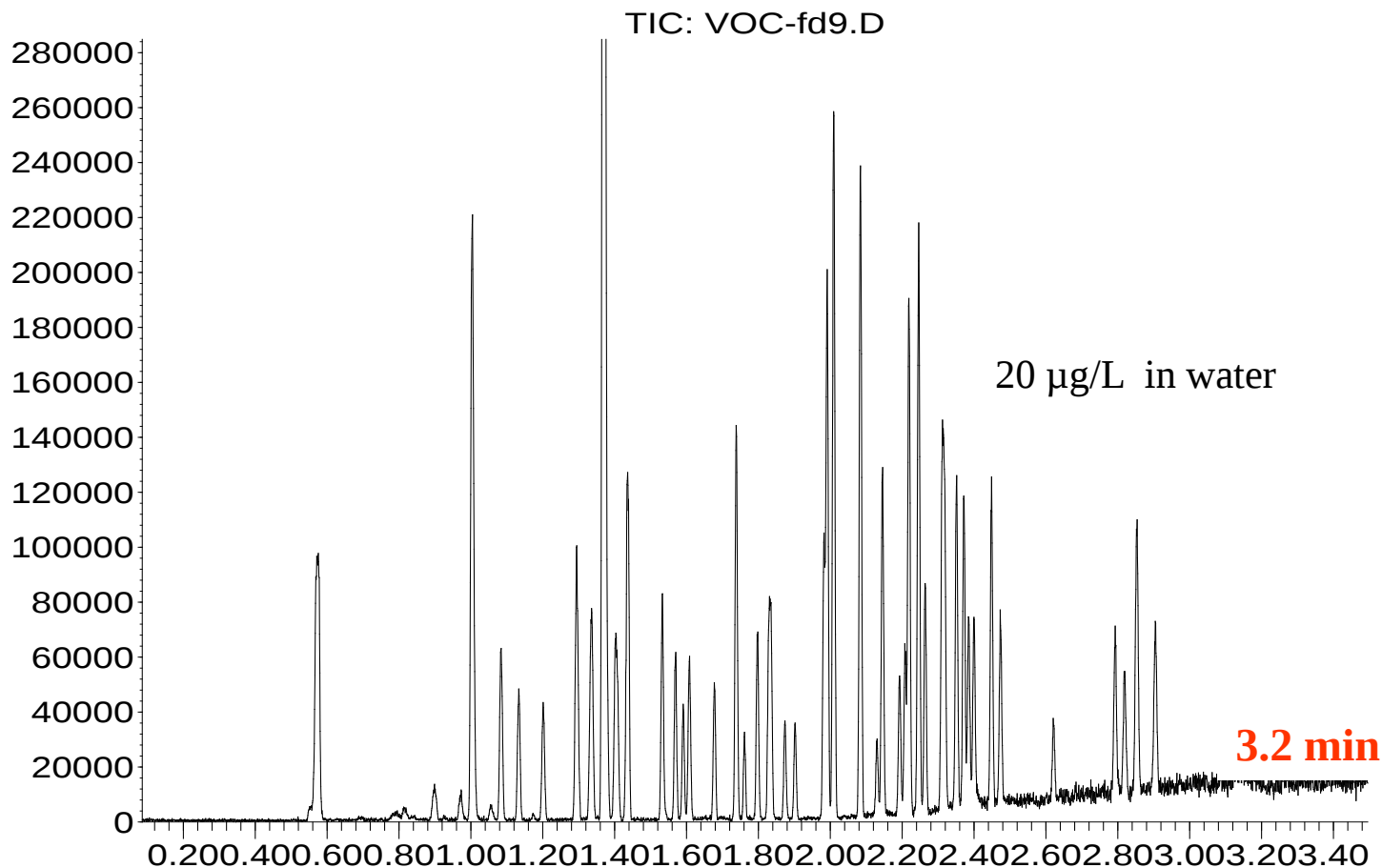


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# Ultra fast SHS-GC-**MSD**: EPA Volatiles

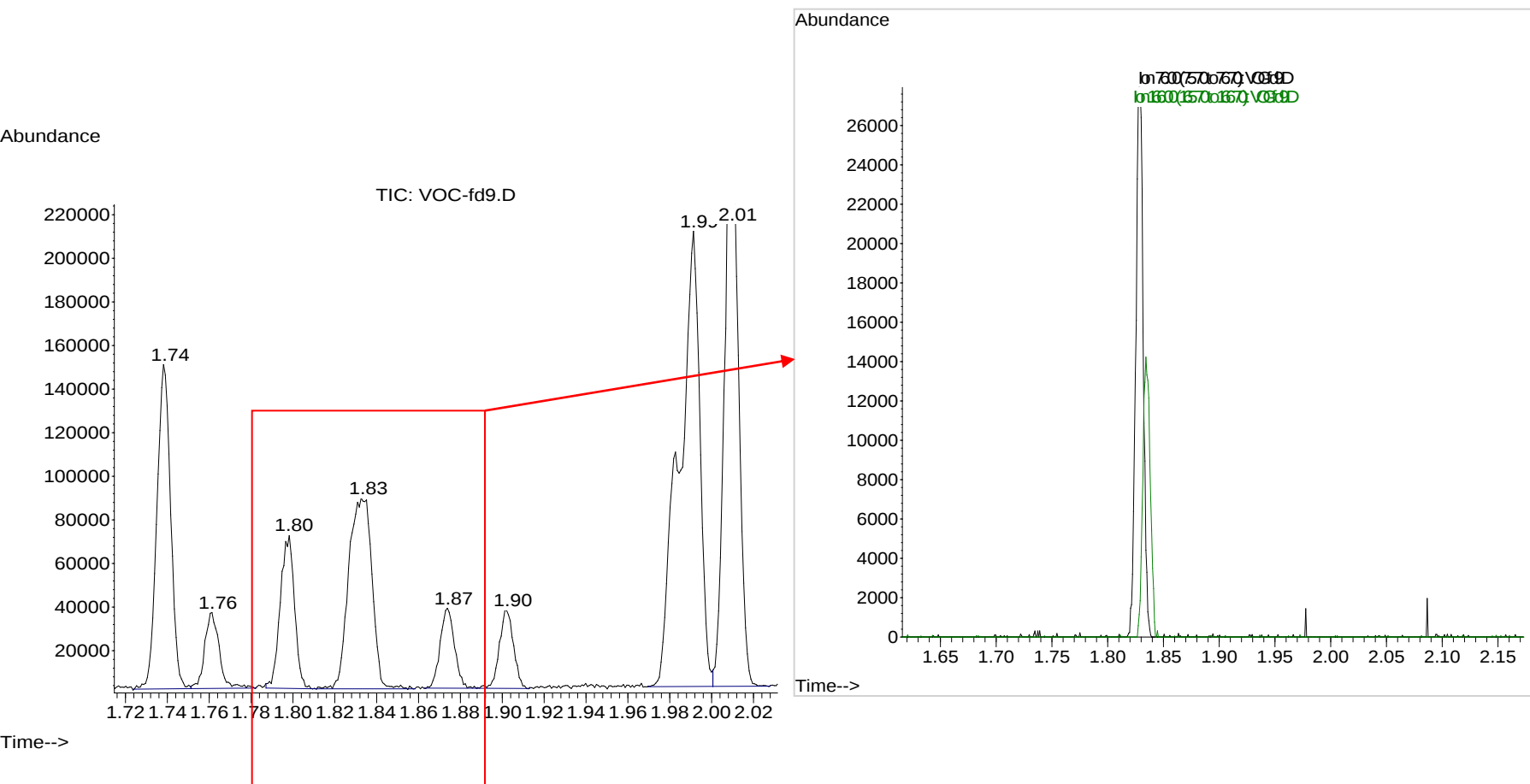
Abundance



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# 'Deconvolution' of critical pairs

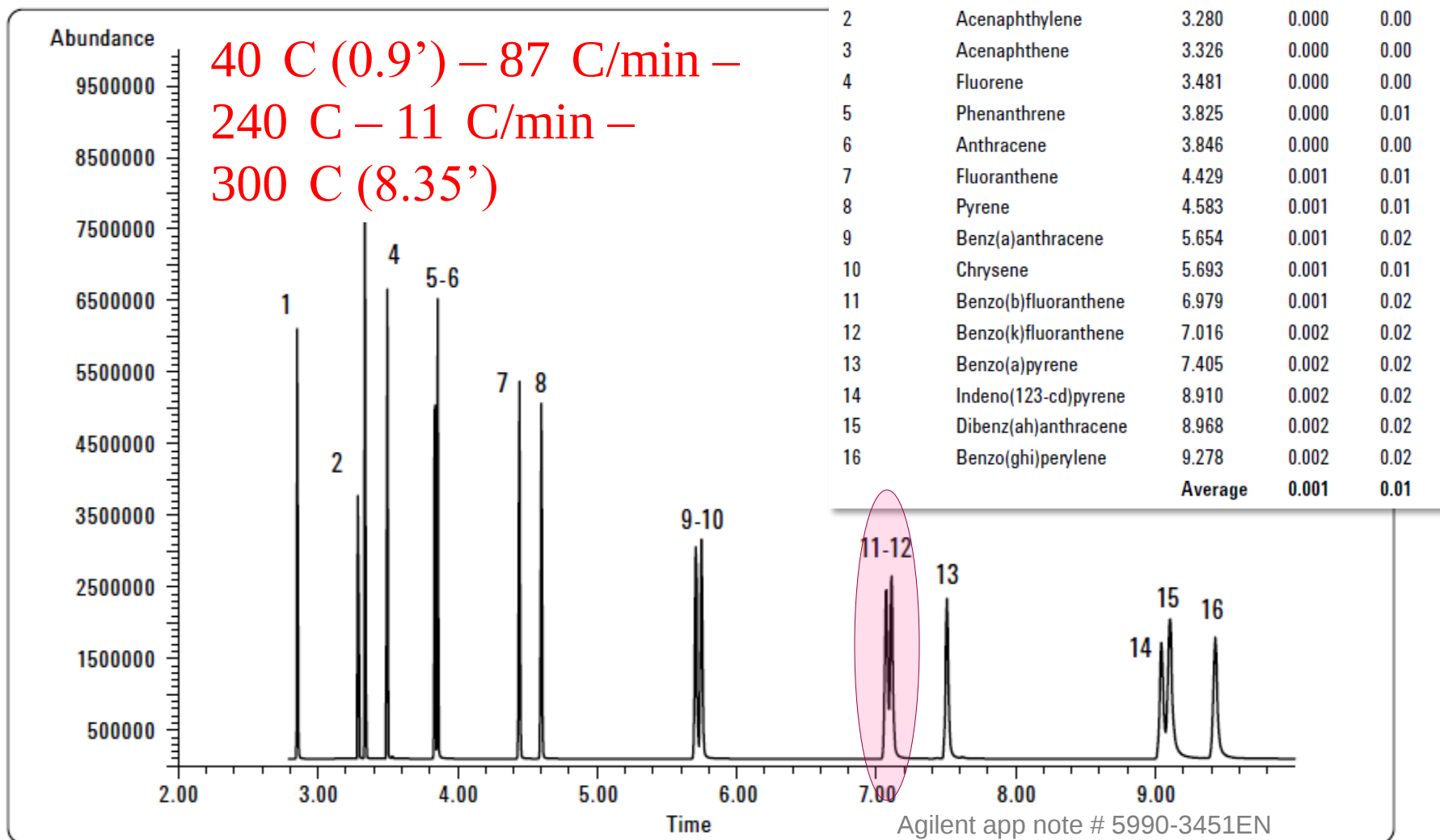


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# Fast LTM analysis of PAH

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



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

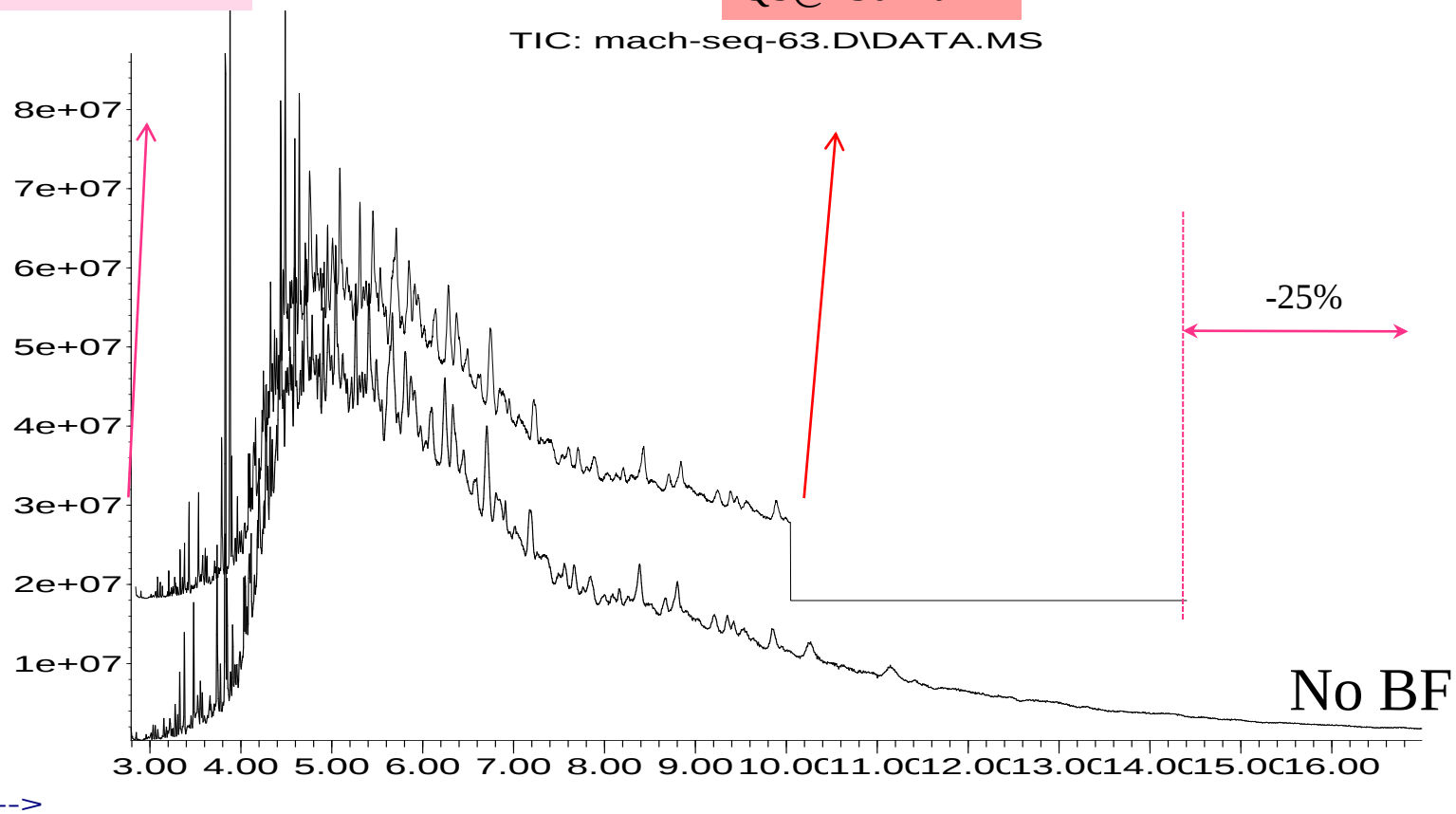
INJ @ 213-392kPa

QS@28 kPa

INJ @ 10kPa

QS@250kPa

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

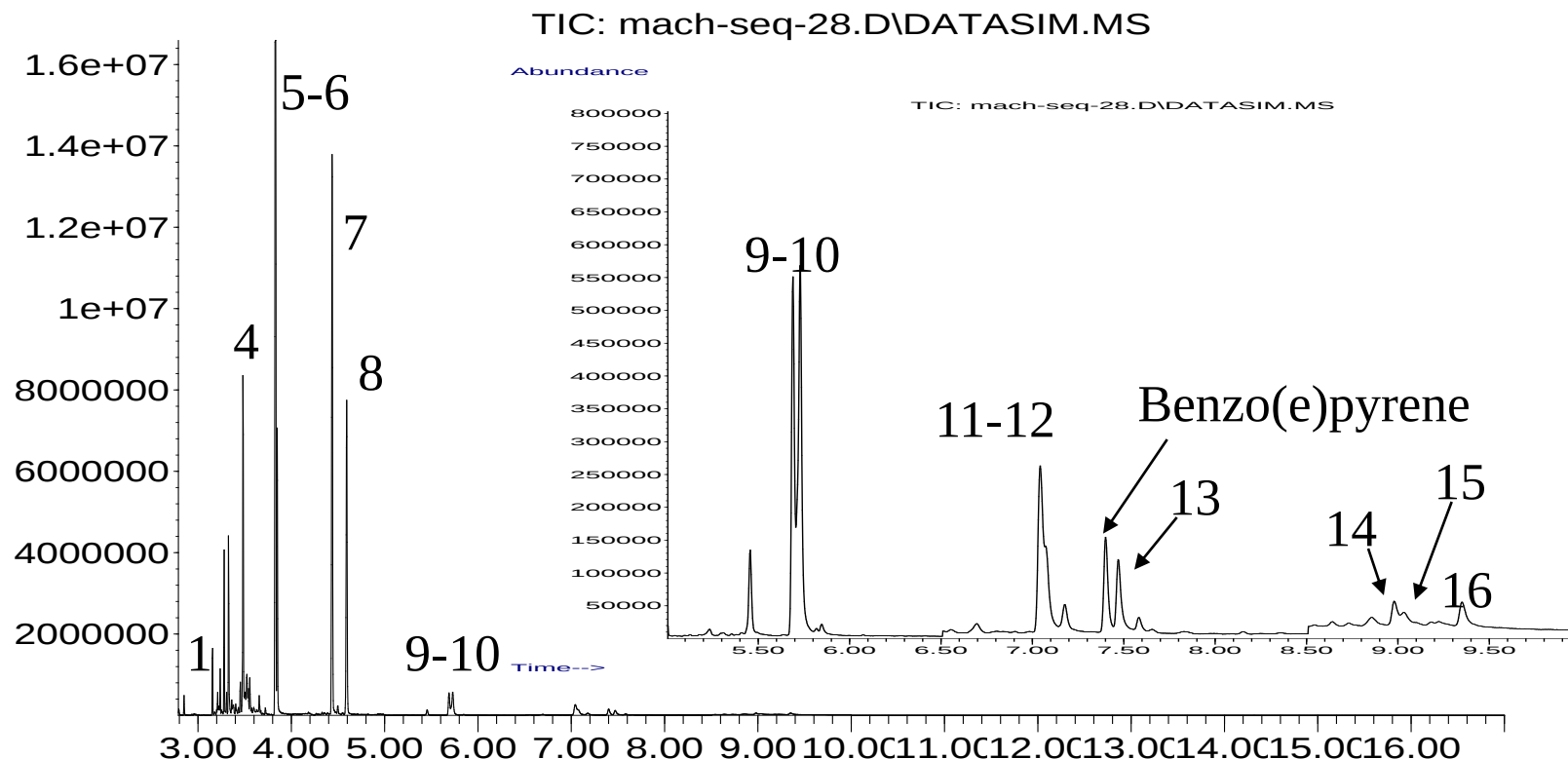


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# Soil extract - GC-MS in scan/SIM – SIM trace

Abundance



Time-->



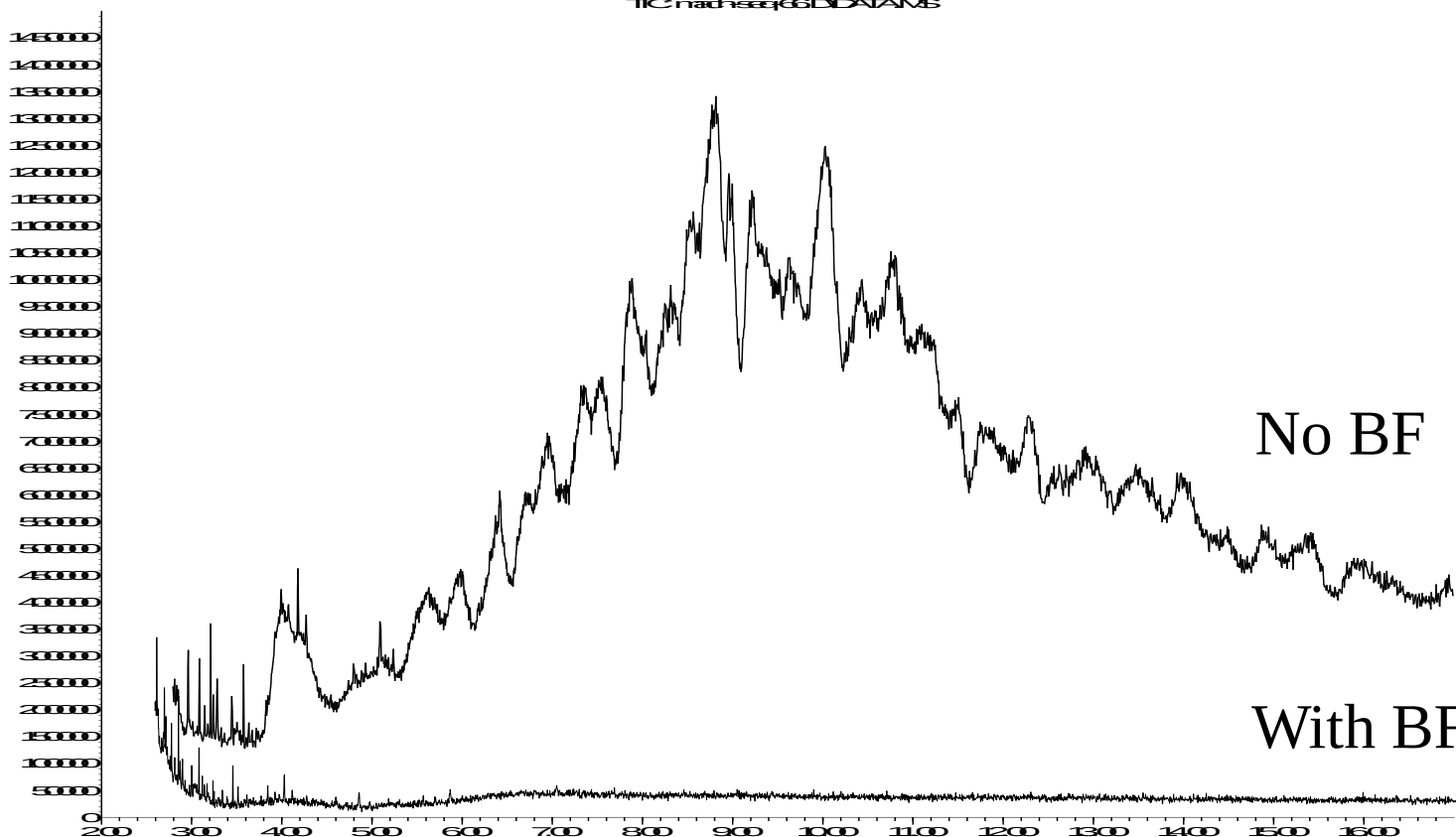
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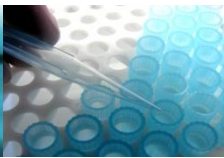
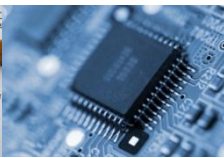
# Blank run after soil extract - scan trace

Abundance

TIC: nashseq66.D\\DATA\\MS



Time-->

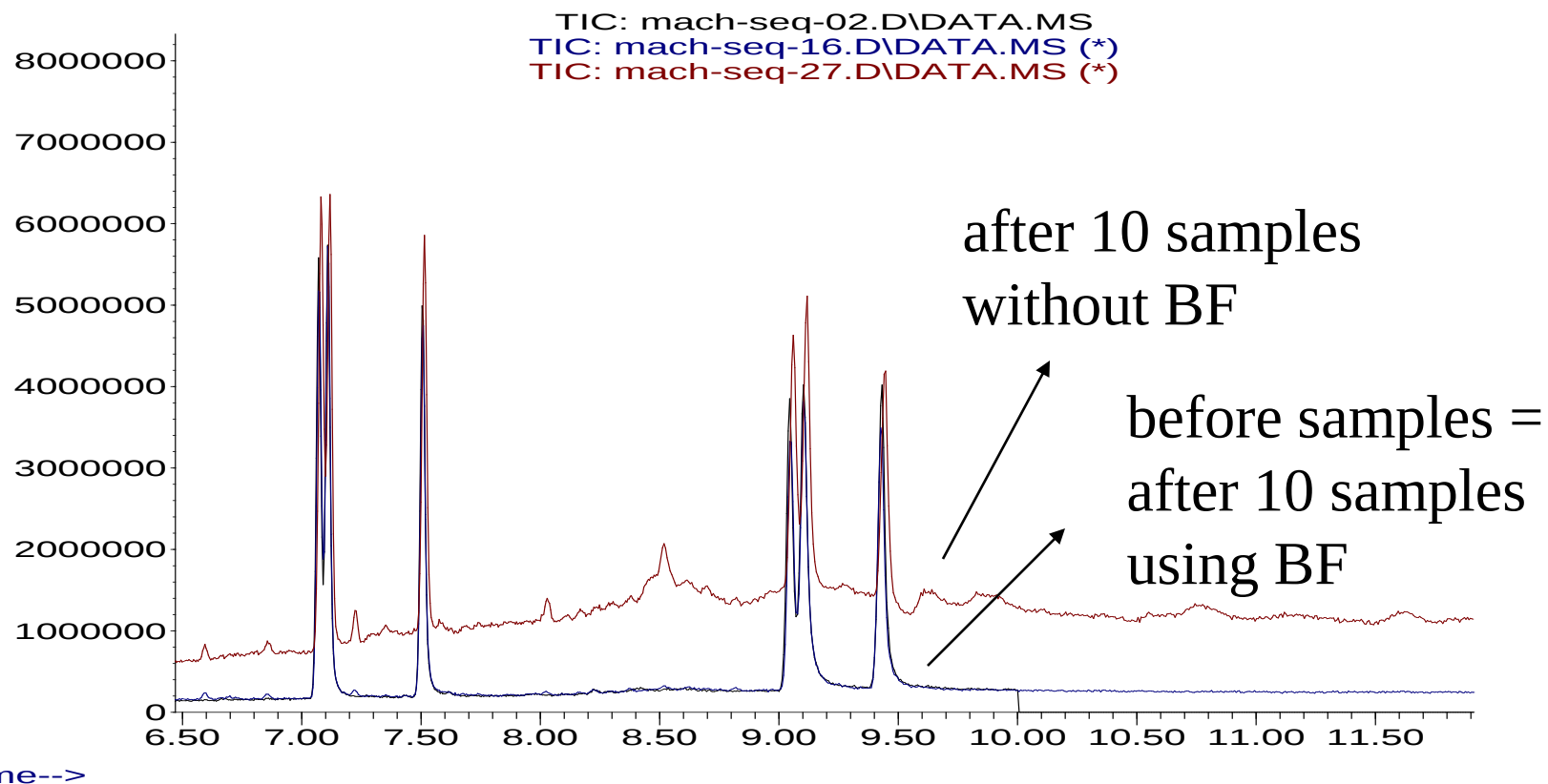


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# Check-out sample after soil extract sequence scan trace

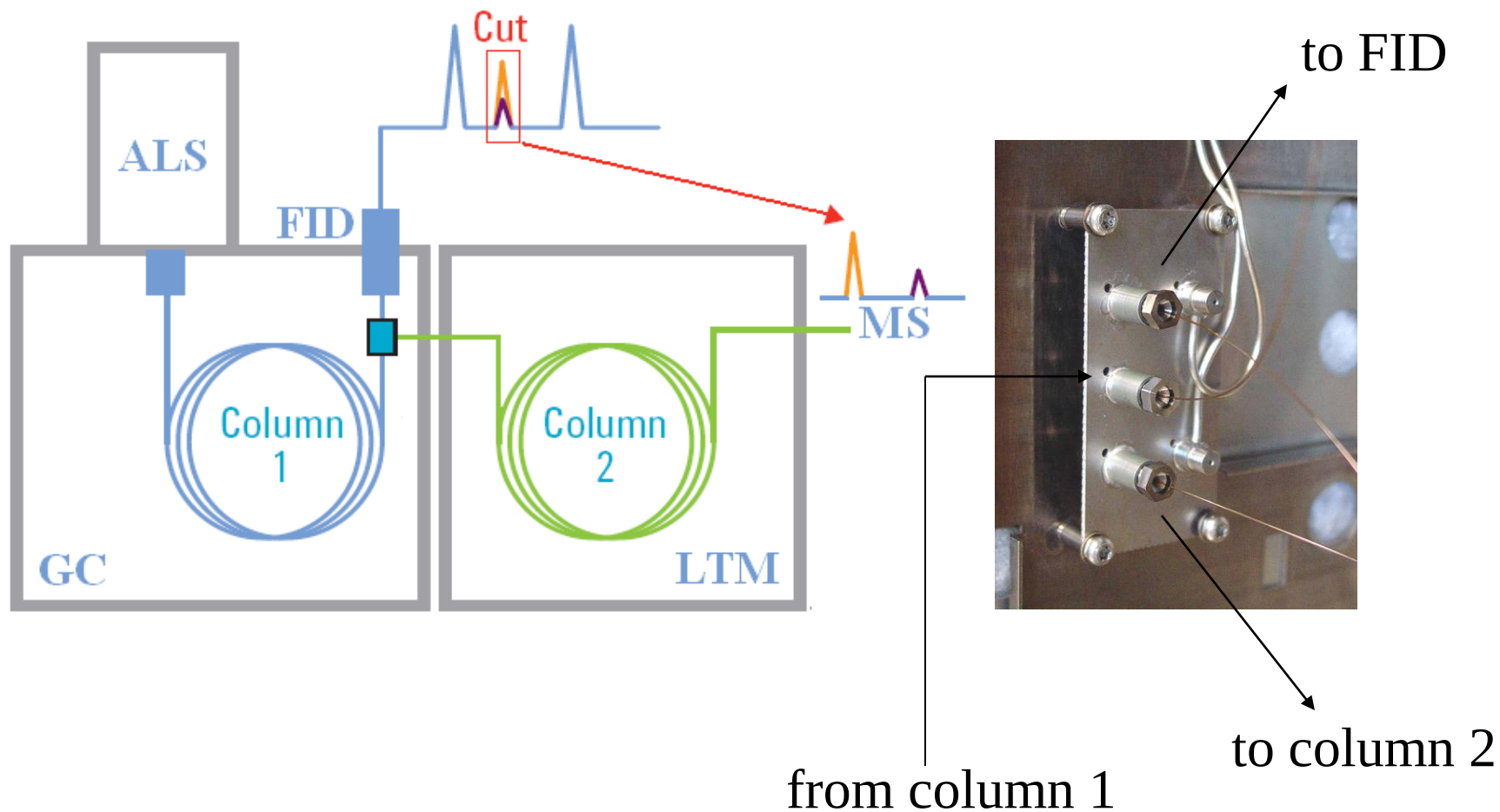
Abundance



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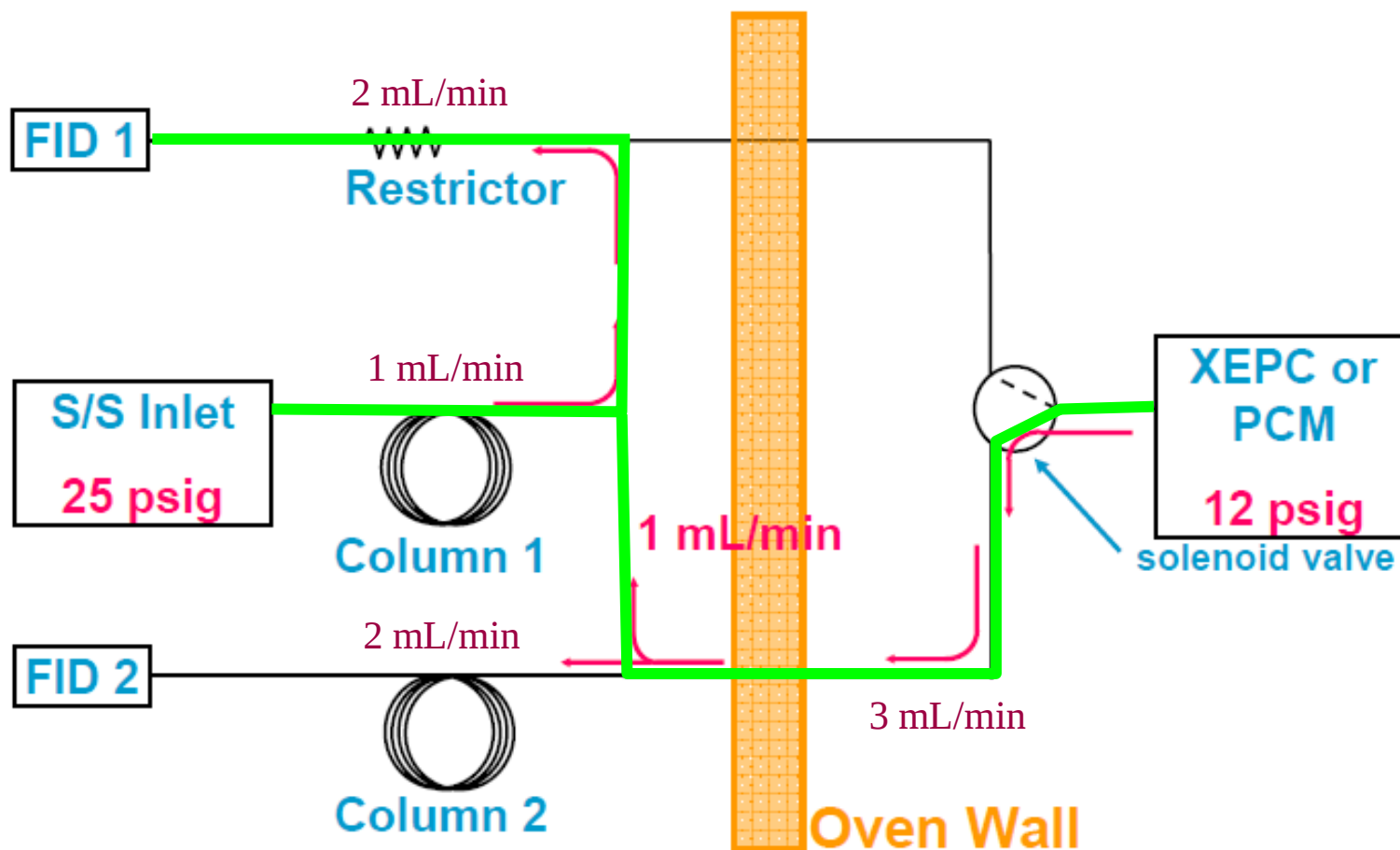
# CFT based 2D-GC



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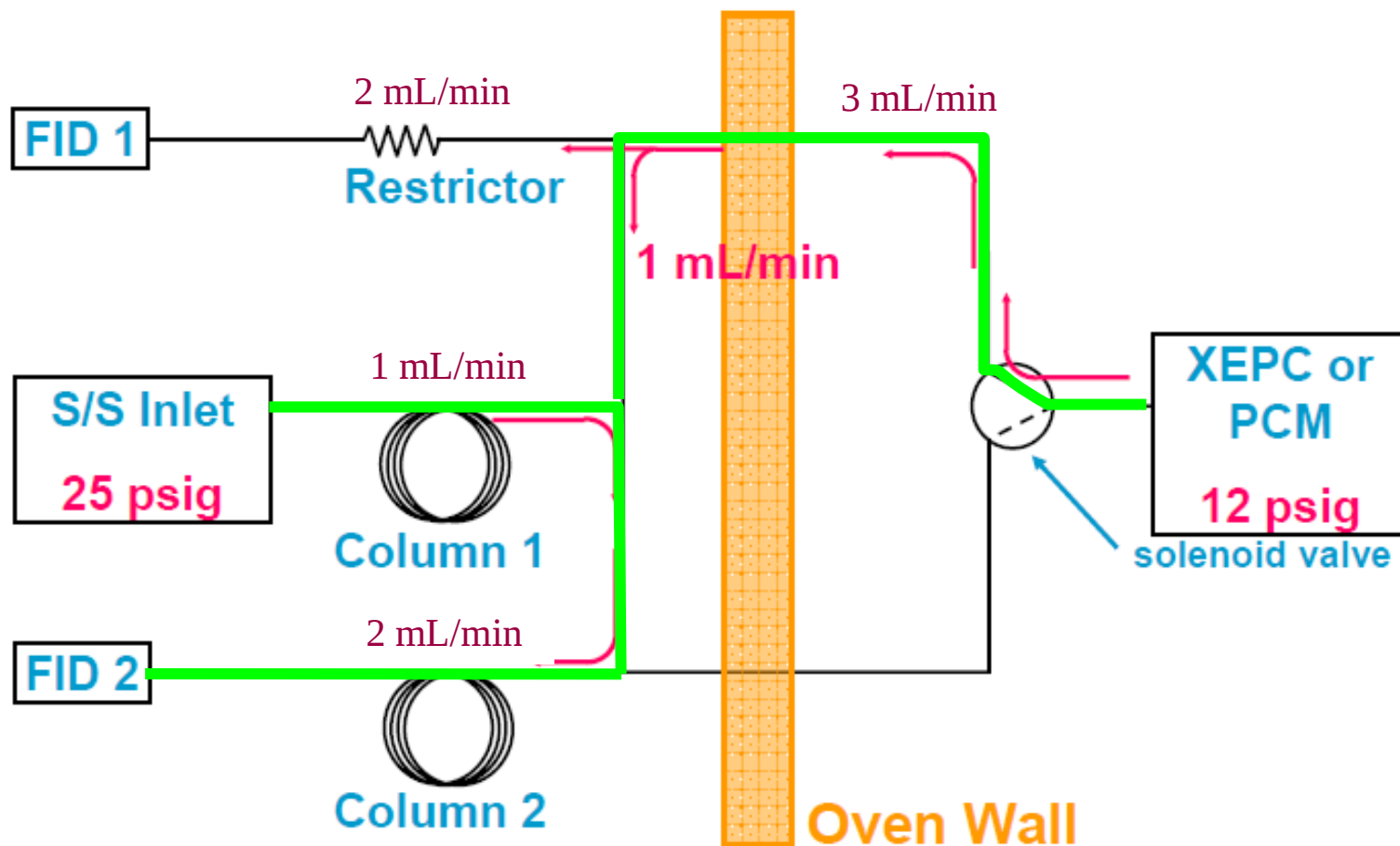
# Deans Switch principle – 1D analysis (FID1)



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

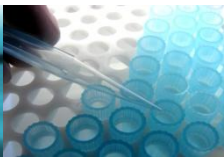
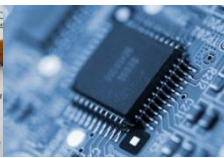
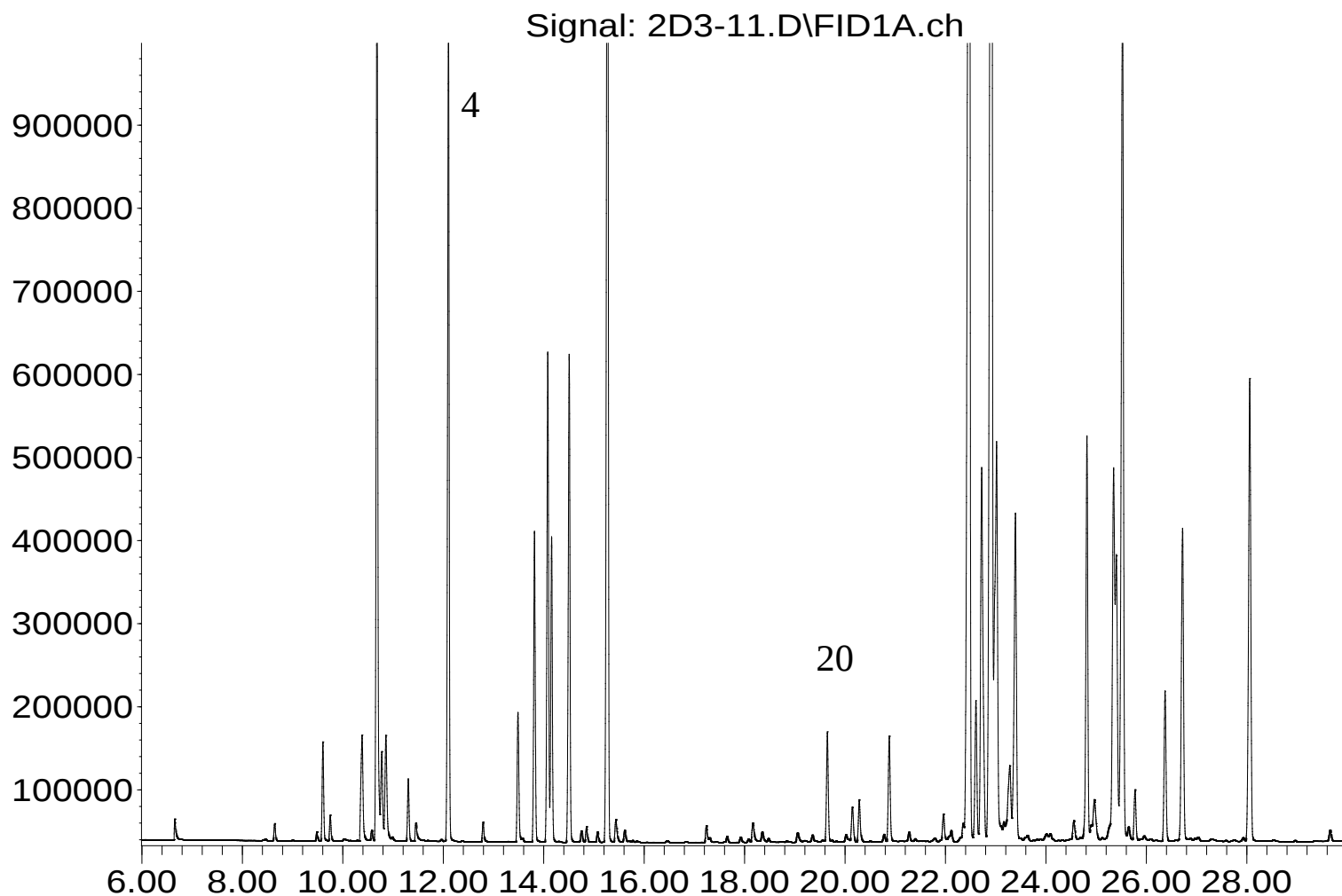


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# Suspected Flavor and Fragrance Allergens in Perfumes

Abundance

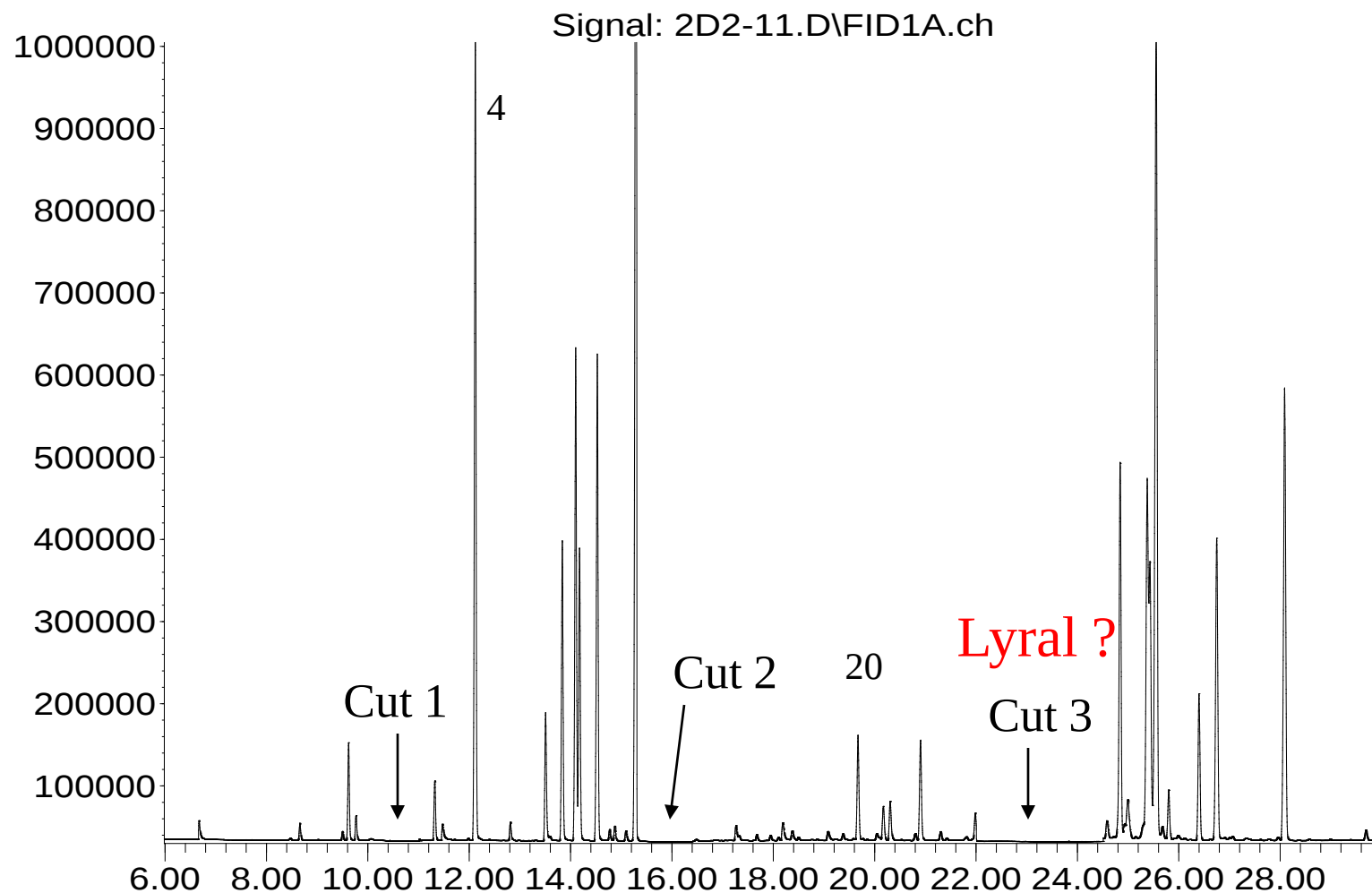


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# Suspected Flavor and Fragrance Allergens in Perfumes

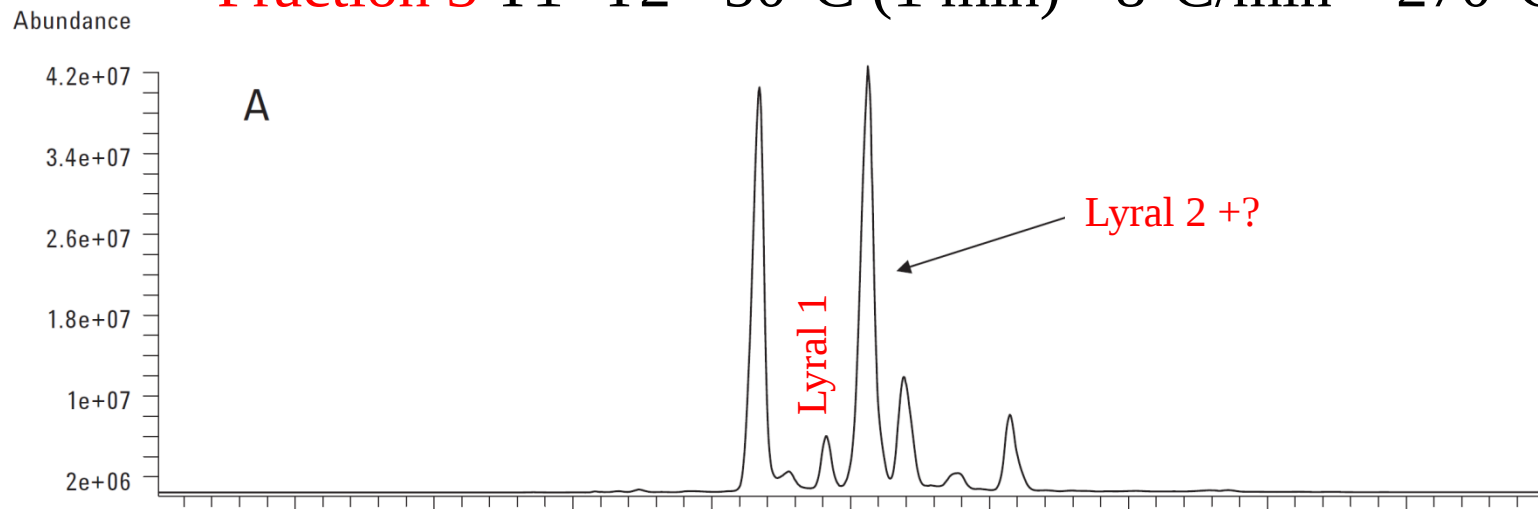
Abundance



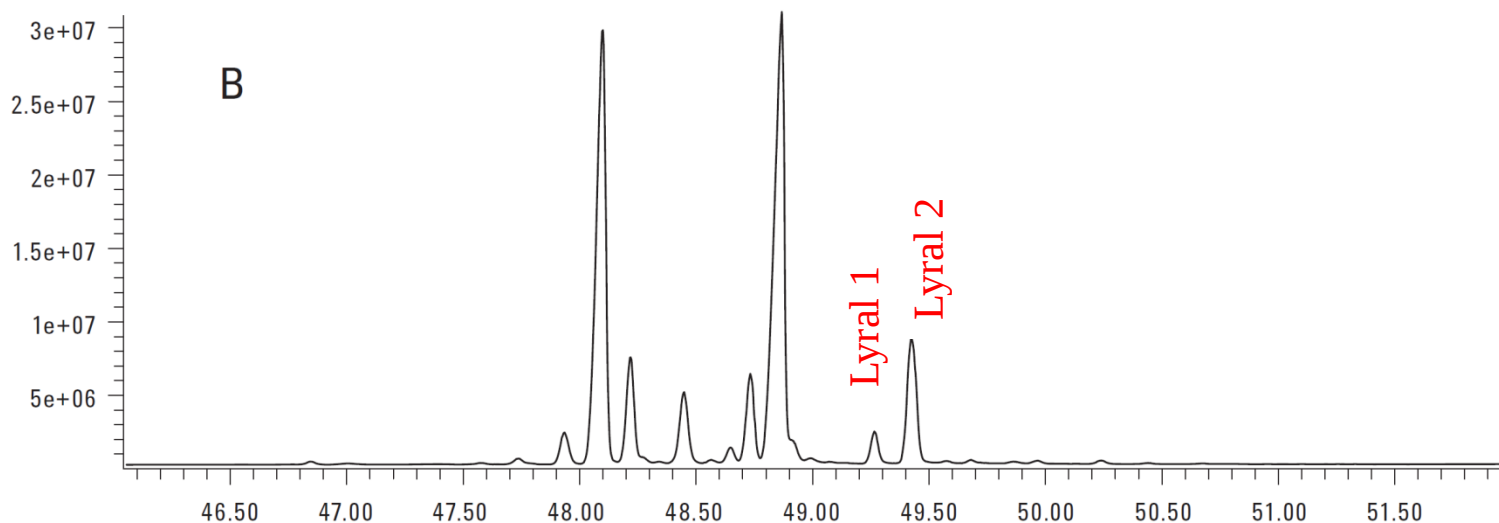
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## Fraction 3 T1=T2= 50 C (1 min) - 8 C/min – 270 C



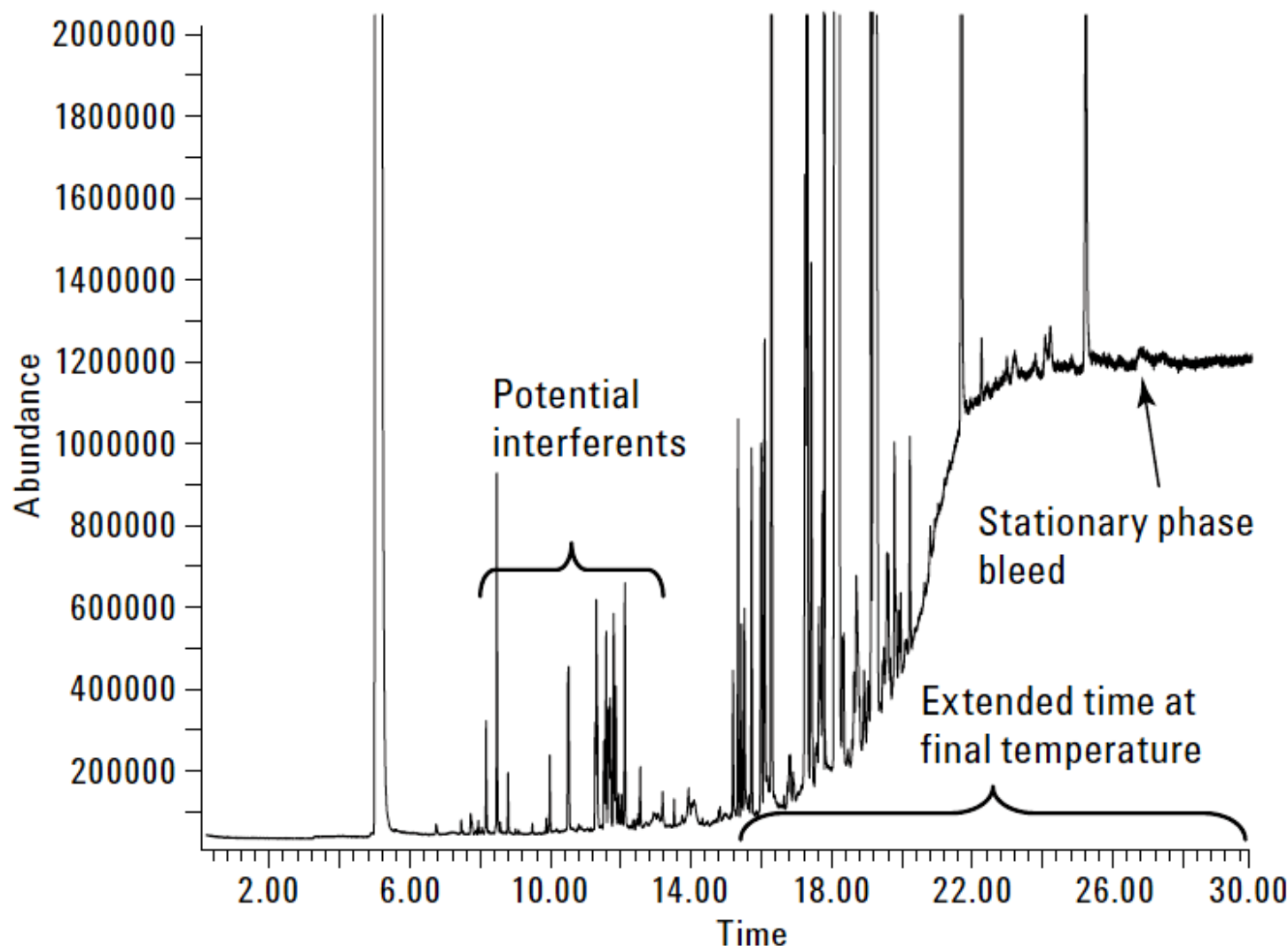
## Fraction 3 T1≠T2; T2= 50 C (25 min) - 6 C/min - 250 C



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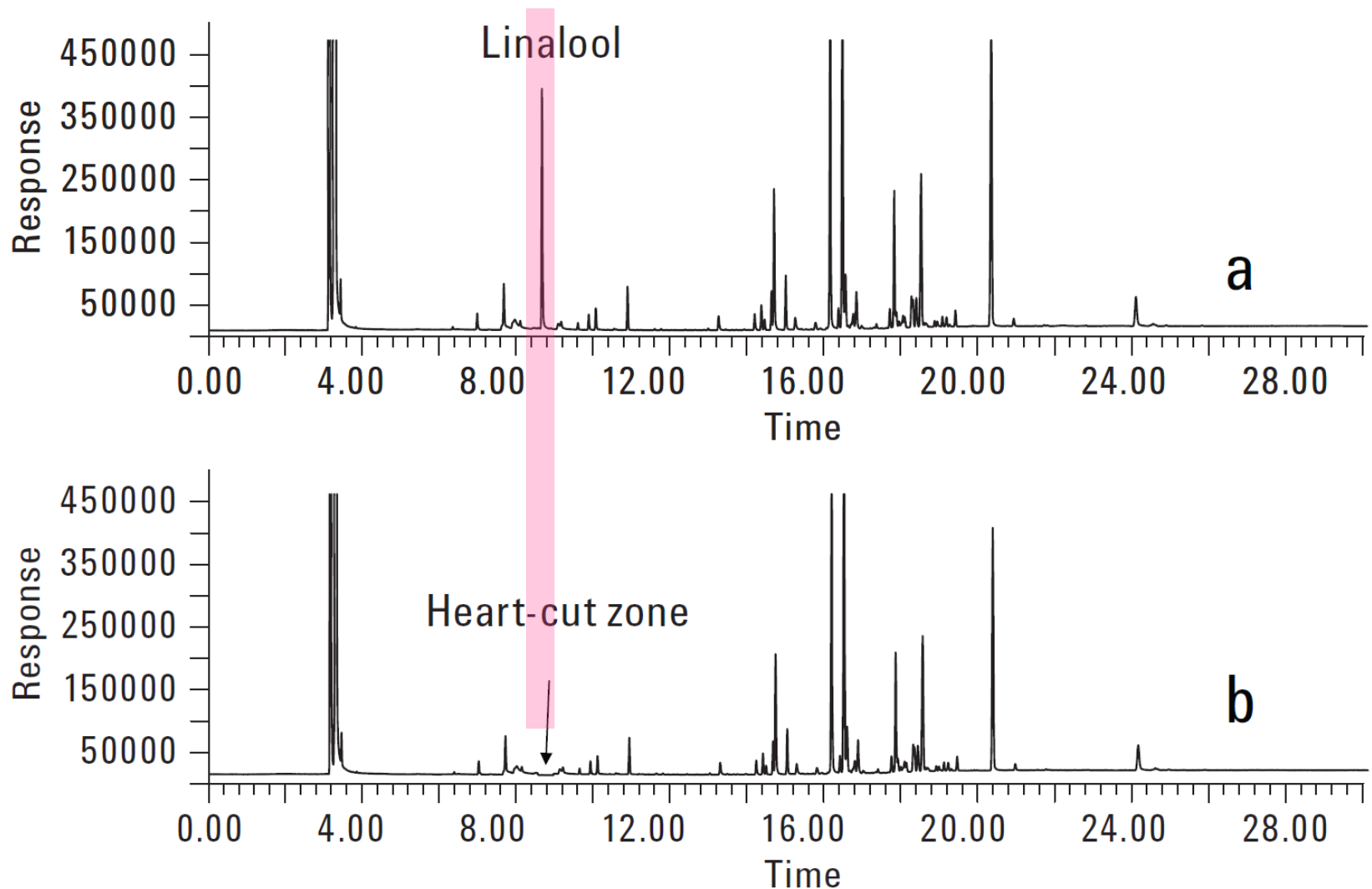
# Linalool analysis in essential oil on chiral GC column (CycloDex-B)



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

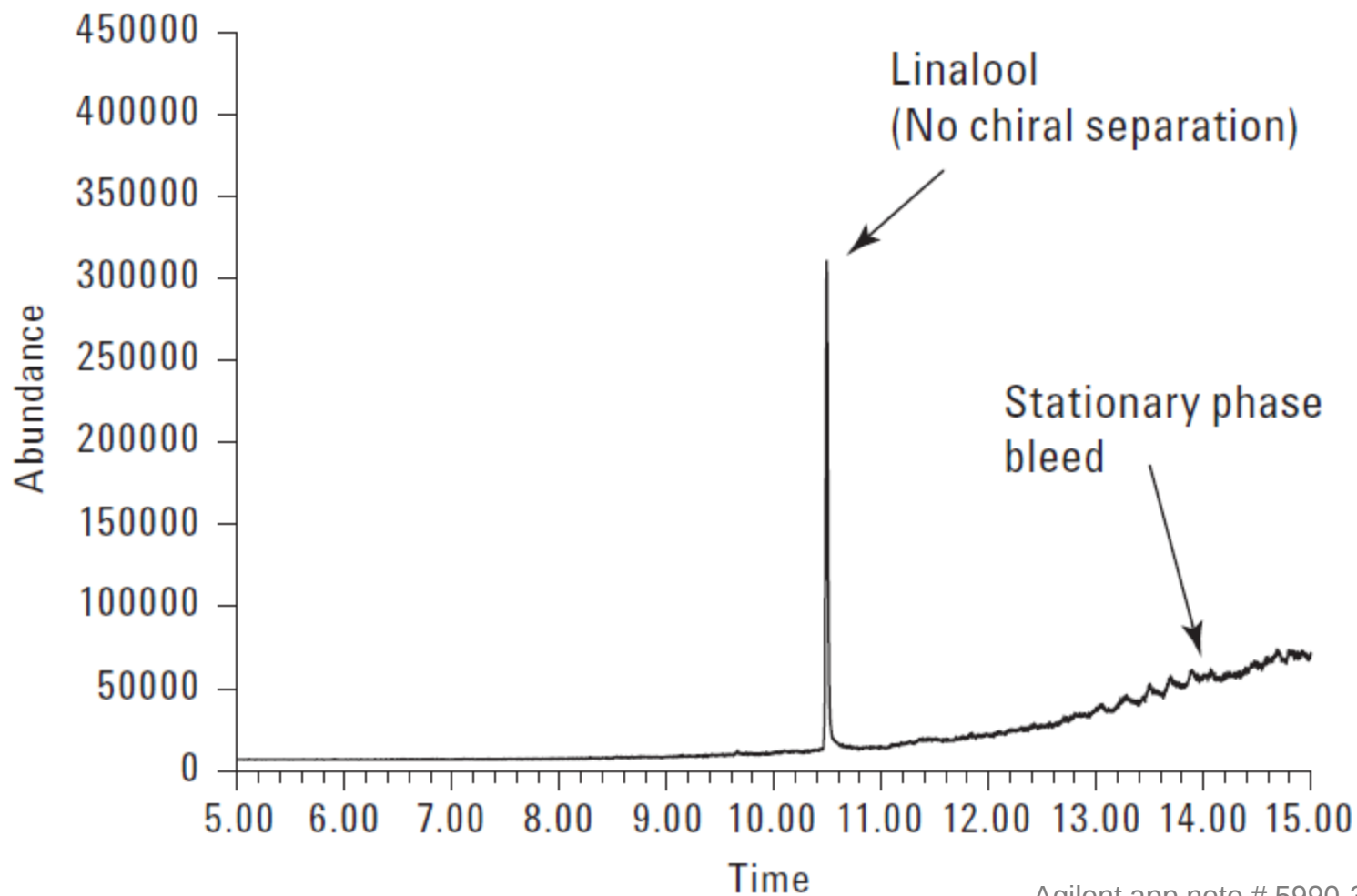


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## 2DGC analysis of fraction 8.5-8.9 min

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



Agilent app note # 5990-3428EN

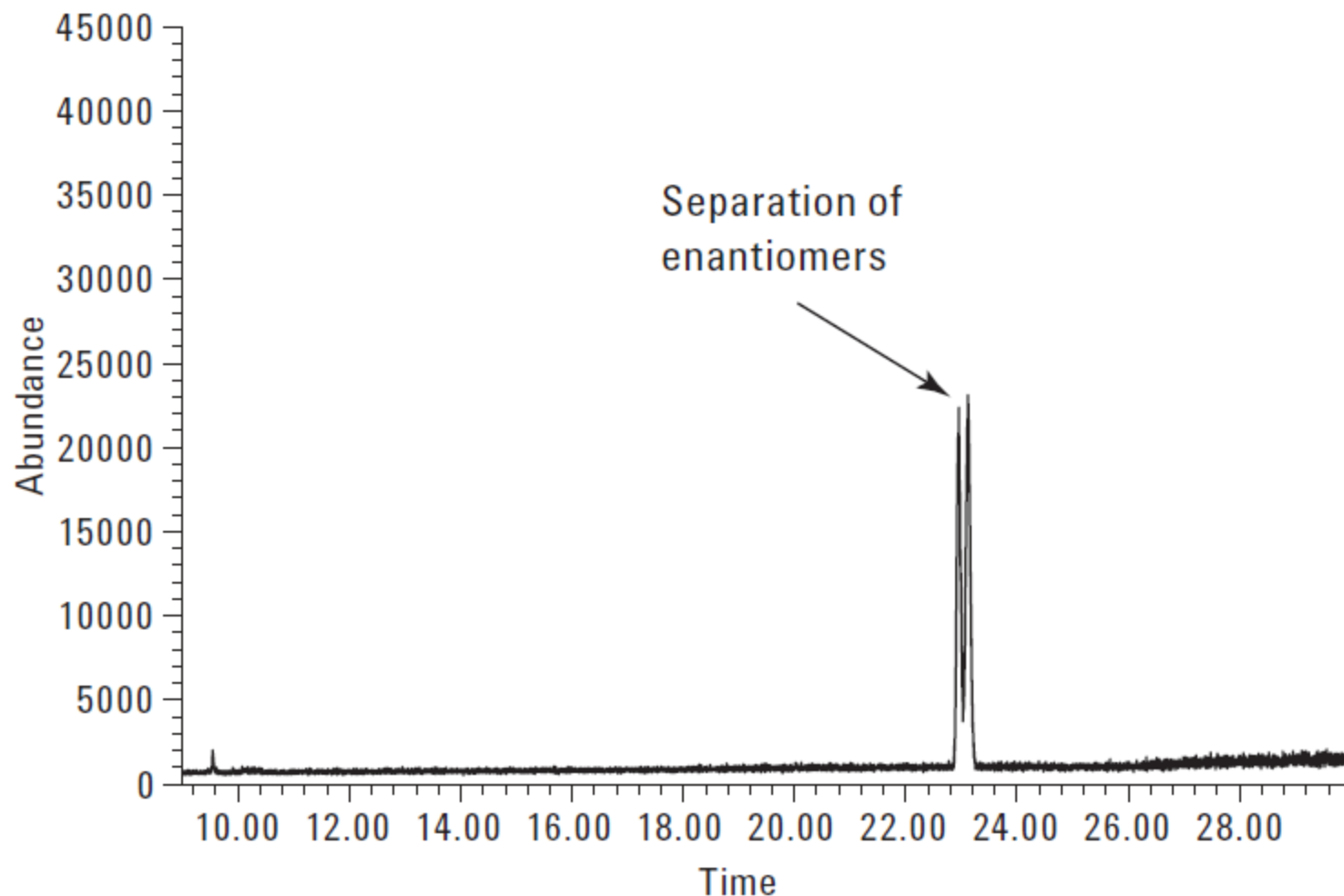


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## 2DGC analysis of fraction 8.5-8.9 min

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



Agilent app note # 5990-3428EN



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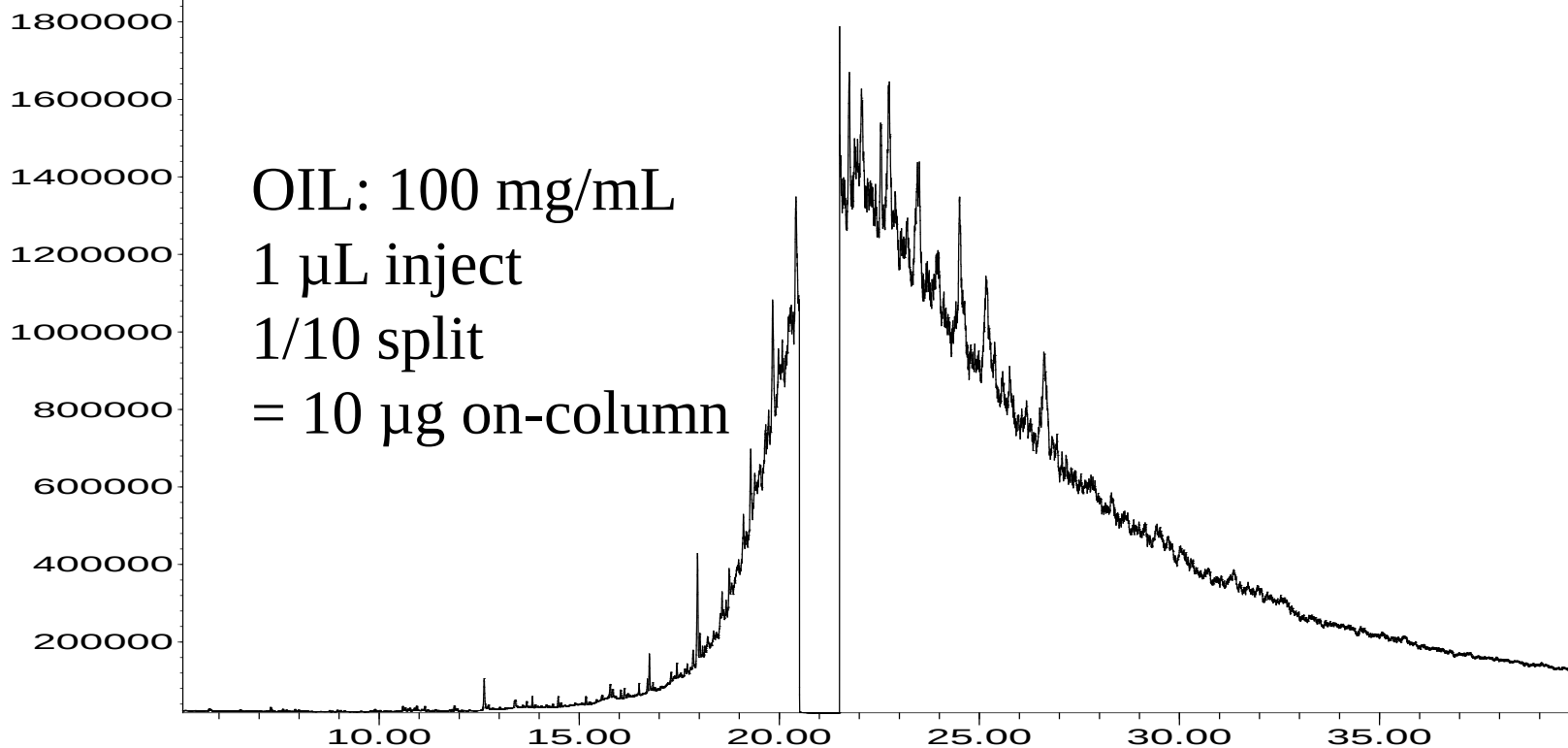
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# Determination of PAHs in Mineral Oil (Food Grade Oil)

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

Abundance

Signal: Find\_Dimension\_10.D\\NDA.ch



Time-->

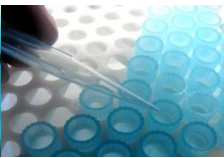
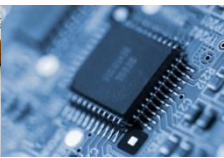
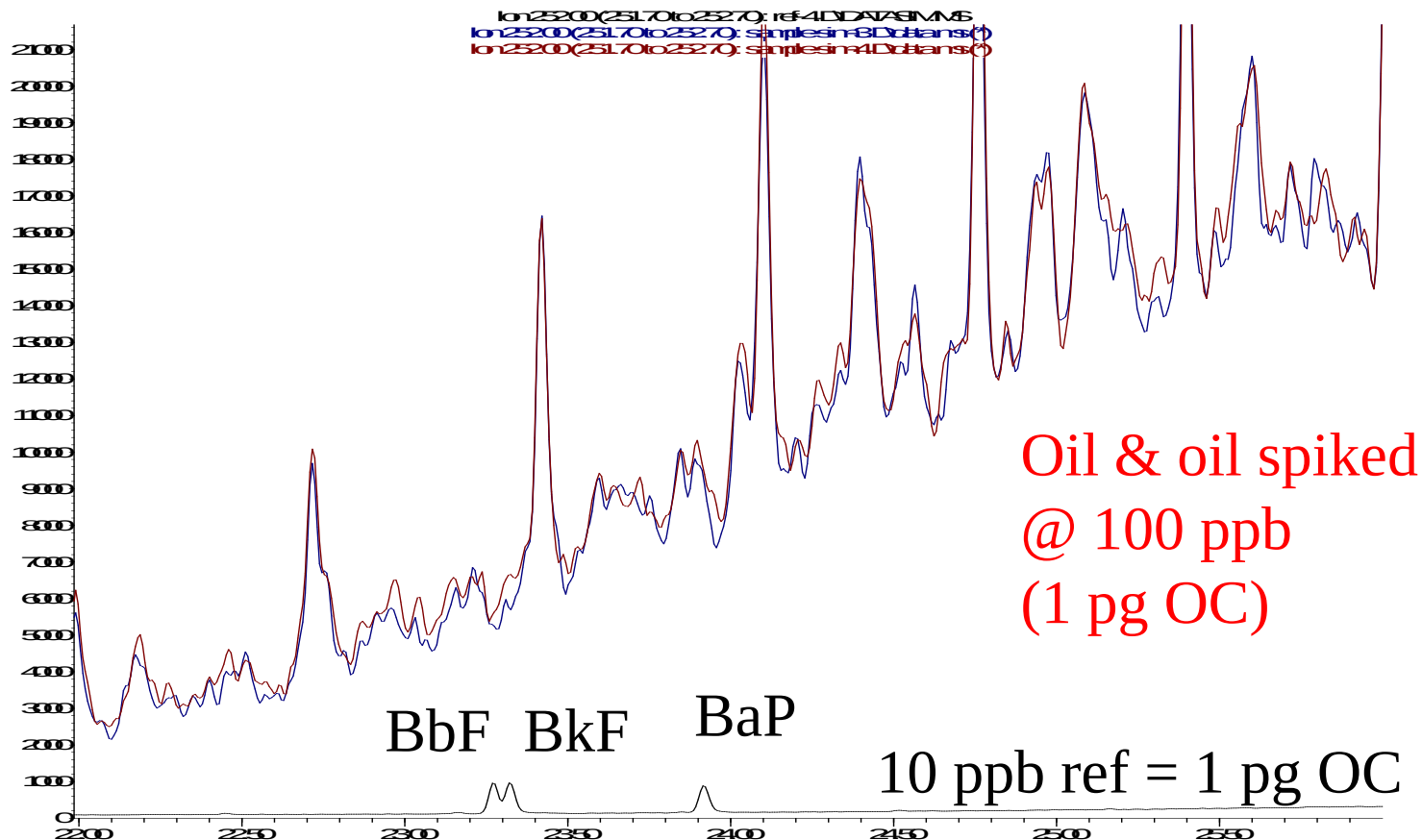


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# Determination of PAHs in Mineral Oil direct analysis by GC-MSD

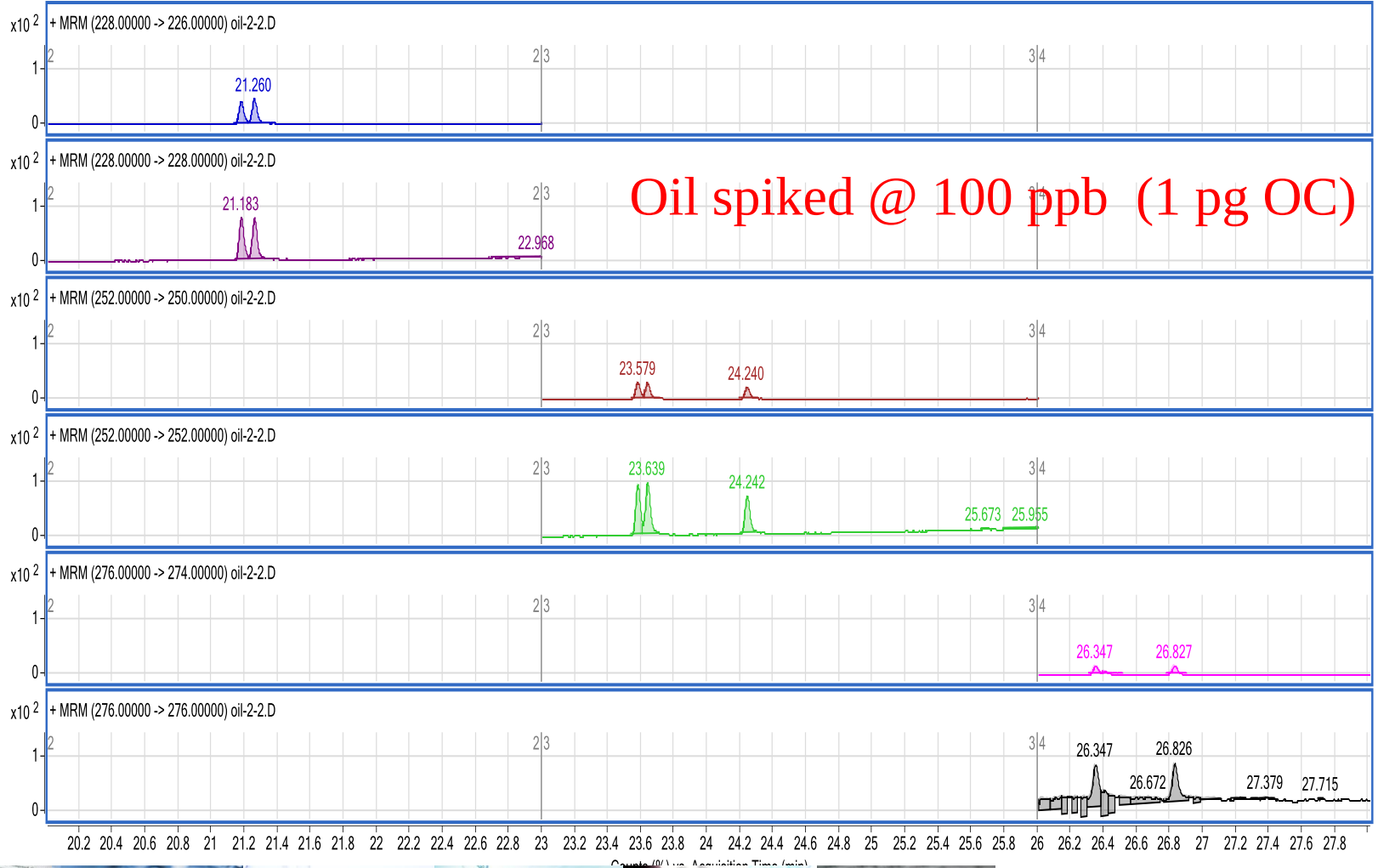
Acordate



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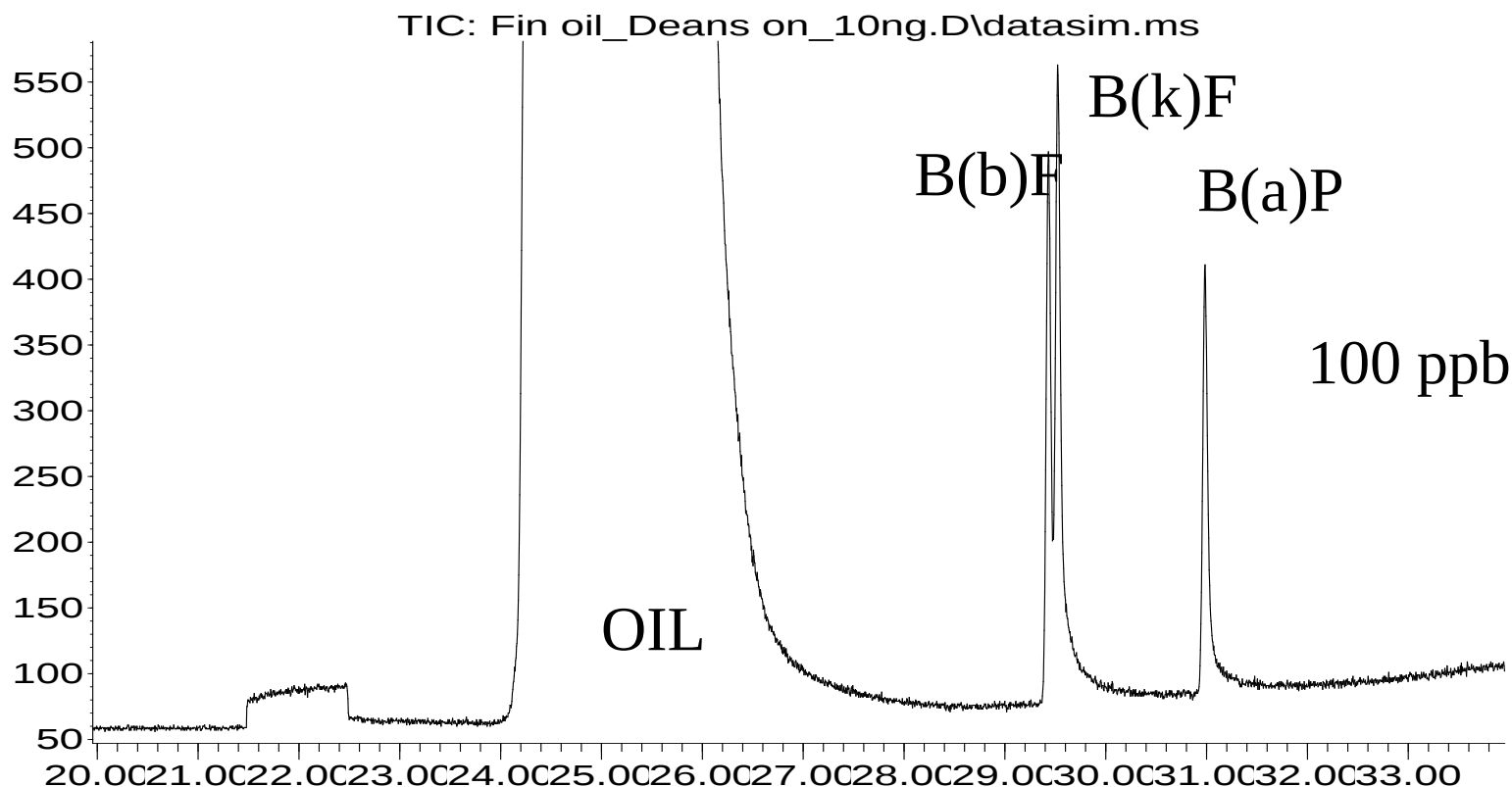
# Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



# 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



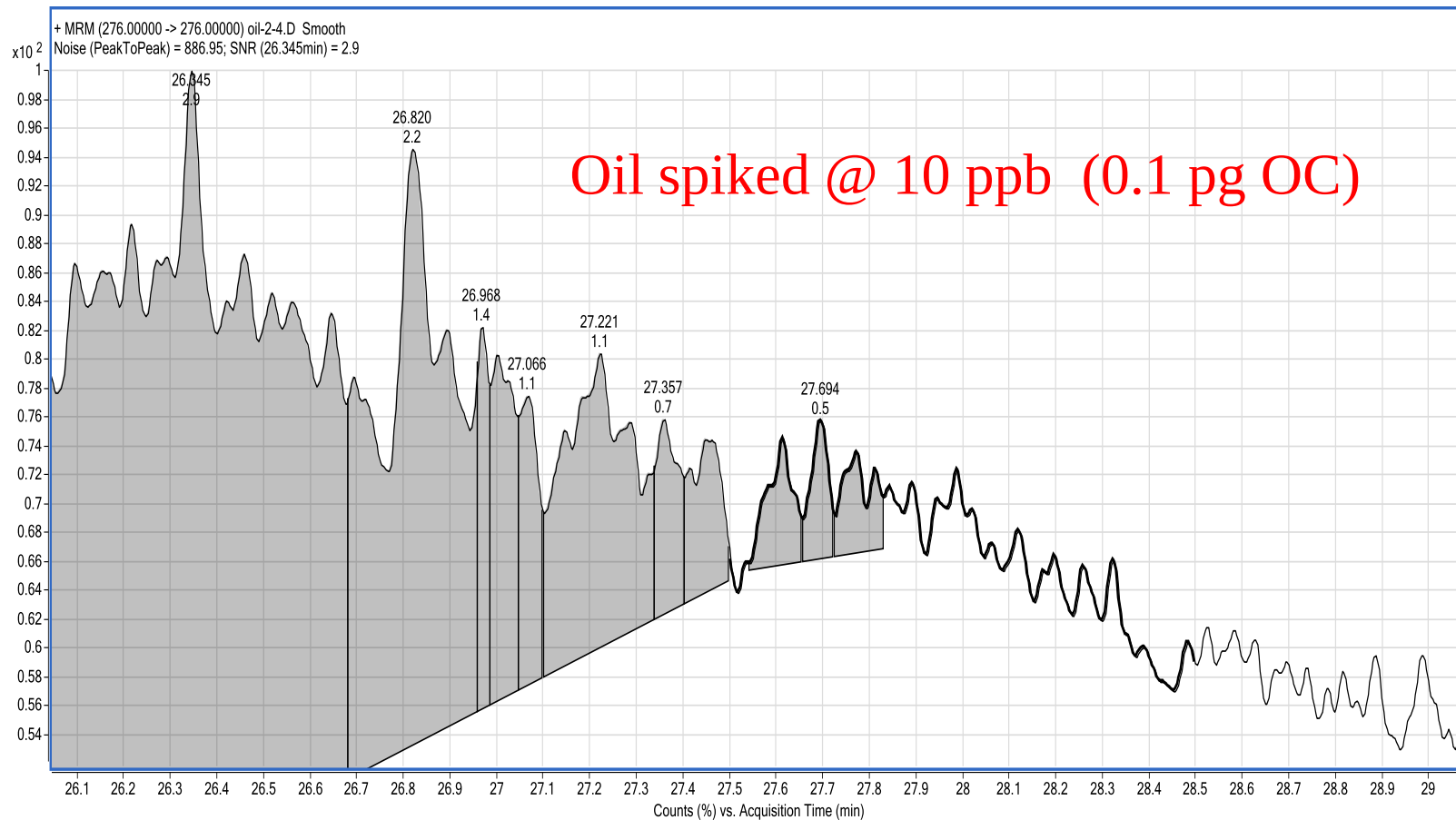
Time-->



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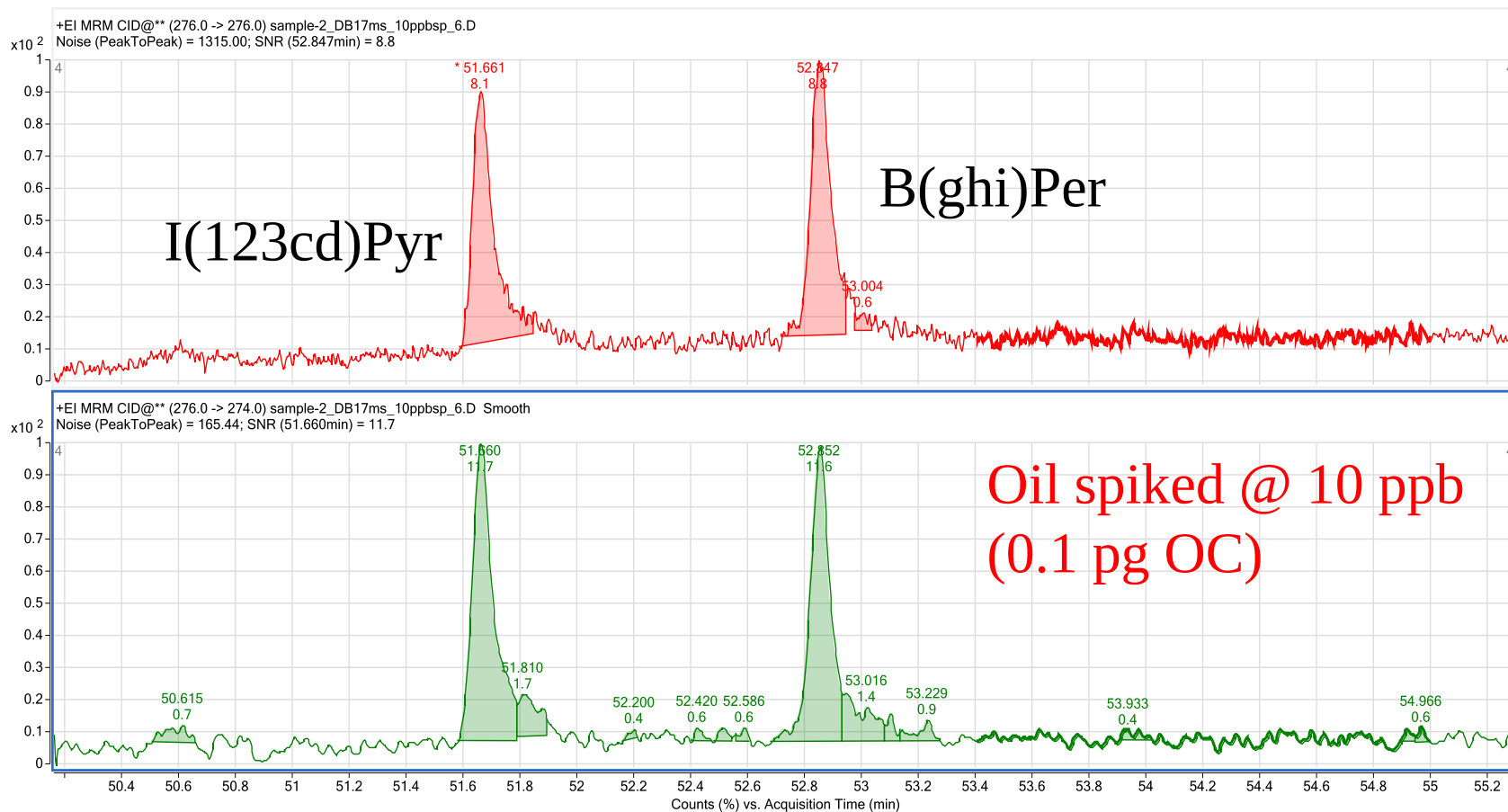
# Determination of PAHs in Mineral Oil direct analysis by GC-QQQ



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



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# 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)



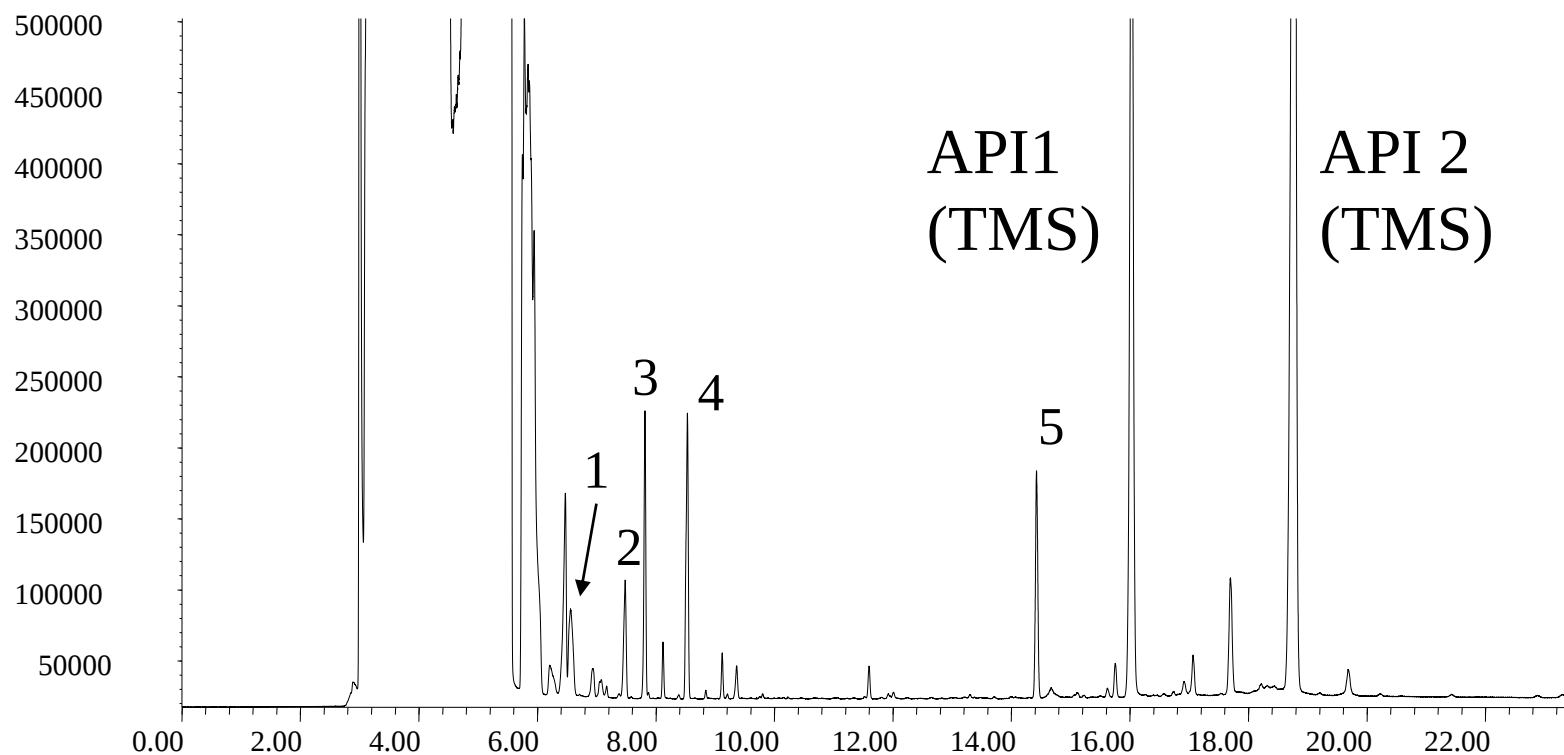
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# Halo-alcohols (TMS der) in Carbamazepine on Dim 1 - scouting run

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

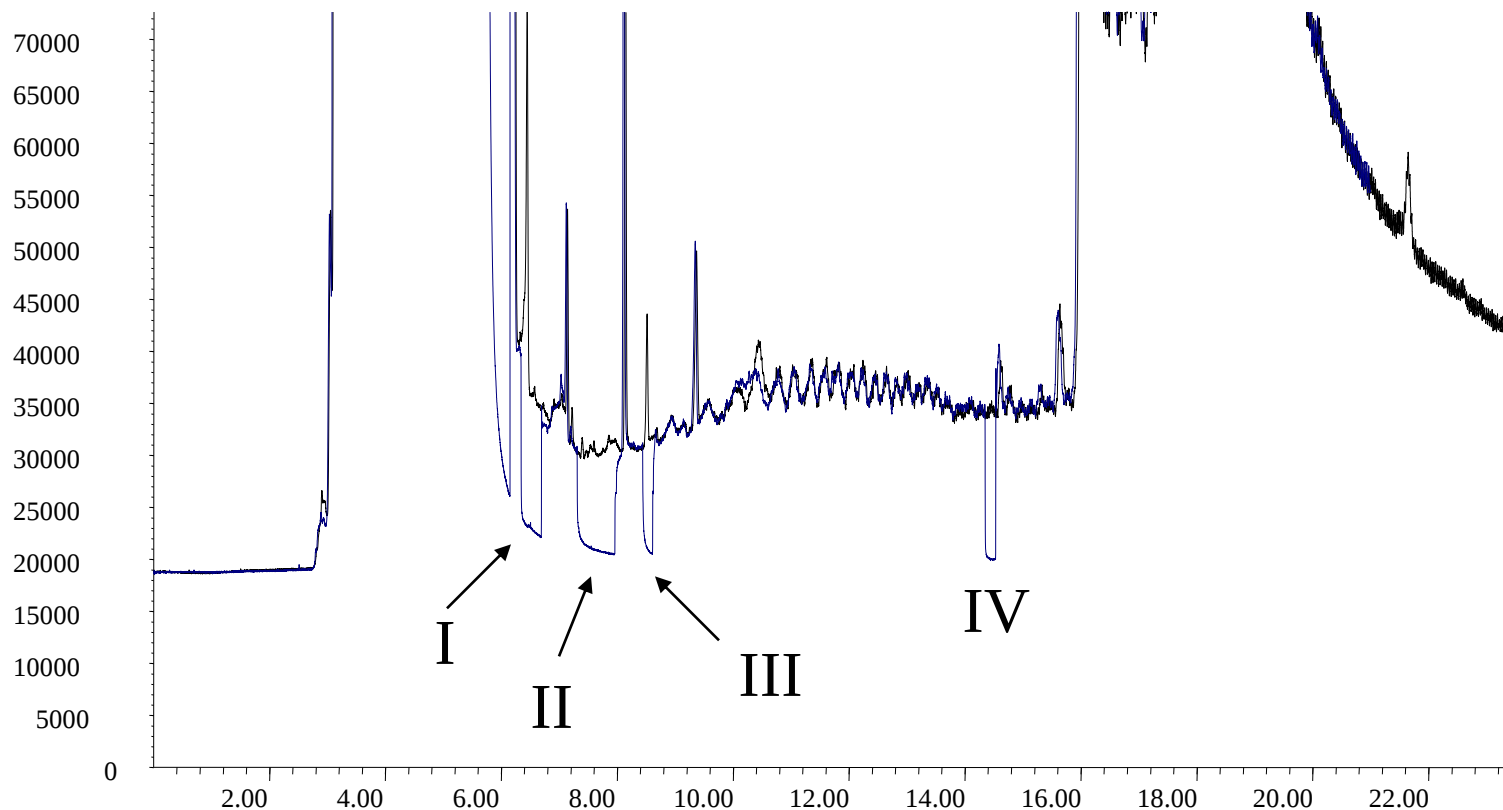


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# Halo-alcohols (TMS der) in Carbamazepine (1 ppm) on Dim 1 with and without heart-cut

Response\_



Time



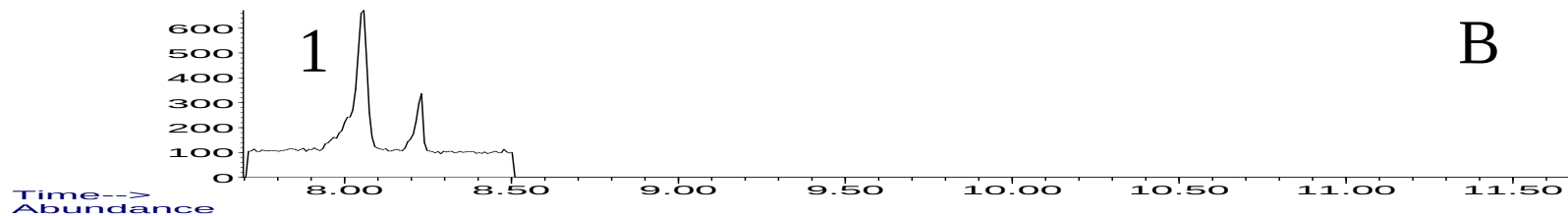
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# Dim 2

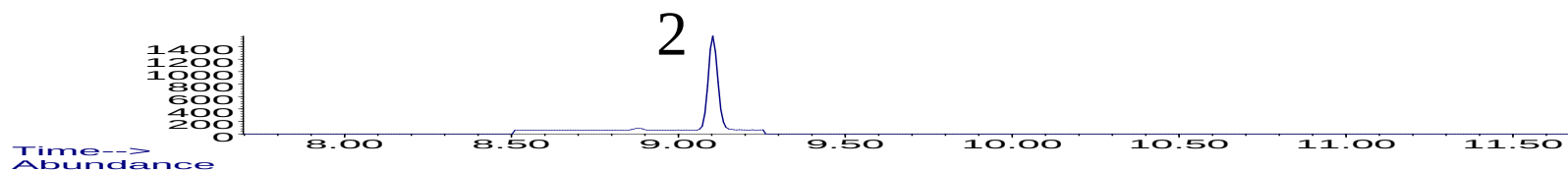
Abundance

Ion1390(137.0to139.0): 5.gAP\_valencasthmpis\_4.D\\DATA\\SIMS

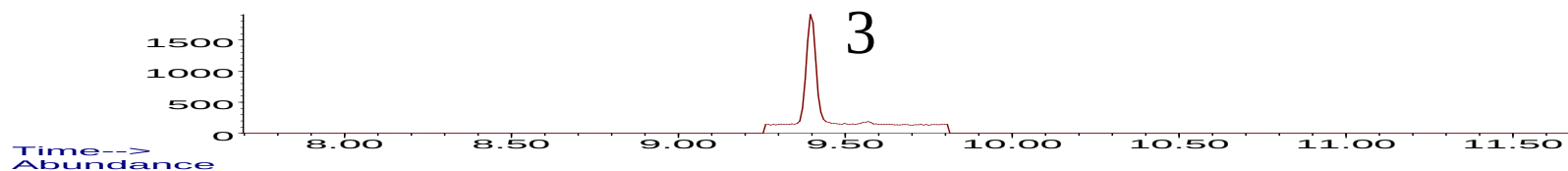


B

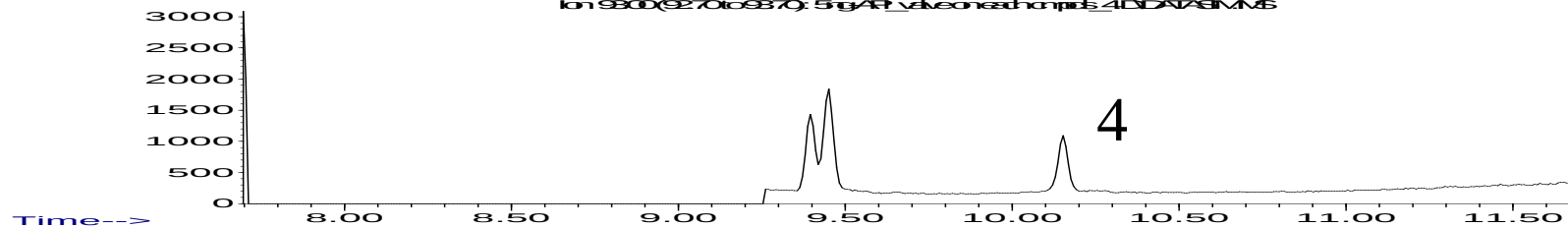
Ion1850(184.0to186.0): 5.gAP\_valencasthmpis\_4.D\\DATA\\SIMS



Ion1230(122.0to124.0): 5.gAP\_valencasthmpis\_4.D\\DATA\\SIMS



Ion980(97.0to99.0): 5.gAP\_valencasthmpis\_4.D\\DATA\\SIMS



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# Dim 2

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

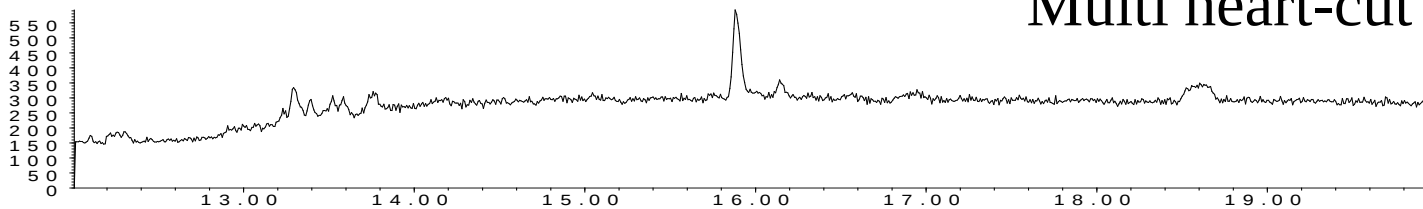
Abundance

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

C

5

Multi heart-cut

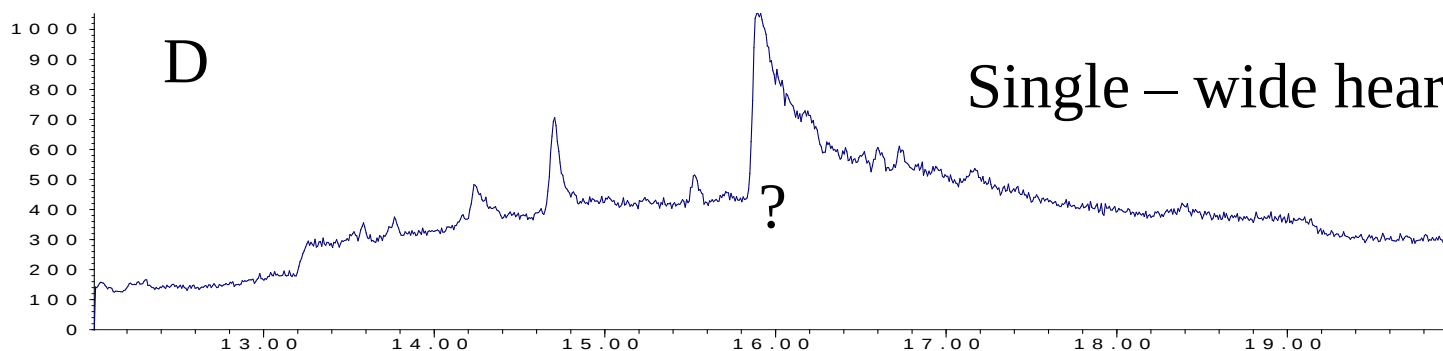


Time-->  
Abundance

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

D

Single – wide heart-cut



Time-->

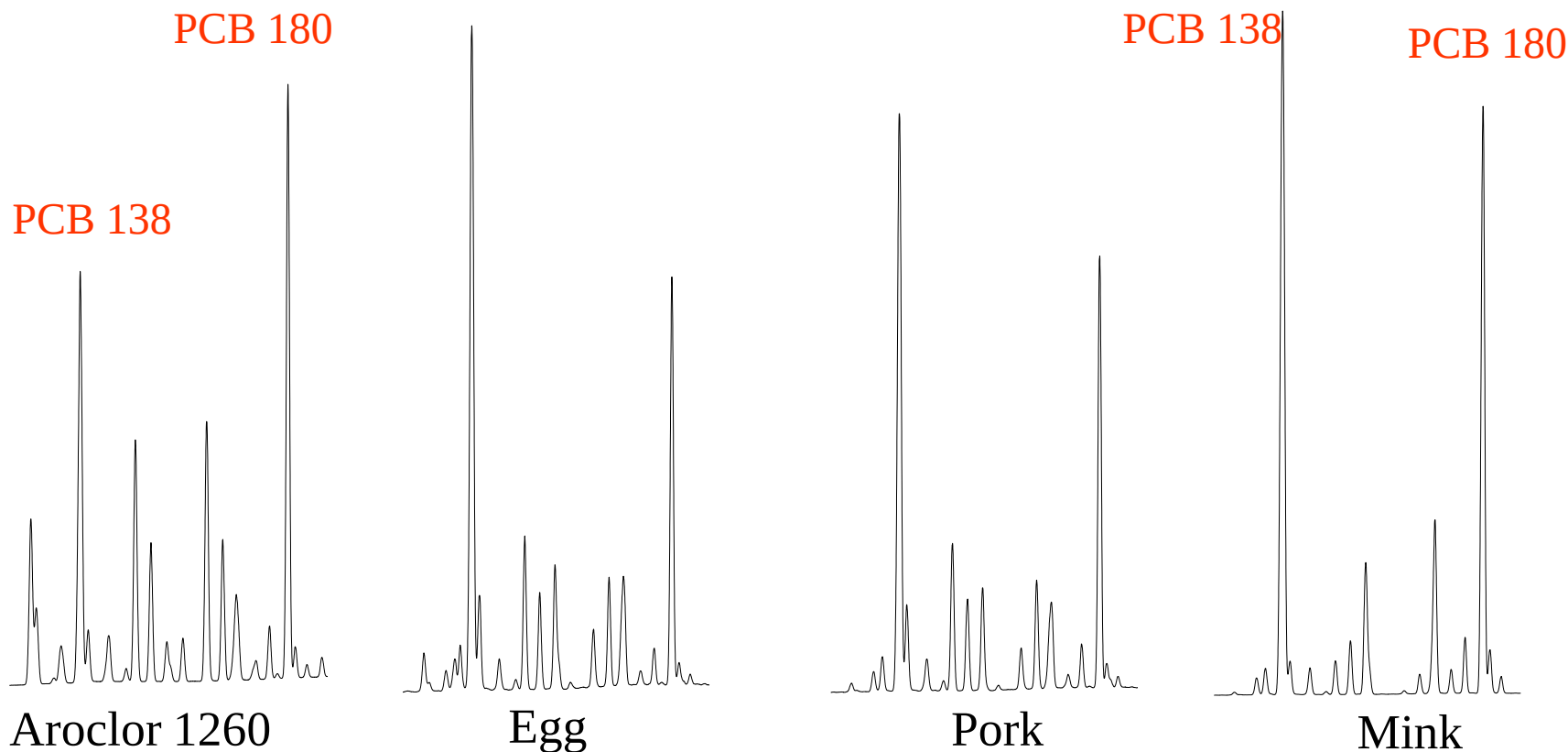


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# PCBs in Food Products

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



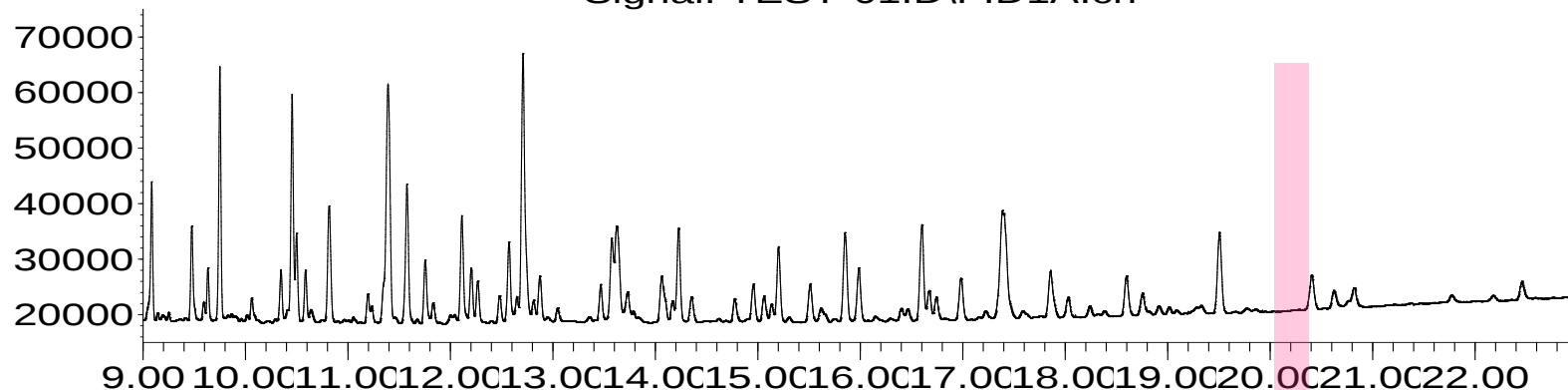
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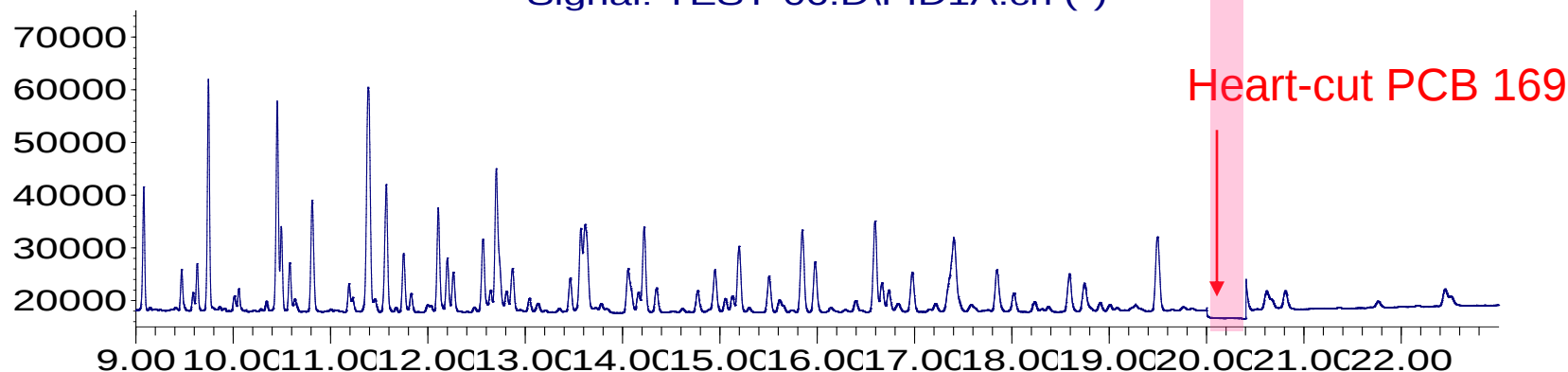
# PCB analysis by GC-GC-MS:

## 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

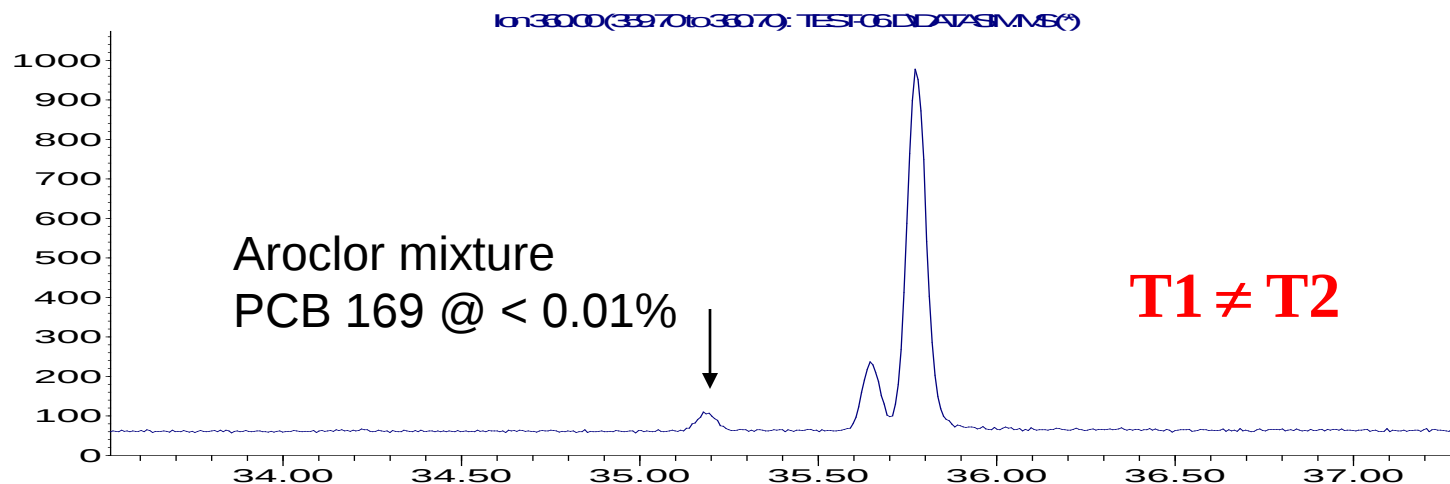
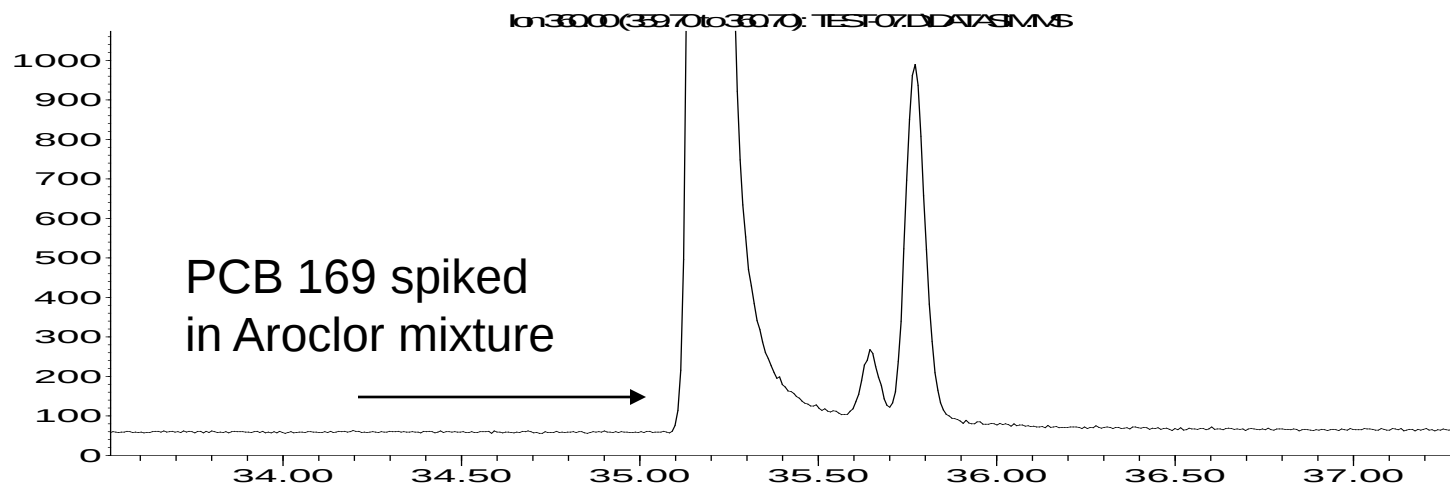


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# PCB analysis by 2DGC-MSD:

## 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



Time-->

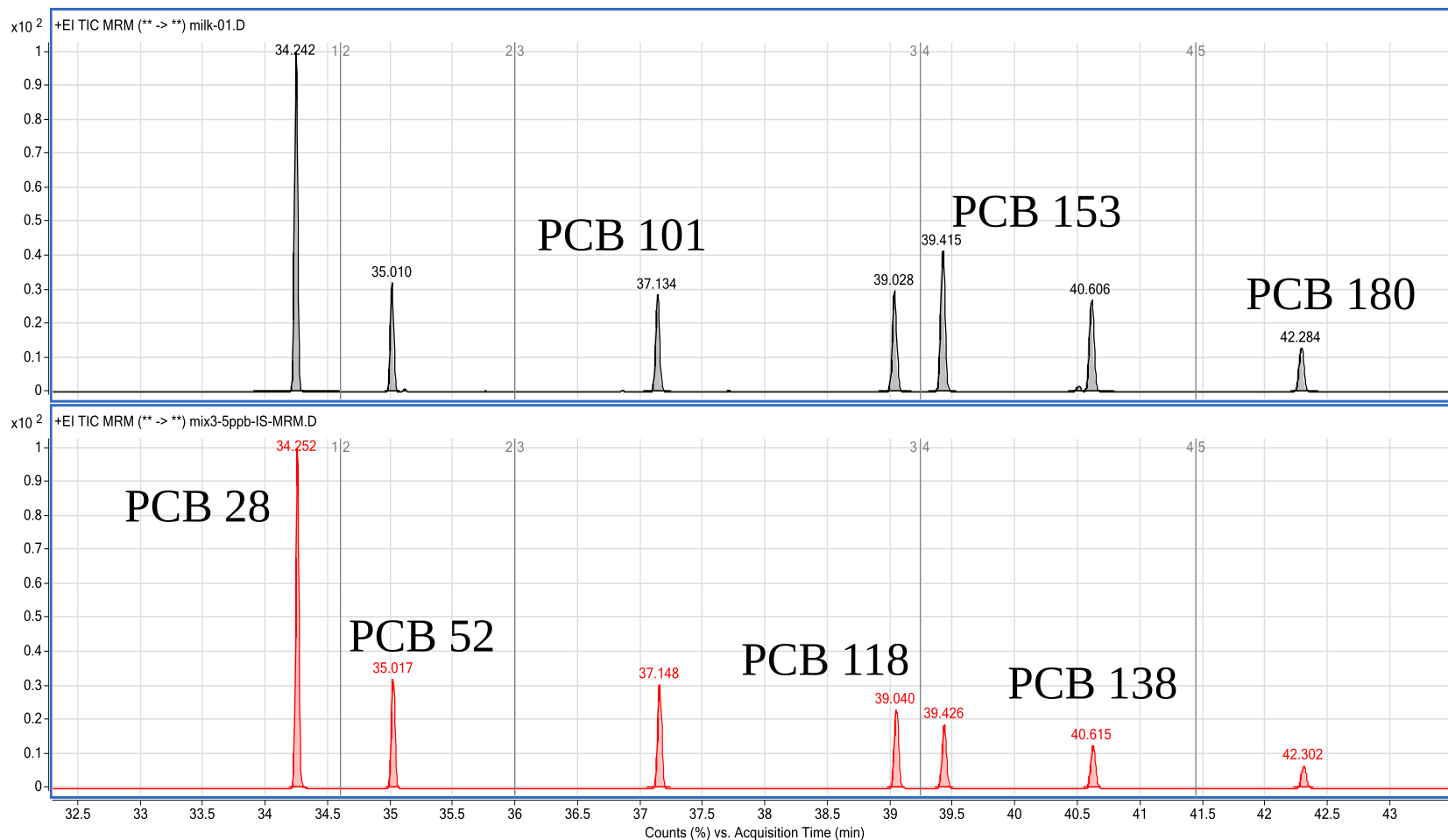


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# PCB analysis in milk powder by 2DGC-QQQ:

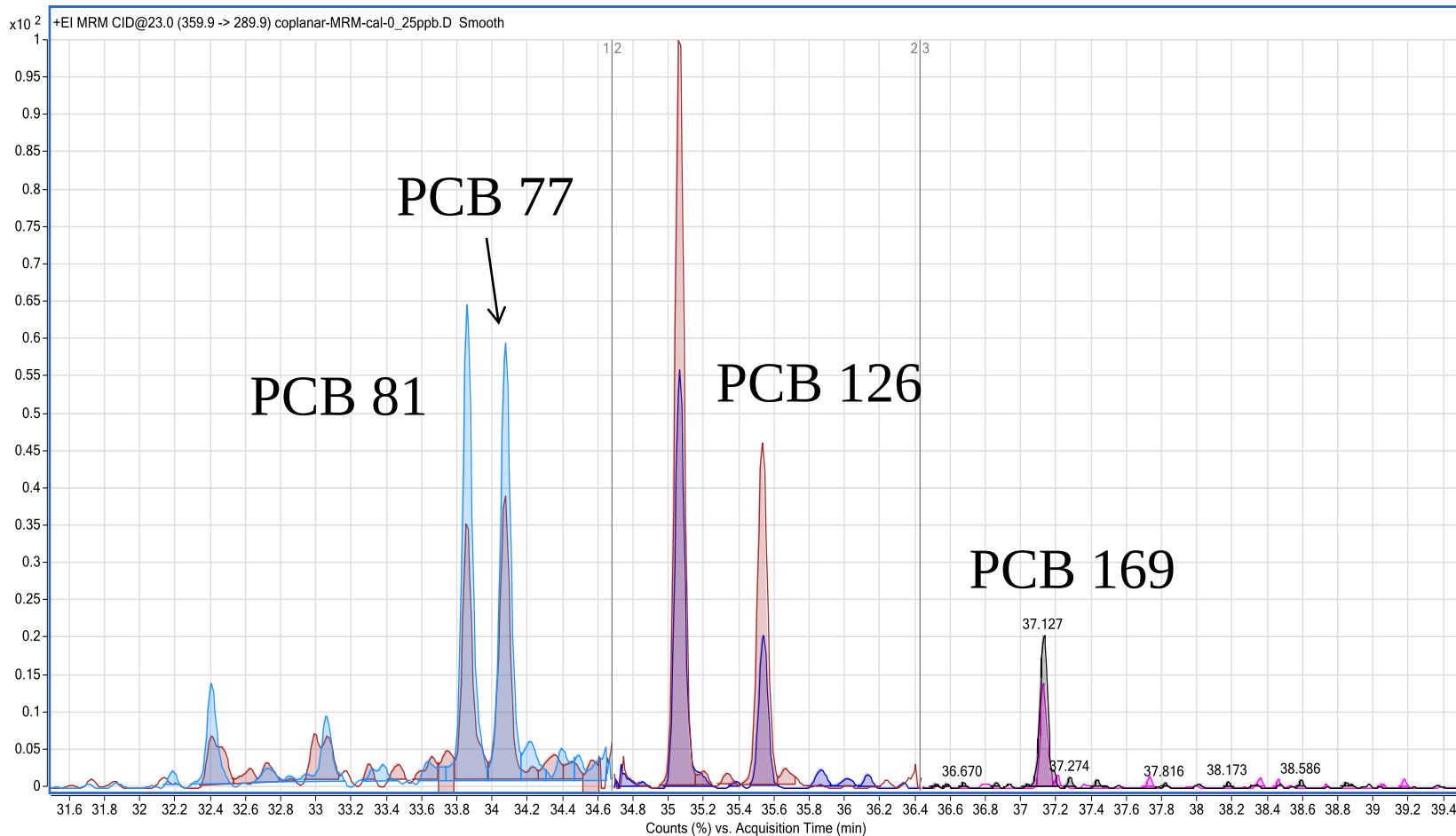
## 30 x 250 HP-5 (FID) + 30 x 250 DB-17 (MS)



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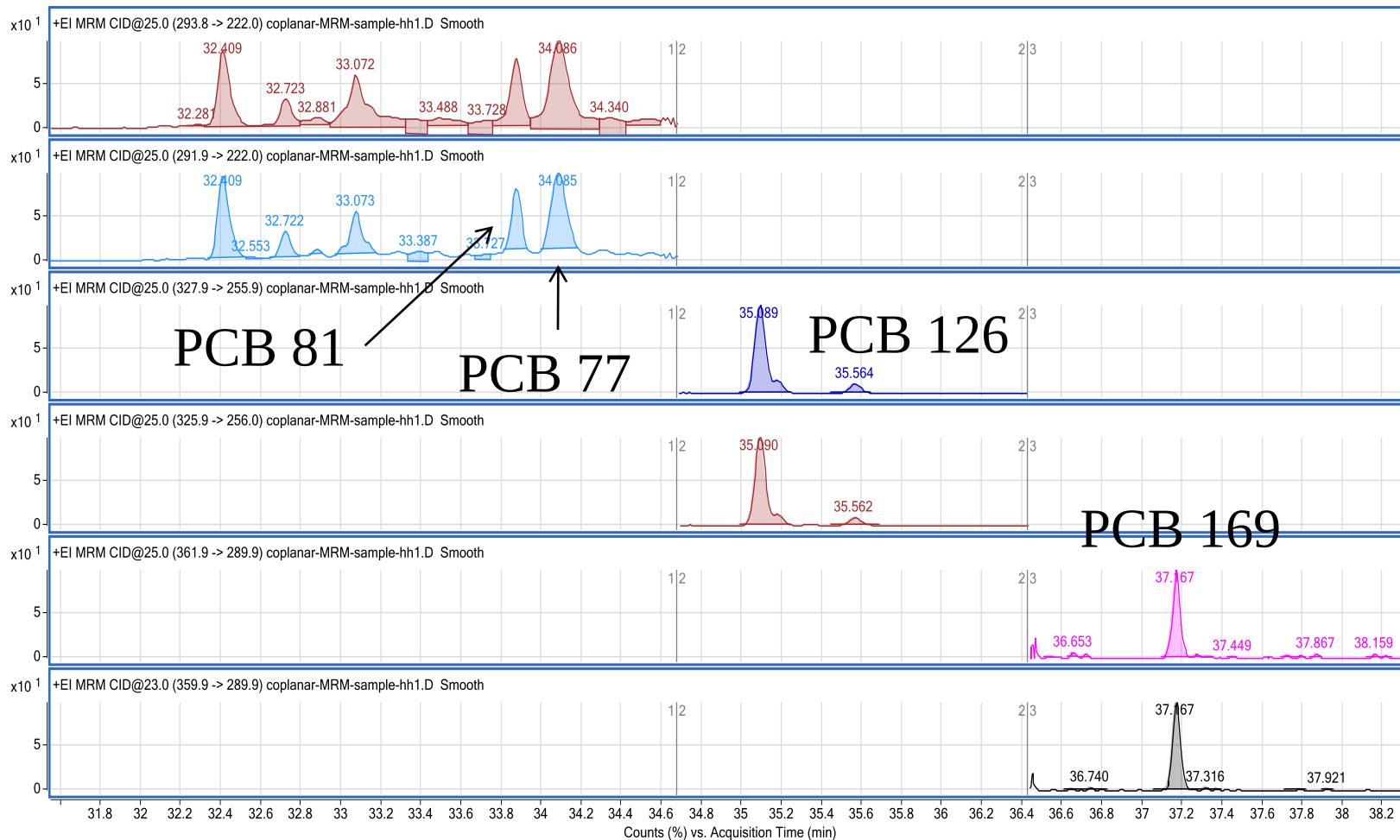
# Coplanar PCBs 0.25 pg/ $\mu$ L



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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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