

# Characterization of Odorous Compounds in Recycled Materials



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**Certech**  
R&D partner in chemistry

16<sup>th</sup> MDCW 2025



**Certechem**  
R&D partner in chemistry

R&D partner and supplier of analytical and technological services for companies involved in activities related to chemicals, plastics, life science



- 573 industrial contracts in 2023
- 33 employees
- > 1700 industrial collaborations since 2000

	<b>ENVIRONMENT</b> Air quality Health & safety Energy Circular Economy
<b>MATERIALS</b> (Bio-based) polymers & composites Emissions and odours from materials Lightweight materials Mechanical Recycling	
	<b>CHEMISTRY &amp; INDUSTRIAL PROCESSES</b> Micro / Meso fluidic technologies Catalysis and synthesis Chemical Recycling
<b>ANALYTICAL &amp; TECHNOLOGICAL SERVICES</b> Extended characterization platform / reverse engineering Pilot equipment Products and processes improvement	

# How to identify the specific components of an odor?

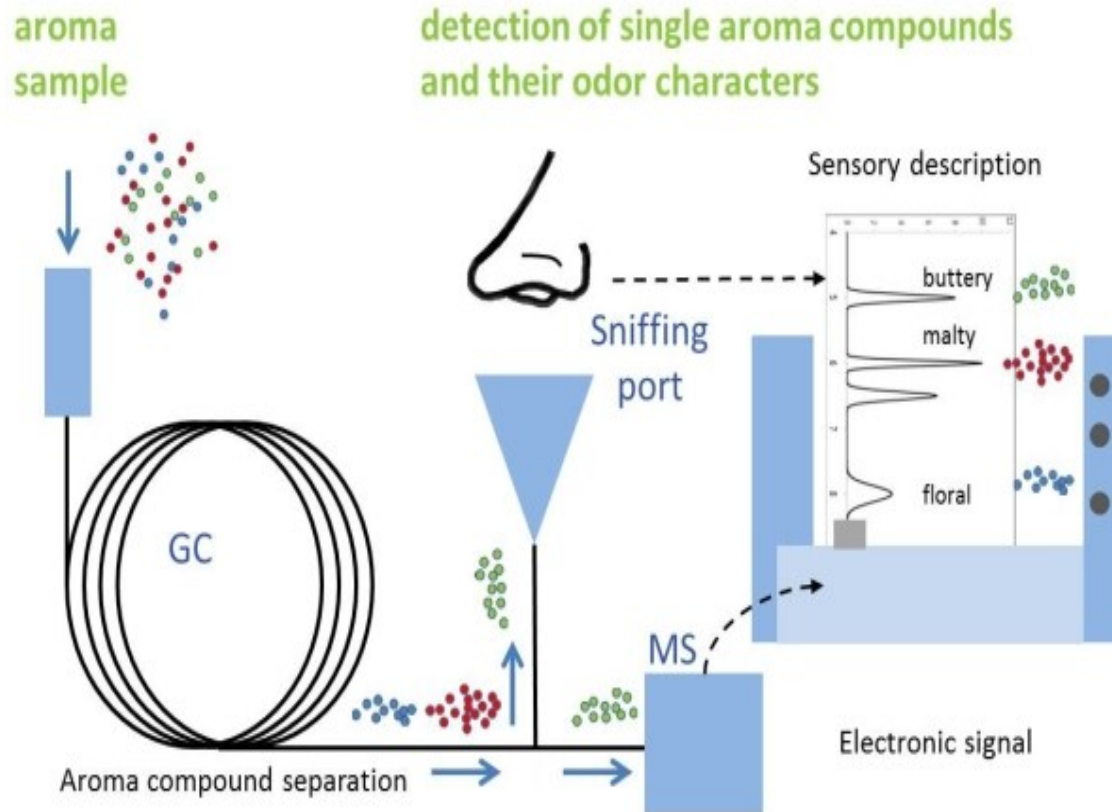


<https://whatsthat smell.wowwee.com/>



- Subjective topic
- Decoding the structure-odor relationship : not straightforward
- Matrix effect
- Complex correlation (synergy, antagonism)

# Correlation chemistry/odor by GC-MS/Olfactometry (« sniffing »)



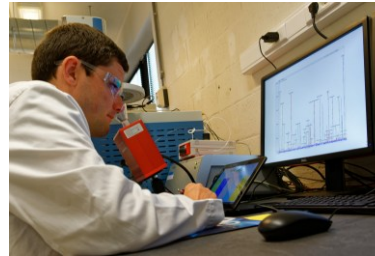
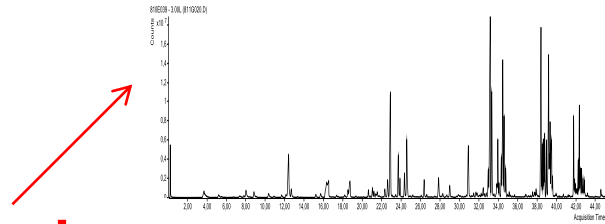
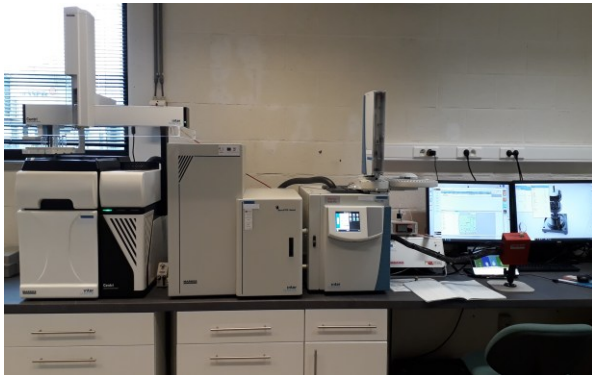
<https://odourobervatory.org>

## ➤ Main challenges :

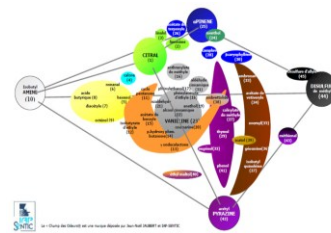
- Odor panelists: methodology & training
- Instrumentation & environment
- Aroma samples vs Material samples
- Minor compounds responsible of odor
- Human nose sensitivity >> detectors
- Complex samples  
(PP, HDPE, recycled materials)

# Correlation chemistry/odor by GC-MS/Olfactometry (« sniffing »)

## ➤ Global odor assessment and decomposition



- Trained sensorial expert (performances recorded)
- Descriptors + Intensity scale
- Continuous on-line sniffing analysis (45 min)
- Sensory room with purified air



R.T. (min.)	Area	N° CAS	Analyte	"Field of odours" References	Evocation	Intensity
7,11			not detected	Diacetyl	fatty, fresh butter- like	1,0
11,52	837794150	431-03-8	2,3-Butanedione	Diacetyl	fatty, fresh butter- like	2,0
13,75	456612394	64-19-7	Acetic acid	Acetic acid	pungent	1,0
16,01	334631686	600-14-6	2,3-Pentanedione	Diacetyl	fatty, fresh butter- like	1,0
16,13	3175053376	80-62-6	Methyl methacrylate	-	acidic, acrylate	1,0
17,90	63966400	624-92-0	Disulfide, dimethyl	Methyl disulfide	sulphured, cabbage -like	1,0
20,41	1158310783	66-25-1	Hexanal	cis-3-Hexenol	grassy green	1,0
22,15			not detected	-	acidic	1,5
23,68	17215867		unknown	Limonene	fresh, citrus fruit	1,0
24,01			not detected	Limonene	fresh, citrus fruit	1,0
24,09	1110398223	95-47-6	o-Xylene	Styrene	aromatic	1,5
24,15	3741702357	100-42-5	Styrene	Styrene	aromatic	1,5
25,35	32327938		unknown	Ethyl isobutyrate	fruity, pineapple- like	1,5
25,50	1693990189		unknown	2-Acetyl pyrazine	roasted, peanuts- like	3,0
27,00	290948609		unknown	Limonene	fresh, citrus fruit	2,0
27,37	1046069439	108-75-8	Pyridine, 2,4,6-trimethyl-	-	unpleasant	2,5
27,95	169040782	124-13-0	Octanal	Limonene	fresh, citrus fruit	2,5
28,16	4152659328	138-86-3	Limonene	Limonene	fresh, citrus fruit	2,5
28,68	3224242966	104-76-7	1-Hexanol, 2-ethyl-	Isobutylquinoline	earthy, asparagus-like	1,0
29,72			not detected	Limonene	fresh, citrus fruit	1,5
30,06			not detected	Isobutylquinoline	earthy, asparagus-like	1,0
30,32	86597502		unknown	Limonene	fresh, citrus fruit	1,0
31,87	53489191		not identified alkane	Nonanal	fatty, waxy	2,0
31,91	87614186		not identified alkane	Nonanal	fatty, waxy	2,0
35,54			not detected	b-Caryophyllene	spicy, cedar wood- like	1,0
35,72	342971958		not identified alkane	Nonanal	fatty, waxy	1,0
37,57	8653014	80-54-6	Lilial	Phenylethyl Alcohol + Vanillin	floral + vanilla	1,0

In blue: odor perceived in the global assessment

# ■ Sampling techniques

## Certech established method:

- Custom Tedlar bag
- Loading factor (weight/air volume)
- Conditioning Temp (max 80°C)
- Global odor assessment
- Tenax sorbent

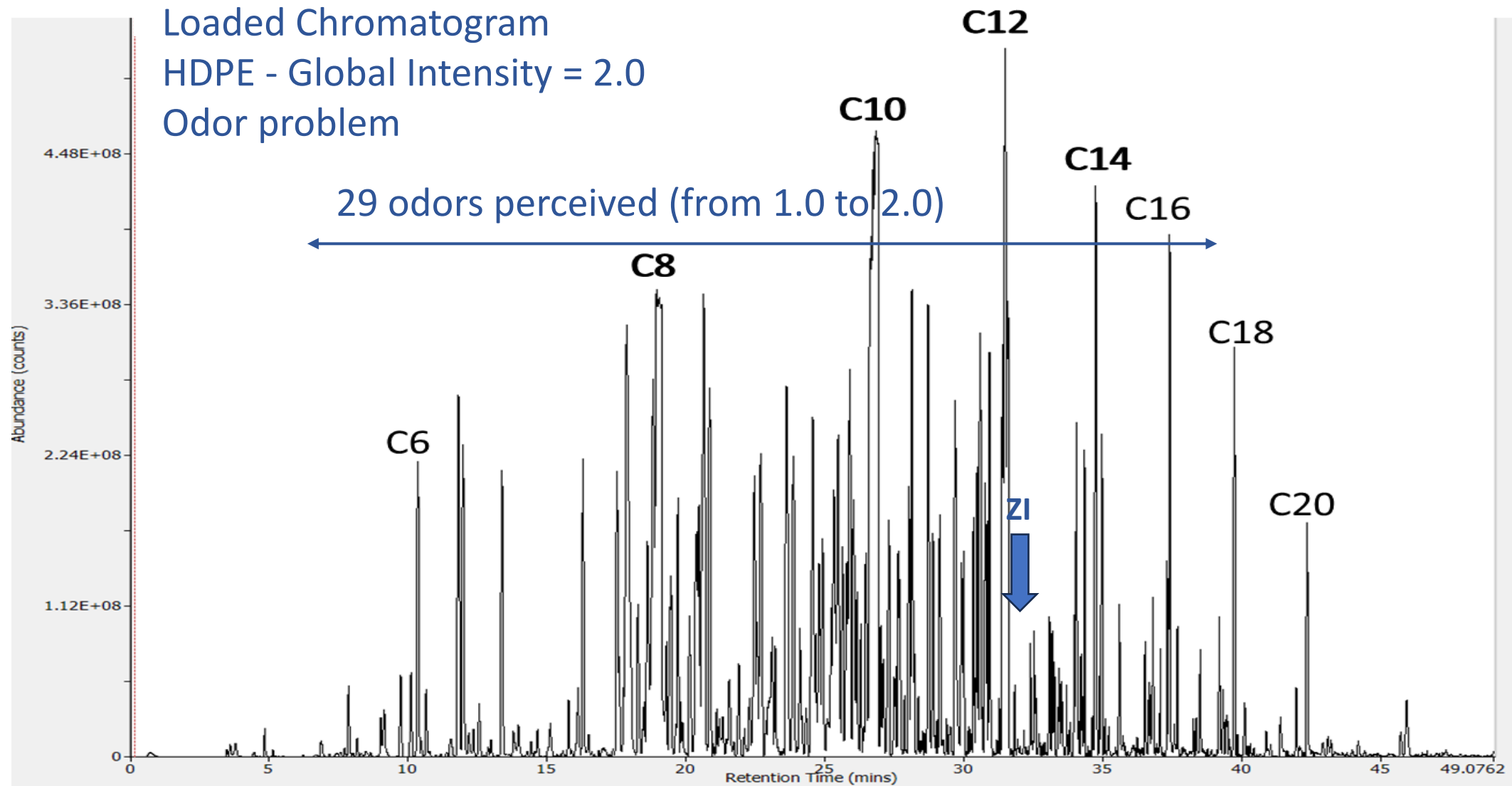


## Analysis :

- TD-GC-MS
- TD-GC-TOFMS/Sniffing
- TD-GC×GC-HRTOFMS/FID



## ■ Example of results



## ■ Sampling techniques



Centri® Platform equipped with:

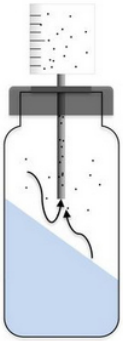
- Thermal Desorption (external sampling)
- Headspace-trap
- High-capacity Sorptive extraction (HiSorb™)

→ automated and closed system

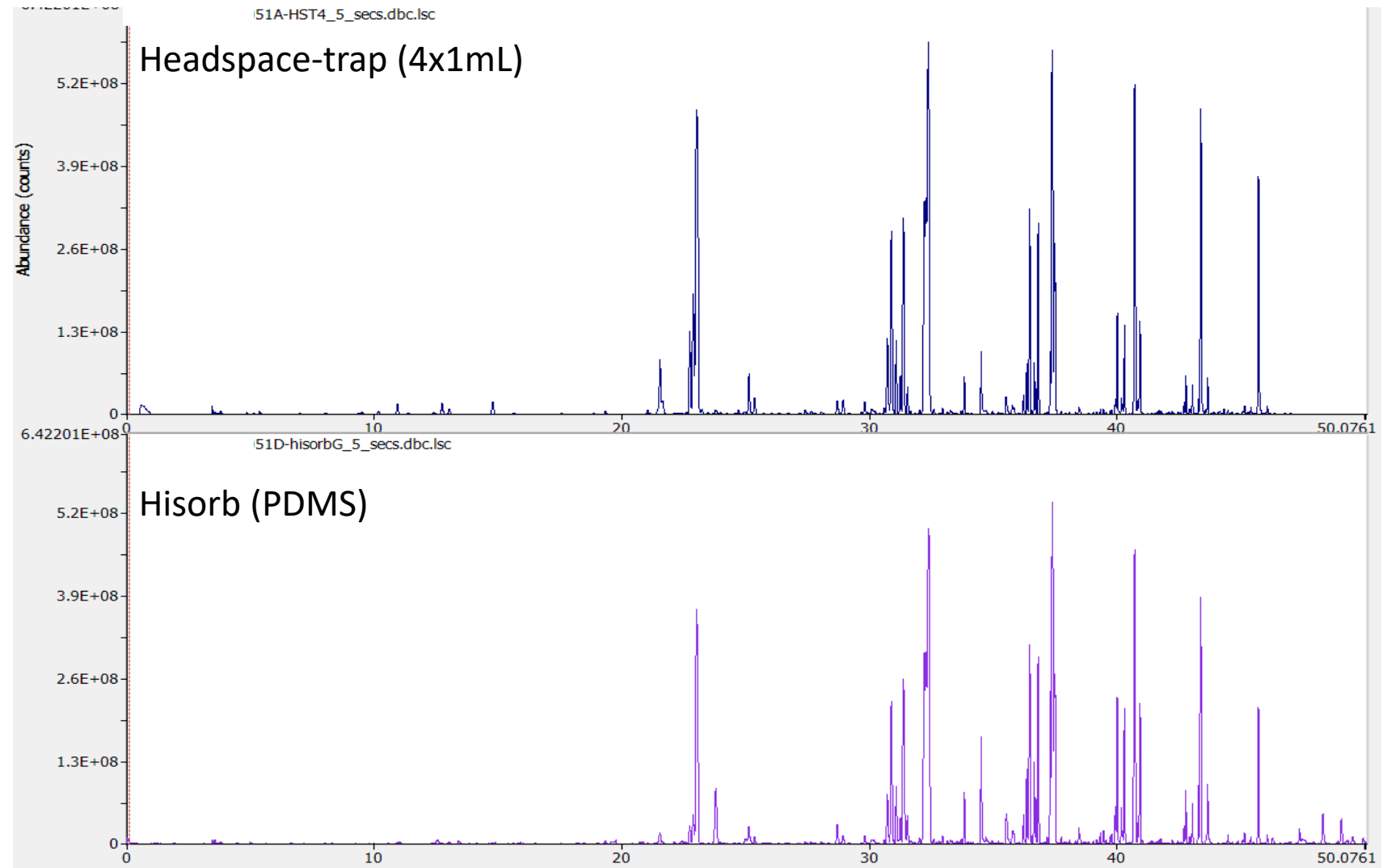
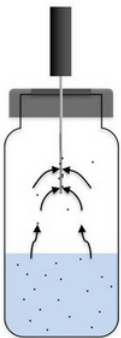


# ■ Sampling techniques: Hisorb

- 1g HDPE pellets
- 150°C 10 min

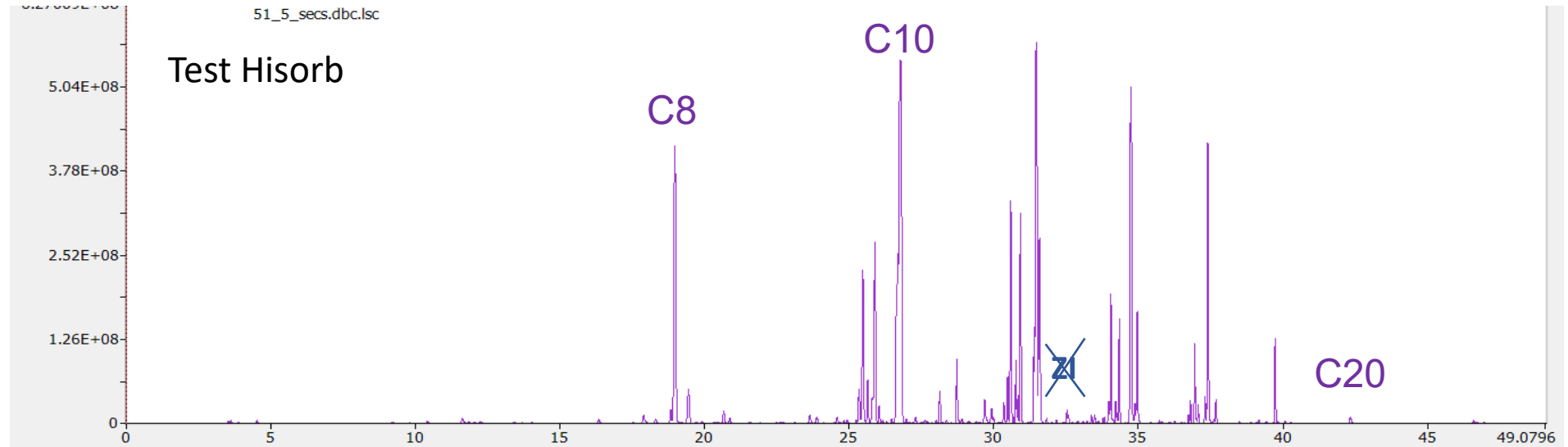


- 1g HDPE pellets
- 70°C 30 min

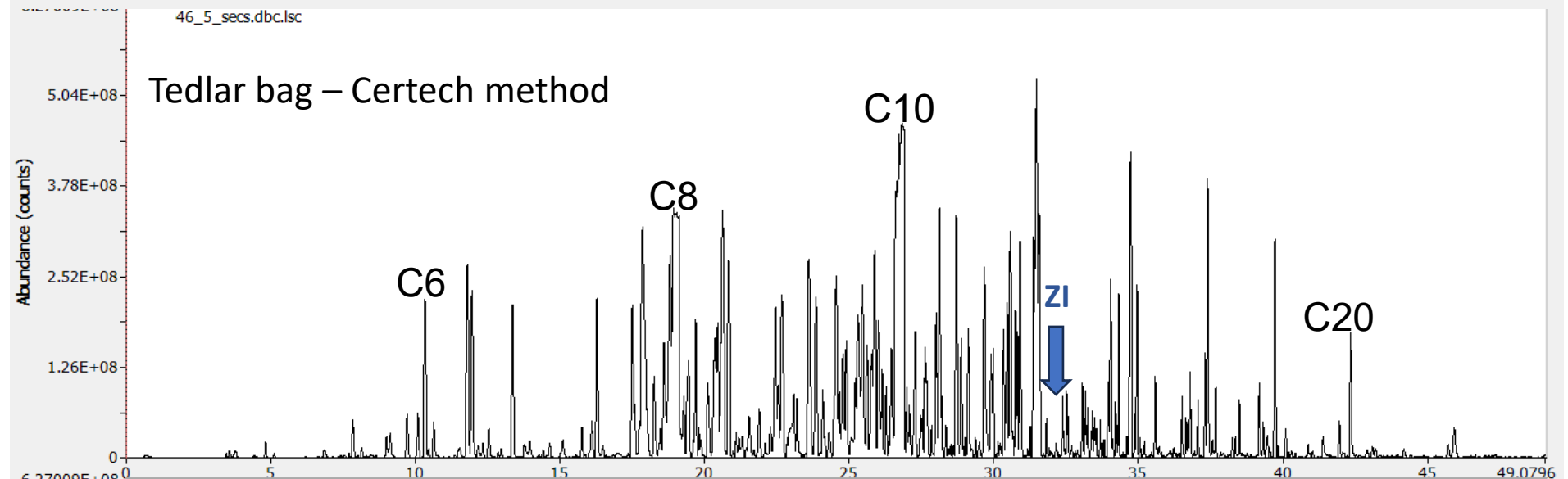


# Sampling techniques: evaluation with sniffing

- 8 odors perceived
- Intensity up to 1.5 (Eq. 1 g)



- 29 odors perceived
- Intensity up to 2.0 (Eq. 30 g)



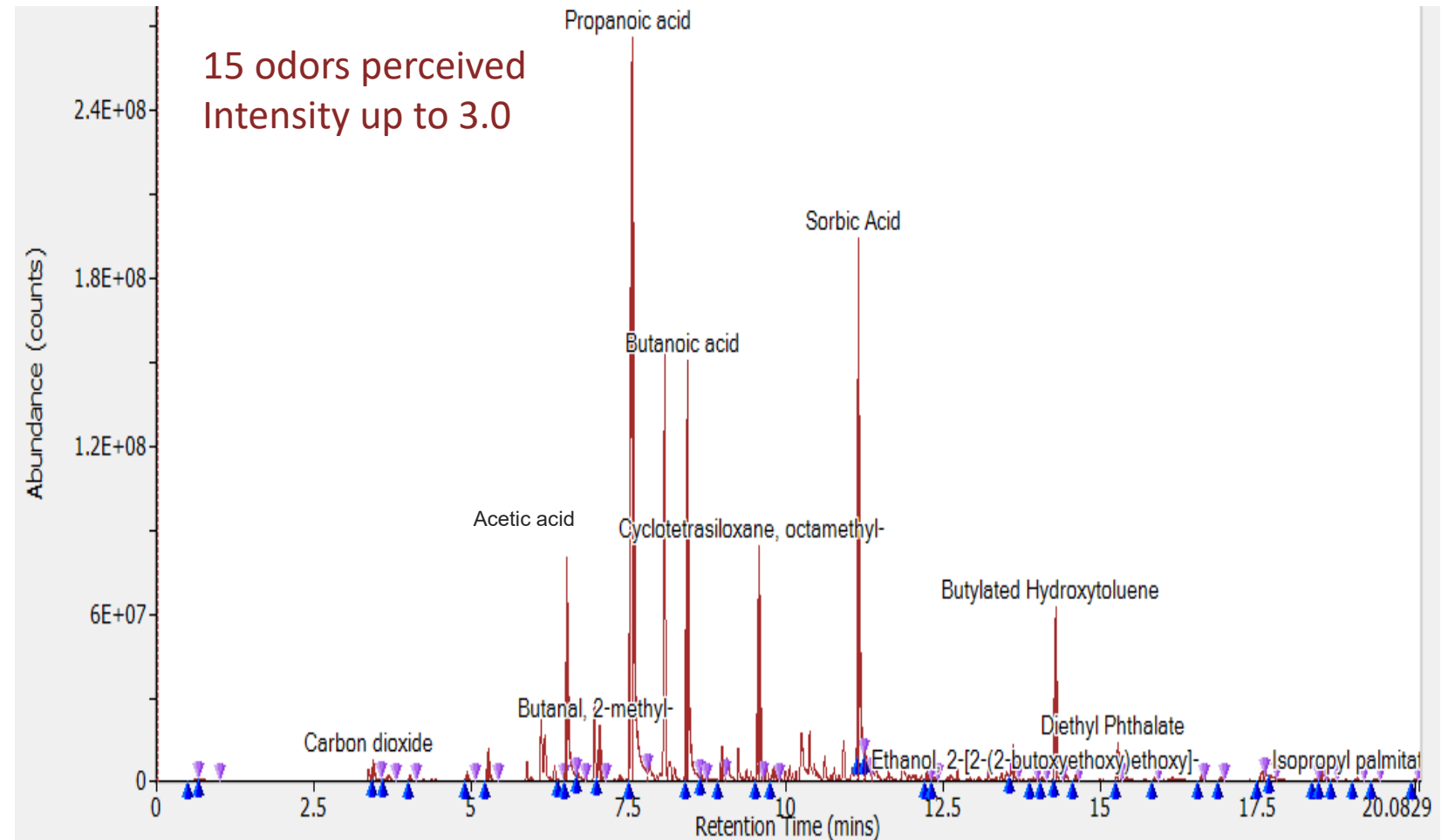
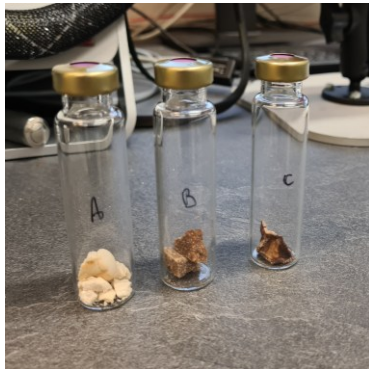
# ■ Sampling techniques: evaluation with sniffing

➤ Analysis of aroma sample\*



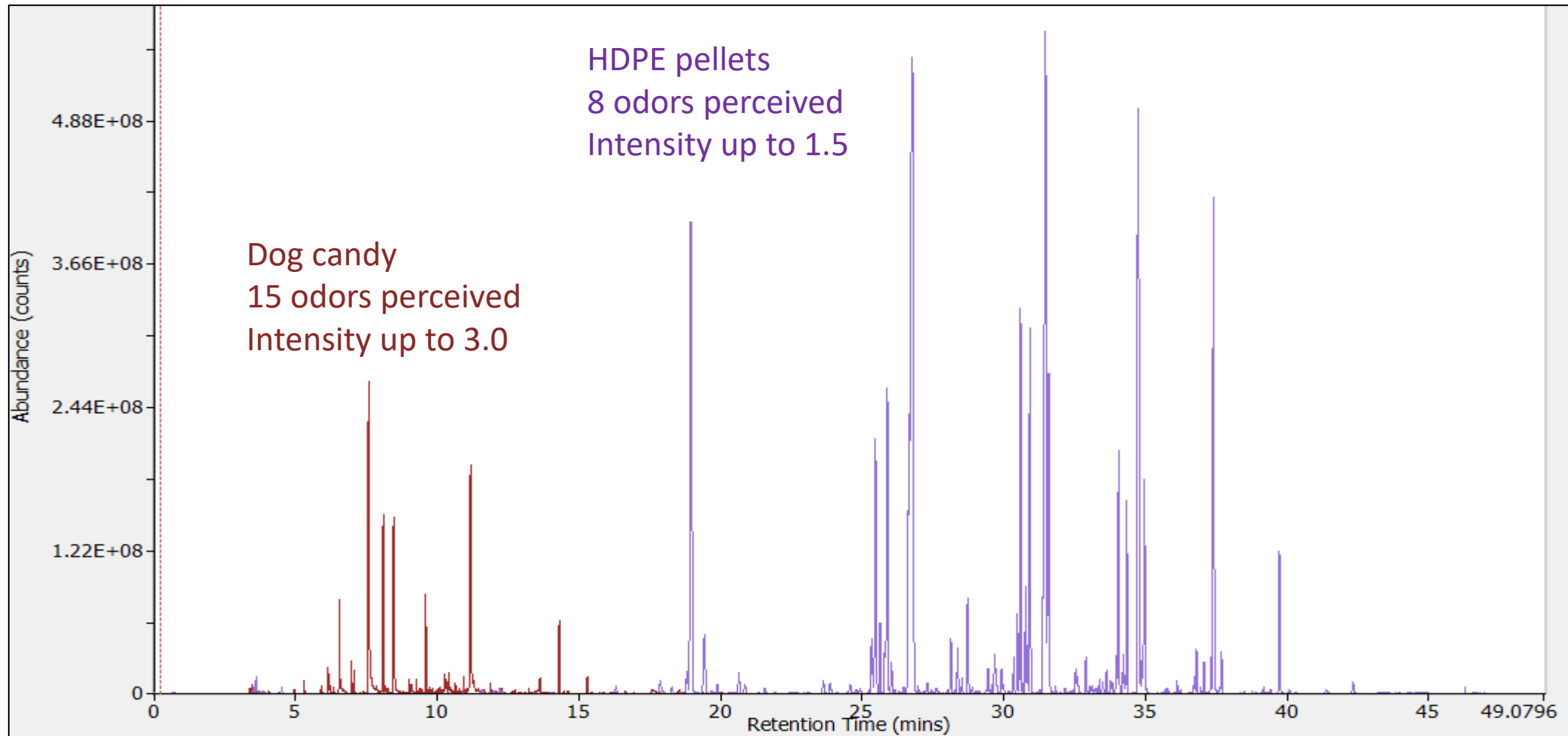
\* Kindly provided

Hisorb – 40°C – 30 min



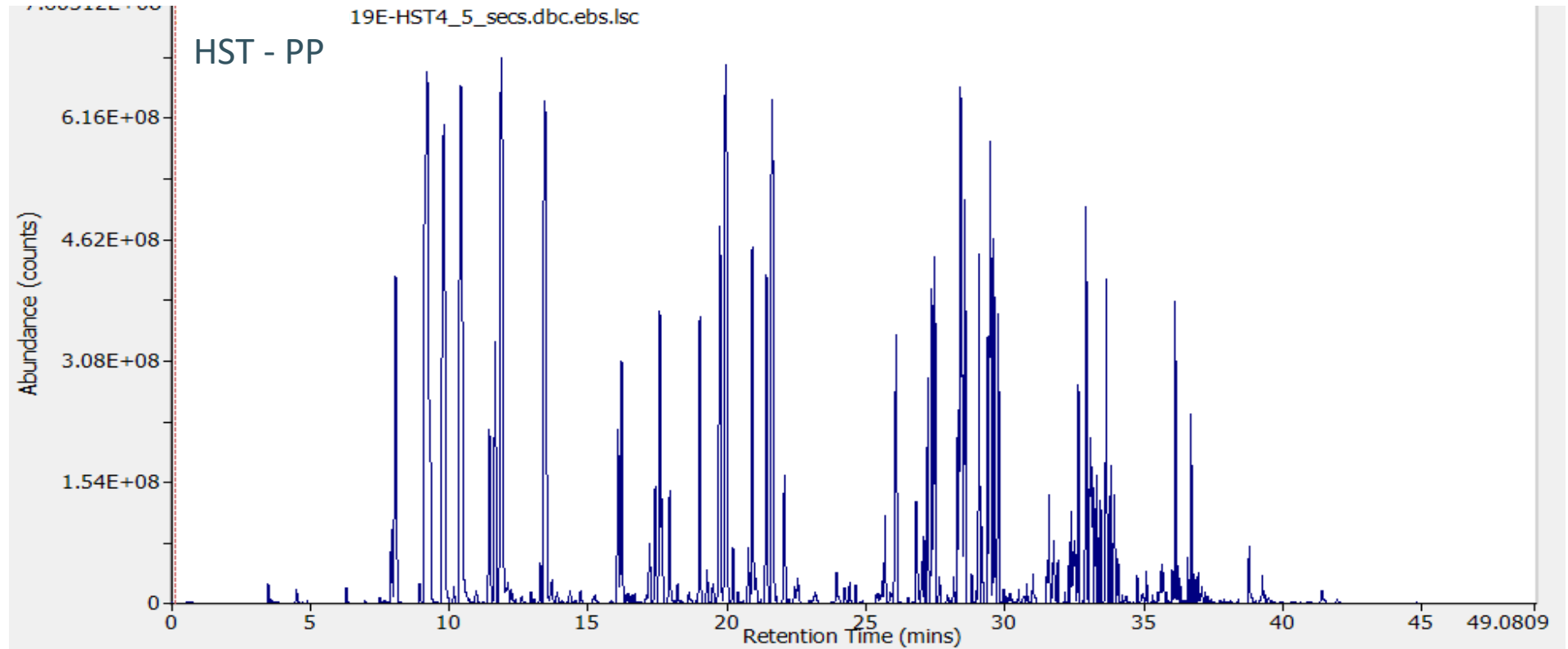
## ■ Sampling techniques: evaluation with sniffing

- Comparison aroma sample vs polymer sample



# ■ Sampling techniques: HST optimization - sniffing

- PP
- HST-4 - 1h @ 120°C
  
- 10 odors perceived
- Intensity up to 2.0 (Eq. 0.2 g)



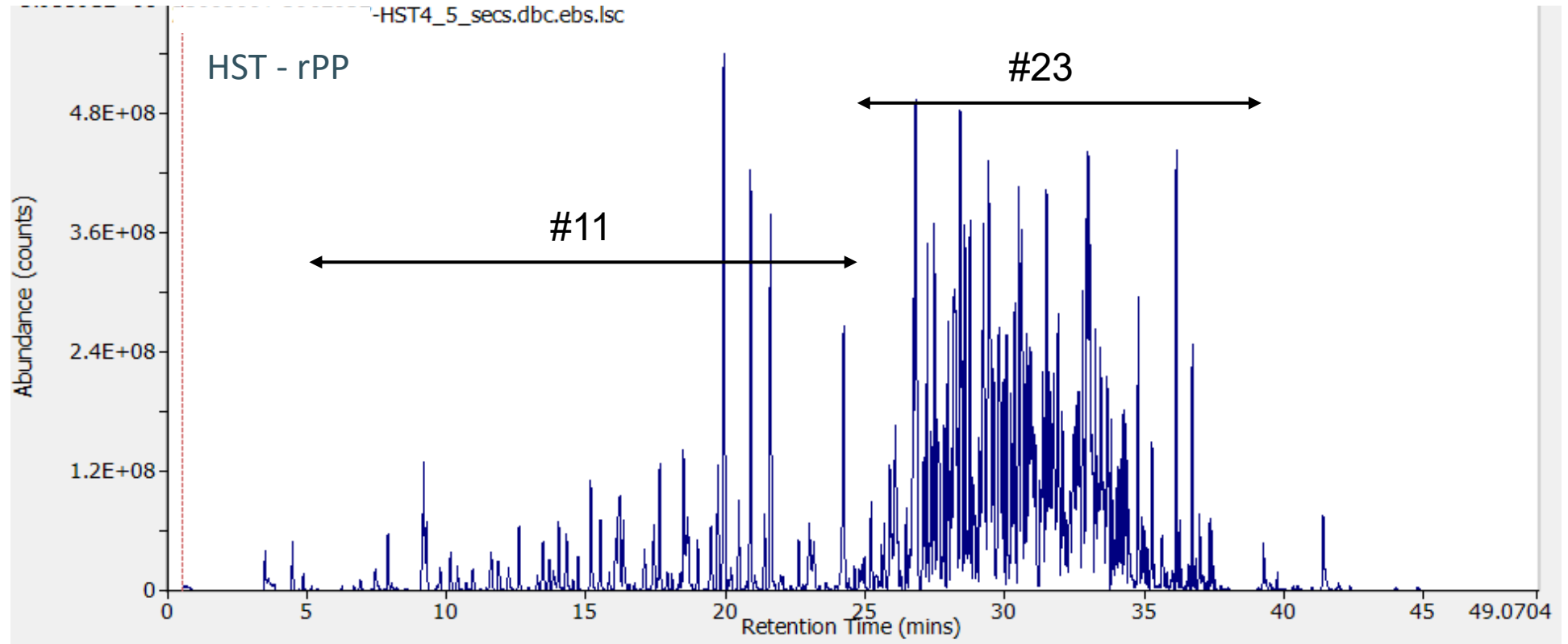
Tedlar bag – Certech method

- 24 odors perceived
- Intensity up to 2.0 (Eq. 10 g)

# ■ Sampling techniques: HST optimization - sniffing

- Recycled PP
- HST-4 - 1h @ 120°C

- 34 odors perceived
- Intensity up to 2.5 (Eq. 0.2 g)

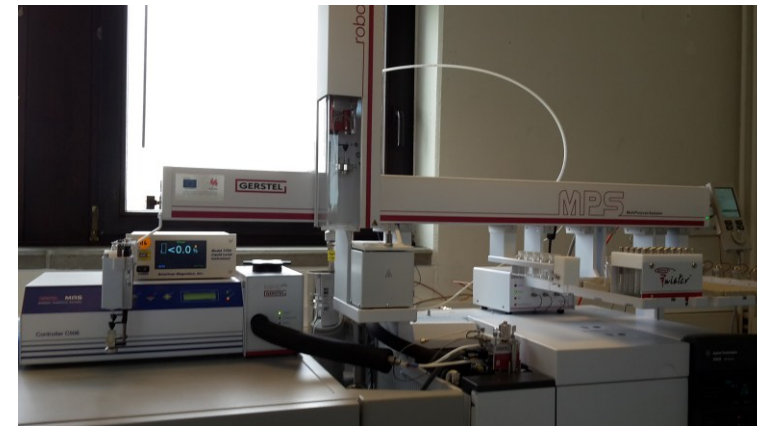
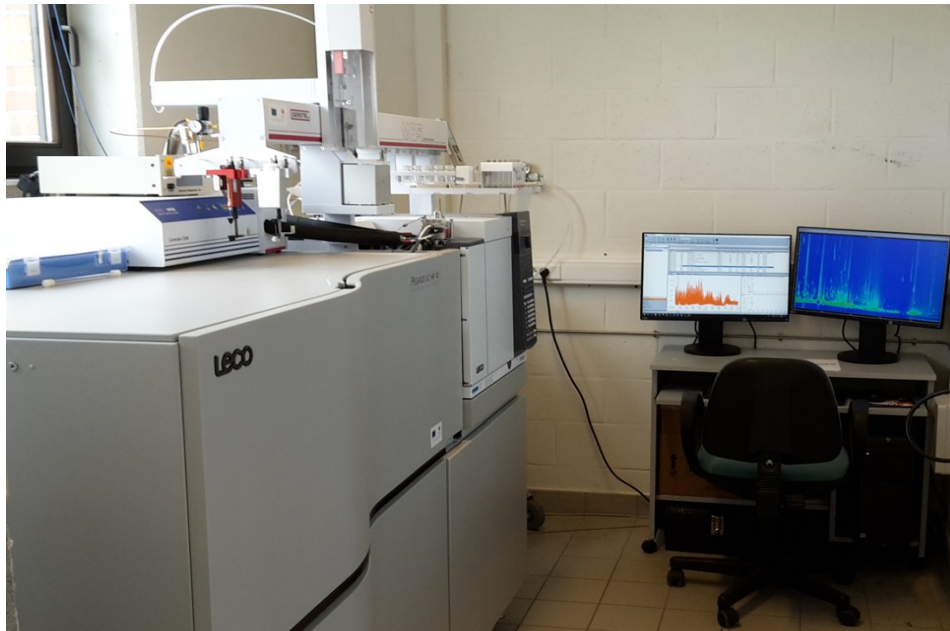


Tedlar bag – Certech method

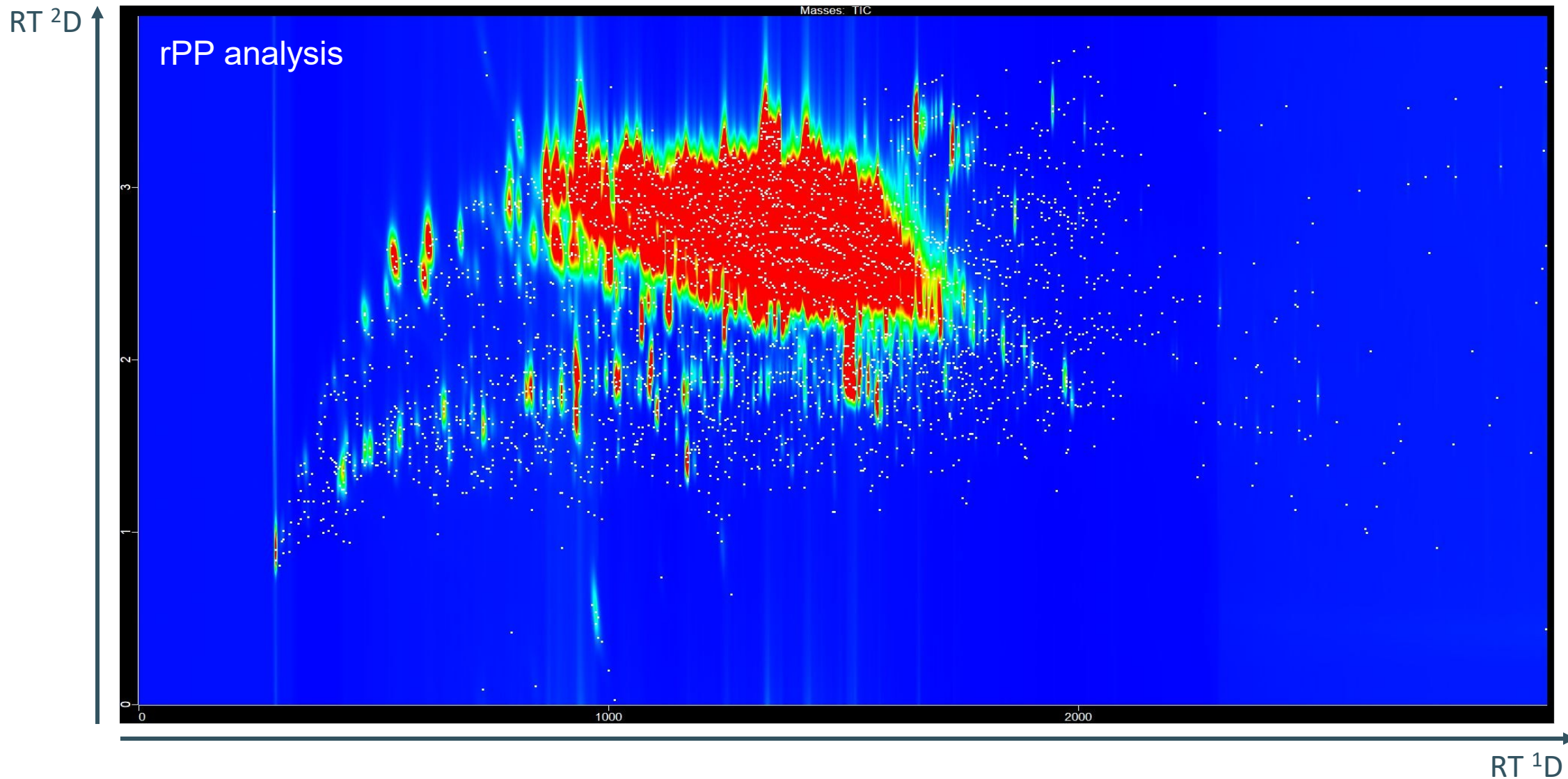
- 46 odors perceived
- Intensity up to 3.0 (Eq. 1 g)

# Analysis of complex samples: GC×GC-HRTOFMS/FID

- Complex VOC profile, co-elutions with matrix, trace level compounds, unknown..
- Complementary technique

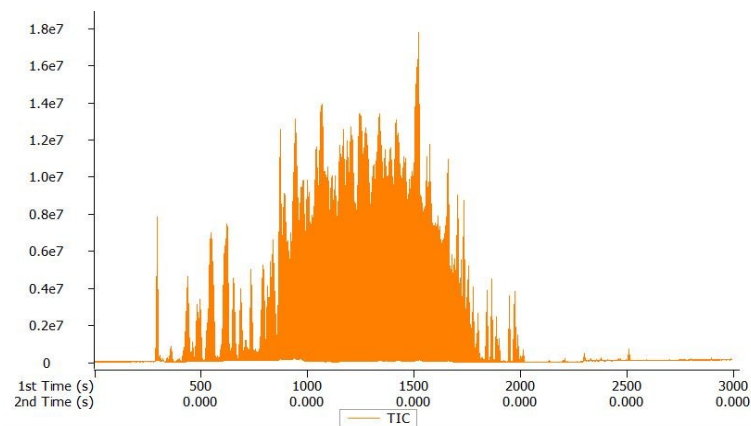


# Analysis of complex samples: GC×GC-HRTOFMS/FID



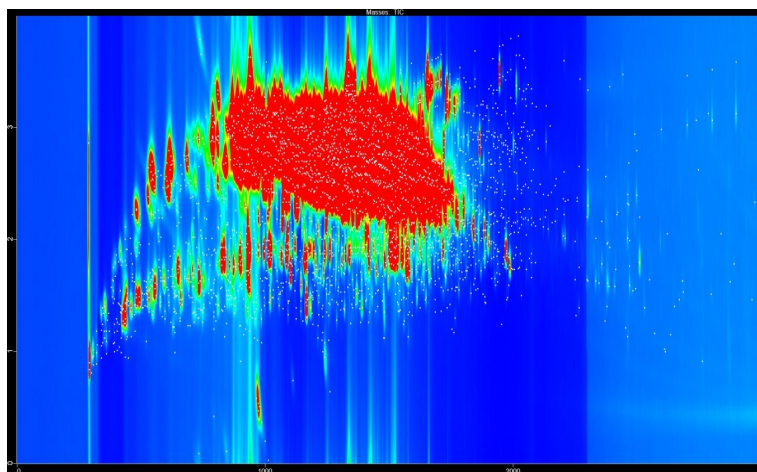
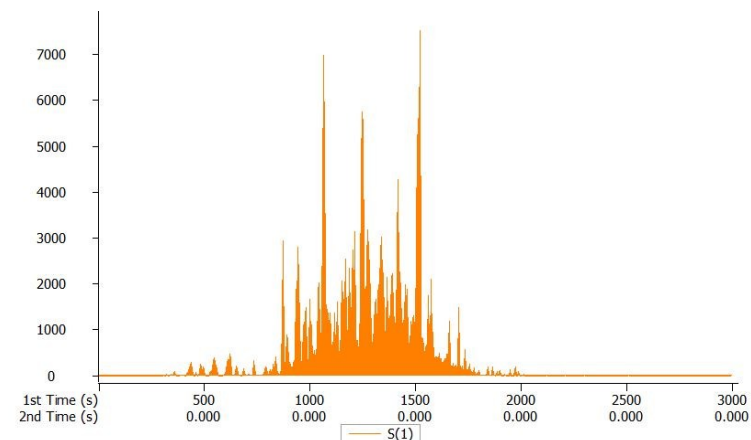
# Analysis of complex samples: GC×GC-HRTOFMS/FID

HRTOFMS signal ( $10^3$ )

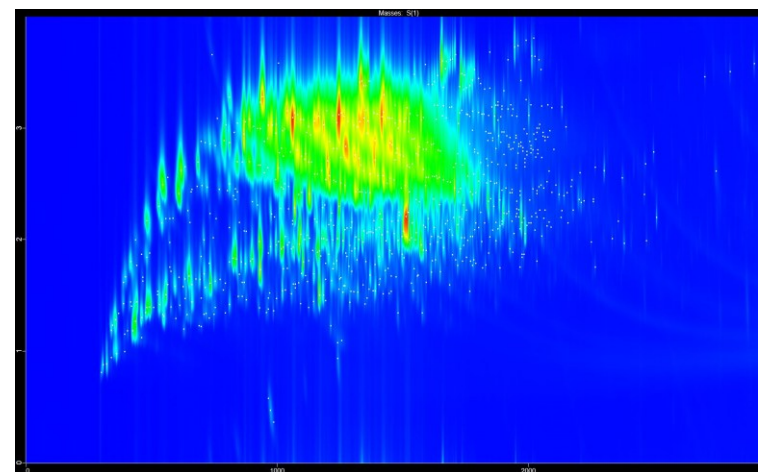


vs

FID signal ( $10^7$ )



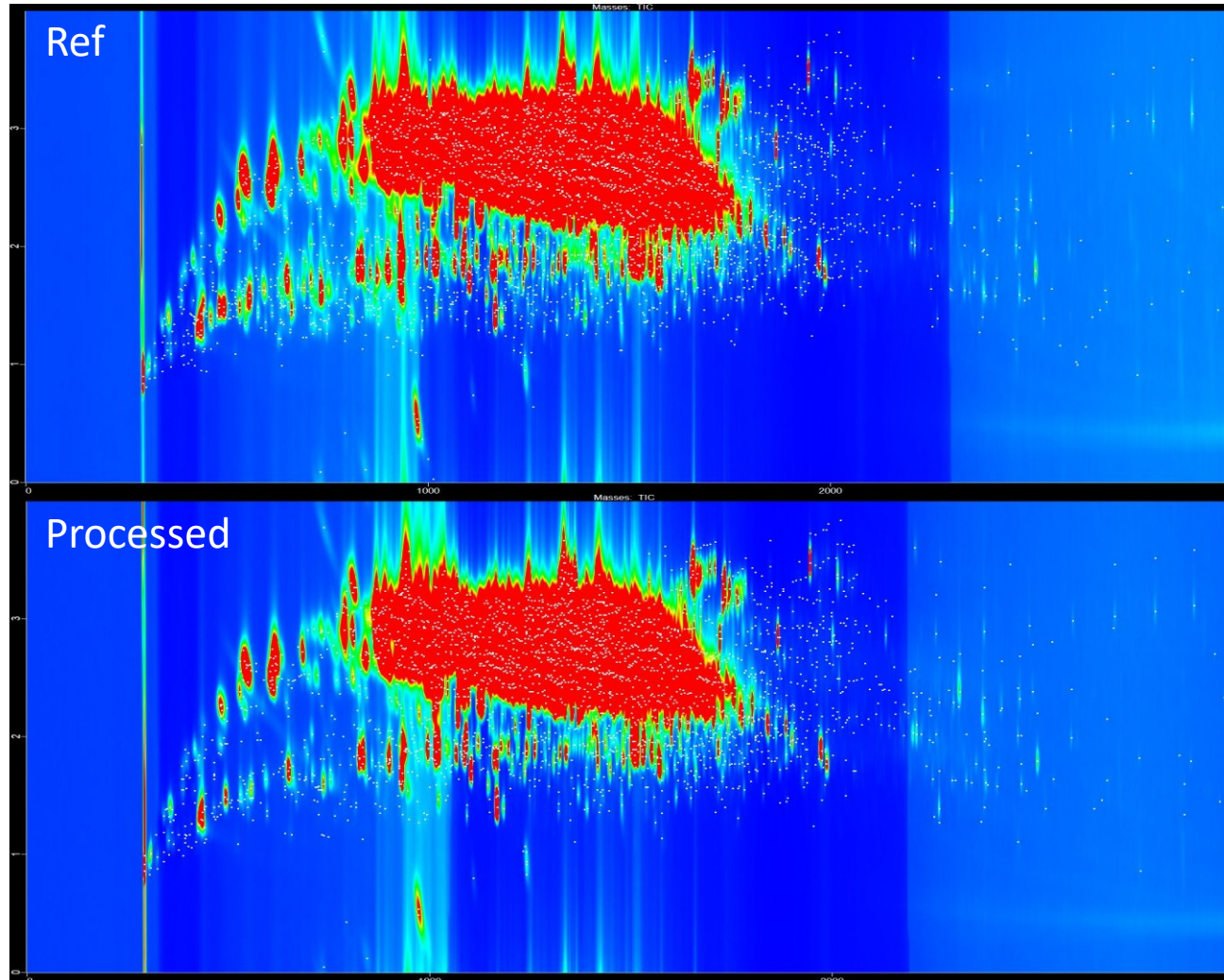
Data processing:  
3186 peaks  
(S/N >50)



Data processing:  
799 peaks  
(S/N >50)

# Analysis of complex samples: GC×GC-HRTOFMS/FID

## Comparison of samples

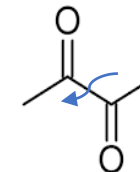


- Example: roasted and fatty notes perceived in the global odor disappeared after the process
- Large amount of data produced  
=> easy check of specific information

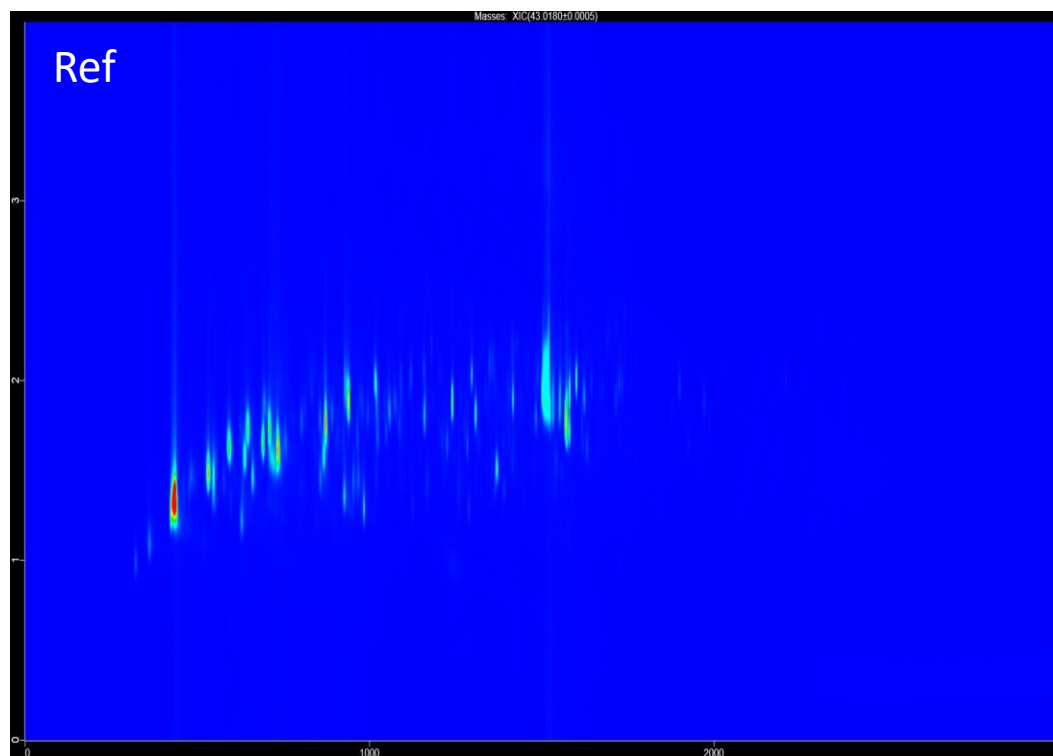
# Analysis of complex samples: GC×GC-HRTOFMS/FID

✓ GC×GC-TOFHRMS ( $R > 25,000$  FWHM, MA  $< 1$ ppm)

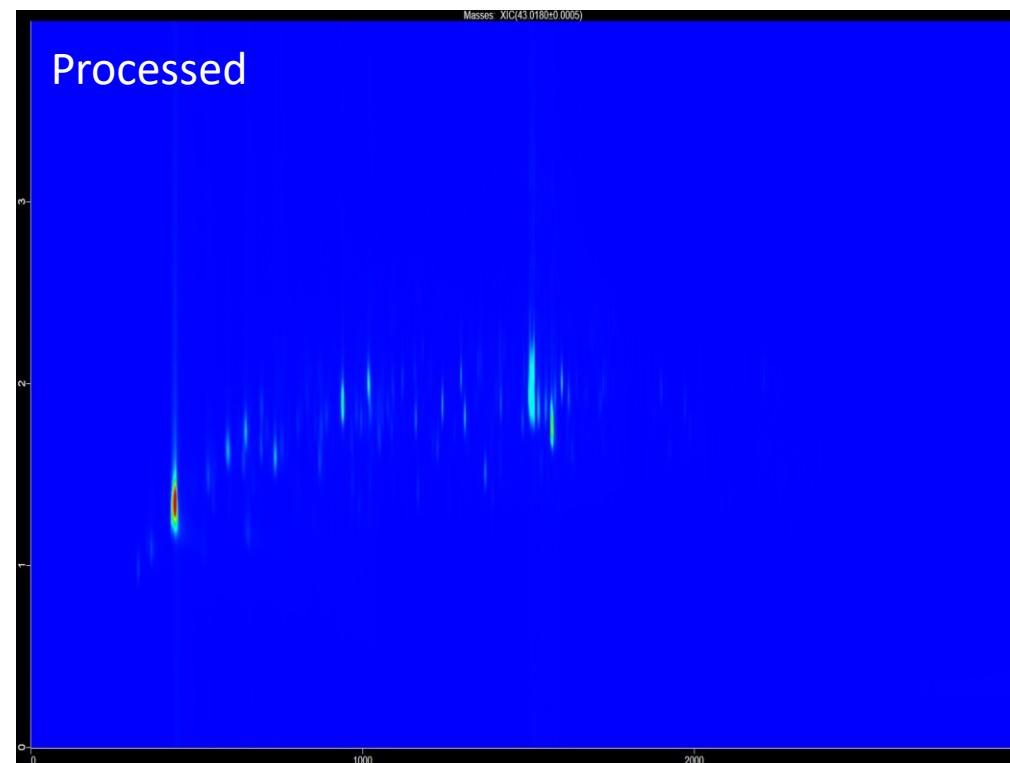
➤ Easy highlight of carbonyl compounds (Fatty notes => diketones)



EIC signal ( $m/z 43,0178 \pm 0,0005$ ) C=O-CH<sub>3</sub>



EIC signal ( $m/z 43,0178 \pm 0,0005$ ) C=O-CH<sub>3</sub>

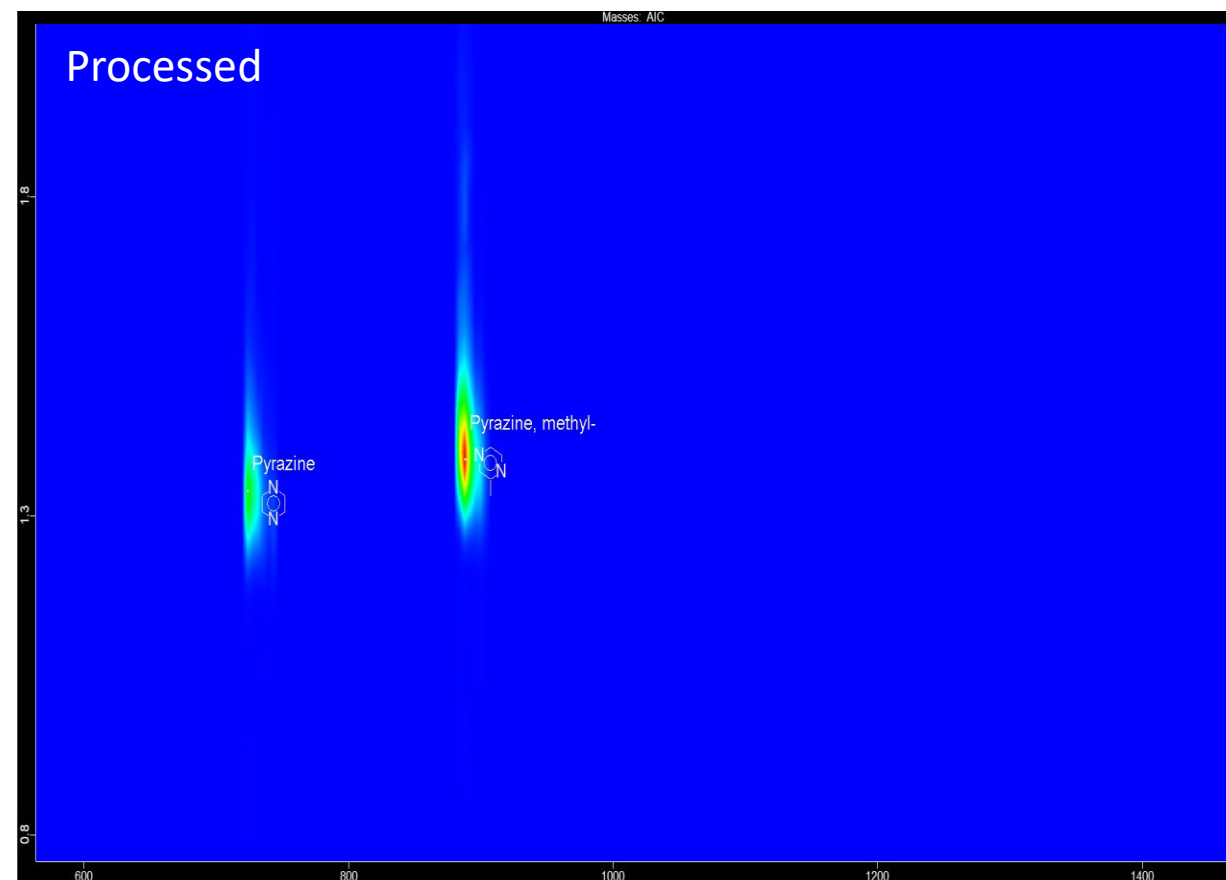
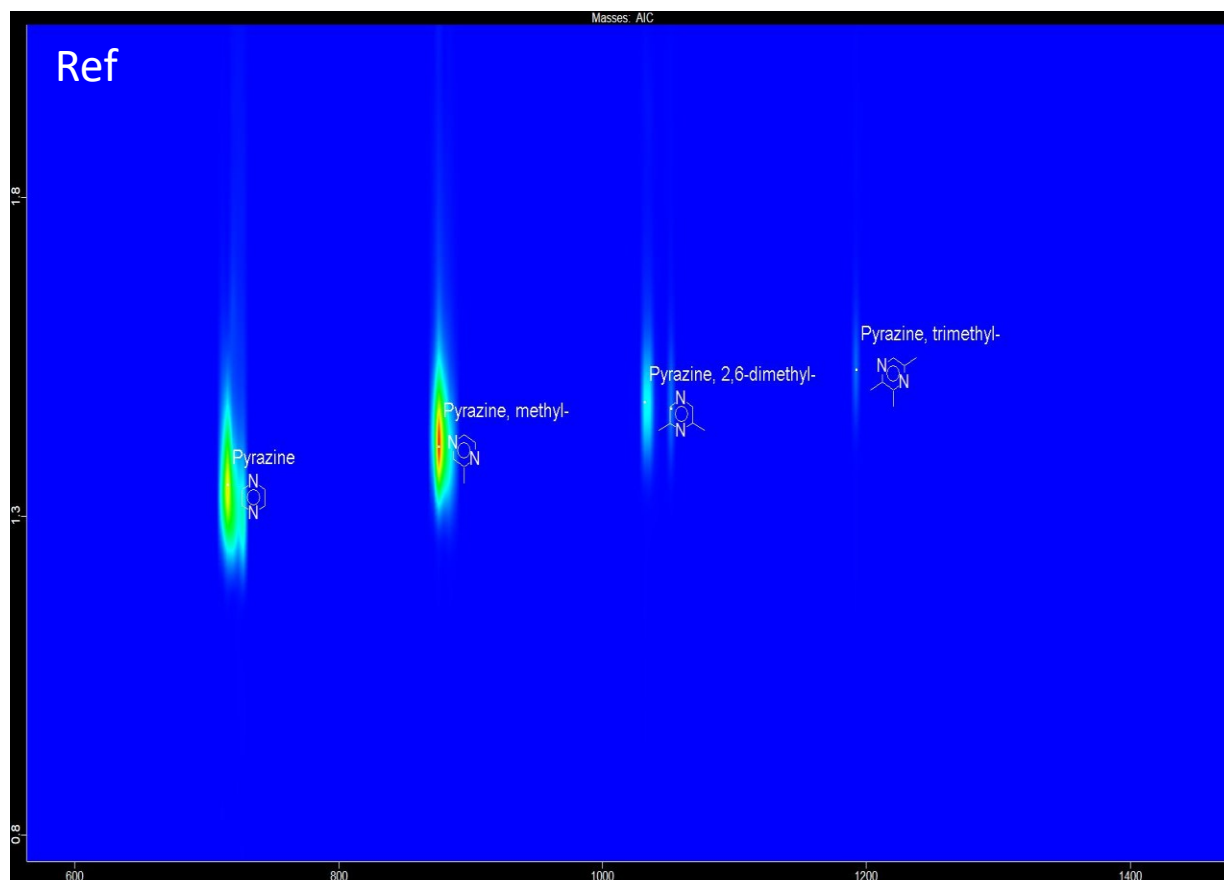


# Analysis of complex samples: GC×GC-HRTOFMS/FID

## Comparison of samples

Filtering criteria (Library search results : name or formula)

Roasted note => search for pyrazines

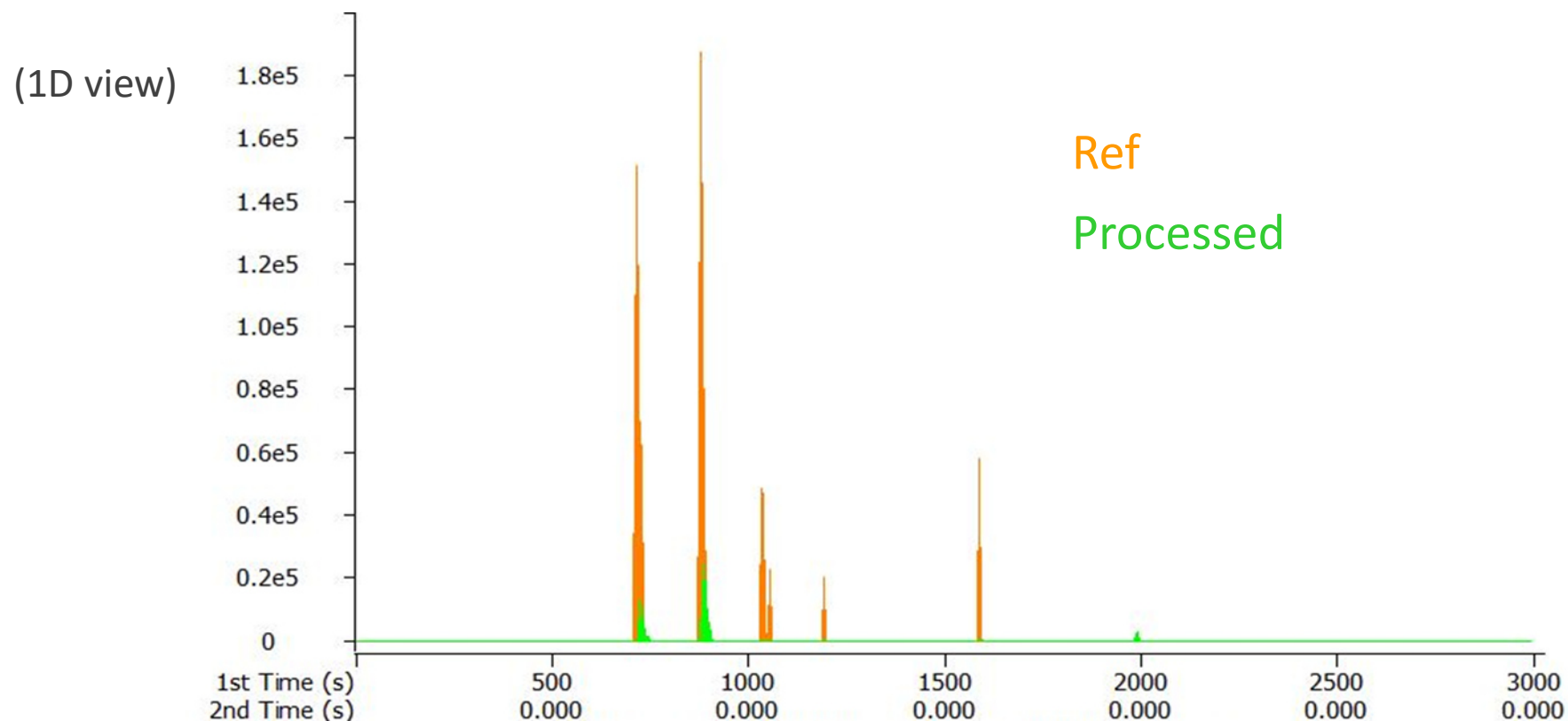


# Analysis of complex samples: GC×GC-HRTOFMS/FID

## Comparison of samples

Filtering criteria (Library search results : name or formula)

Roasted note => search for pyrazines

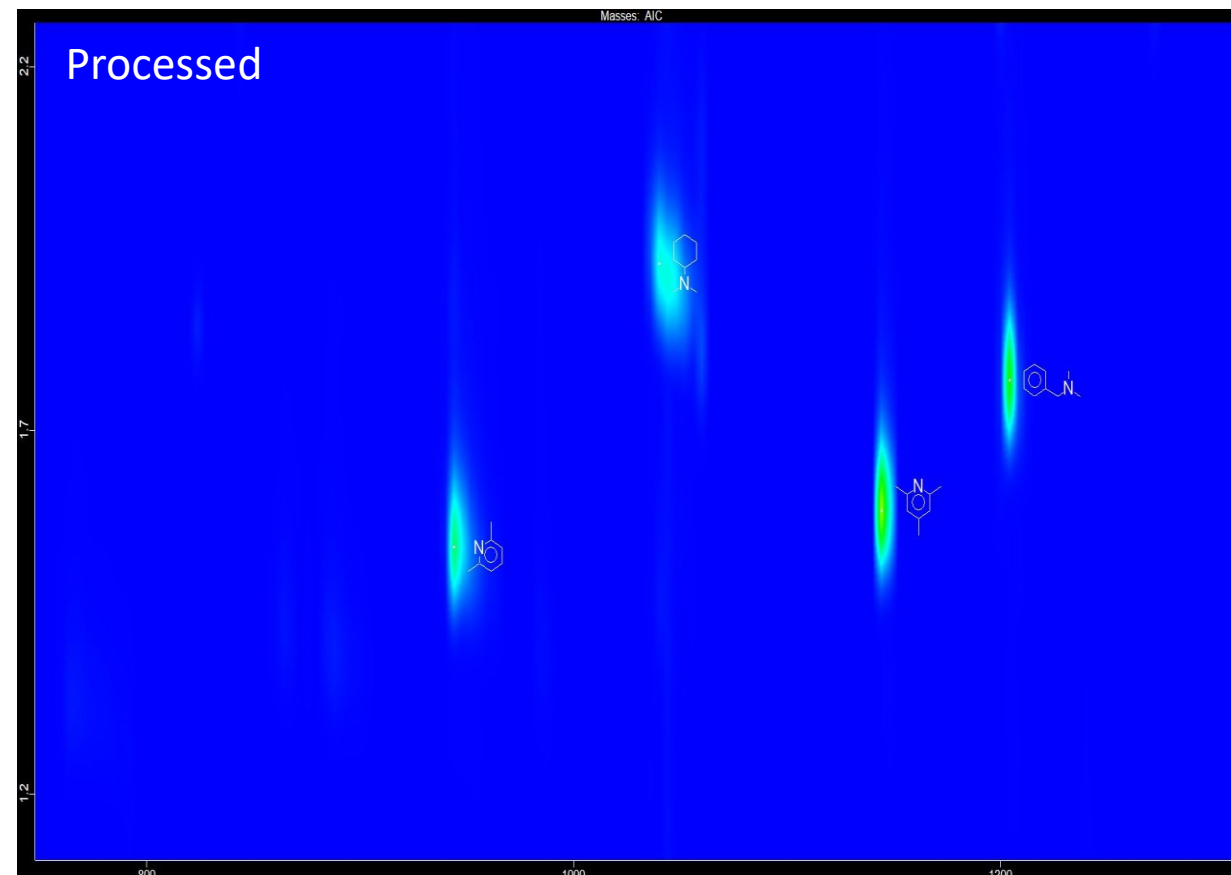
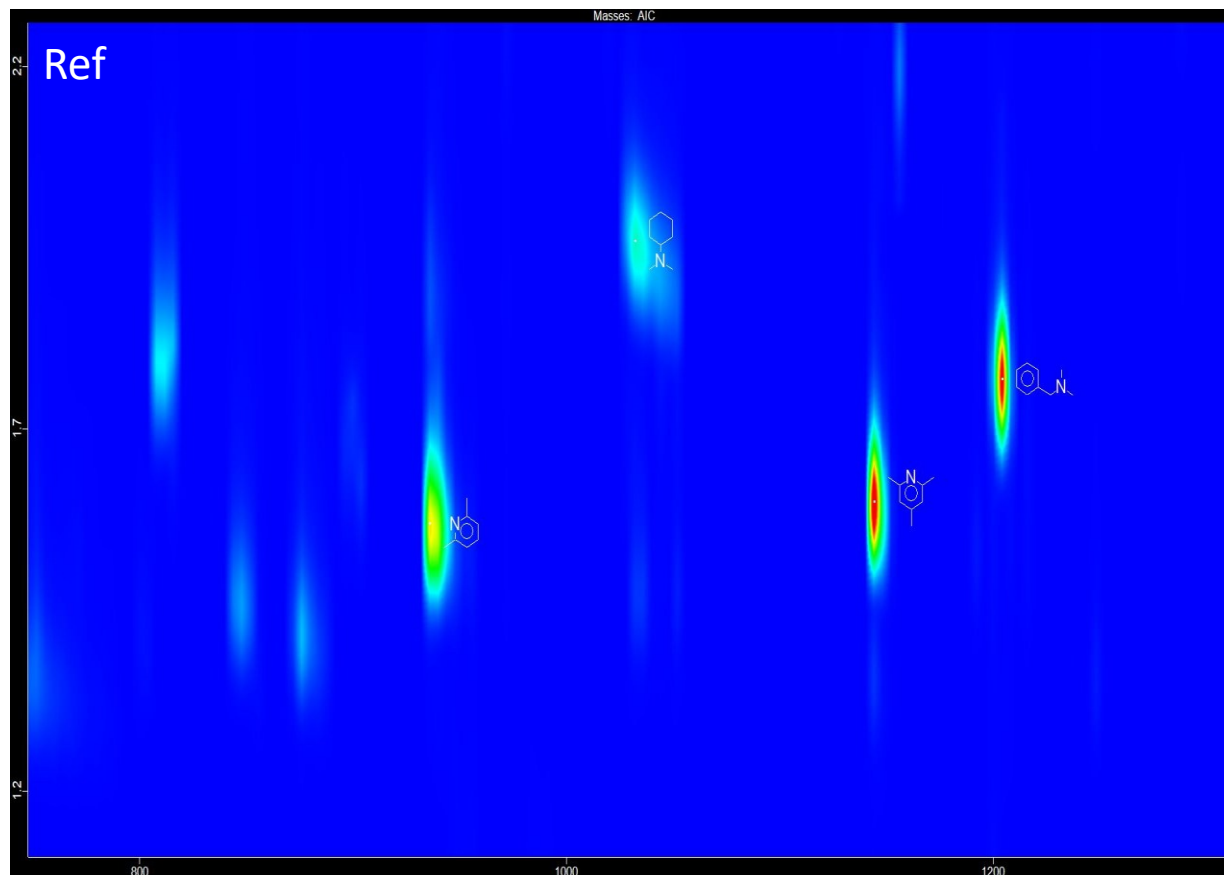


# Analysis of complex samples: GC×GC-HRTOFMS/FID

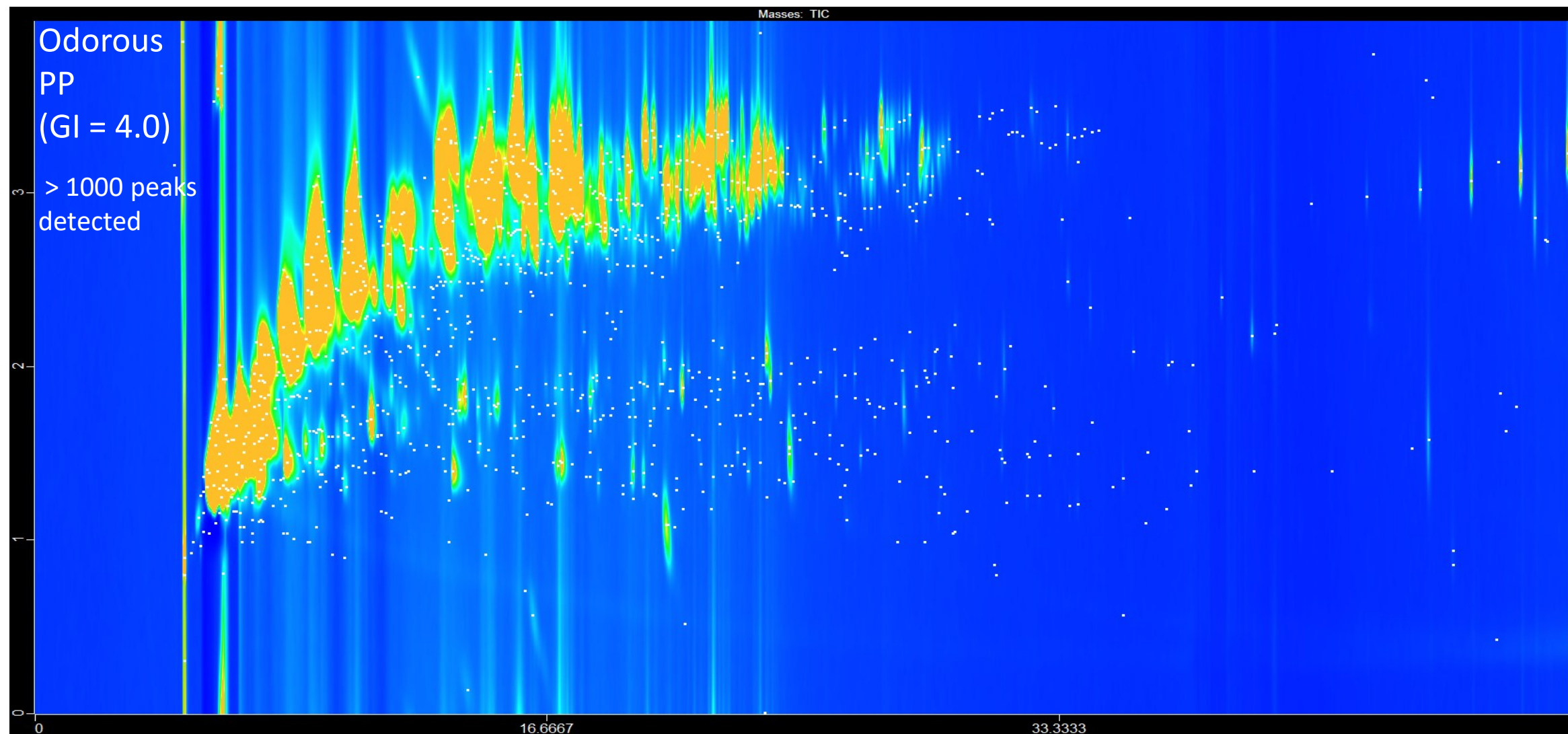
## Comparison of samples

Filtering criteria (Library search results : name or formula)

Roasted note => search for Nitrogen

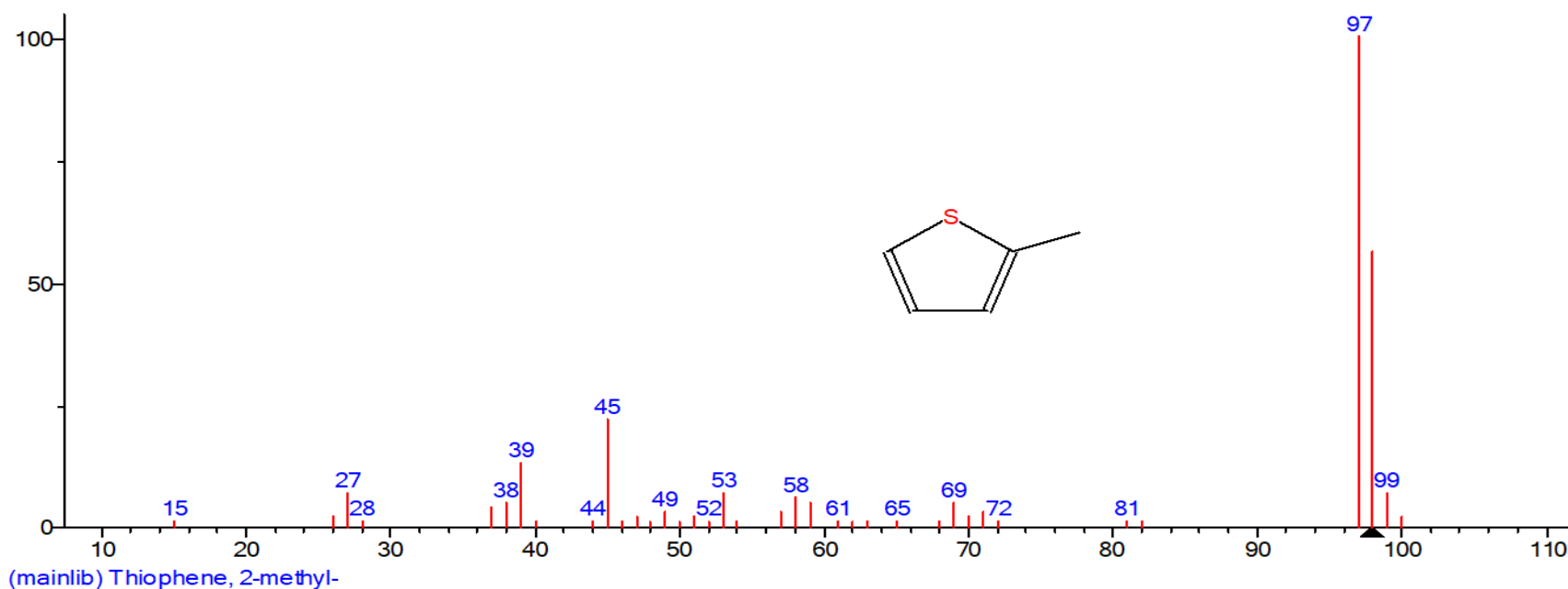


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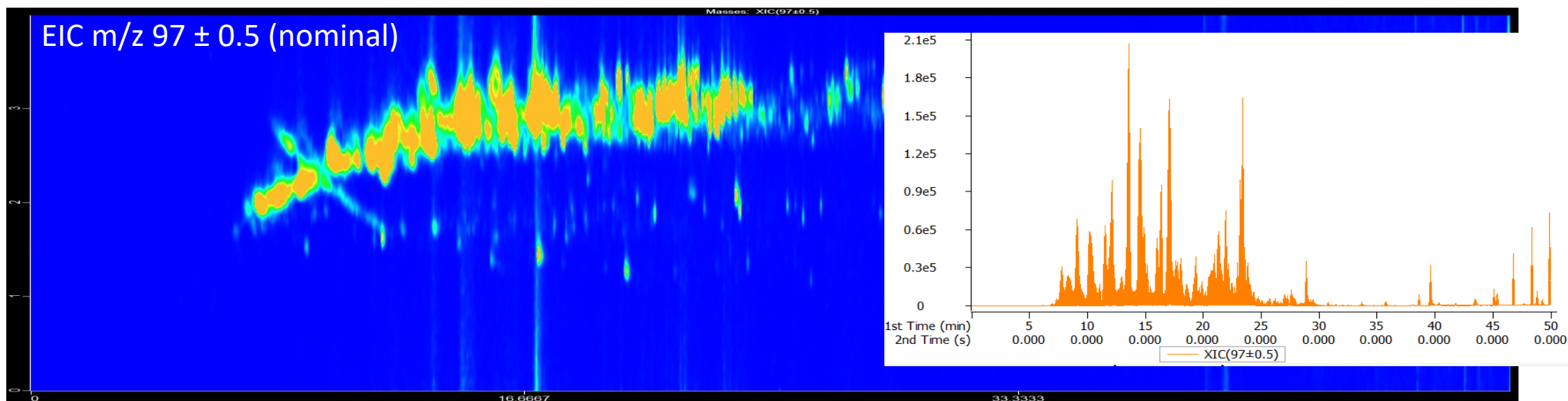
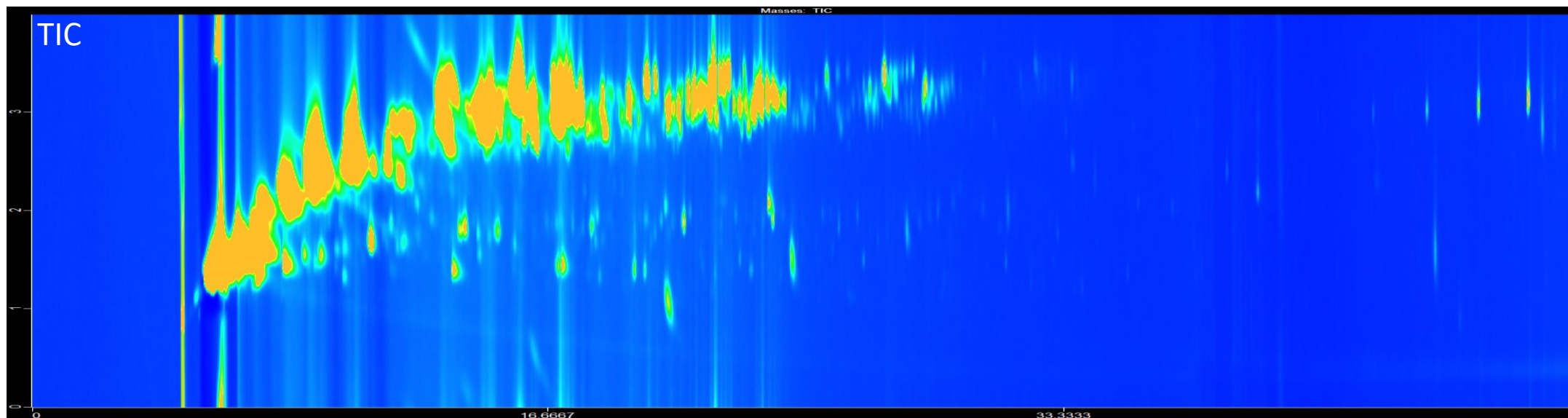


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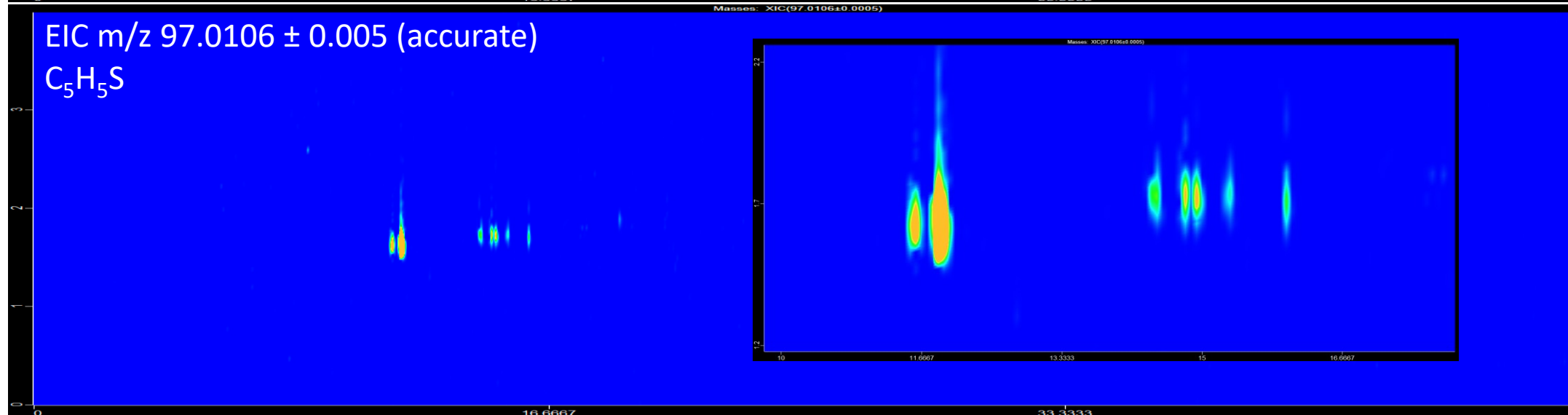
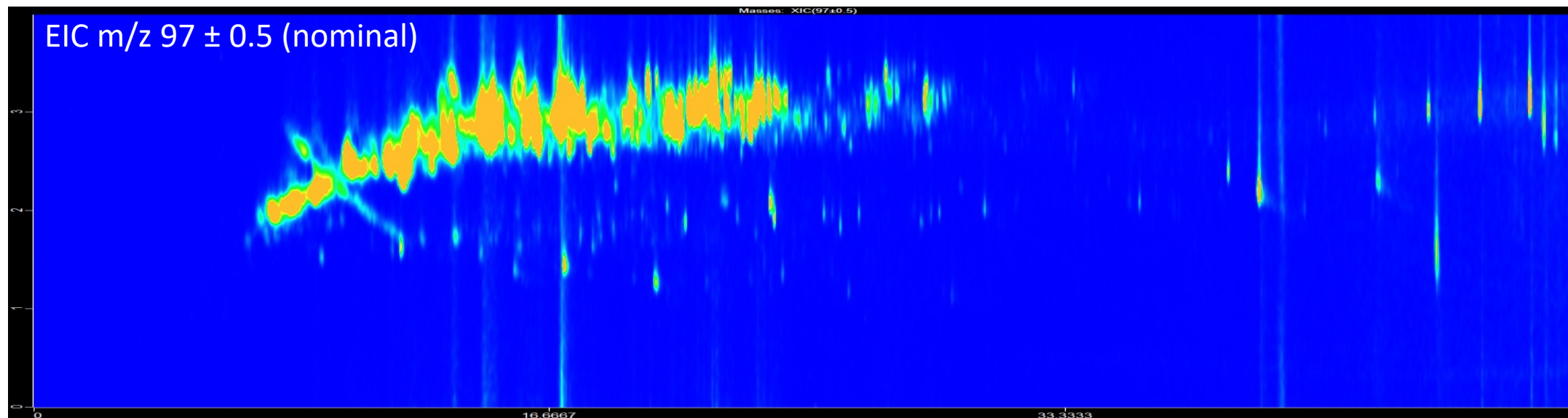
=> search for thiophene derivatives



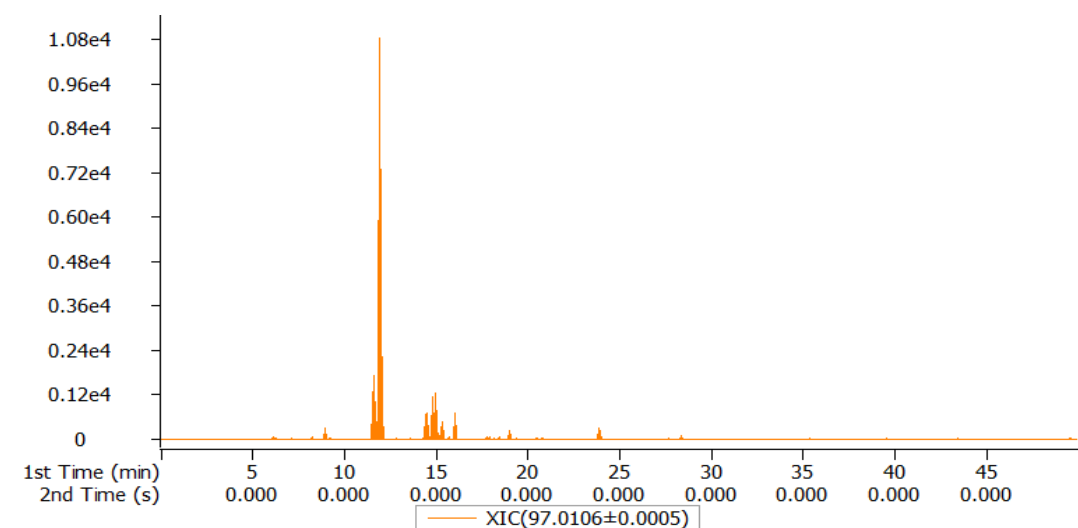
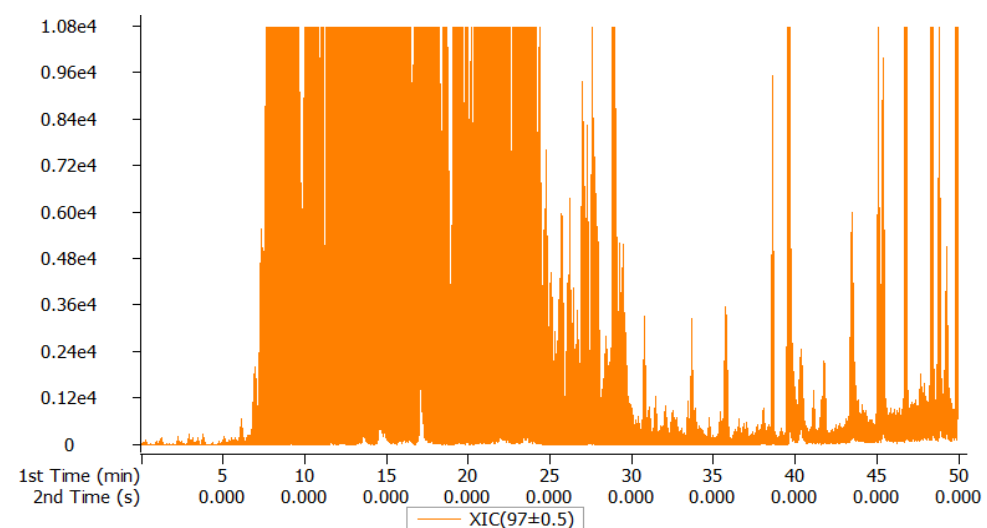
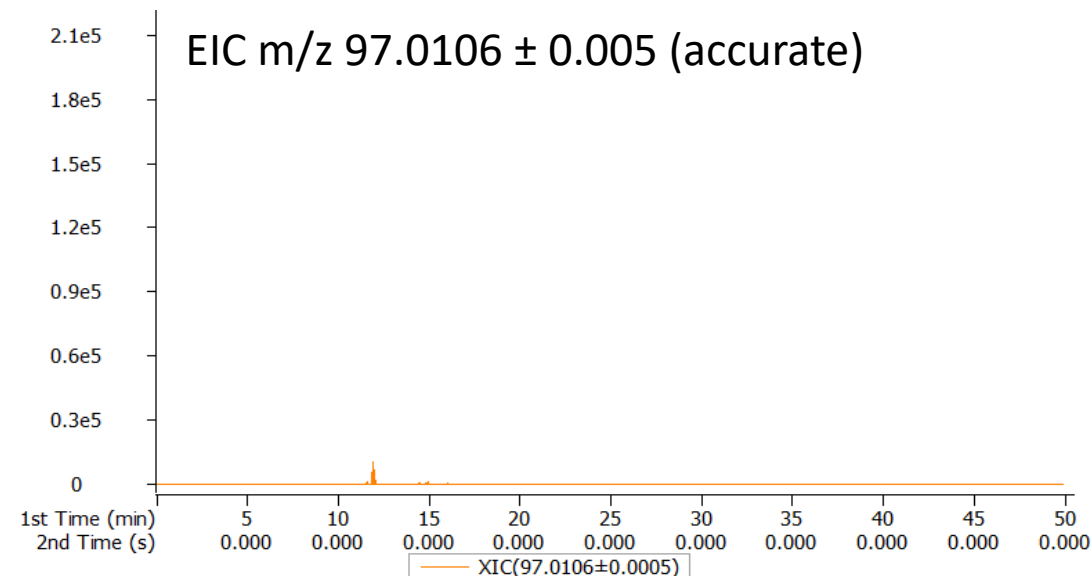
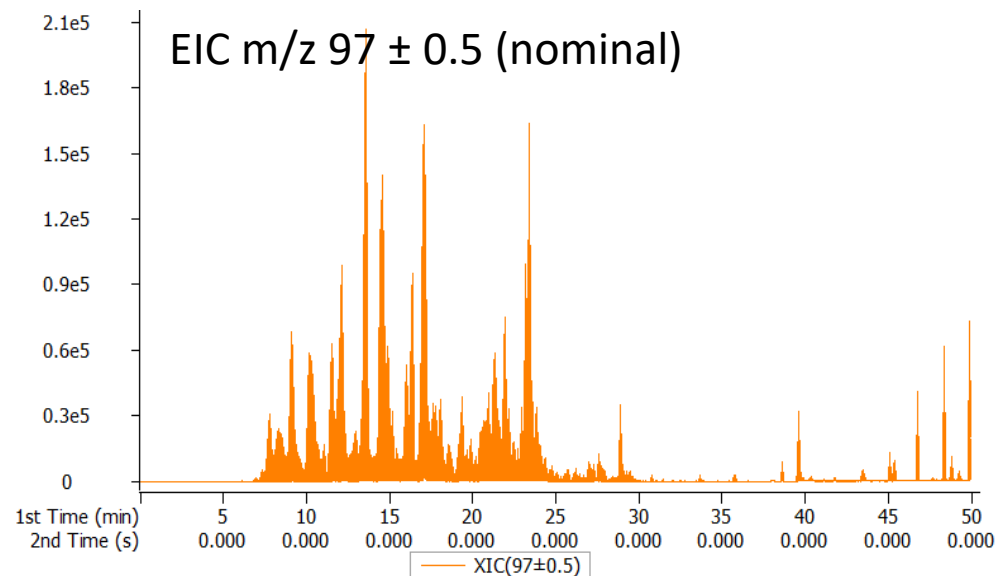
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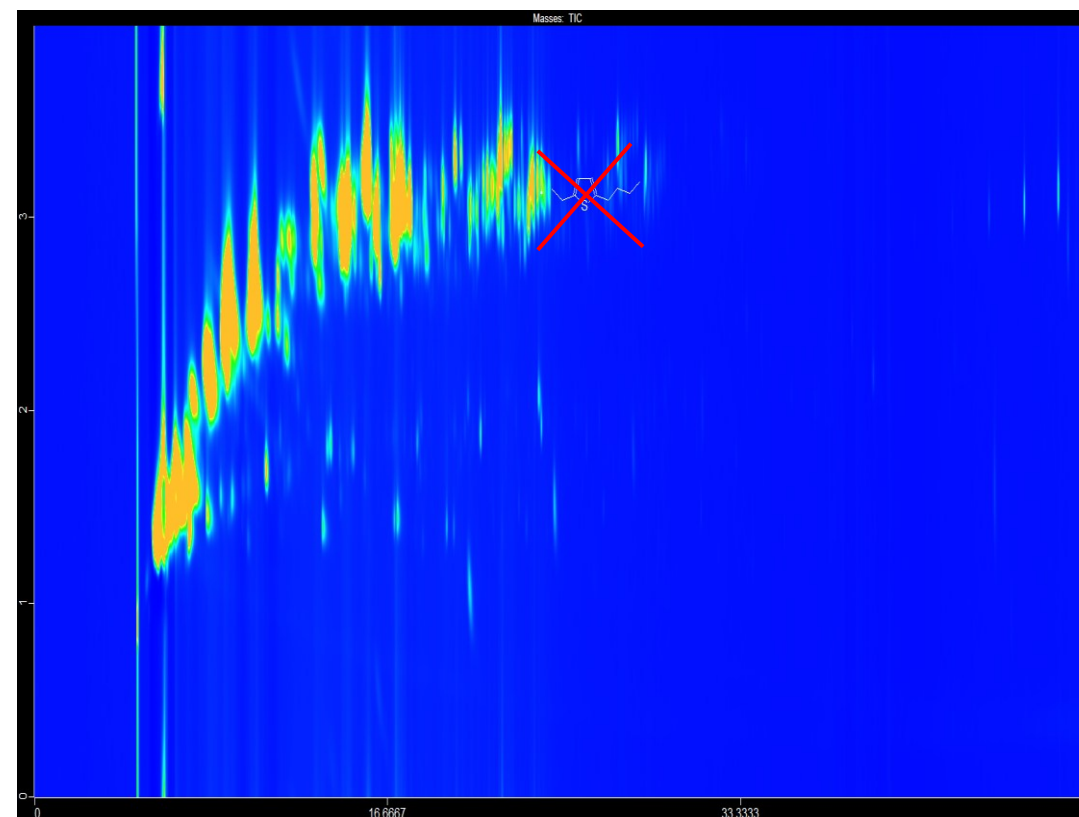
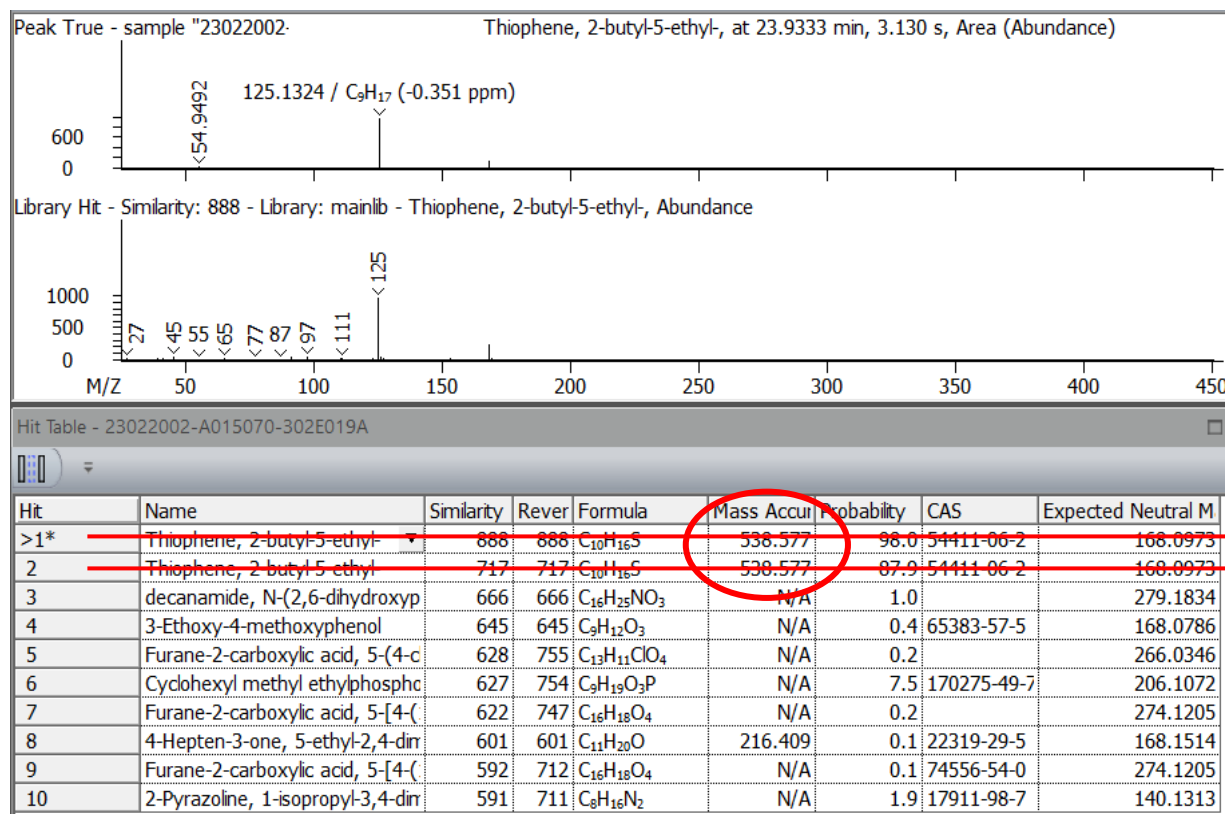


# Analysis of complex samples: GC×GC-HRTOFMS/FID



# Analysis of complex samples: GCxGC-HRTOFMS/FID

=> search for thiophene derivatives  
Challenge the library search results!



- Characterization of VOC and Odorous compounds from materials:
- Sampling techniques
  - Detectors (MS/Human nose)
  - Complementary techniques (GC×GC-HRTOFMS)

## Analytical & Technological Services

