



The role of GC×GC in oils & fats analysis

Giorgia Purcaro

Gembloux Agro Bio-Tech, University of Liège, Belgium

gpurcaro@uliege.be

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Can you summarize in a single sentence the benefits of comprehensive two-dimensional chromatography for **Fats and oils analysis**?



Comprehensive two-dimensional chromatography provides enhanced resolution and detailed profiling of complex lipid mixtures, enabling precise identification and quantification of fatty acids and other components in fats and oils.

Can you summarize in a single sentence the benefits of comprehensive two-dimensional chromatography for **Fats and oils analysis**?



Comprehensive two-dimensional gas chromatography enhances oil and fats analysis by providing exceptional separation, resolution, and sensitivity, enabling detailed profiling of complex lipid mixtures and contaminants ¹ ².

¹ www.aocs.org

² www.leco.com



Comprehensive two-dimensional chromatography provides enhanced resolution and detailed profiling of complex lipid mixtures, enabling precise identification and quantification of fatty acids and other components in fats and oils.

Can you summarize in a single sentence the benefits of comprehensive two-dimensional chromatography for **Fats and oils analysis?**



- Separation <> Resolution
- Selectivity
- Sensitivity
- Speed in terms of number of compounds separated for unit of time
- Structured chromatograms

5s

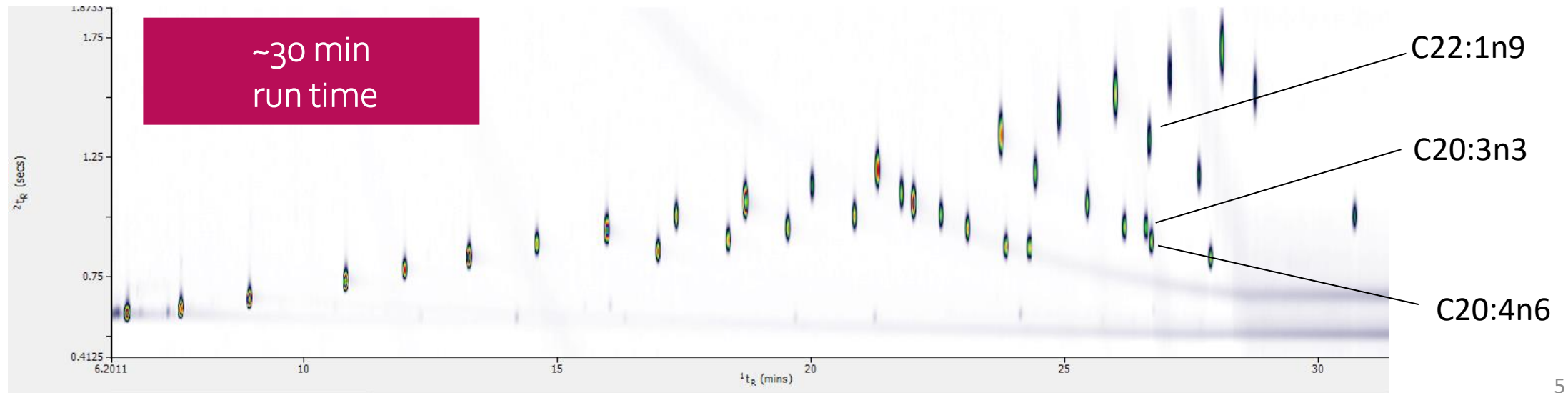
GCxGC-FID

FAMES



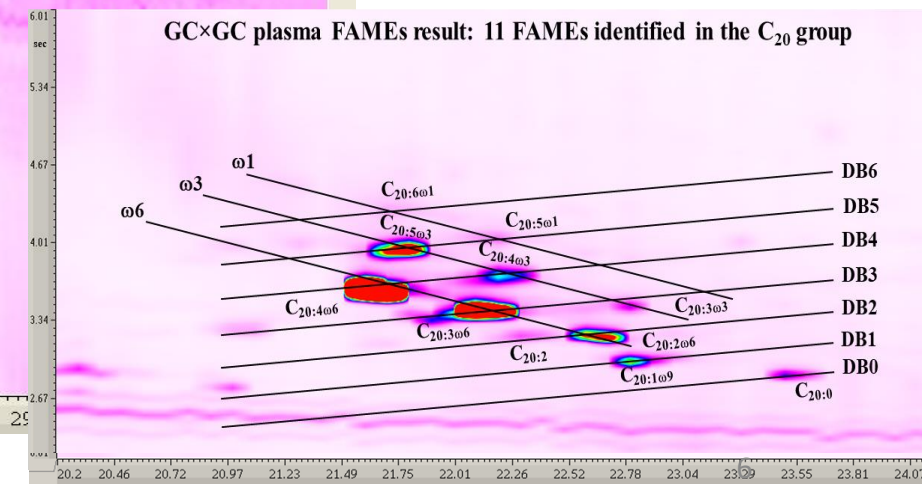
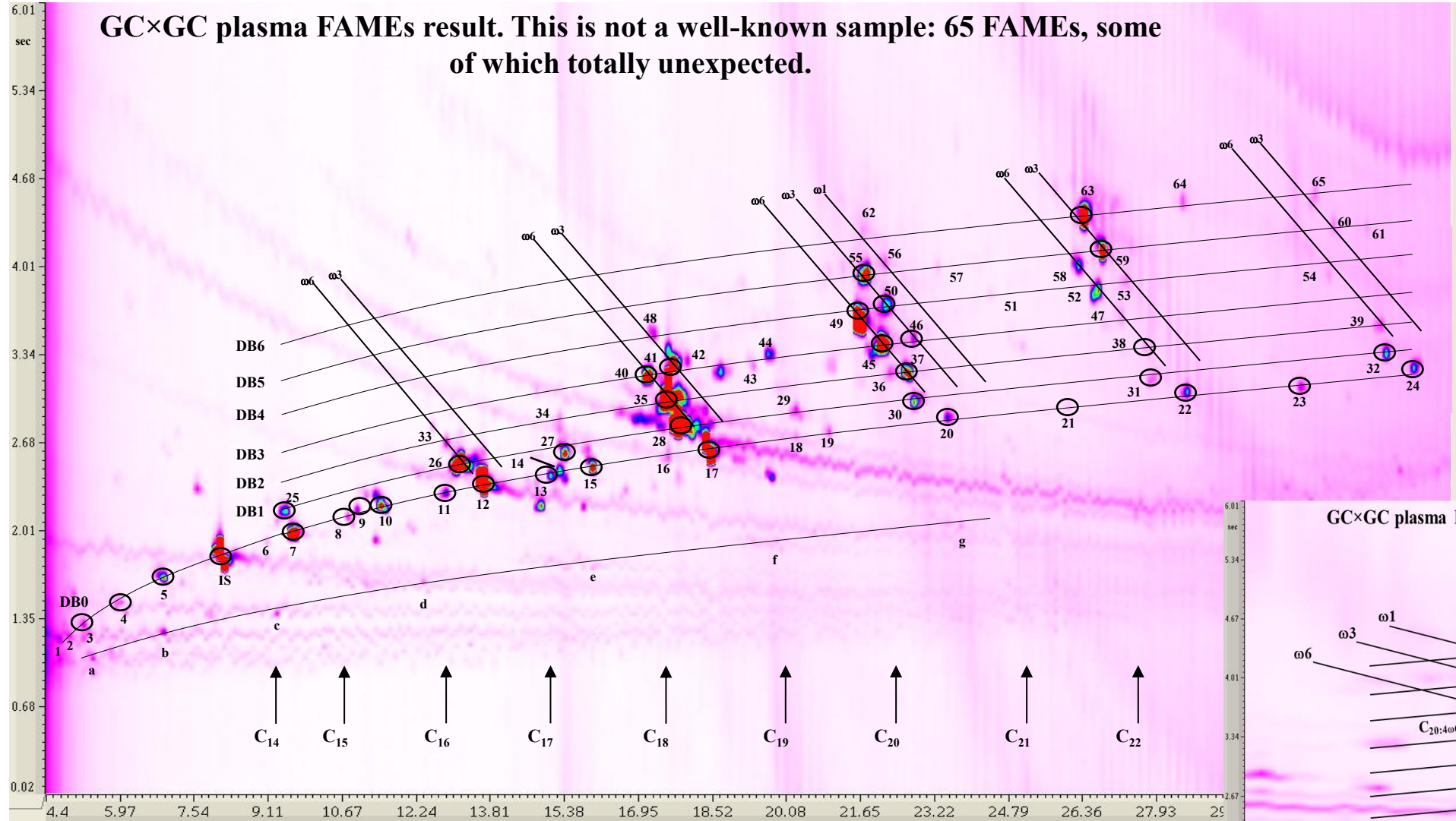
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ADVANTAGES OF GCxGC



Analysis of FAME:

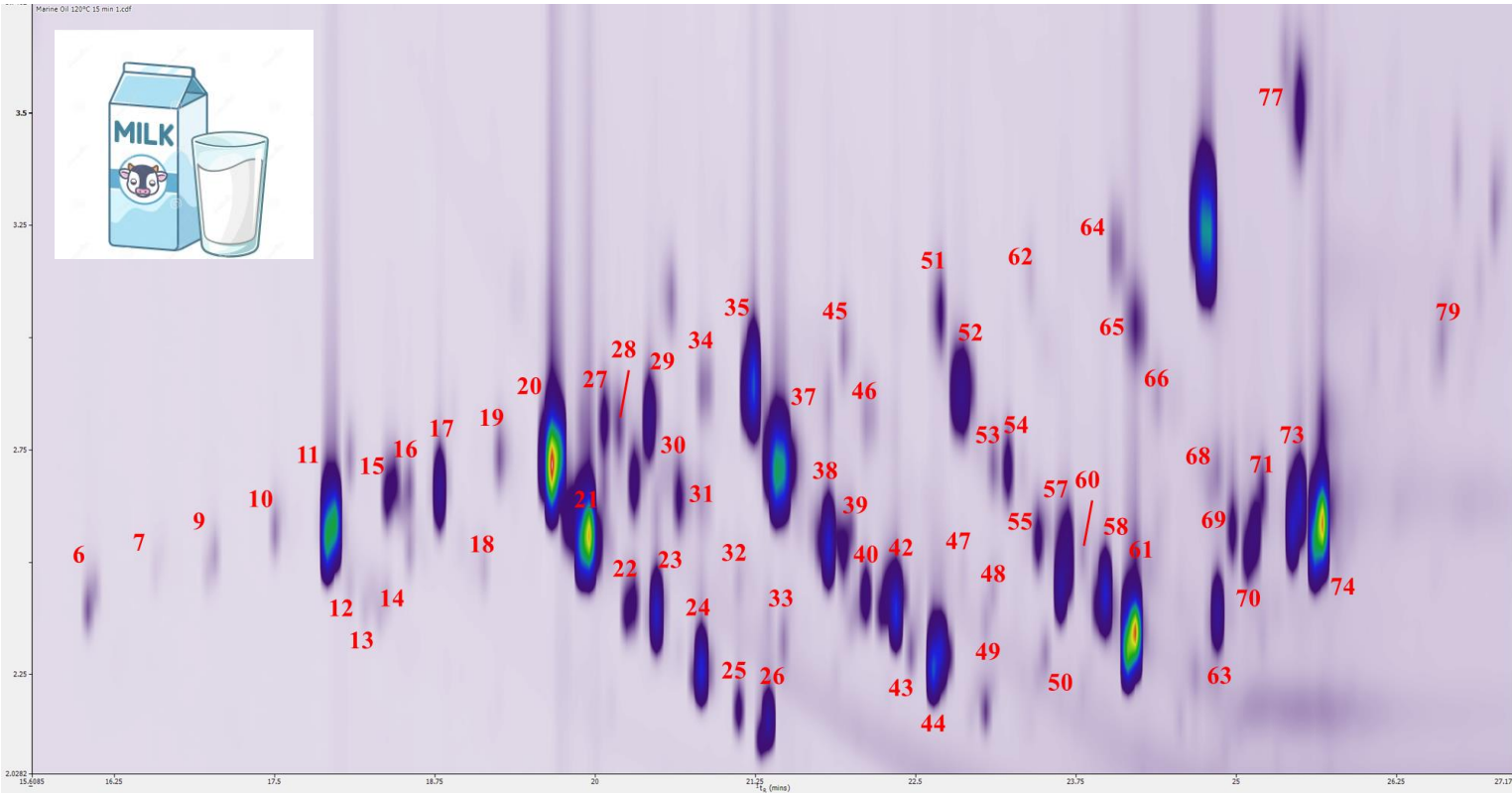
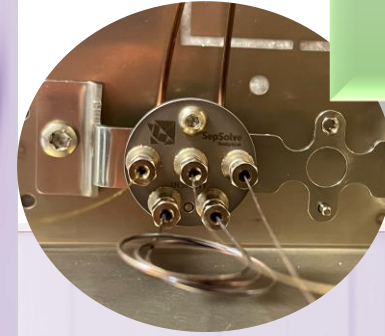
GC×GC



Analysis of FAME:

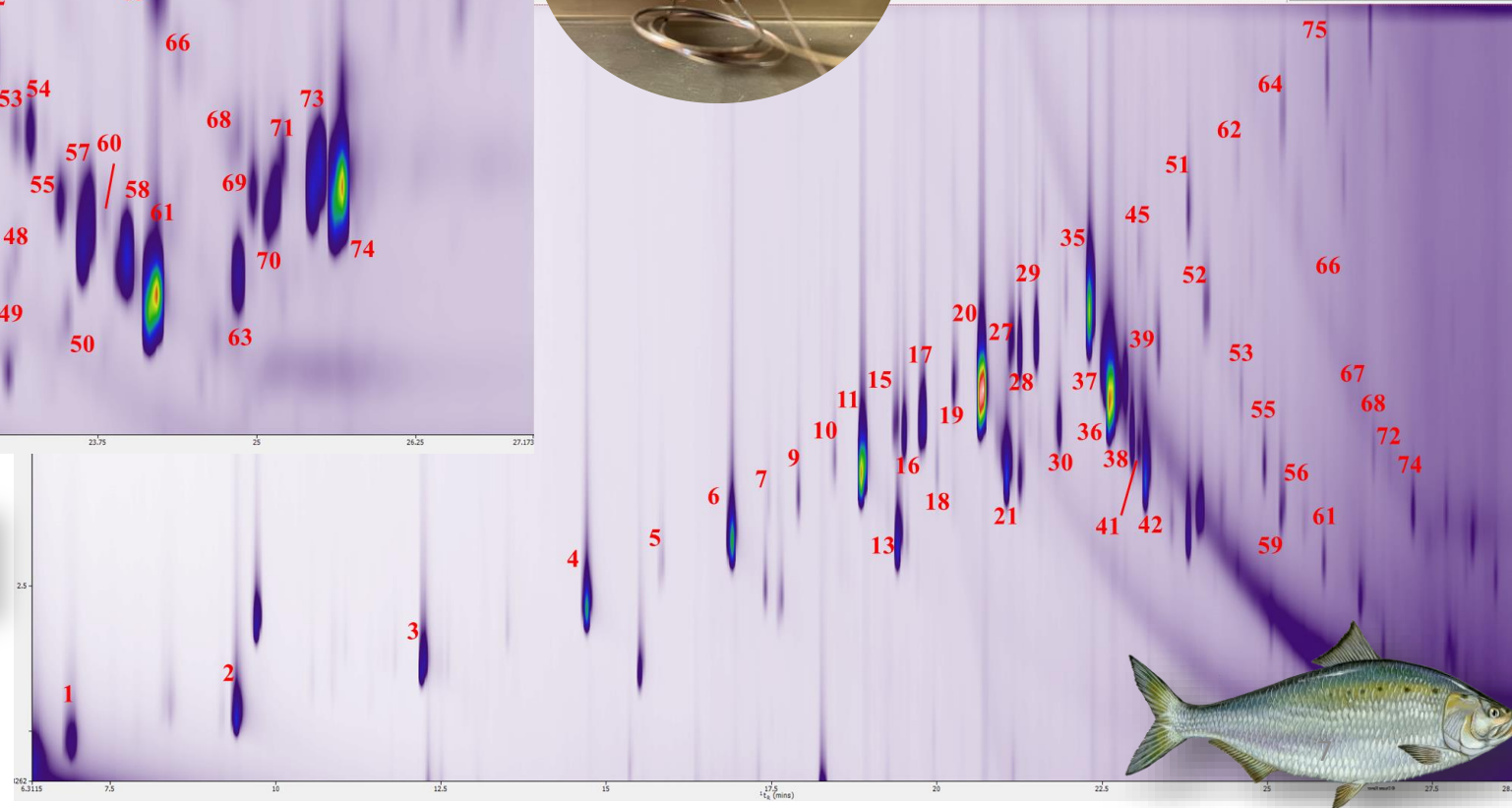
GC×GC-FID

~30 min
run time



79 different FAMES!

- comparable with a cryogenic separation
- superior to previously reported FFF-GC×GC



FAMEs

GC × GC–FID



- Separation <> Resolution
- Selectivity
- Sensitivity
- Speed in terms of number of compounds separated for unit of time
- Structured chromatograms

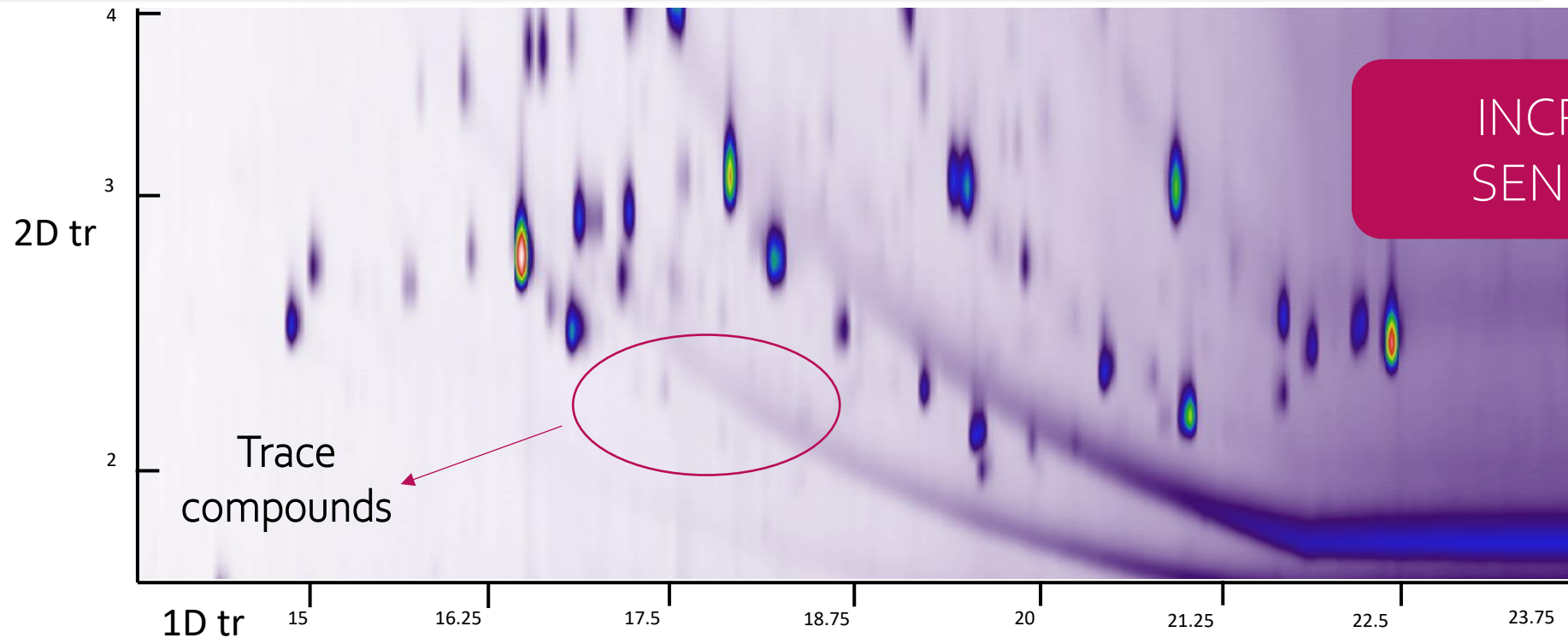


FAMES

GC × GC–FID



- Separation \leftrightarrow Resolution
- Selectivity
- Sensitivity
- Speed in terms of number of compounds separated for unit of time
- Structured chromatograms



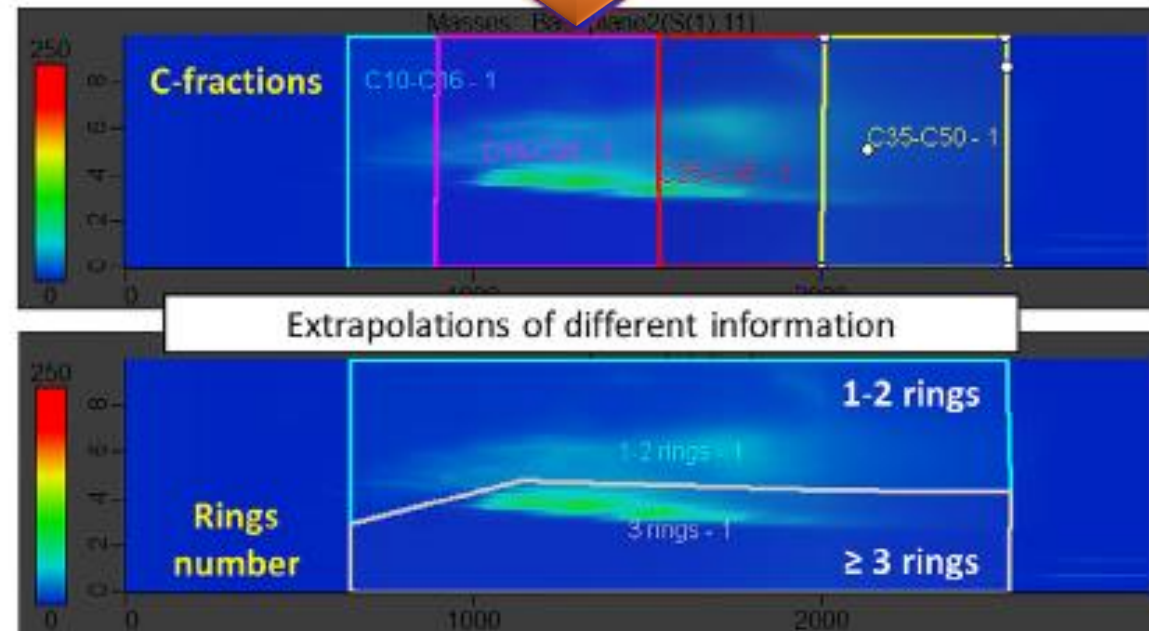
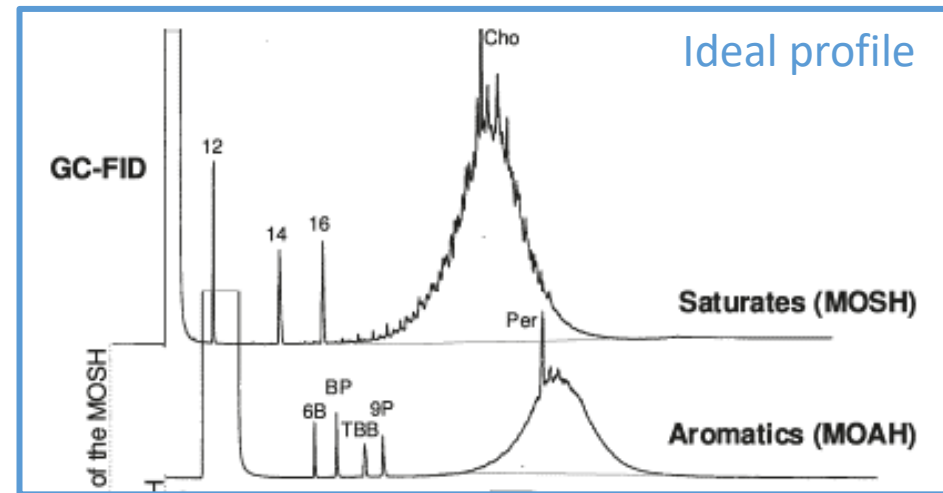
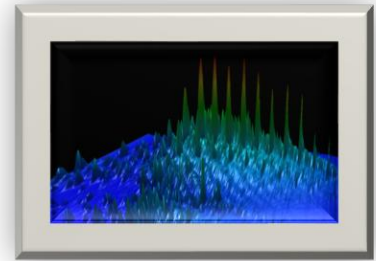
Can you summarize in a single sentence the benefits of comprehensive two-dimensional chromatography for **Fats and oils analysis**?



- Separation <> Resolution
 - Selectivity
 - Sensitivity
 - Speed in terms of number of compounds separated for unit of time
 - Structured chromatograms
-
- Support and simplify sample preparation and data interpretation

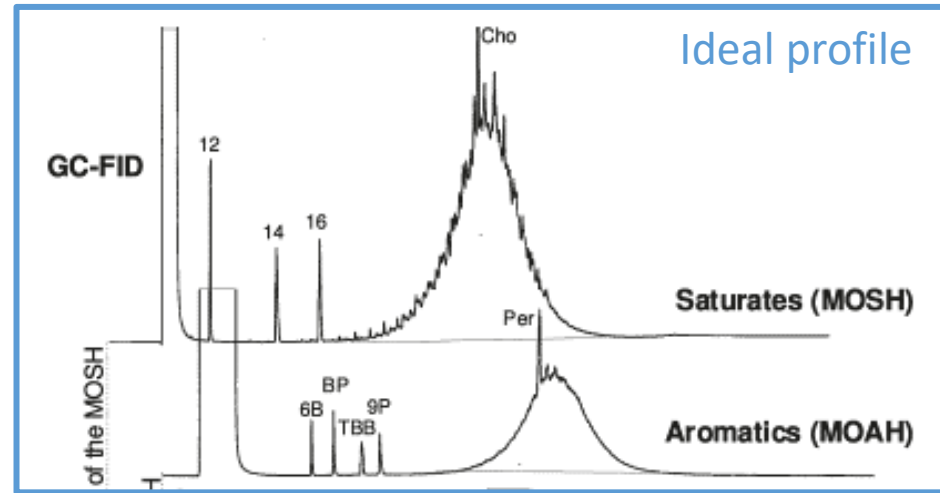
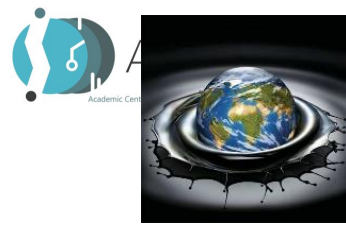
5s

GC×GC benefits for MOSH and MOAH analysis



MOSH & MOAH
sub-classes
quantification

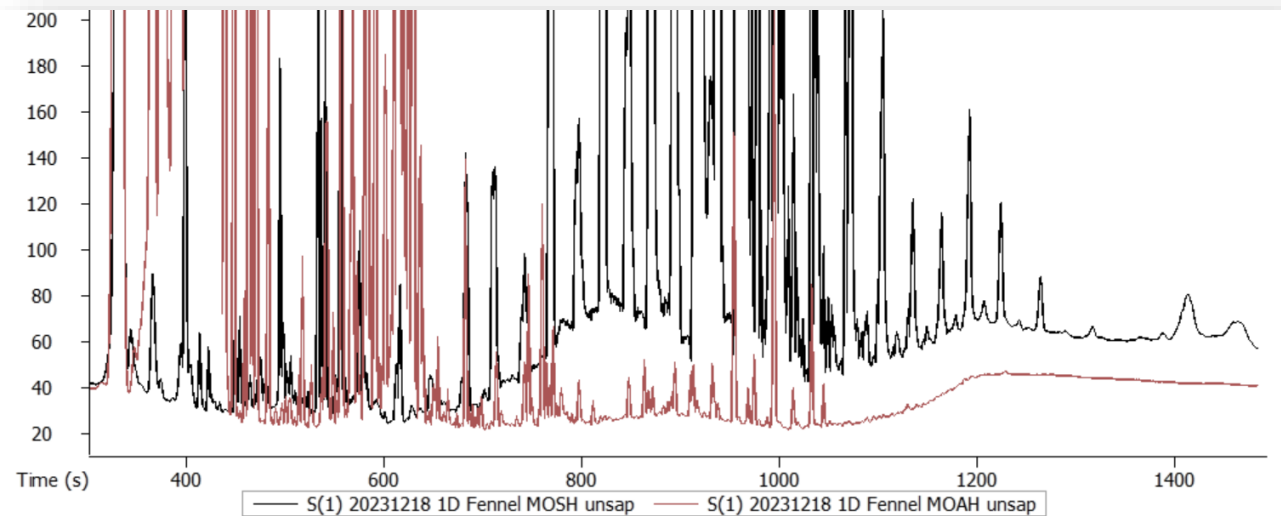
GCxGC benefits for MOSH and MOAH analysis



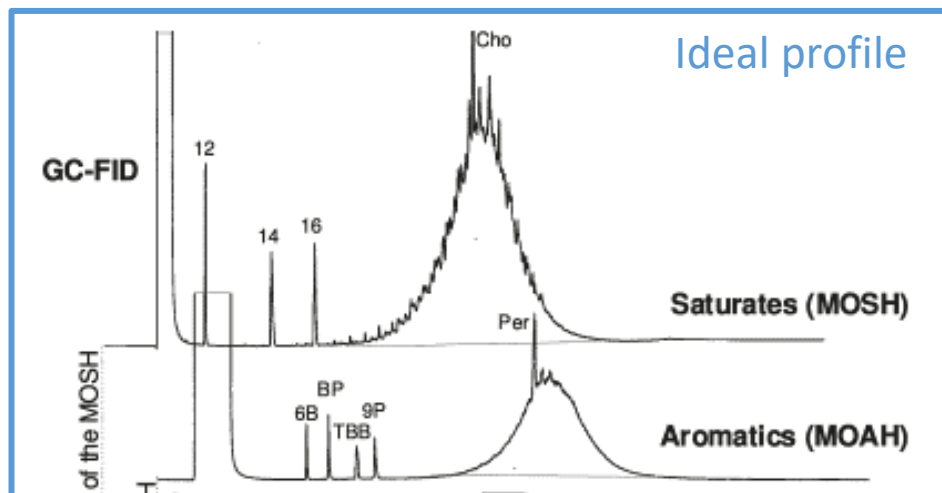
➤ **Baseline**

➤ **Riding peaks subtraction**

MOSH vs MOAH – No purification



GCxGC benefits for MOSH and MOAH analysis

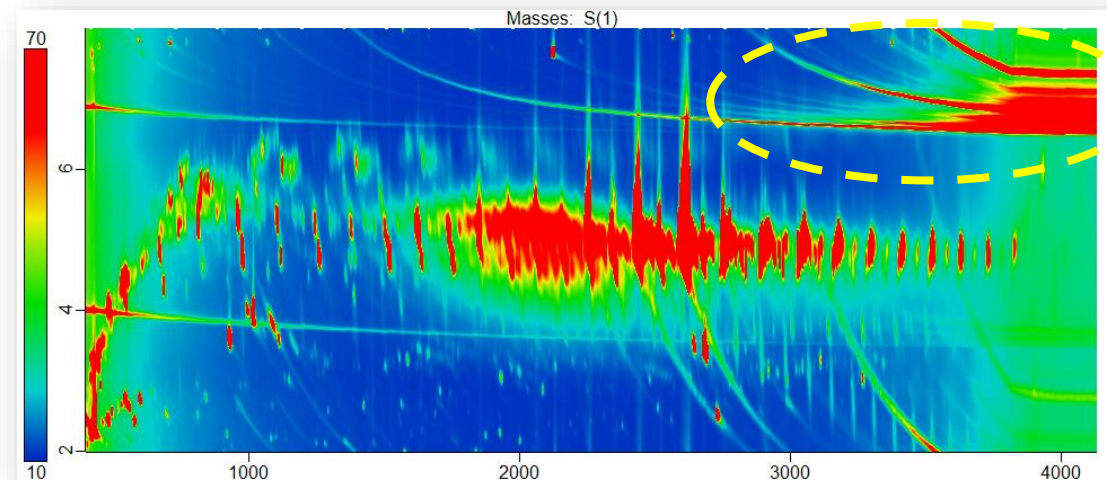
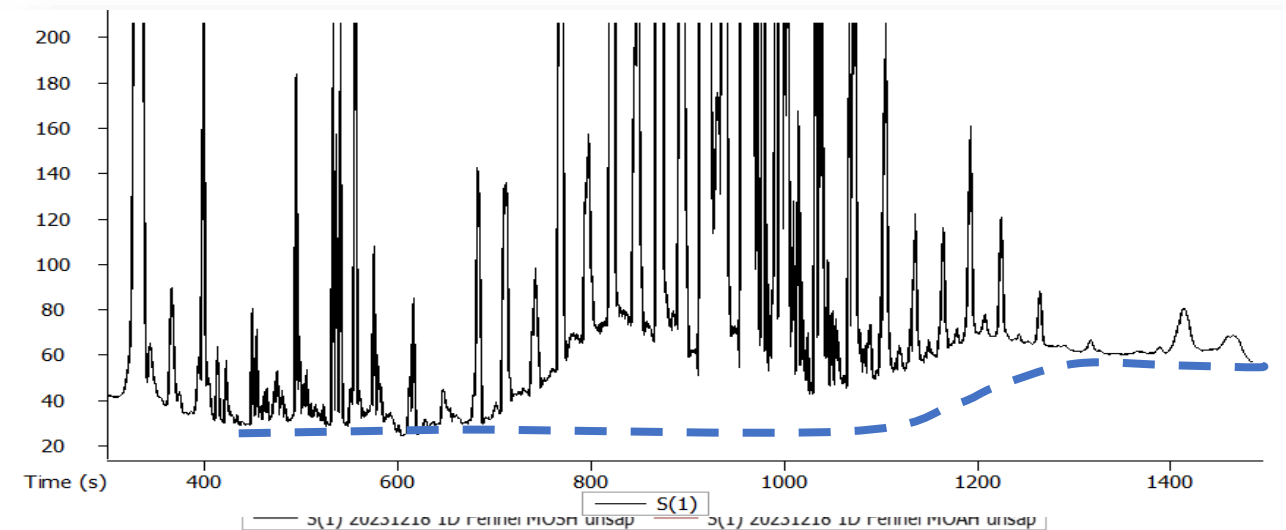


➤ **Baseline**

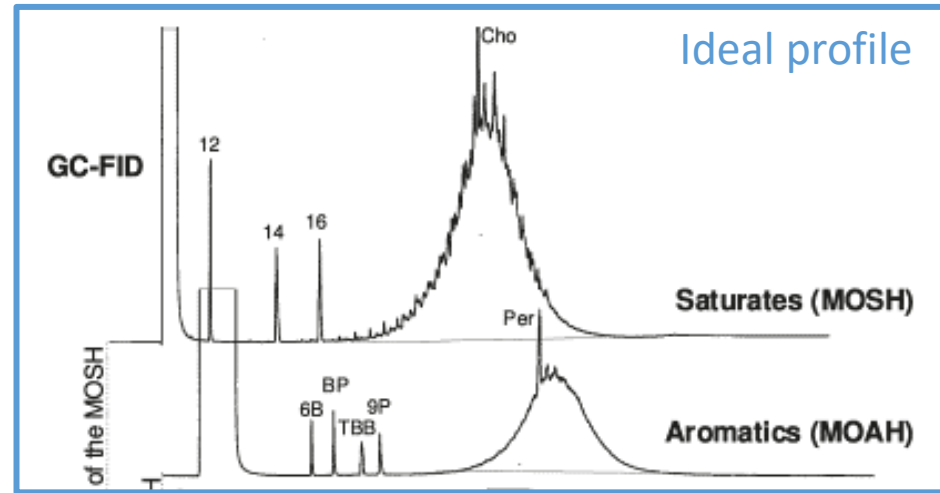
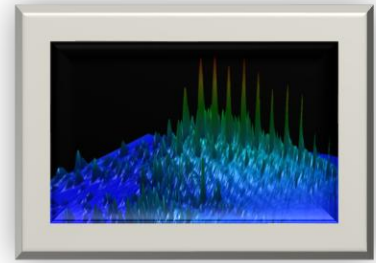
➤ **Riding peaks subtraction**

More accurate quantification

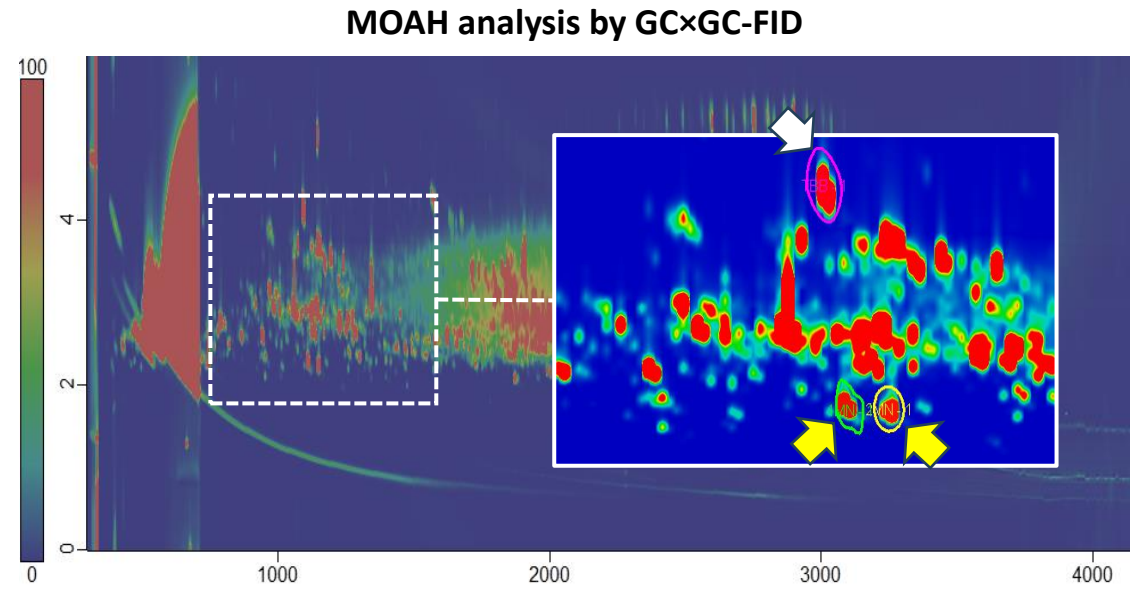
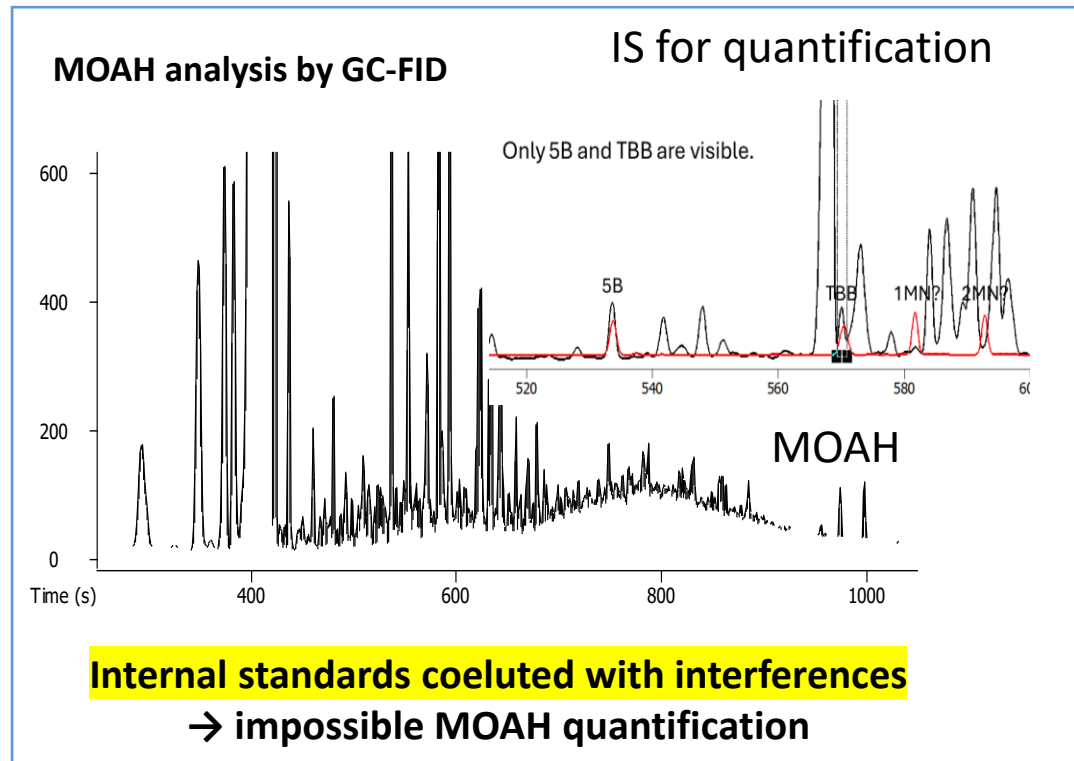
MOSH vs MOAH – No purification



GC×GC benefits for MOSH and MOAH analysis

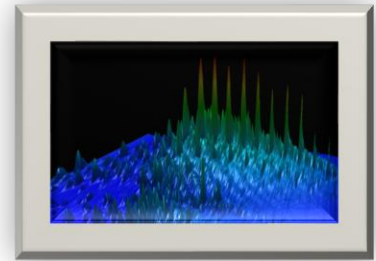


**More accurate
quantification**

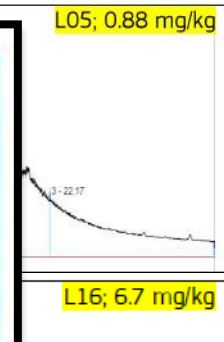
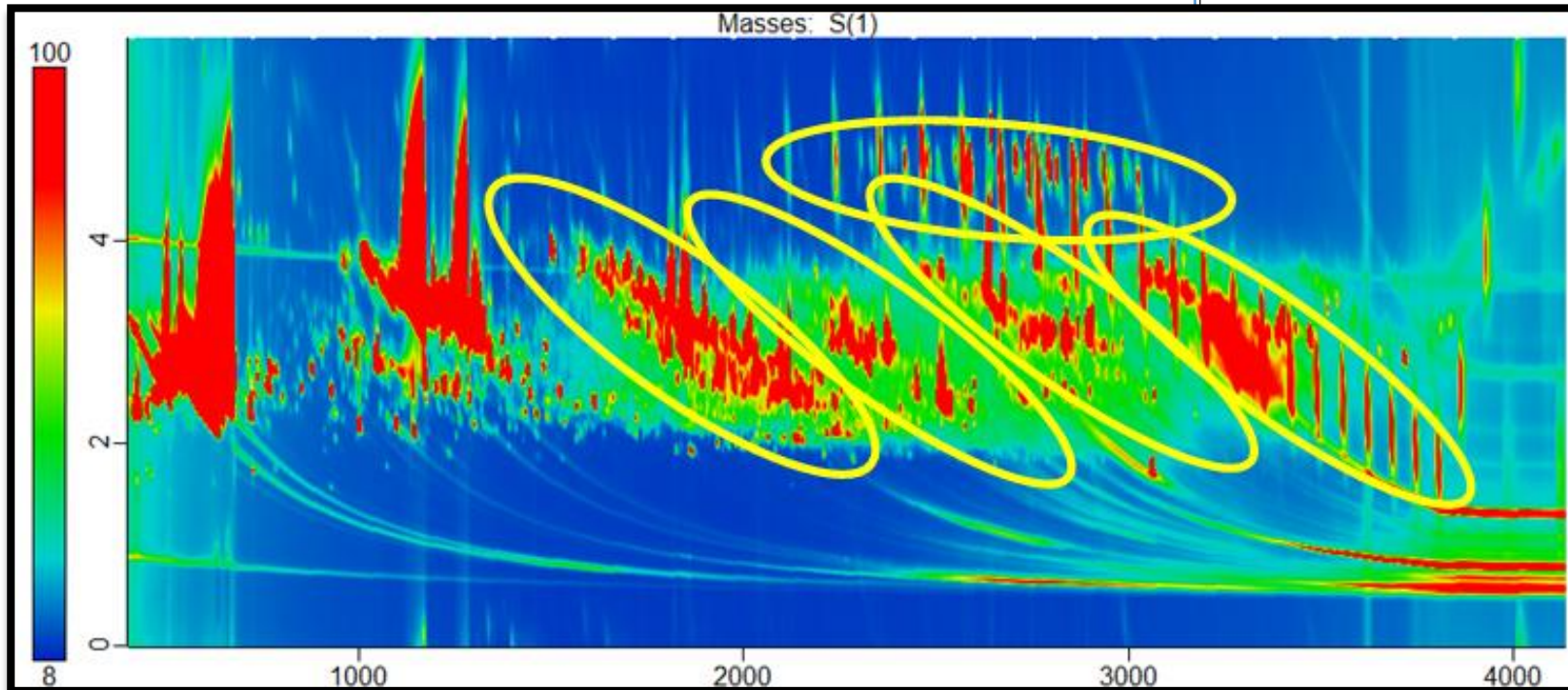
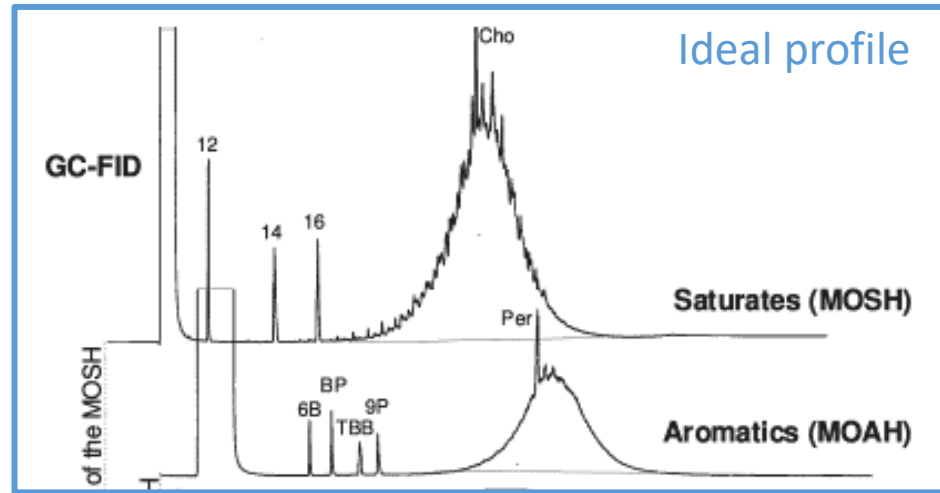


**Internal standards resolved from the interferences
→ quantification of MOAH is possible**

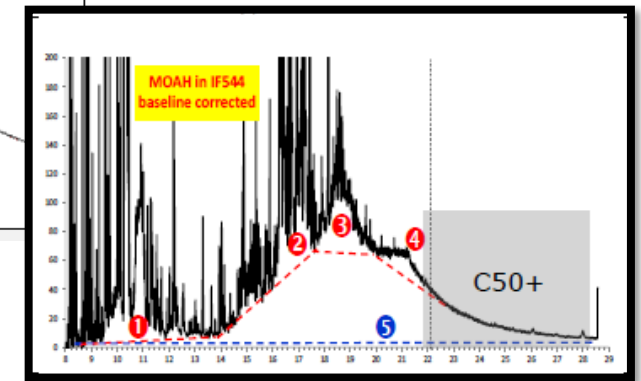
GCxGC benefits for MOSH and MOAH analysis



Easier interpretation & more accurate quantification



Coeluted interferences to be removed
→ High uncertainty of the result



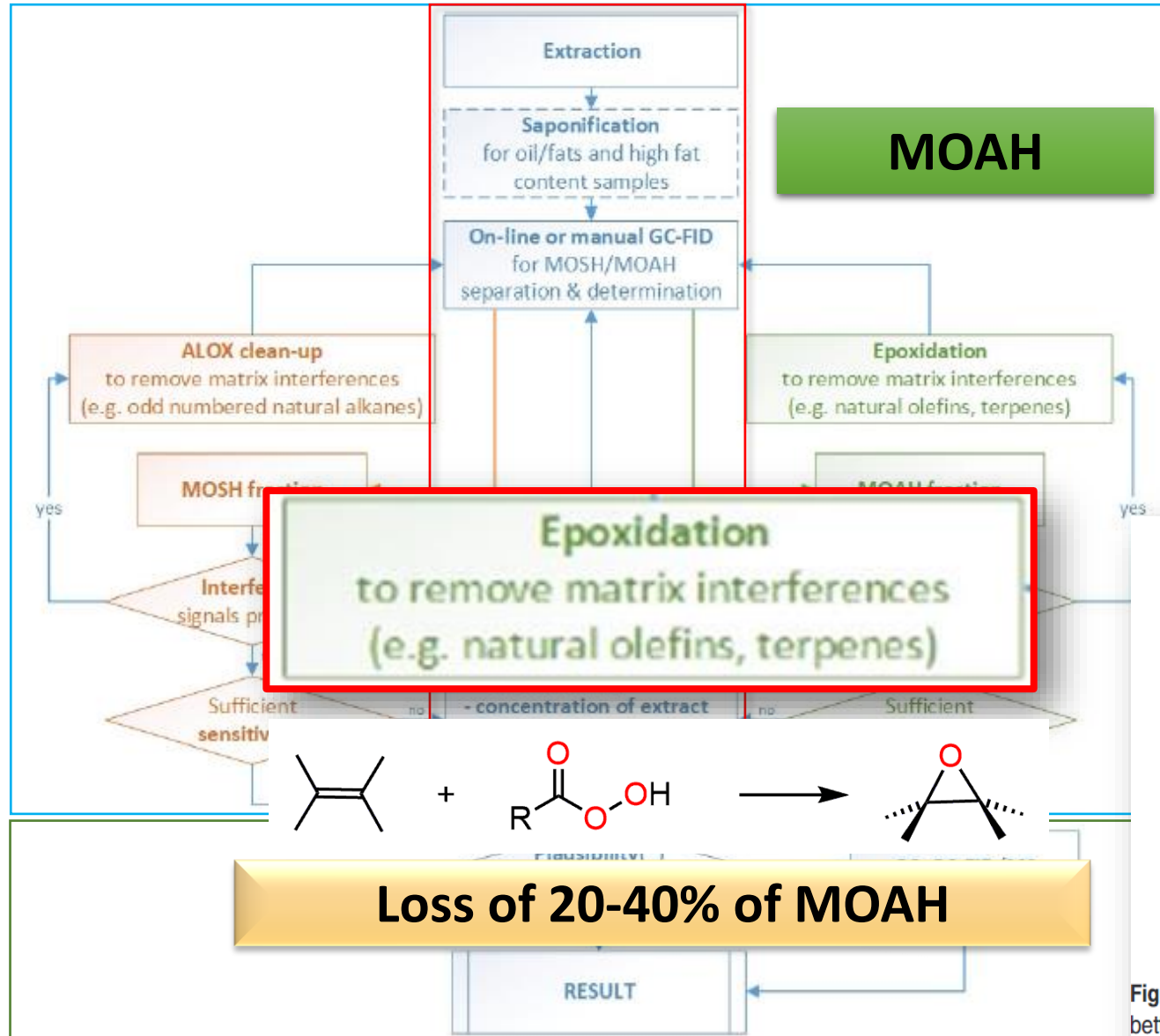


Figure 5 Decision tree on the use of auxiliary methods.

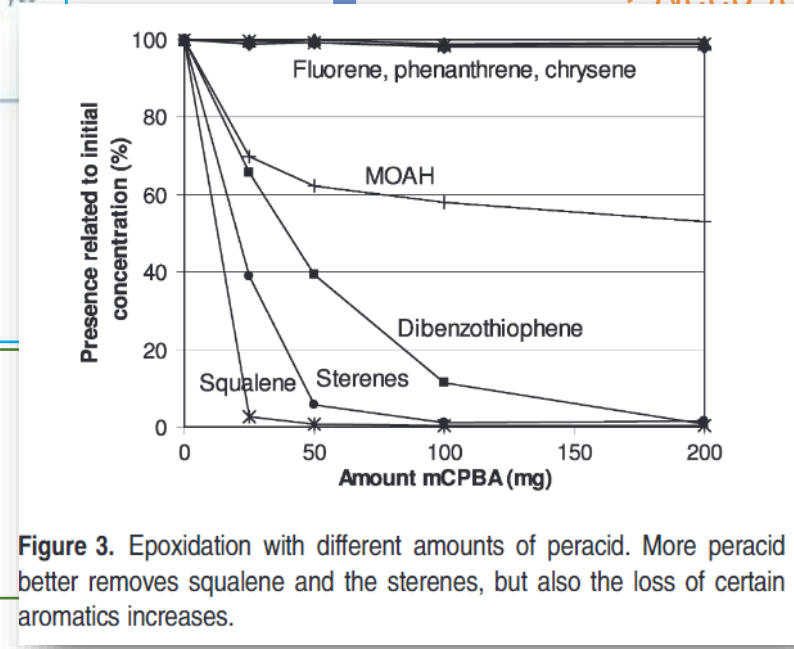


Figure 3. Epoxidation with different amounts of peracid. More peracid better removes squalene and the sterenes, but also the loss of certain aromatics increases.

✓ Need for matrix-tailored prep protocols

% of uncertainty!

Data Integration

An alternative purification method

Journal of Chromatography A 1743 (2025) 465684

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Purification of mineral oil aromatic hydrocarbons and separation based on the number of aromatic rings using a liquid chromatography silica column. An alternative to epoxidation

Aleksandra Gorska ^a, Grégory Bauwens ^a, Marco Beccaria ^b, Giorgia Purcaro ^{a,*}

LC-GC×GC-FID/MS



Very good removal of carotenoids and squalene

Other terpenoids are less well removed



MOAH Recovery LC Purification:

94% ± 2%

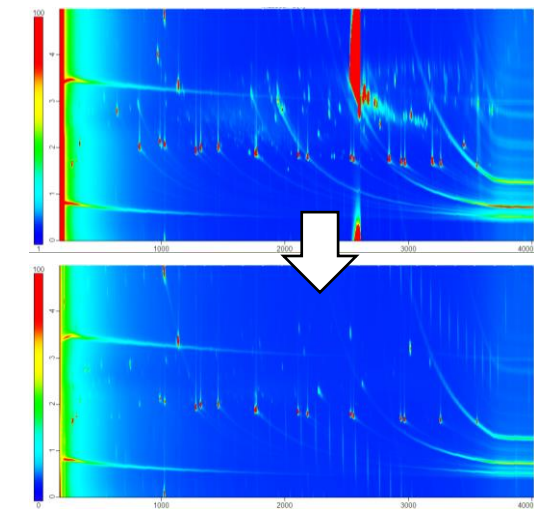
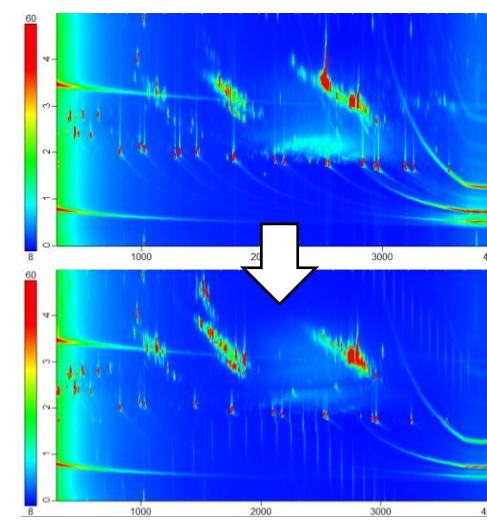
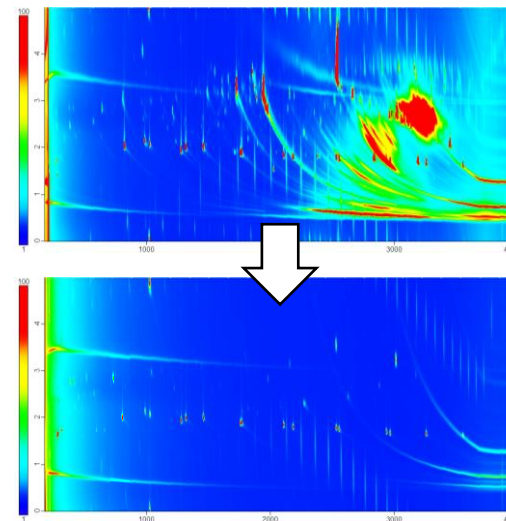
Epoxidation

mCPBA

82% ± 10%

performic acid

71 ± 16%

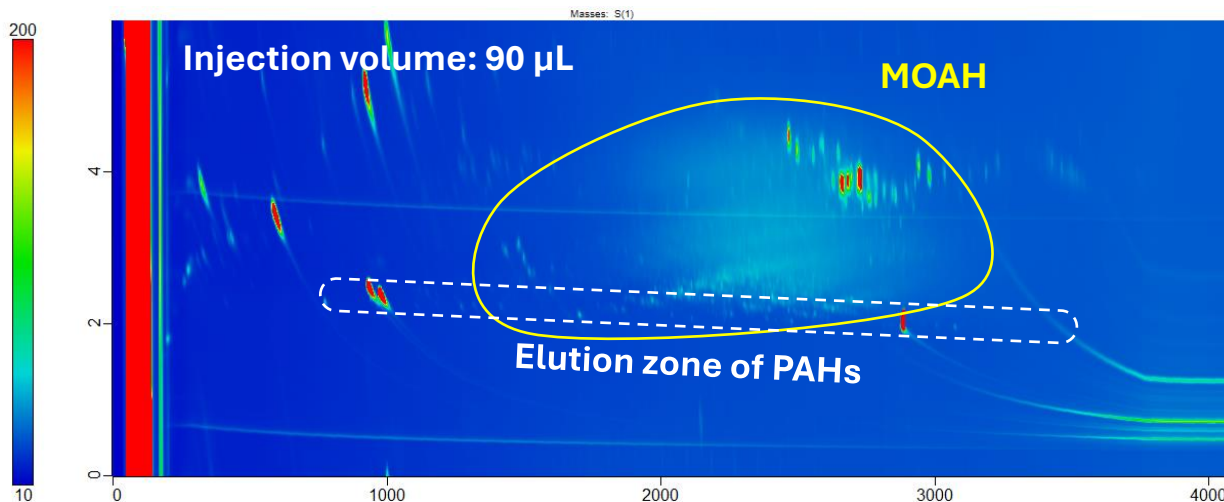


Simultaneous analysis of MOAH and PAHs



Example of MOAH and PAHs analysis in soya lecithin

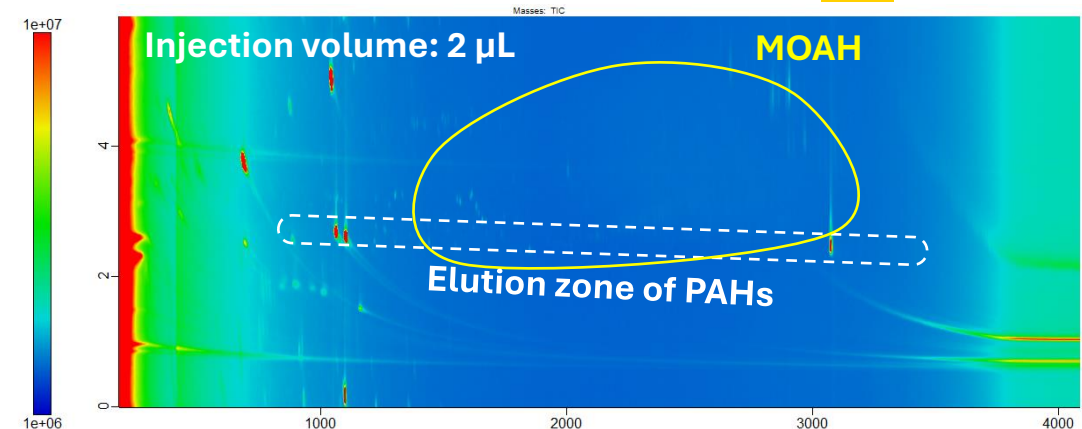
HPLC/GC×GC-FID



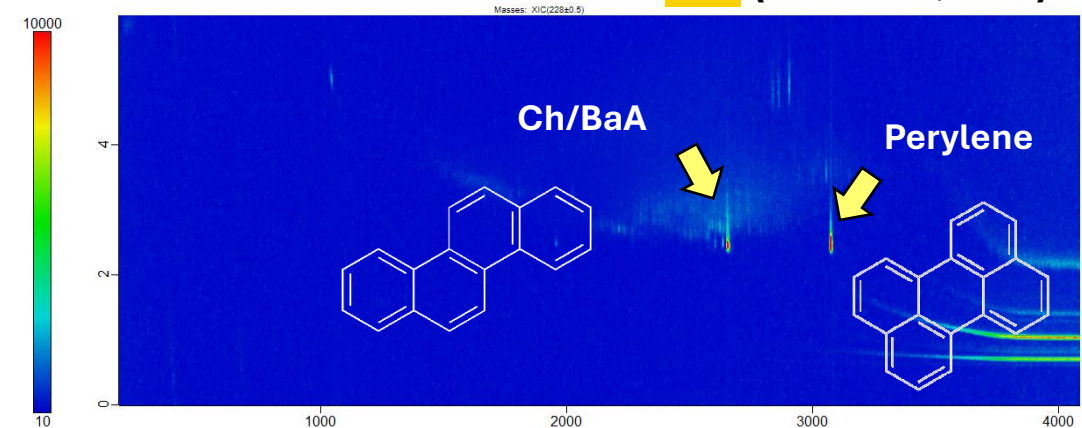
MOAH fraction of a soya lecithin spiked with **0.5 mg/kg MOAH** and **1 to 2 µg/kg PAHs** (16 compounds)

In FID, we see the MOAH, but not the 16 PAHs

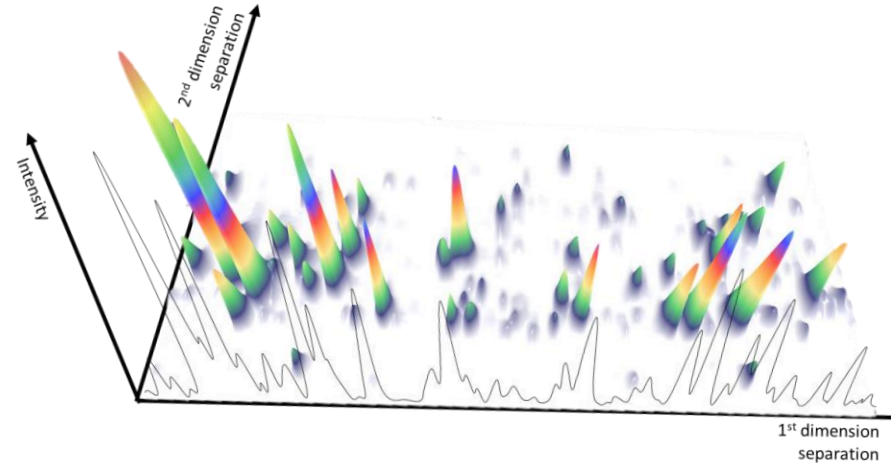
HPLC/GC×GC-TOFMS – TIC



HPLC/GC×GC-TOFMS – EIC (m/z 228; 252)



In MS, we see the PAHs using EIC



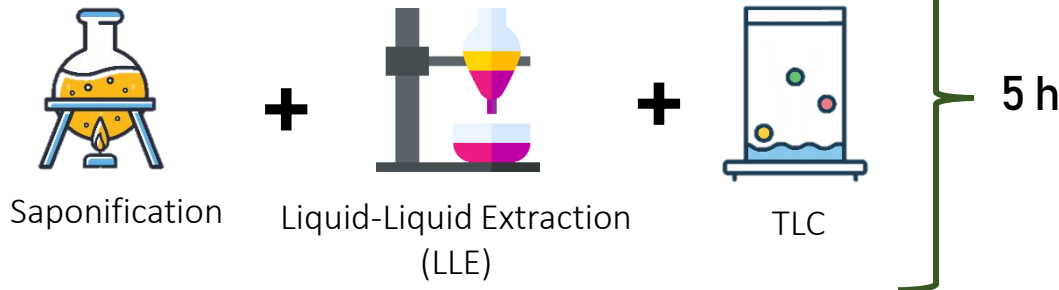
Support and Simplify Sample Prep



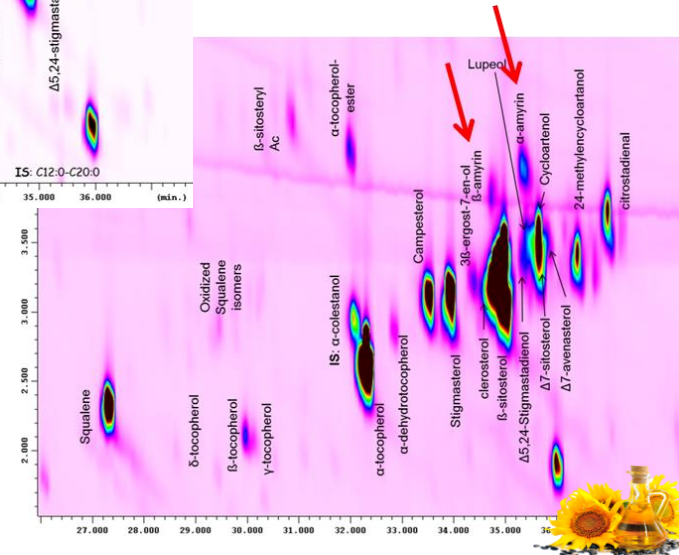
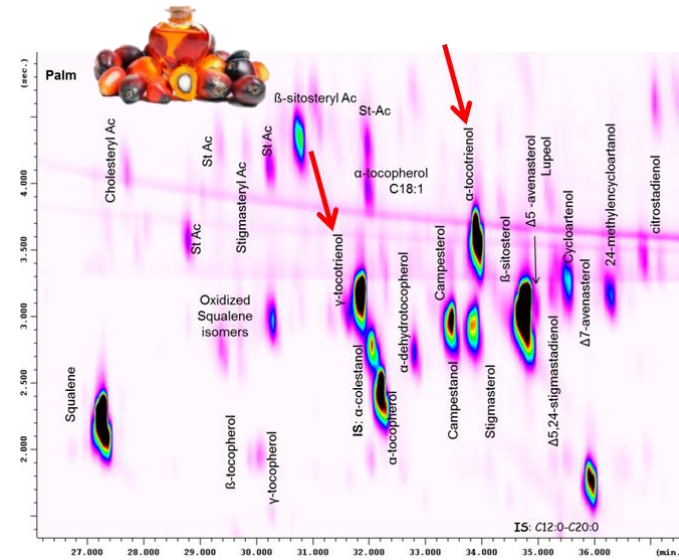
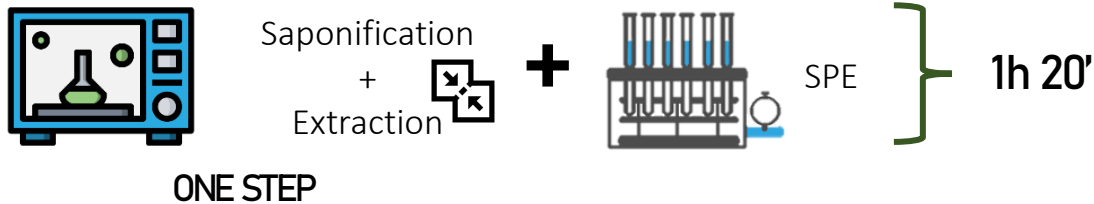
STEROLS



International Olive Council method (IOC)



Microwave-Assisted Saponification and Extraction (MASE)



STRUCTURED ANALYSIS

ENHANCED SEPARATION

INCREASED SENSITIVITY

REDUCED CO-ELUTION



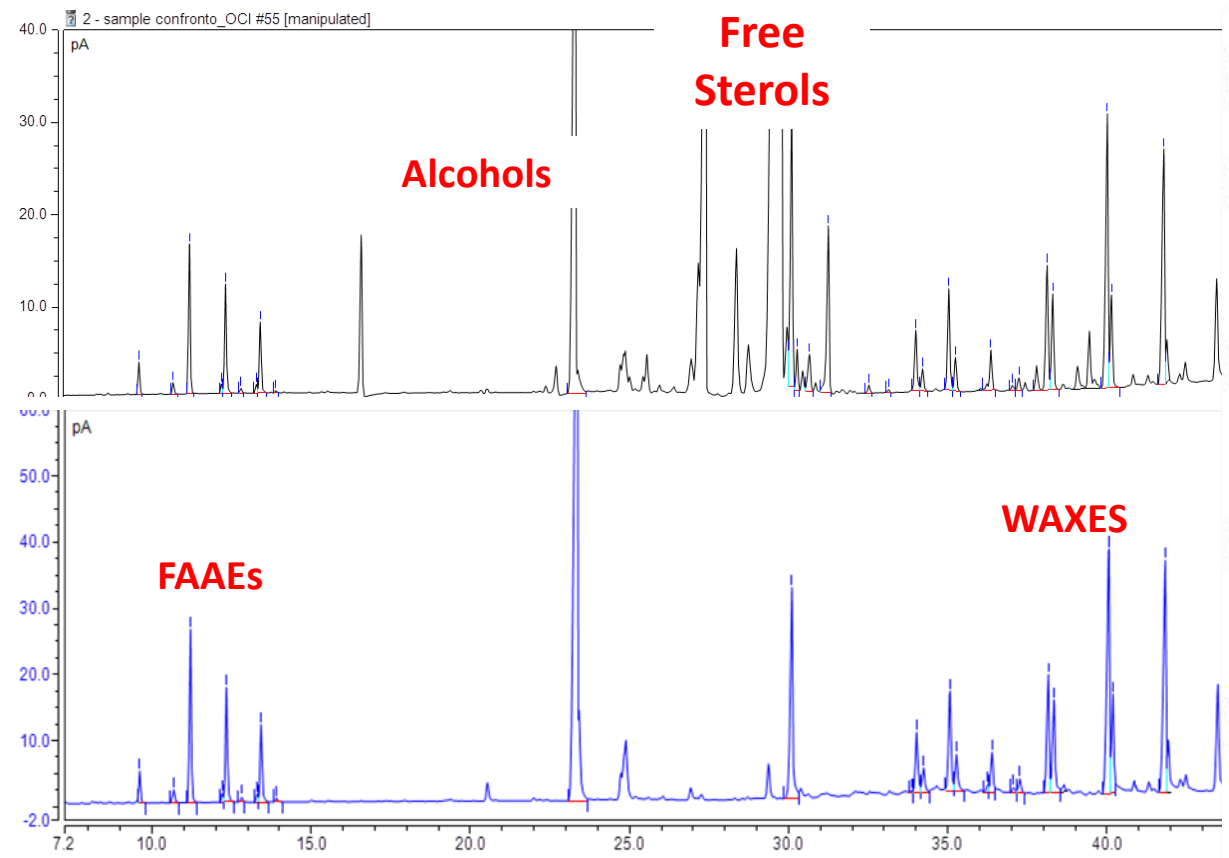
ANALYTICAL METHODS



Slight modification compared to the official method for waxes and FAAE

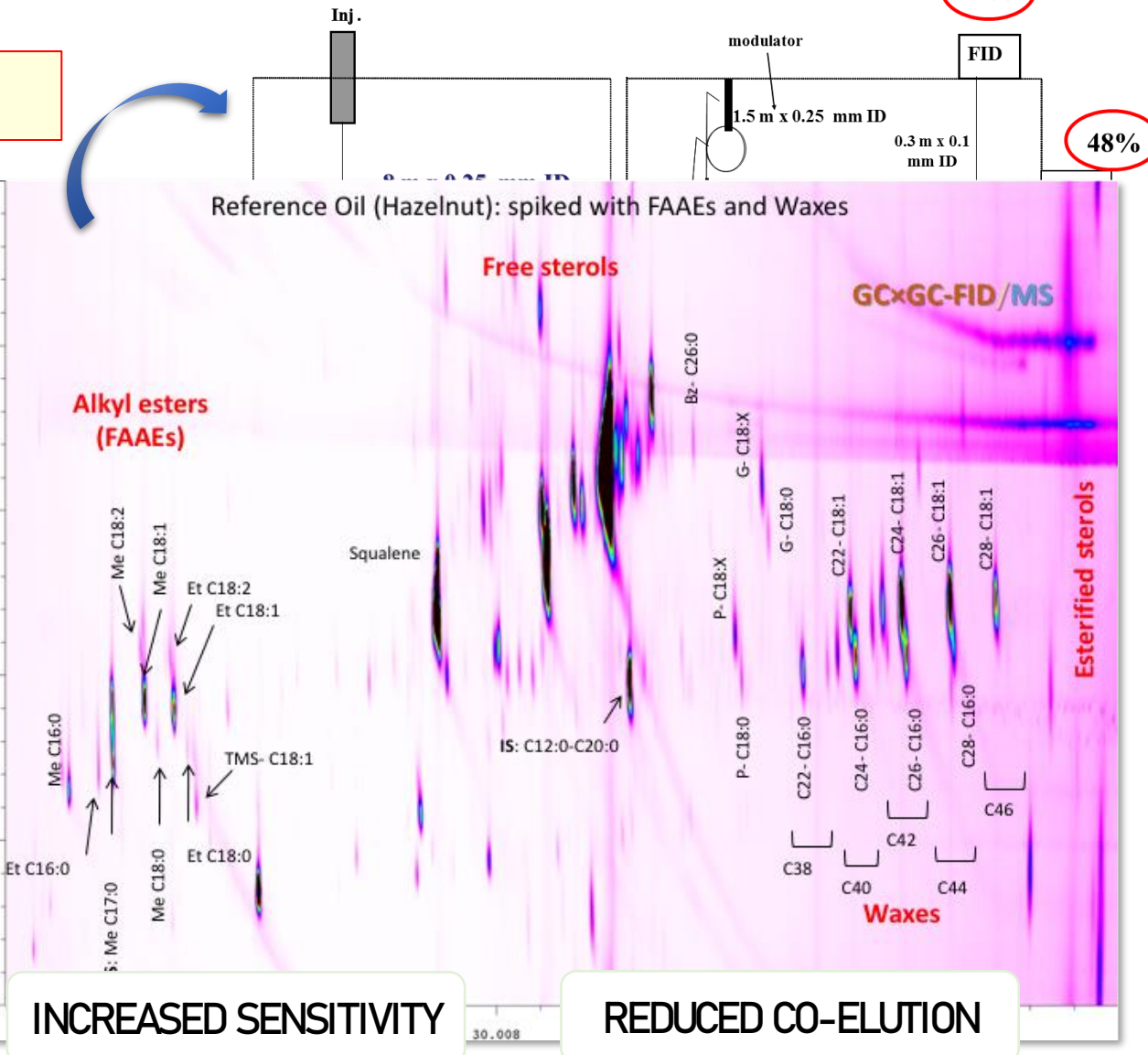


Silylation before loading the oil



STRUCTURED ANALYSIS

ENHANCED SEPARATION



INCREASED SENSITIVITY

REDUCED CO-ELUTION

52%

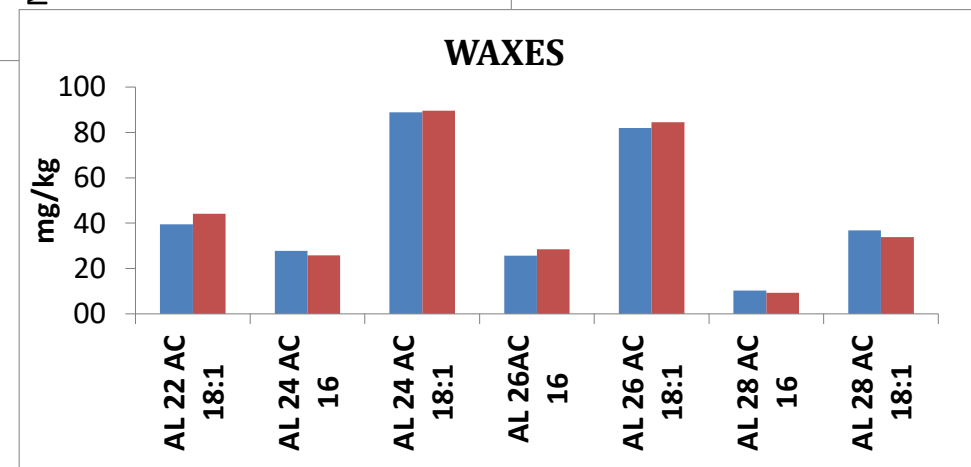
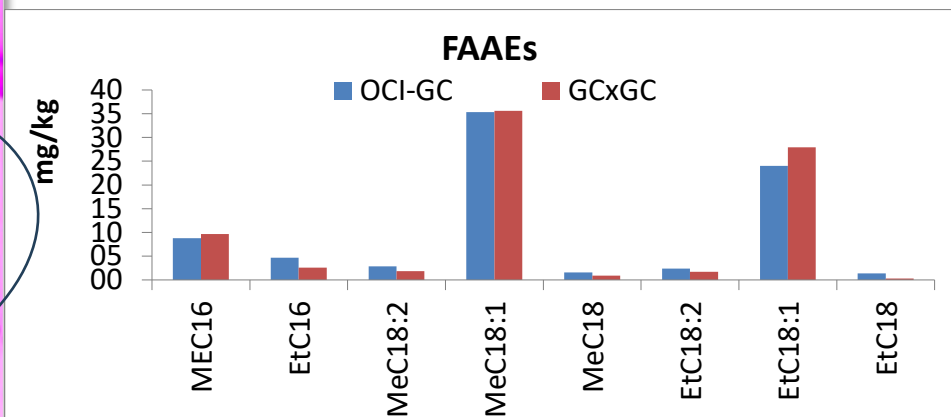
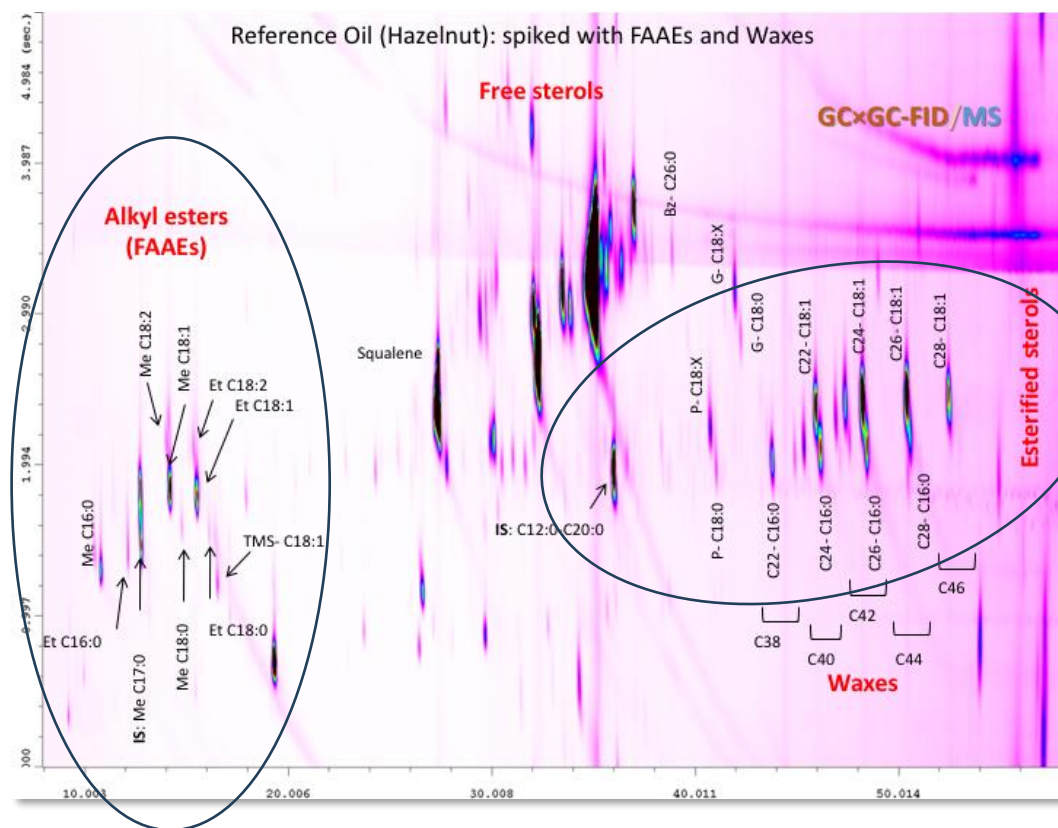
48%

MINOR COMPOUNDS



a-comparison with the Official method

(FAAEs and Wax) GC×GC-FID/MS

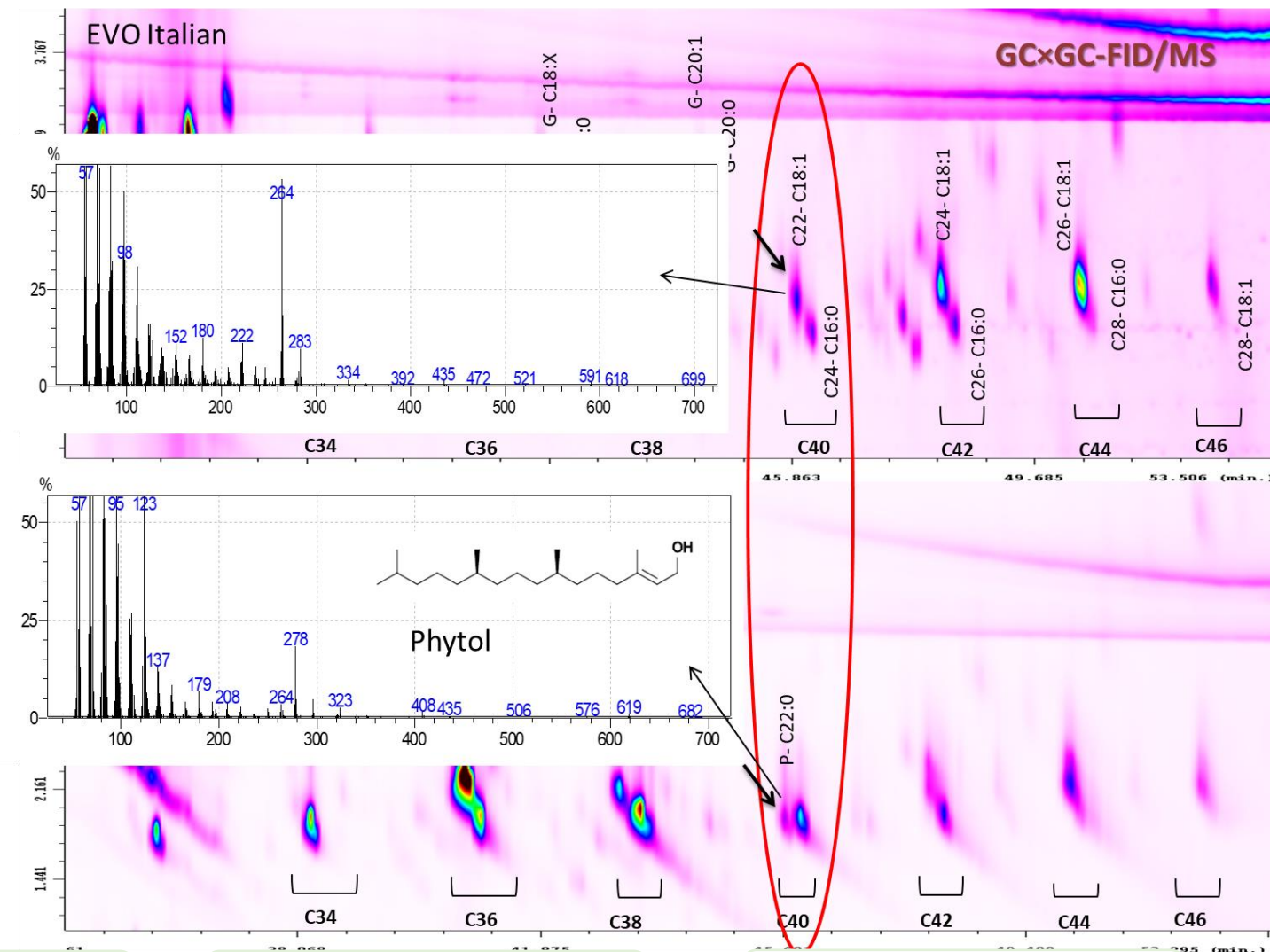
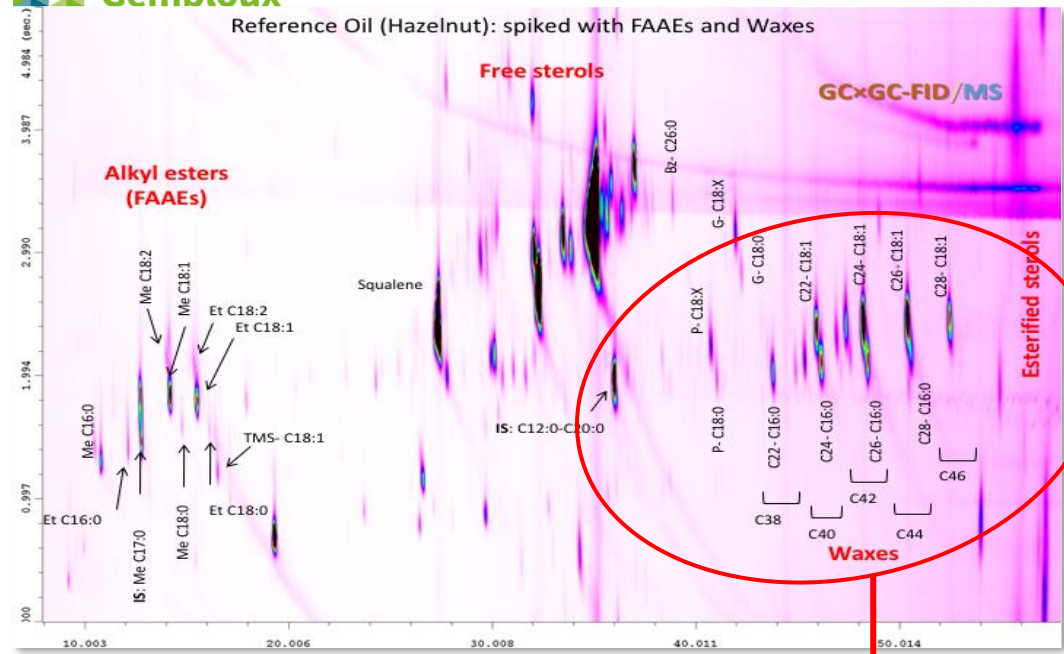


G. Purcaro*, L. Barp, L. Conte, *J. Sep. Sci.* 38 (2015) 2278-2285

G. Purcaro*, L. Barp, M. Beccaria, L. Conte, *Anal Bioanal Chem* 407 (1) (2015) 309-319.

L. Barp, F. A. Franchina, G. Purcaro*, P. Q. Tranchida, L. Mondello, *Talanta* 165 (2017) 598-603.

GC×GC-FID/MS



STRUCTURED ANALYSIS

ENHANCED SEPARATION

INCREASED SENSITIVITY

REDUCED CO-ELUTION

MINOR COMPOUNDS



a-comparison with the Official method

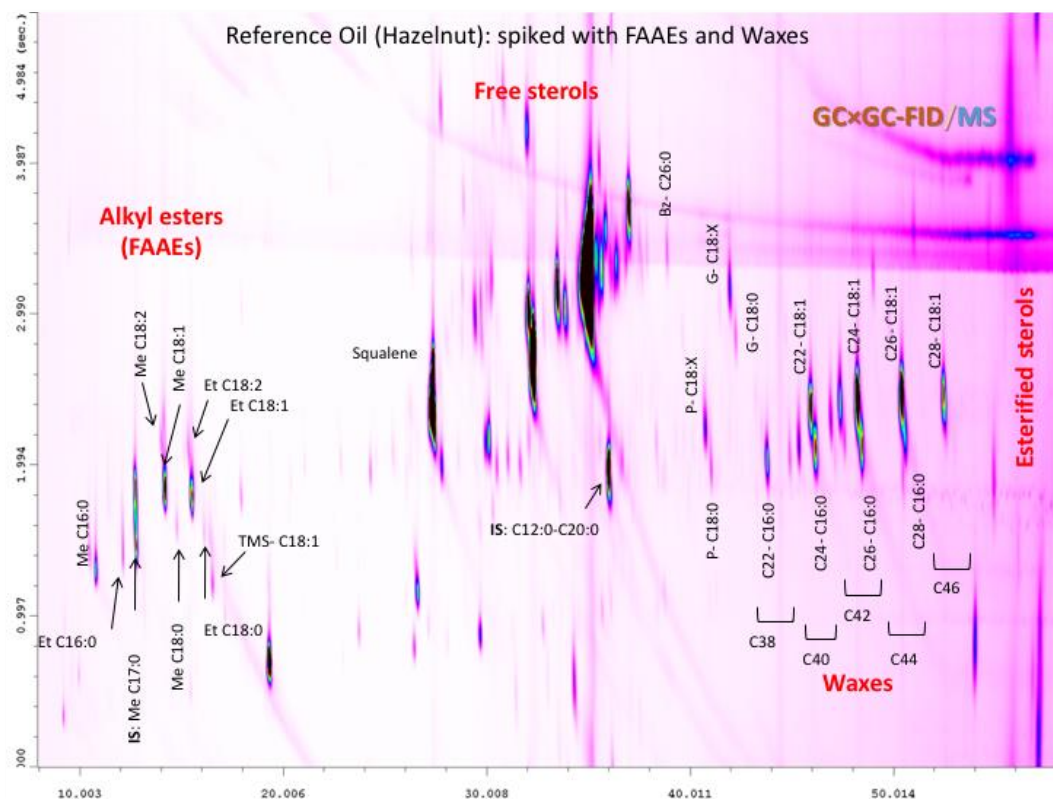
(FAAEs and Wax)

GC×GC-FID/MS

b- Detailed investigation

Structured chromatogram

GC×GC-FID/MS



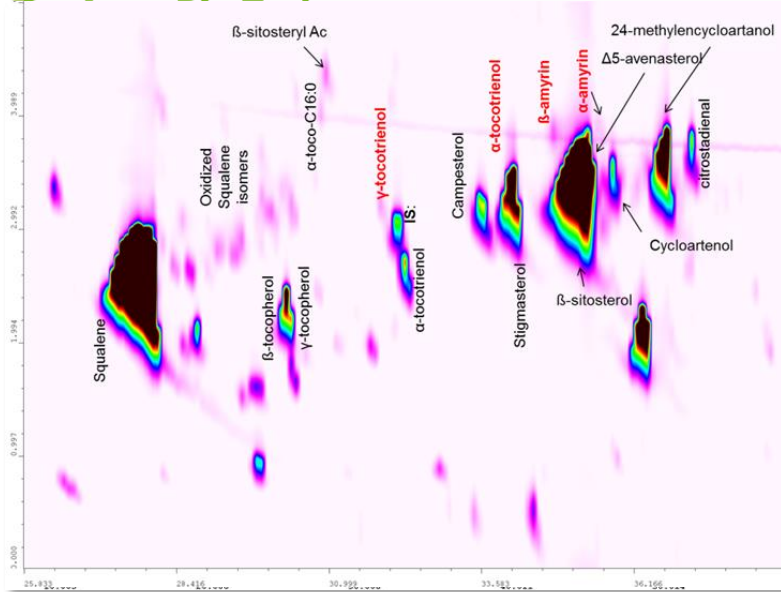
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b- Detailed investigation GC×GC-FID/MS



It was clearly highlighted the presence of the following sterols, not present in EVO

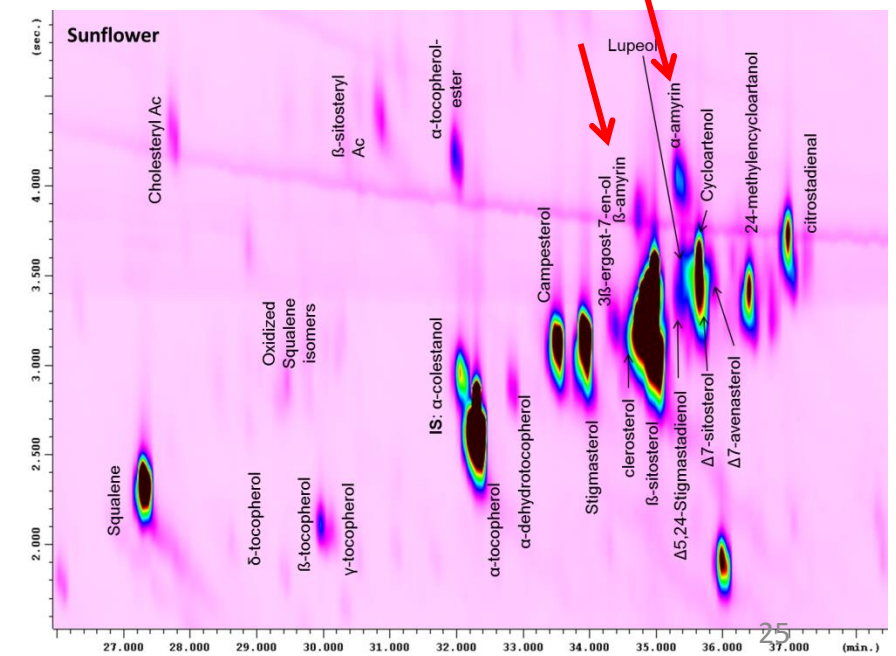
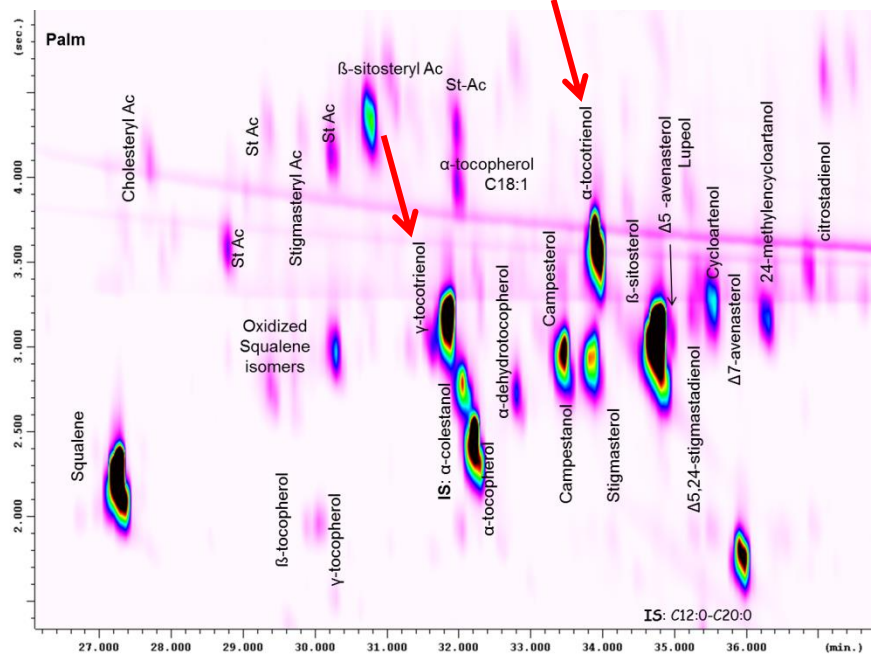
α- and γ-tocotrienol

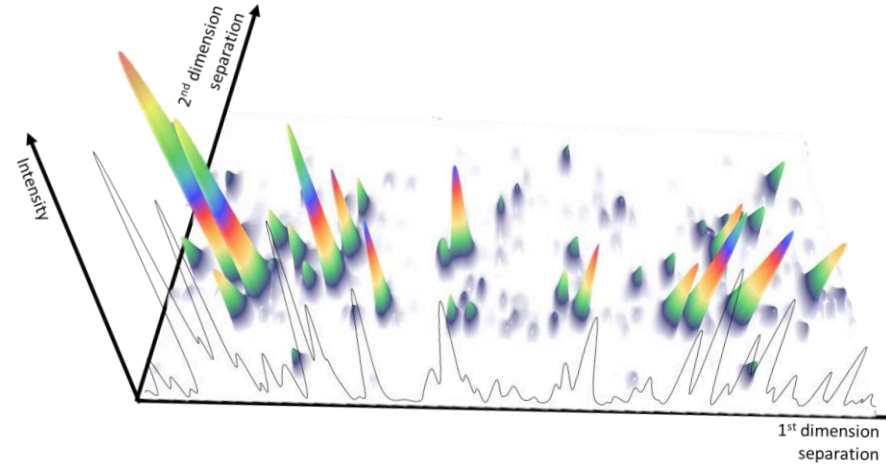
α- and β-amyirin.



Palm

Sunflower





Support and Simplify Sample Prep

Unveil and explored sample multidimensionality



SAFETY



QUALITY



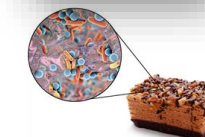
NUTRITION



AUTHENTICITY



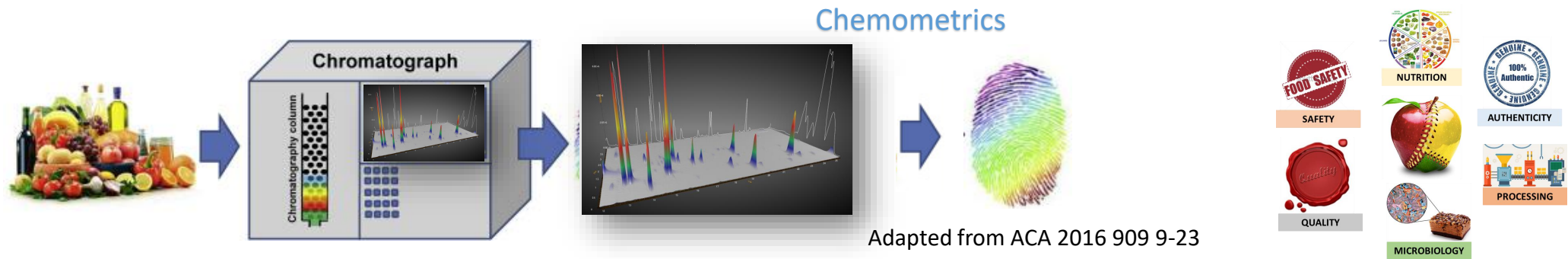
PROCESSING



MICROBIOLOGY

Chromatographic fingerprints

provide a unique situation where the data generated can be processed either following fingerprinting or profiling methodologies, providing multi-level information



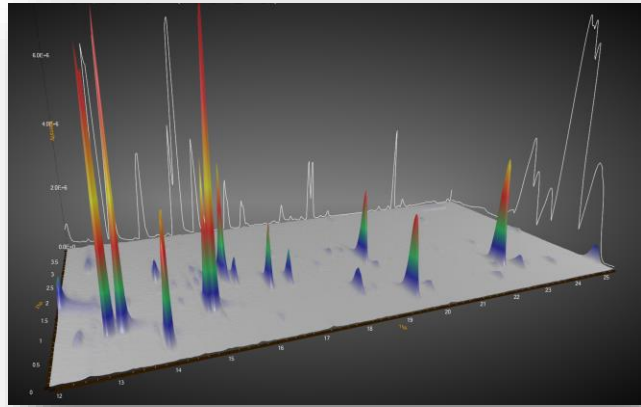
Profiling:

detailed analysis (either targeted or untargeted) to obtain quali- and quantitative information on the compounds present in a sample

Fingerprinting:

High throughput method capable of capturing relevant information but not necessarily achieving quantitative information





➤ Intensity data from the whole (or selected zone) chromatogram

FINGERPRINT
Unspecific information

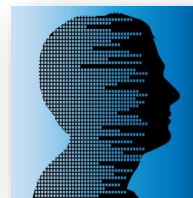


➤ Measurement of peaks areas

Component PROFILE

➤ Determination of individual components

Peak PROFILE



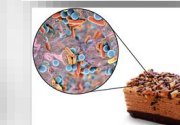
SAFETY



QUALITY



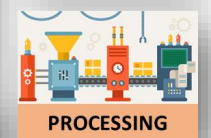
NUTRITION



MICROBIOLOGY

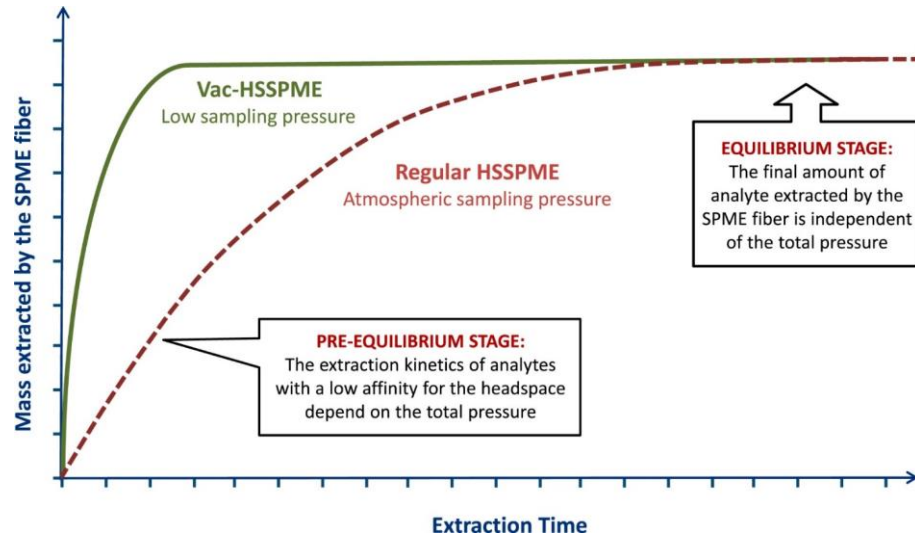
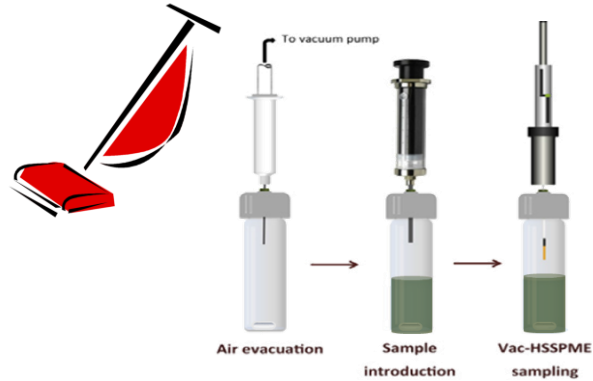
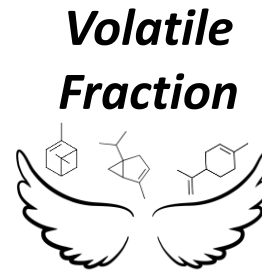


AUTHENTICITY



PROCESSING

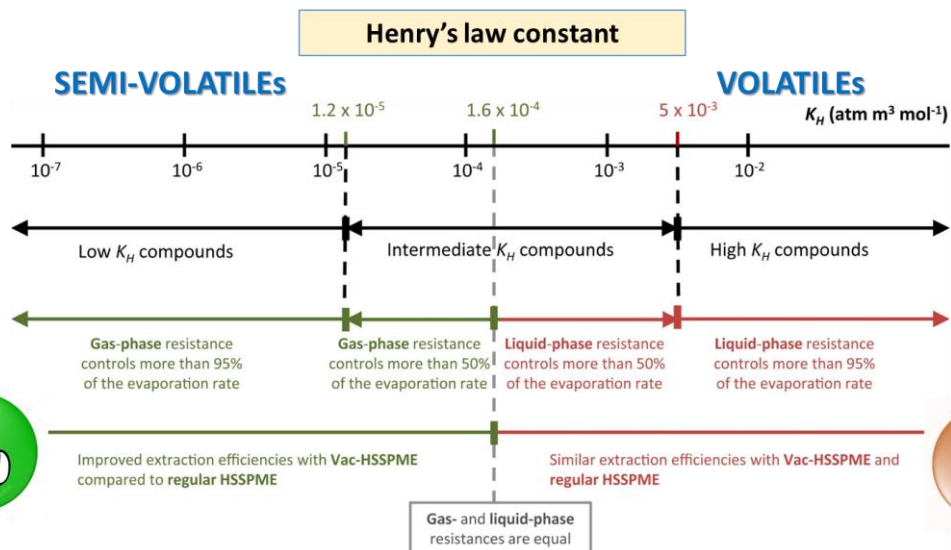
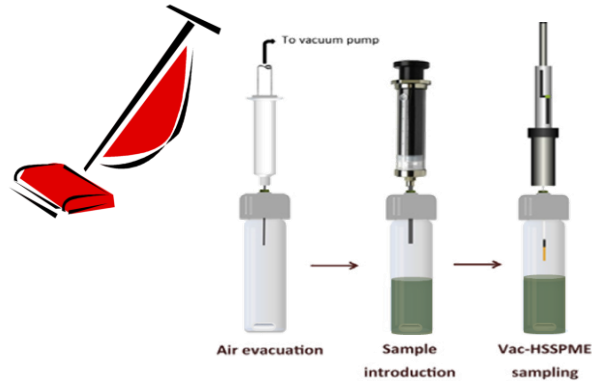
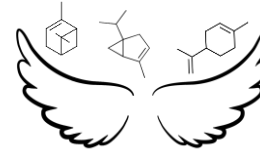
Vacuum-assisted extraction (Vac)-HS-SPME



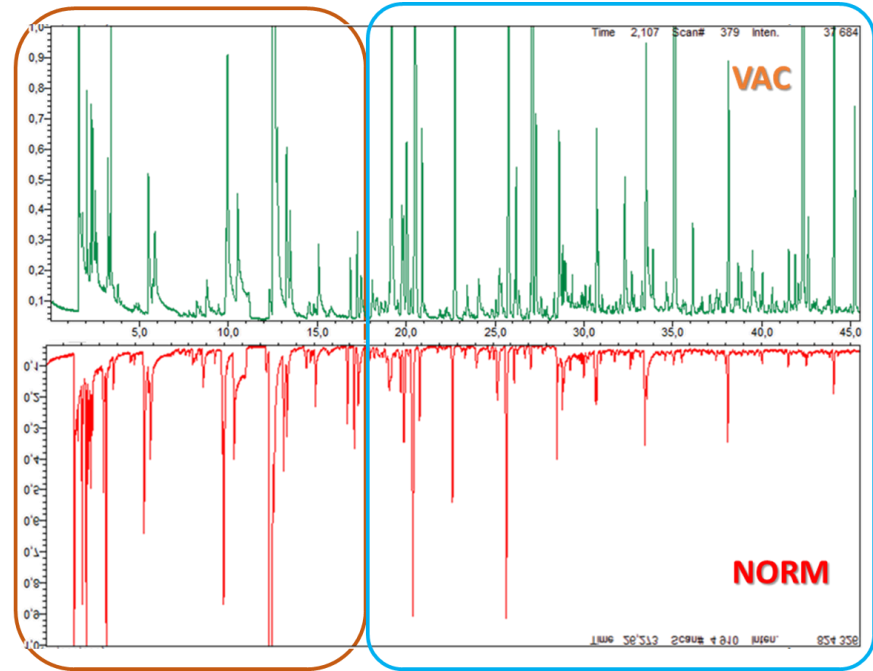
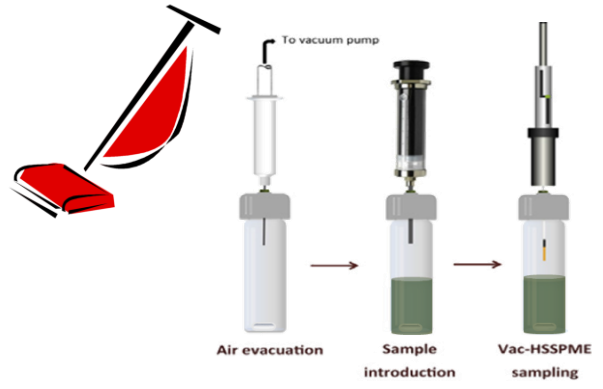
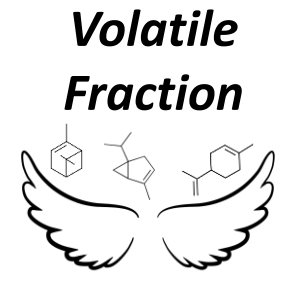
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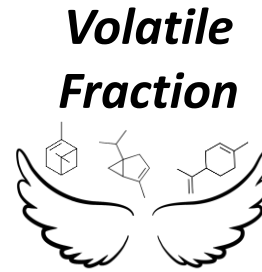
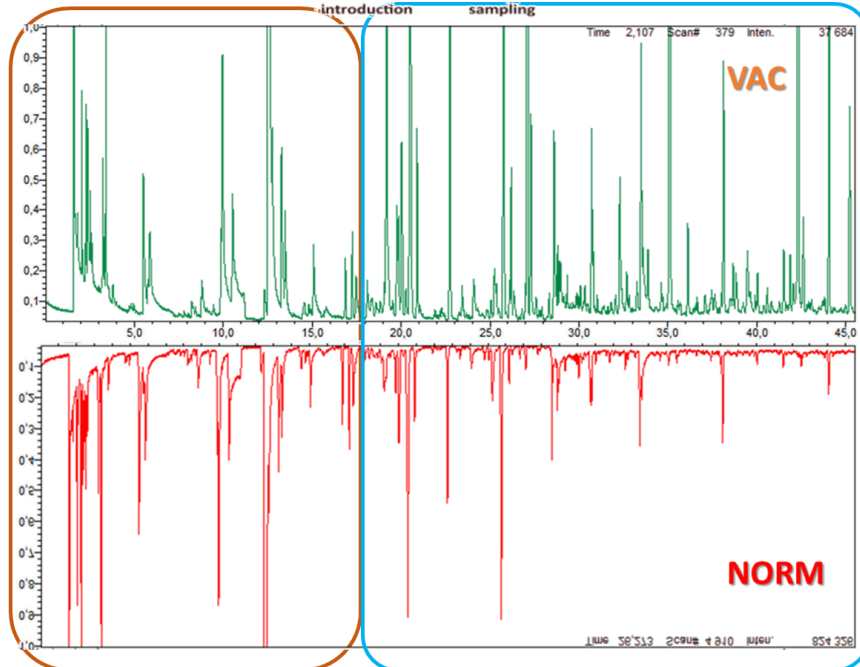
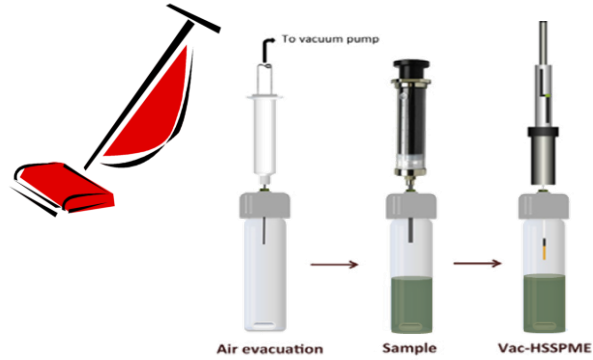
*Volatile
Fraction*



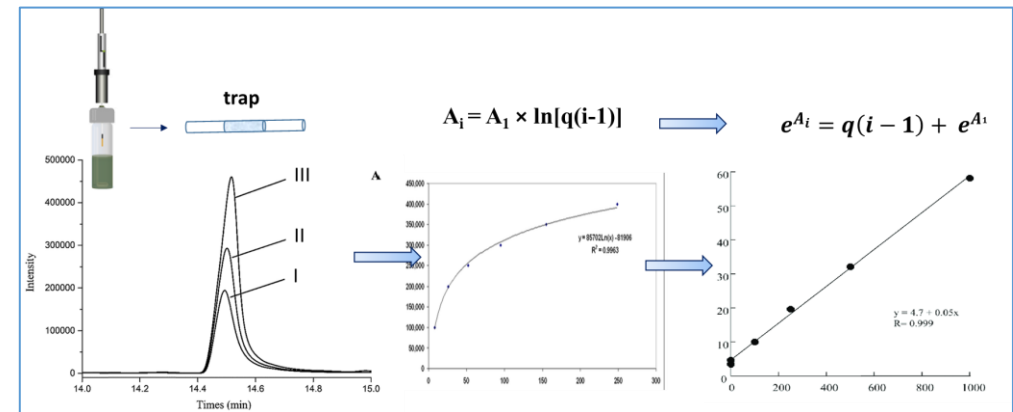
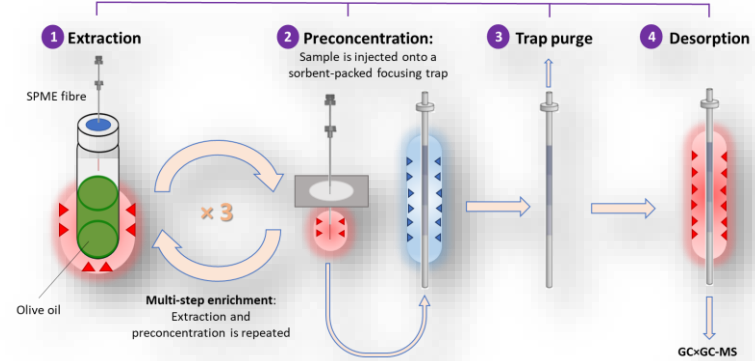
Vacuum-assisted extraction (Vac)-HS-SPME



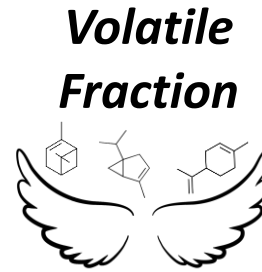
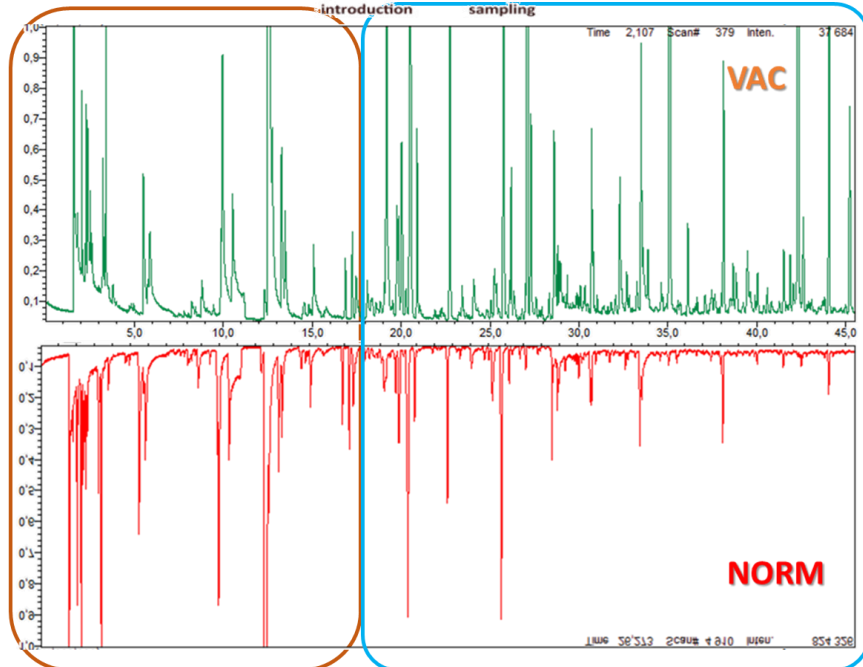
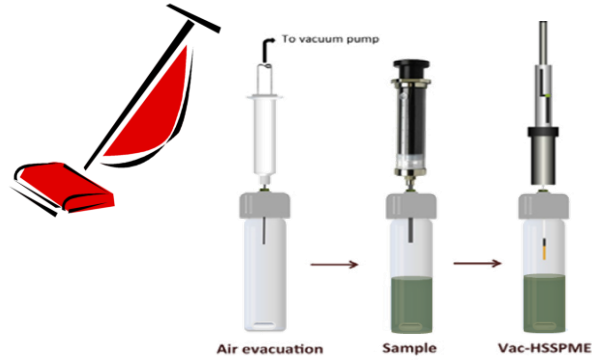
Vacuum-assisted extraction (Vac)-HS-SPME



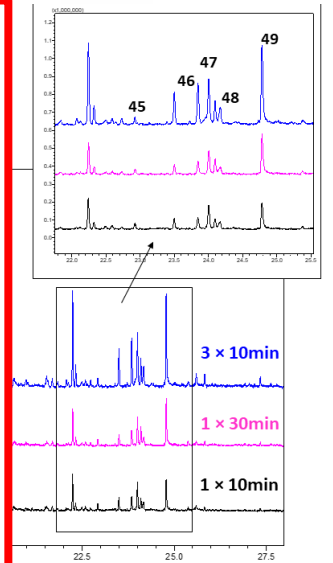
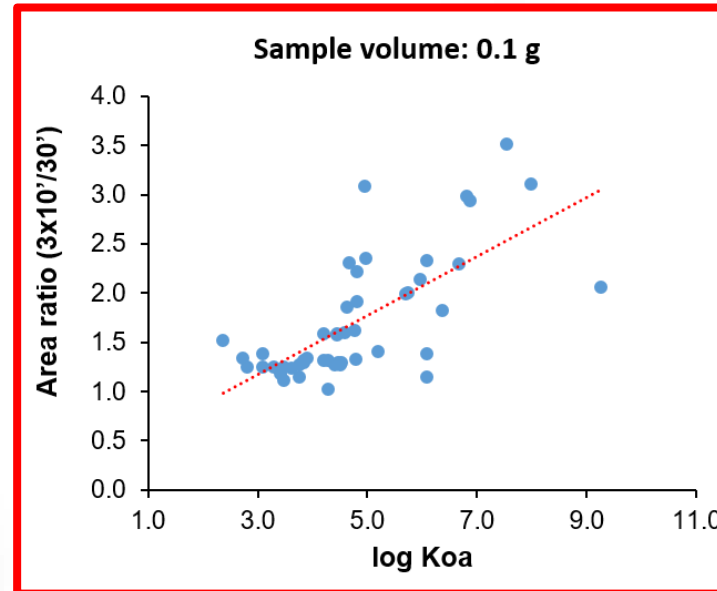
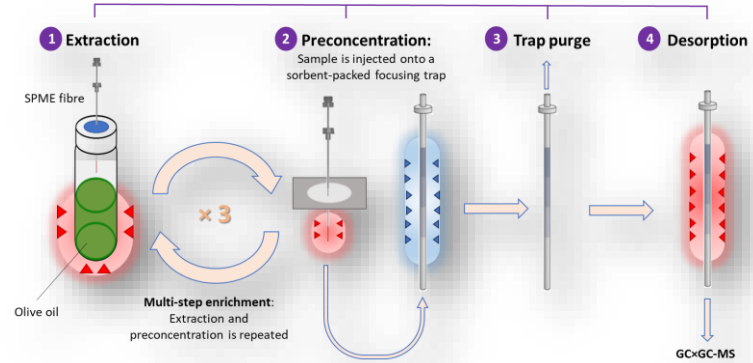
Multiple Cumulative Extraction (MCT)- HS-SPME



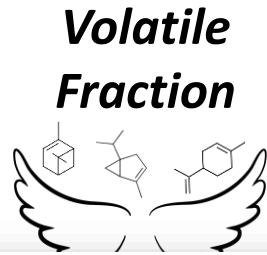
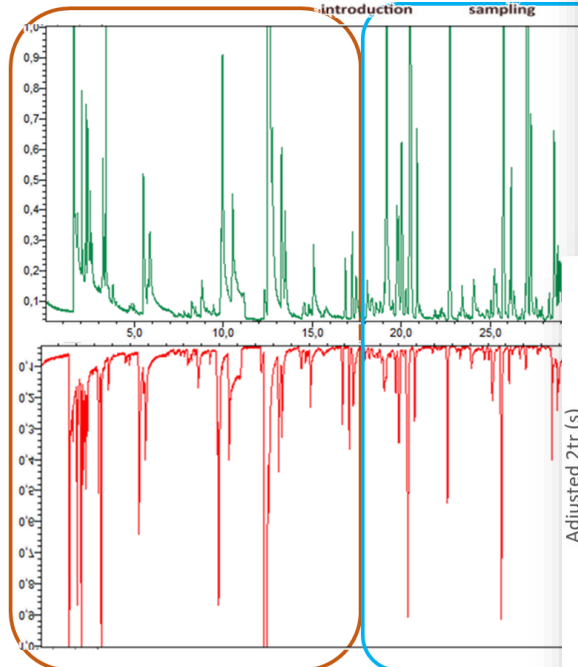
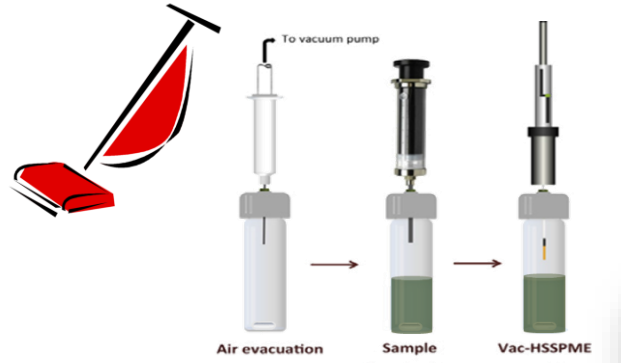
Vacuum-assisted extraction (Vac)-HS-SPME



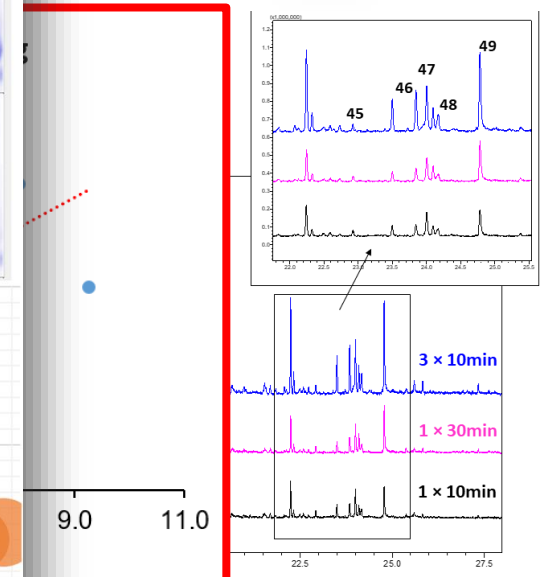
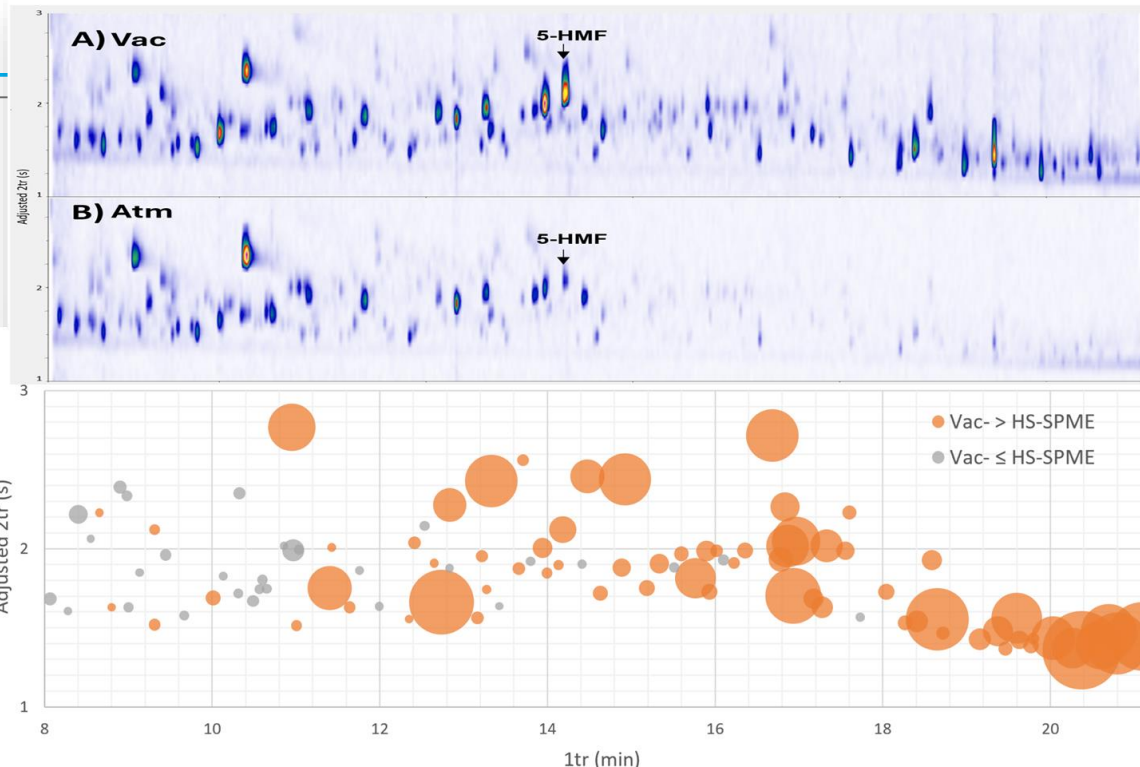
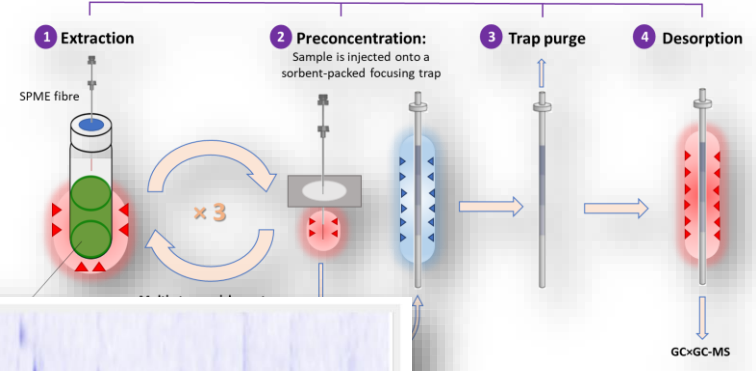
Multiple Cumulative Extraction (MCT)- HS-SPME

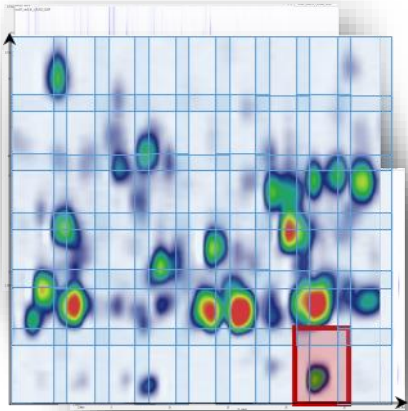


Vacuum-assisted extraction (Vac)-HS-SPME

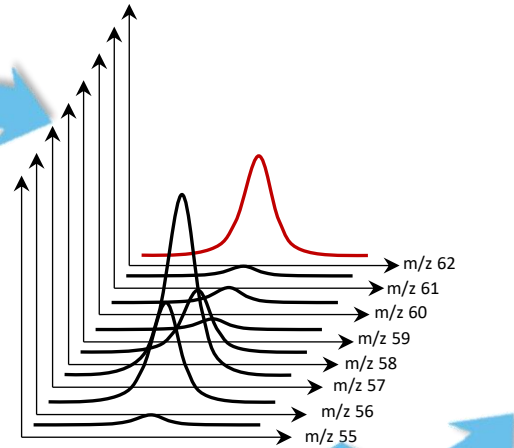


Multiple Cumulative Extraction (MCT)- HS-SPME

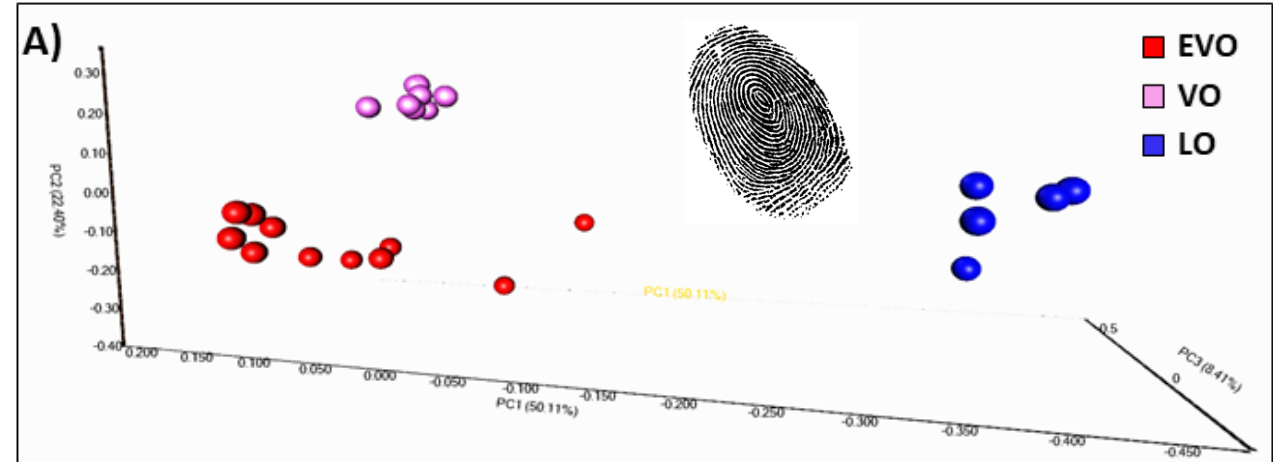




Aligned chromatograms are automatically split in to sections

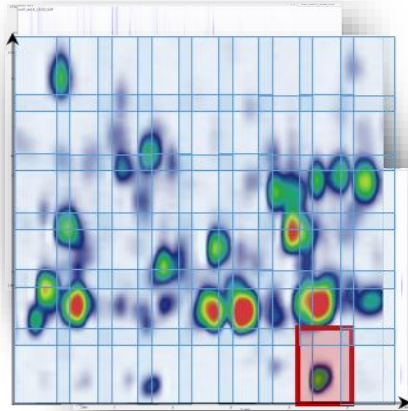


Every m/z channel for every section of every datafile is compared

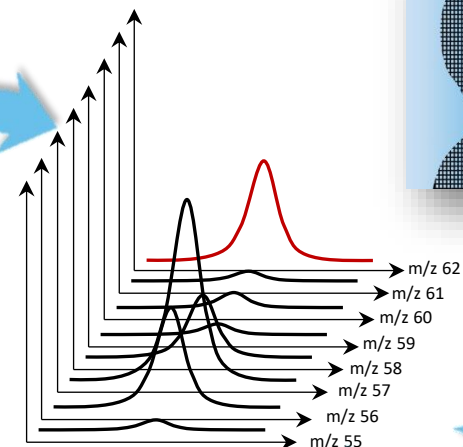


Data visualization

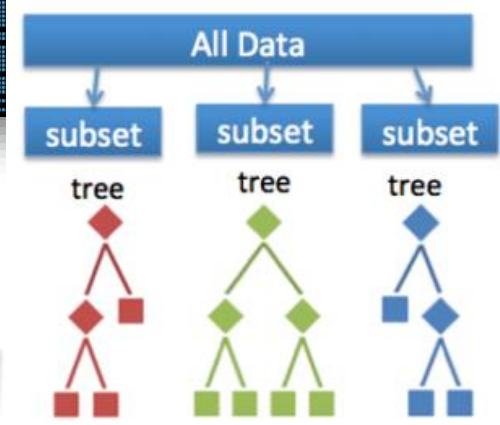
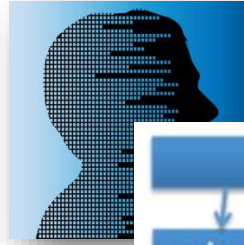
Using all of the raw data



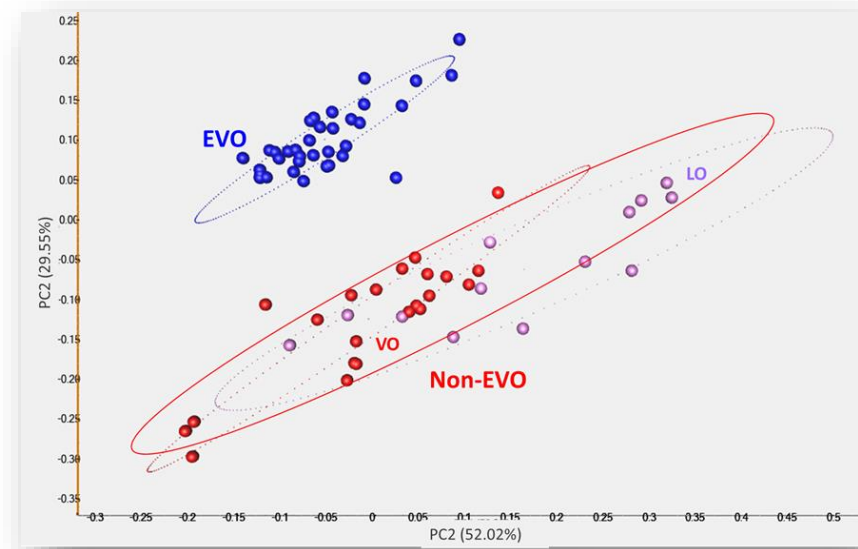
Aligned chromatograms are automatically split in to sections



Every m/z channel for every section of every datafile is compared



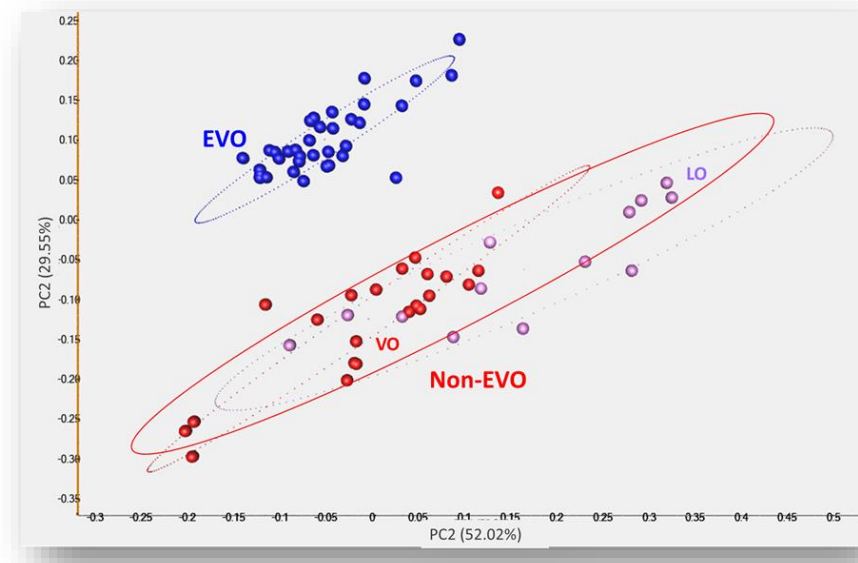
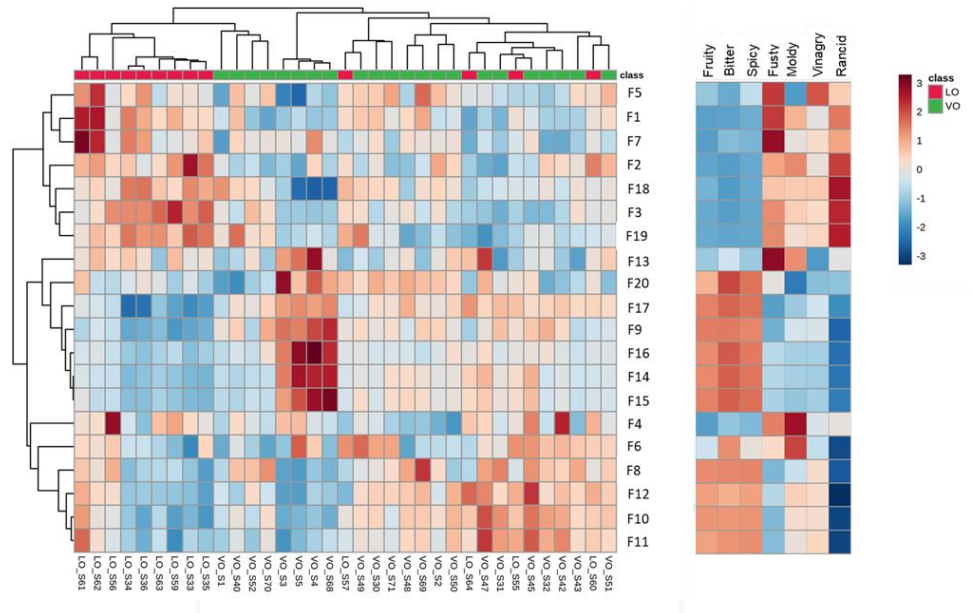
Data reduction highlights key differences between sample classes



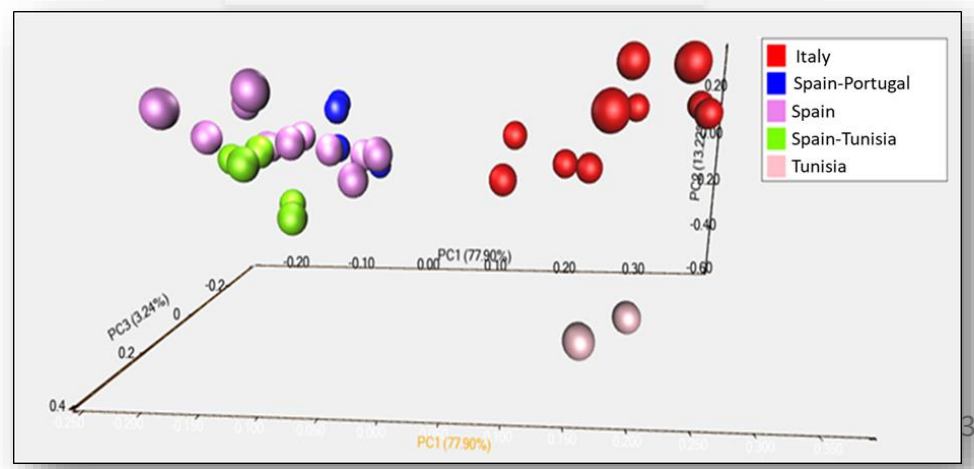
Data visualization

Using all of the raw data
The tile-approach

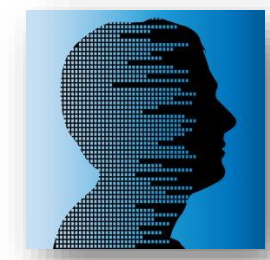
VO vs LO

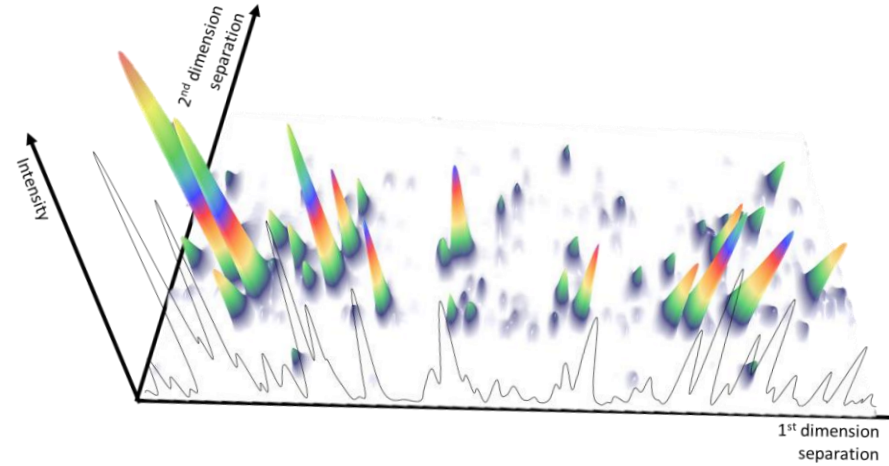


EVO geographical origin



Data visualization





Optimize and Simplify Sample Prep

Unveil and explored sample multidimensionality



SAFETY



QUALITY



NUTRITION



MICROBIOLOGY



AUTHENTICITY



PROCESSING

Convince a sceptic scientist on the benefits of using GC×GC for **Fats and oils analysis!**



1. Superior Separation:

- Two dimensions = untangled, clean separations
- Distinguishes co-eluting compounds easily

2. Enhanced Sensitivity:

- Modulation concentrates analytes
- Improved signal-to-noise = better trace detection

3. Structural Organization:

- Chemical classes form distinct clusters
- Easy fingerprinting & authenticity checks

4. Regulatory-Ready Data:

- High-resolution, defensible results for QA and compliance

5. Modern Practicality:

- Robust, automated, user-friendly systems
- Advanced software for smart data processing

Takeaway:

GC×GC doesn't just add information — it *rescues* critical insights you miss with 1D-GC.
It's not hype — it's physics

June, 12-13

3rd ADVANCES IN SEPARATION SCIENCE WORKSHOP

Gembloux, Belgium



J. Pawliszyn



C. Cordero



G. Hopfgartner



H. Mol



B. Bojko



E. Leitner



P. Tranchida



E. Rosenberg



K. Schug



H.-G. Janssen



E. Gionfriddo

June, 11: 1-day course in

- SPME
- GC



My research group:

Sophie Vancaenenbroeck

Paula Albendea

Damien Eggermont

Aleksandra Gorska

Donatella Ferrara

Damien Pierret

Carlo Bellinghieri



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