GC-BASED HYPHENATED TECHNIQUES IN FOOD ANALYSIS

Giorgia Purcaro

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Evolution of FOOD ANALYSIS

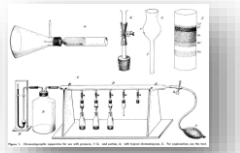
"reductionist approach"

"interactionism" and -omics sciences"

1800s **Wet chemistry era**
main components

1900s **Instrumental era**
minor and trace components

1906



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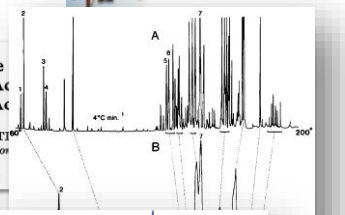

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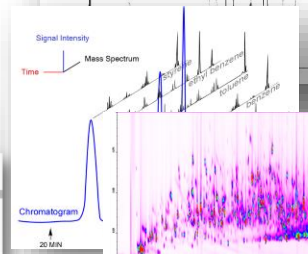

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Journal of Chromatographic Science, Vol. 18, October 1980
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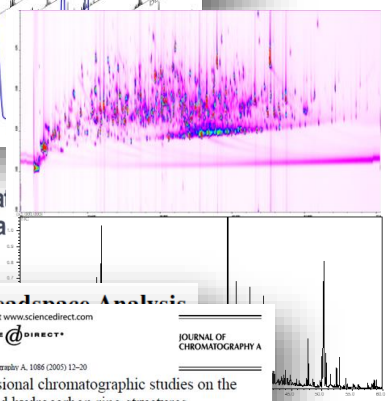

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Comprehensive LC × CC for Enhanced Headspace Analysis
Wes W. C. Quigl
Department of Chemistry,
University o

2005 **LC-GC×GC**

Available online at www.sciencedirect.com
SCIENCE DIRECT
JOURNAL OF CHROMATOGRAPHY A
ELSEVIER
Journal of Chromatography A, 1086 (2005) 12–20
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[a] University of Amsterdam, Van 't Hoff Institute for Molecular Sciences, Amsterdam, The Netherlands
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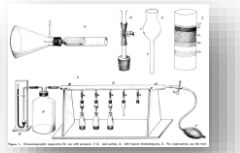

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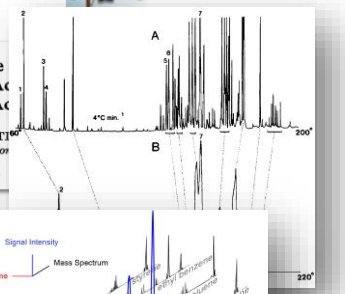

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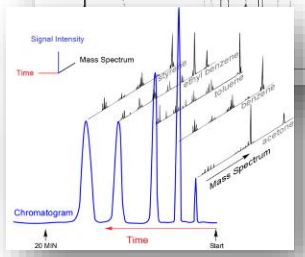

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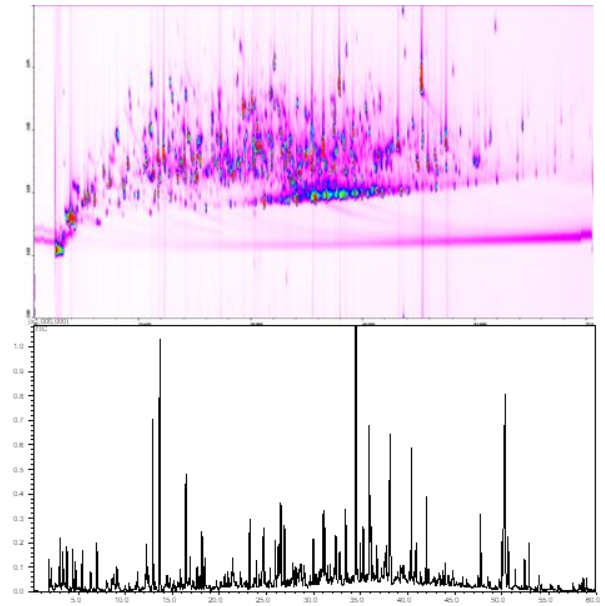

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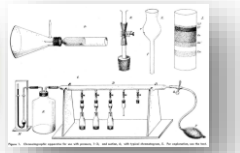

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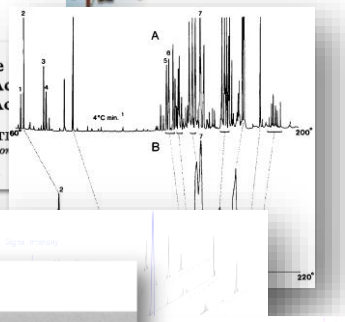

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Journal of Chromatography, 295 (1984) 55–61
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 16,706

1984

COUPLING OF HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH CAPILLARY GAS CHROMATOGRAPHY

K. GROB, Jr.*, D. FRÖHLICH, B. SCHILLING, H. P. NEUKOM and P. NÄGELI
Kantonales Labor, P.O. Box, CH-8030 Zürich (Switzerland)
(Received March 6th, 1984)

Mineral oil

LC-GC

Received: 19 August 2020 | Revised: 4 October 2020 | Accepted: 26 October 2020
DOI: 10.1002/jssc.202000901
JOURNAL OF SEPARATION SCIENCE

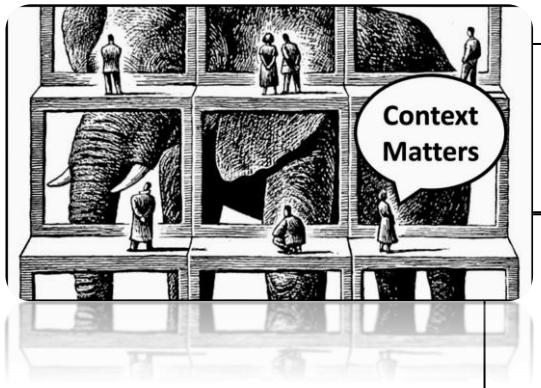
REVIEW ARTICLE

Evolution of hyphenated techniques for mineral oil analysis in food

Nicola Sdrigotti[1,2] | Maurine Collard[1] | Giorgia Purcaro[1]

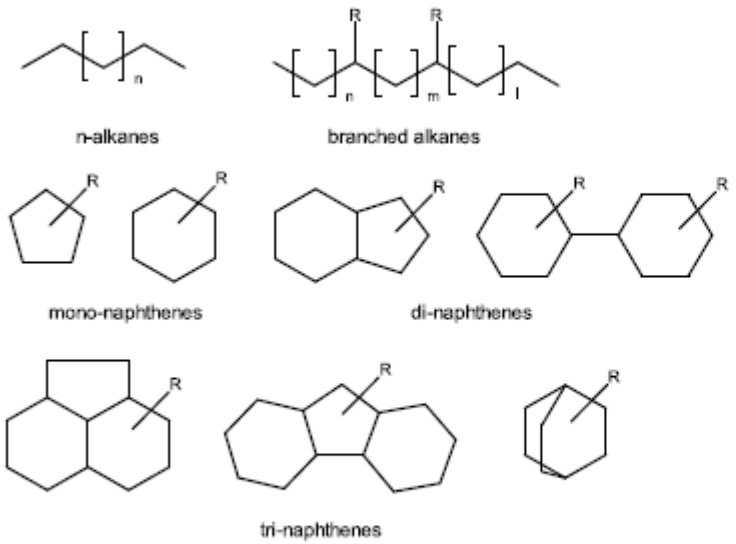
MINERAL OIL HYDROCARBONS (MOH): DEFINITION*

a wide range of products deriving from petroleum distillation fractions



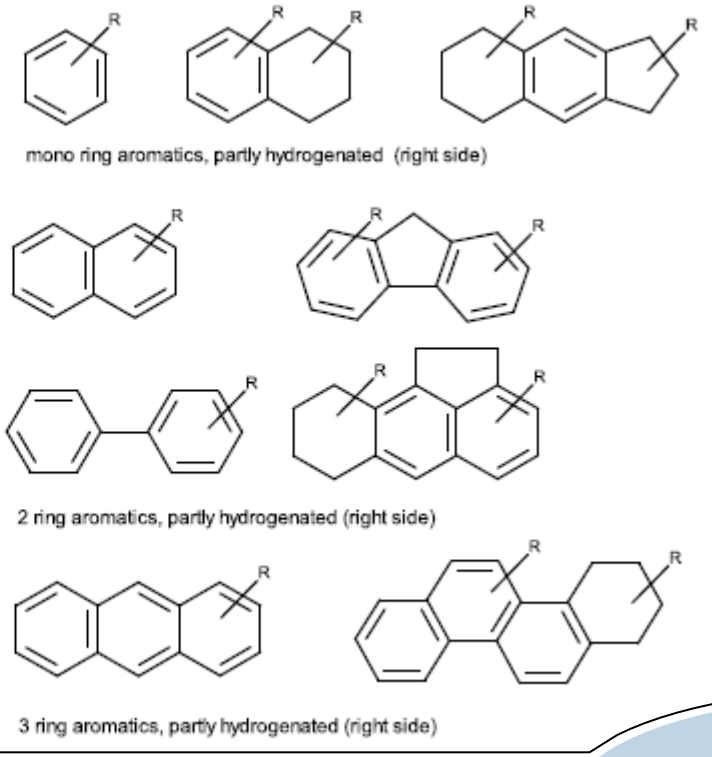
MOSH

Mineral oil saturated hydrocarbons



MOAH

Mineral oil aromatic hydrocarbons



-n-alkane
- isoalkane
- cycloalkane

Aromatic hydrocarbons, mainly alkylated

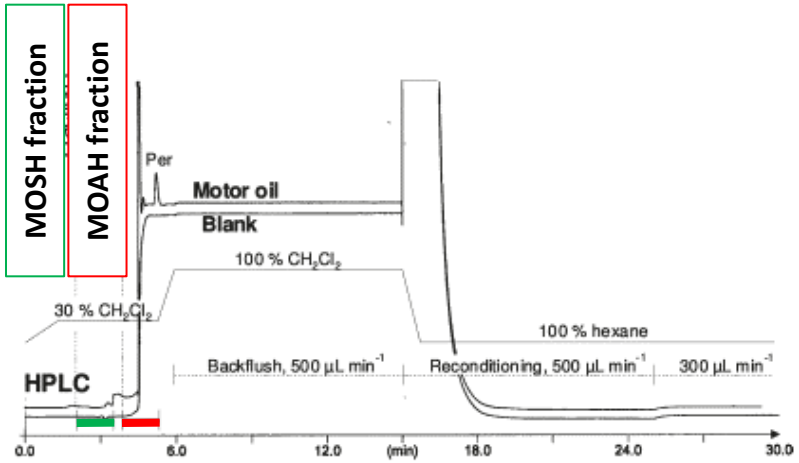
LC-GC

LC-GC-FID

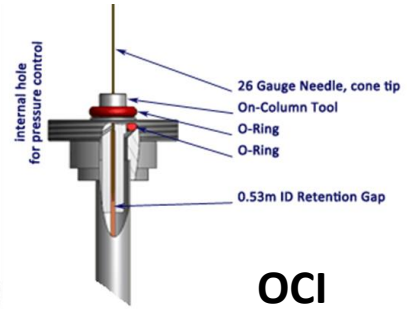
LC

Sample preparation step

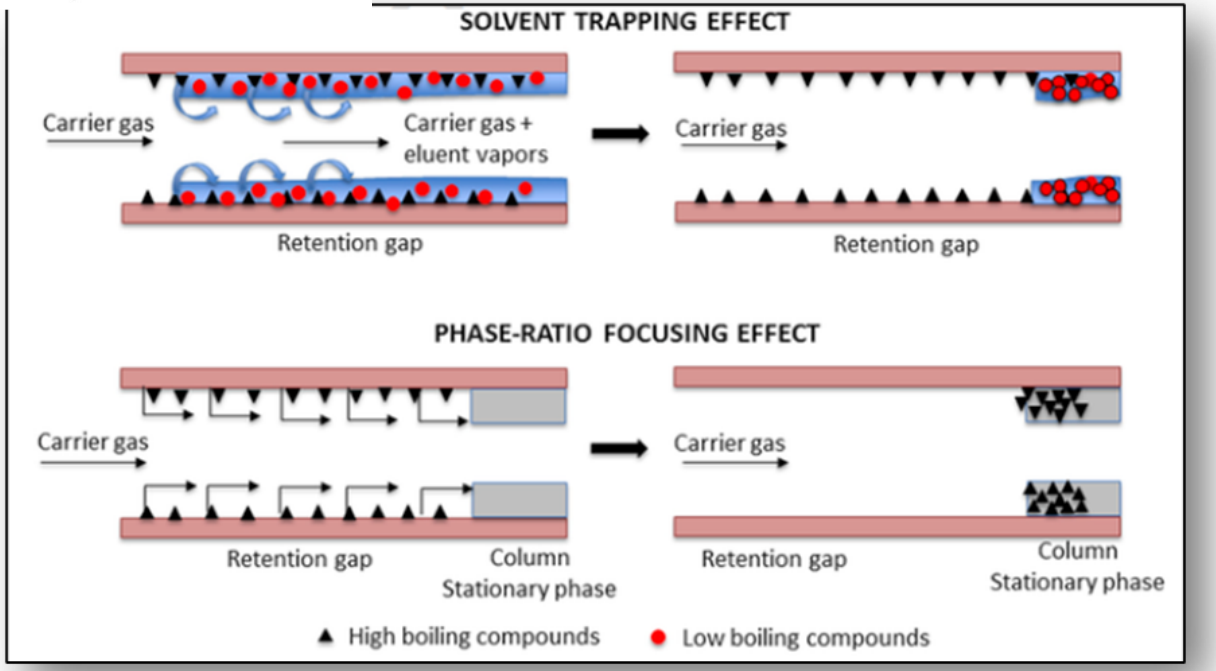
- ✓ Purification form TAGs
- ✓ Pre-fractionation



Biedermann et al, J Agric Food Chem, 2009, 57, 8711-8721



OCI



C10-C50

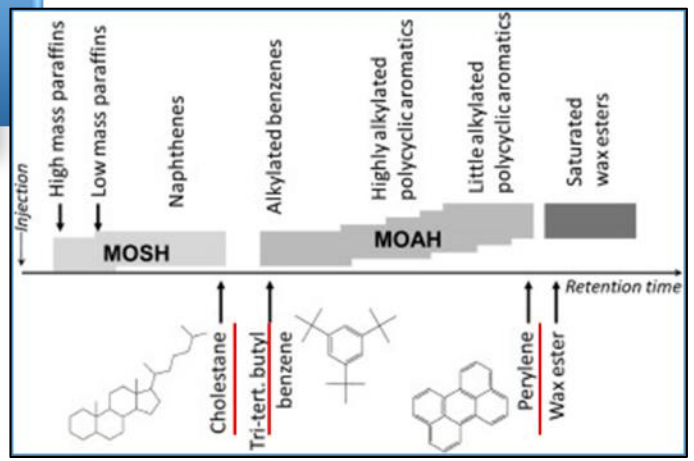
ROUTINE METHOD FOR MOH DETERMINATION

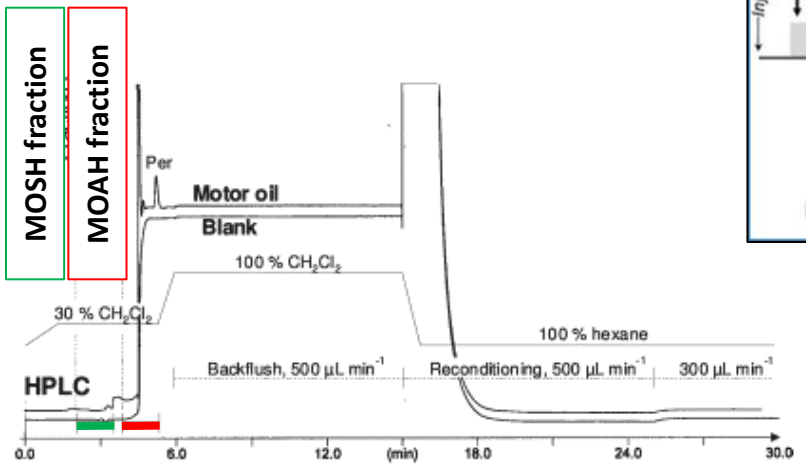
LC-GC-FID

LC

Sample preparation step

- ✓ Purification form TAGs
- ✓ Pre-fractionation





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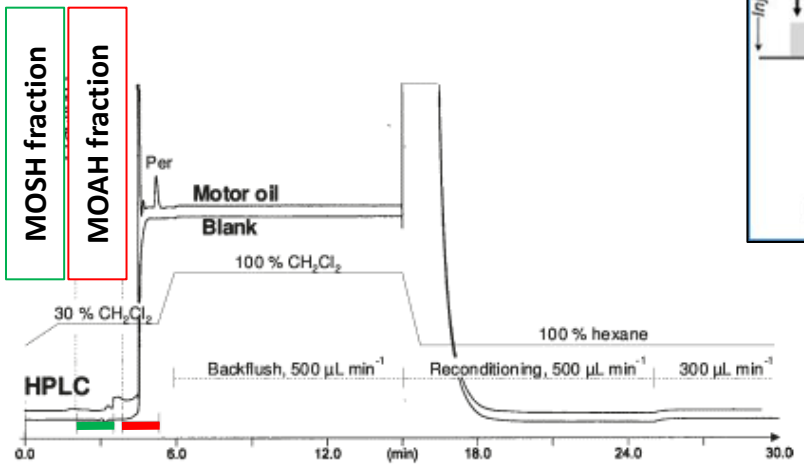
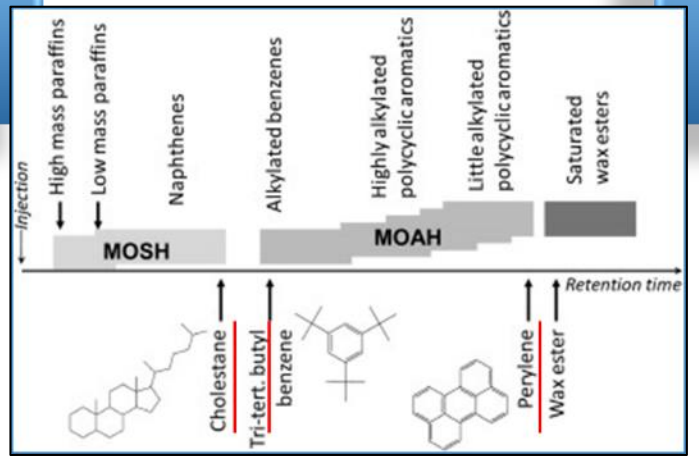
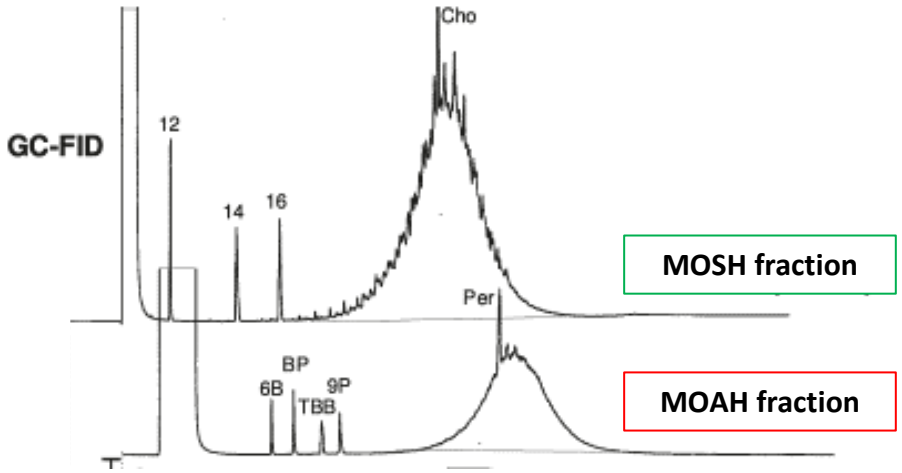
Analytical Determination

✓ Analytical Separation

✓ Quantification







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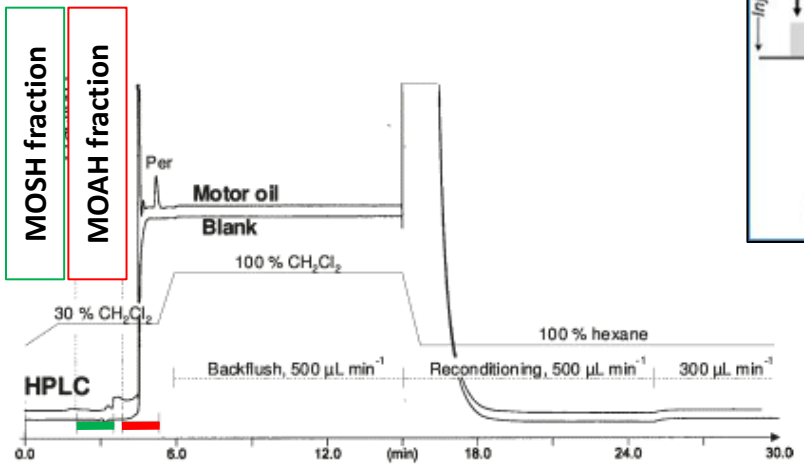

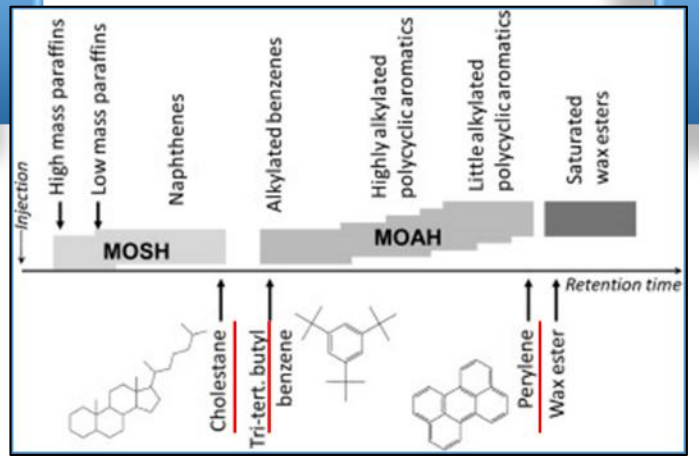

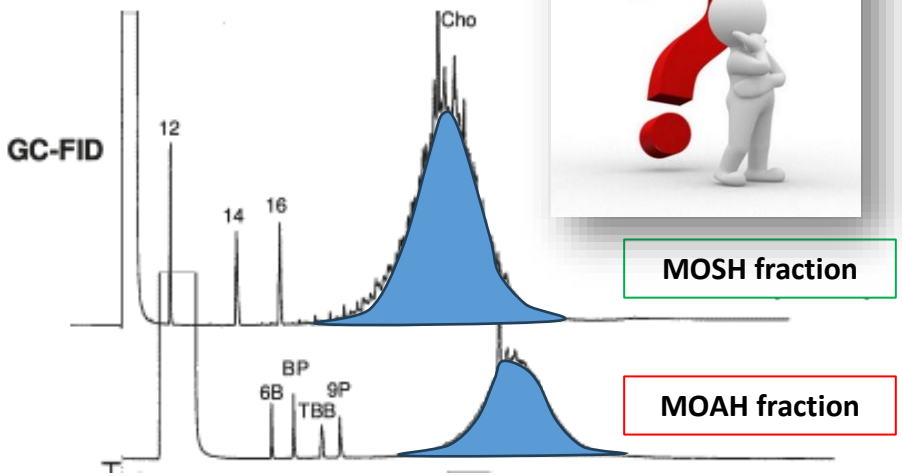

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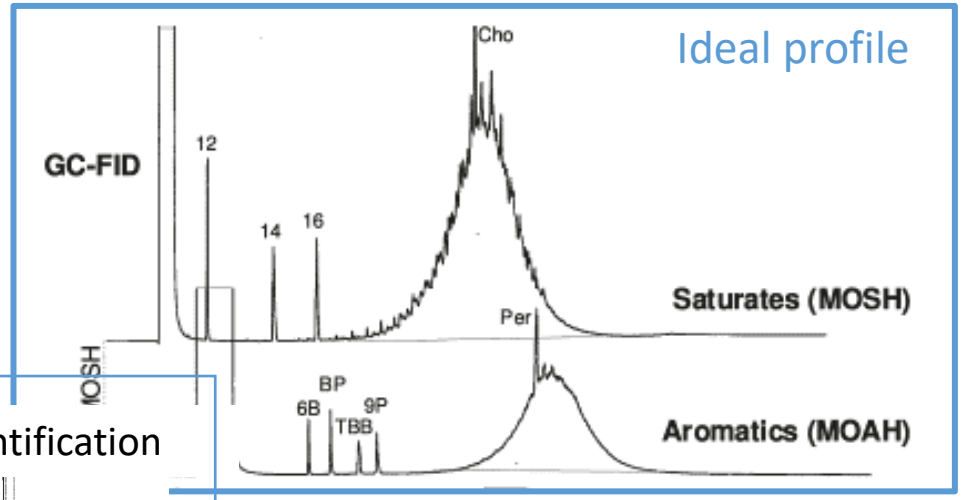
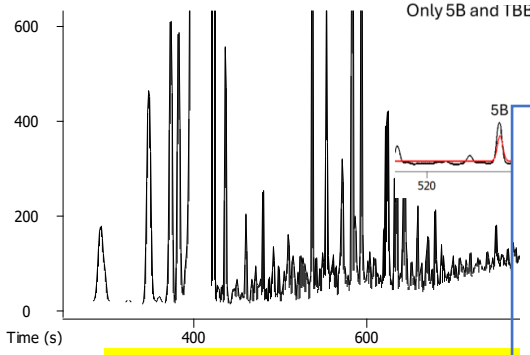
CHALLENGES in MOSH/MOAH analysis

Ideal profile



MOAH analysis by GC-FID

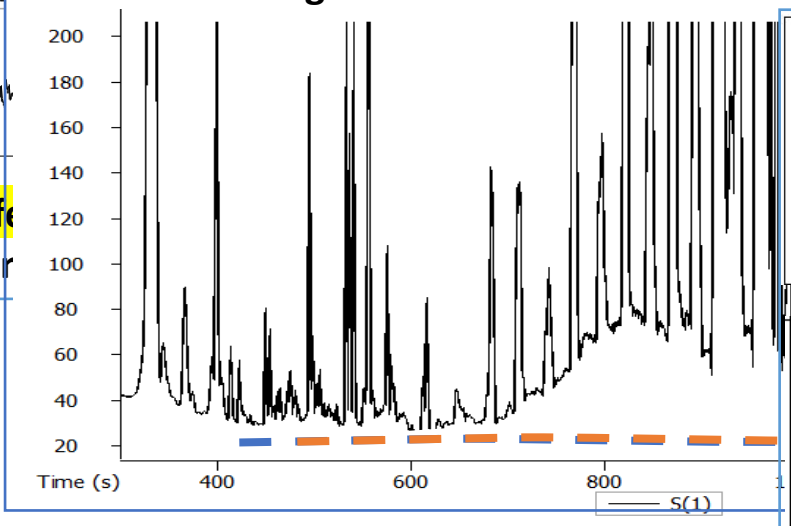
IS for quantification

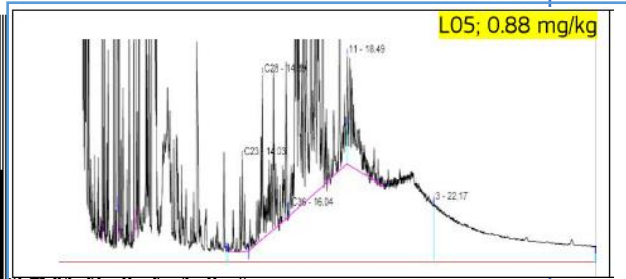


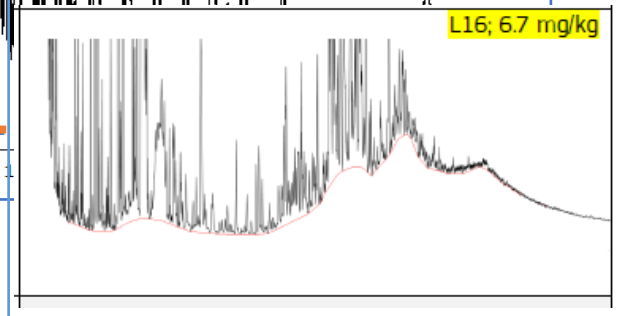
ISs coeluted with interfe

→ impossible MOAH quar

Baseline drawing

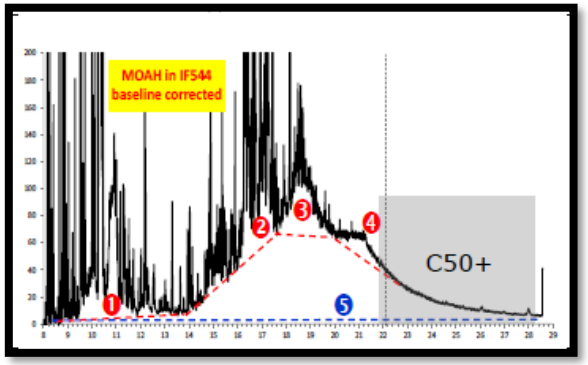






Coeluted interferences to be removed

→ High uncertainty of the result



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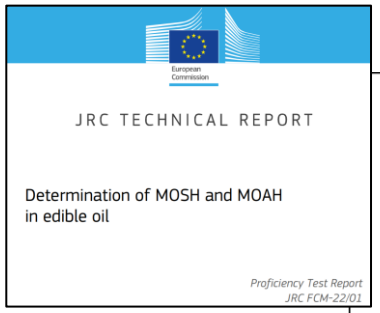
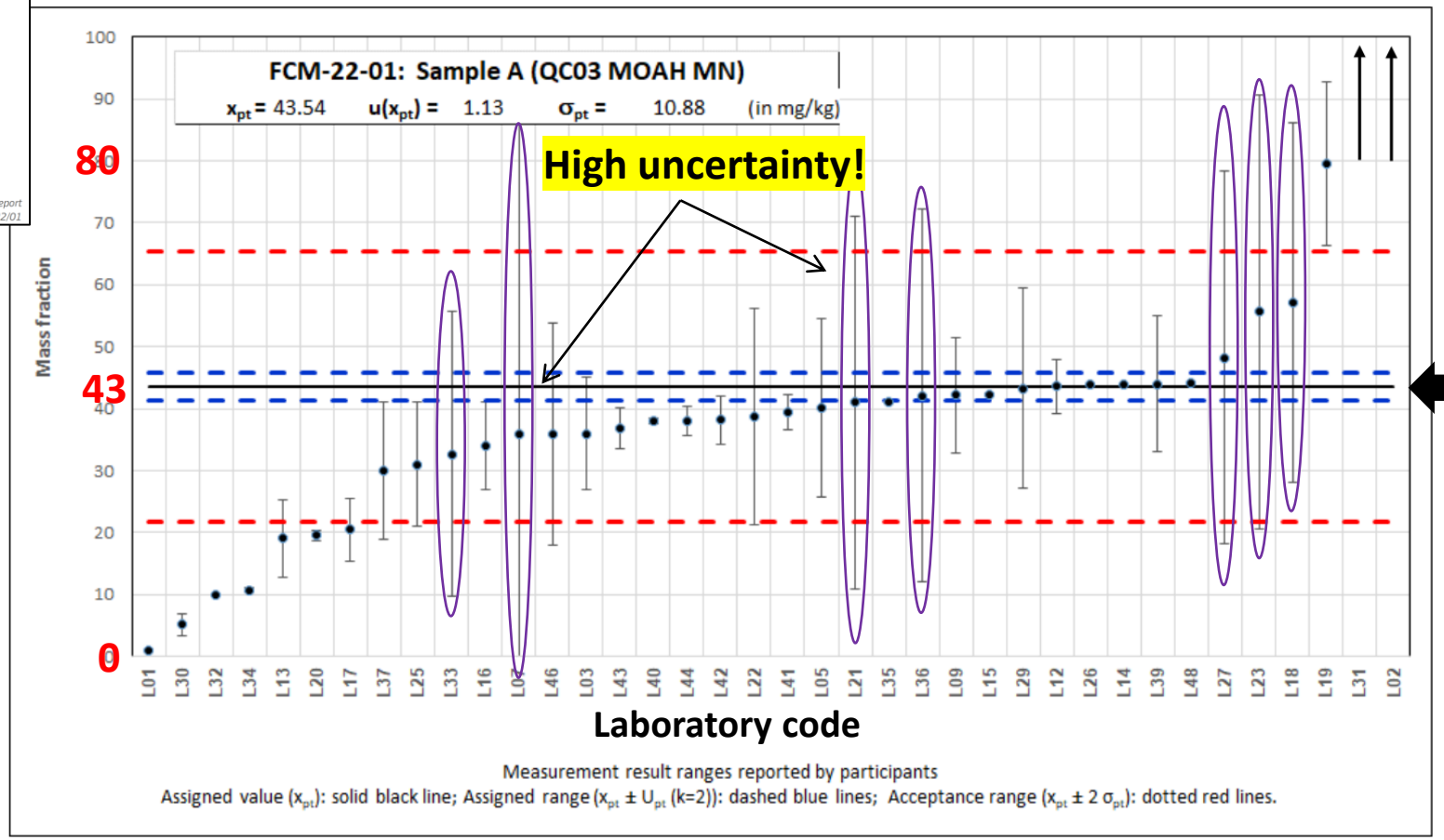


JRC proficiency test 2023 on olive oil



Expected value

Bratinova, S., Robouch, P., Cordeiro Raposo, F., Beldi, G., Senaldi, C., Karasek, L. and Hoekstra, E., Determination of MOSH and MOAH in edible oil, EUR 31478 EN, Publications Office of the European Union, Luxembourg, 2023, ISBN 978-92-68-02137-8, doi:10.2760/208184, JRC133284.

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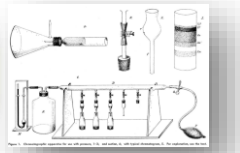

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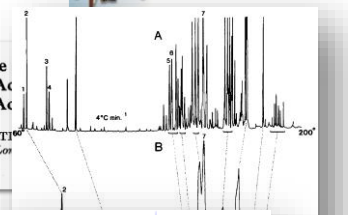

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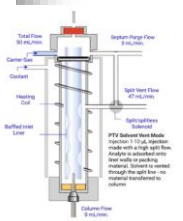

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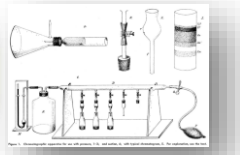

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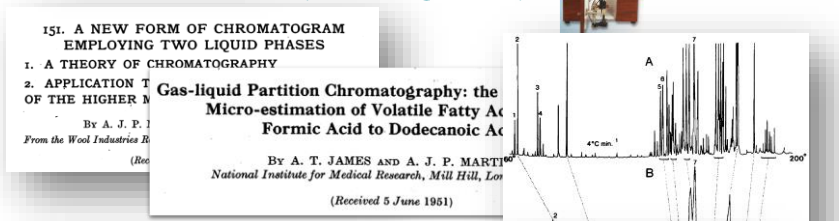

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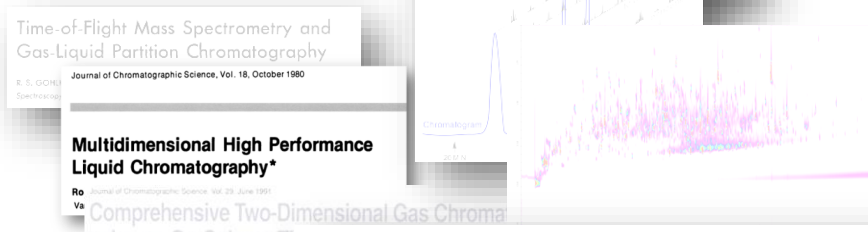

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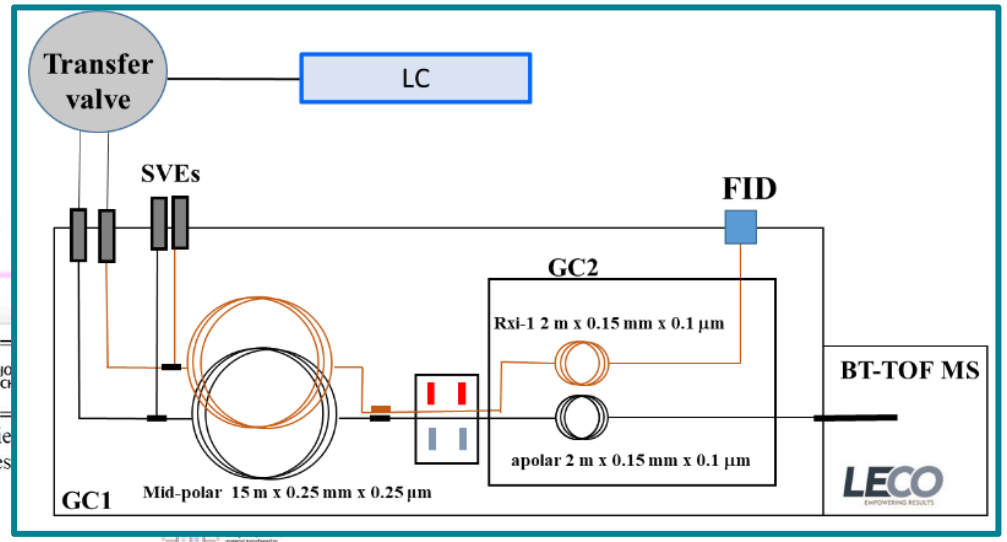
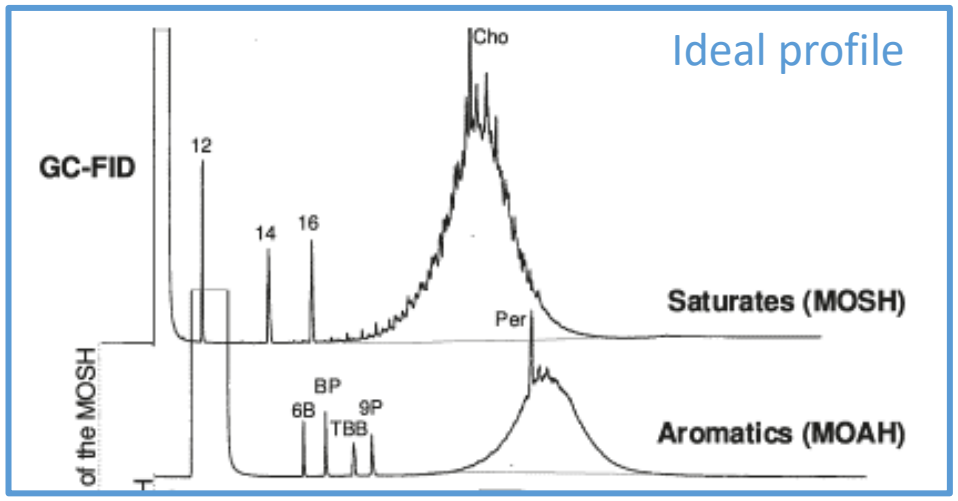
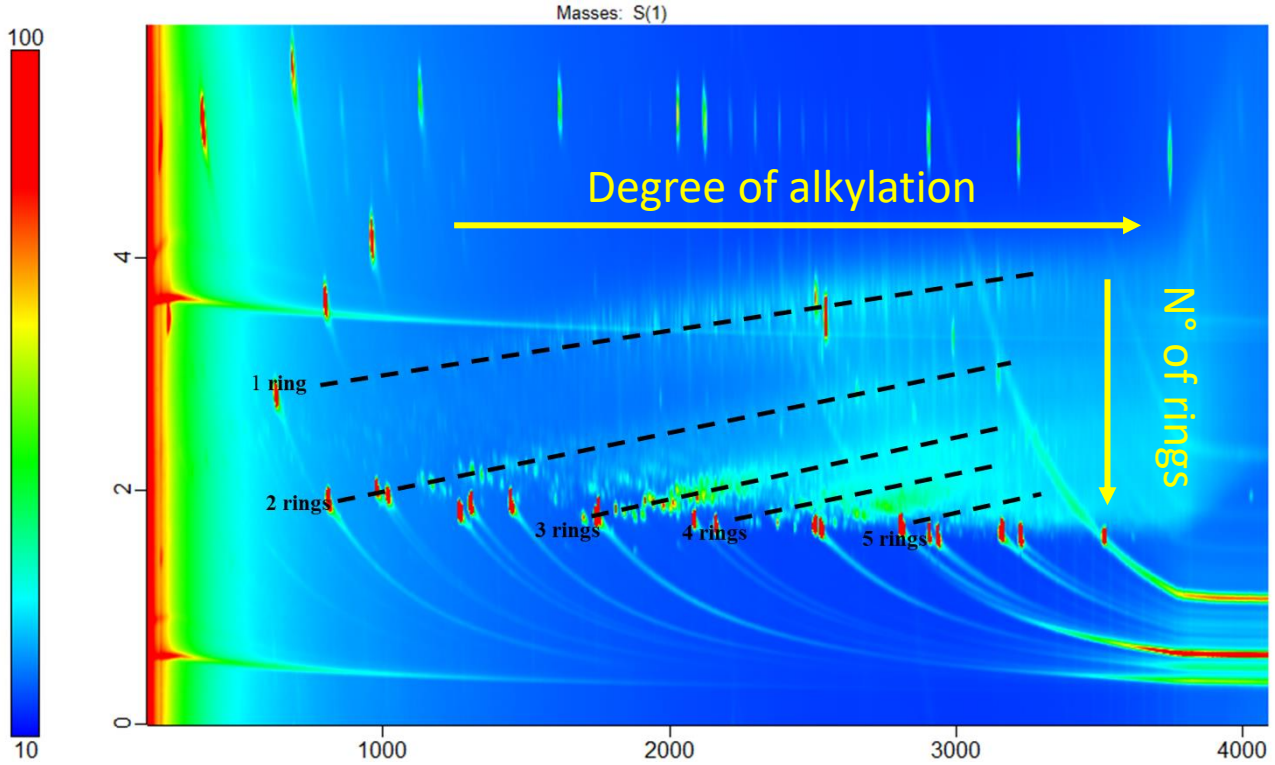
2021

LC-2GC×GC-FID/MS

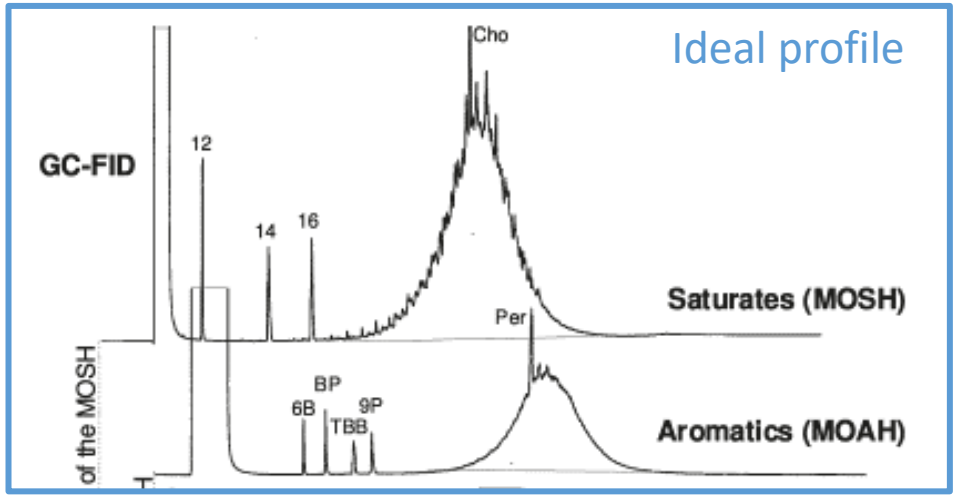


GC×GC benefits for MOSH and MOAH analysis





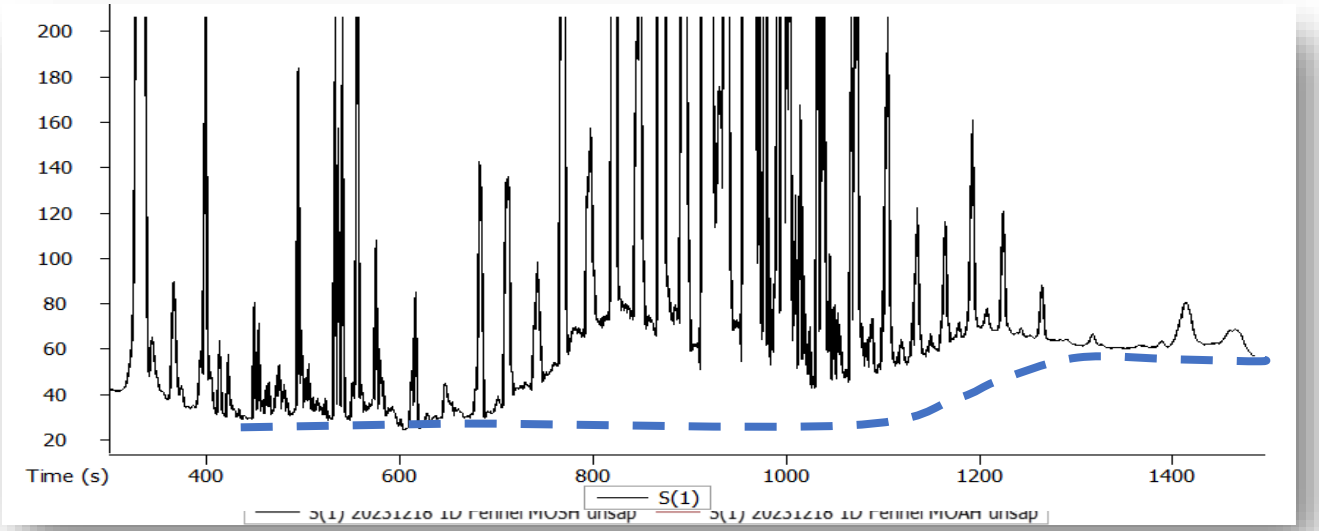
GC×GC benefits for MOSH and MOAH analysis

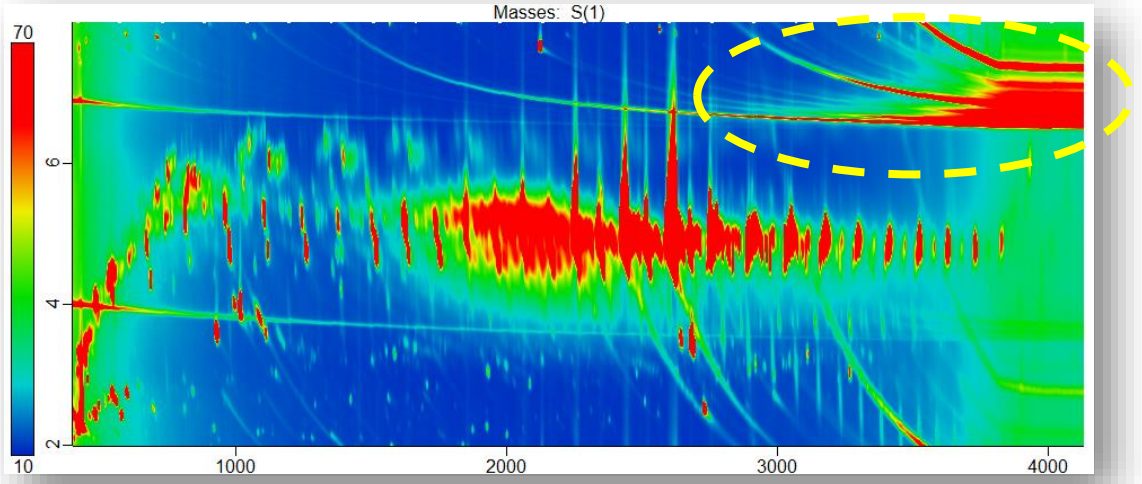


- **Baseline**
- **Riding peaks subtraction**

More accurate quantification

MOSH vs MOAH – No purification





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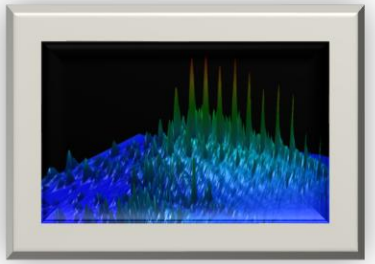
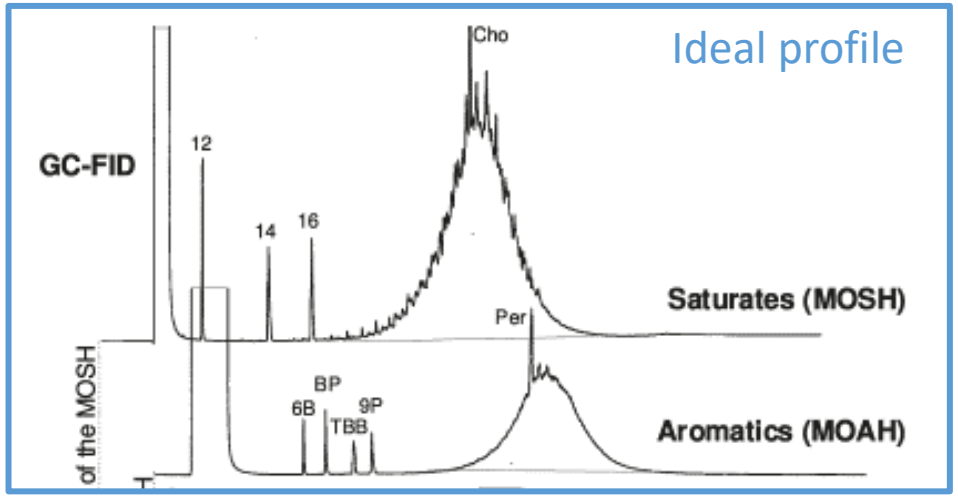
GC×GC benefits for MOSH and MOAH analysis

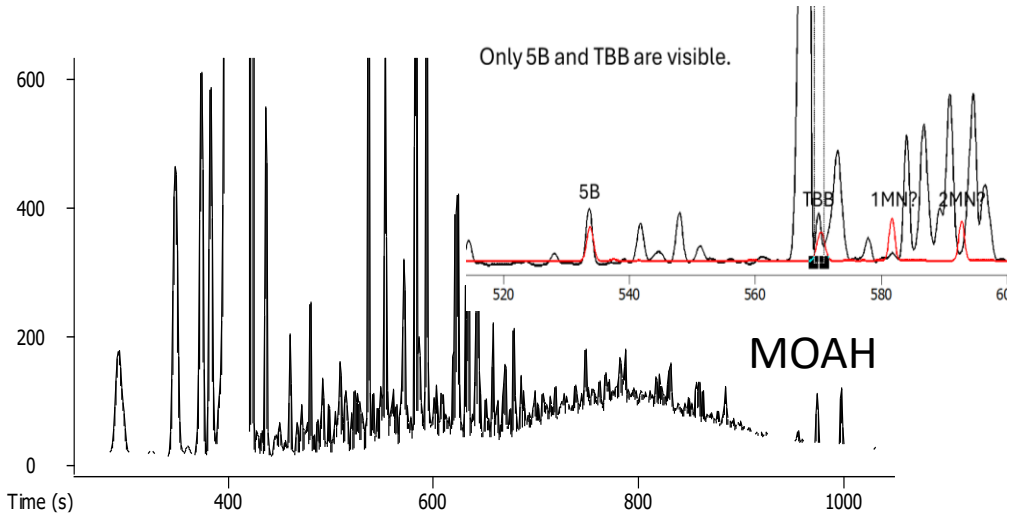




More accurate quantification

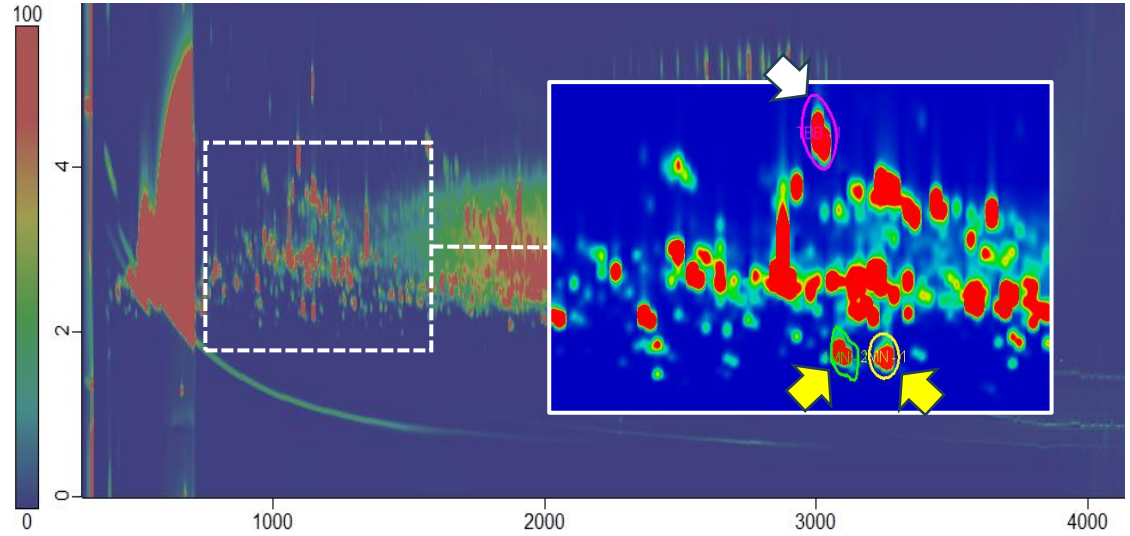
MOAH analysis by GC-FID

IS for quantification



Internal standards coeluted with interferences
→ impossible MOAH quantification

MOAH analysis by GC×GC-FID



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

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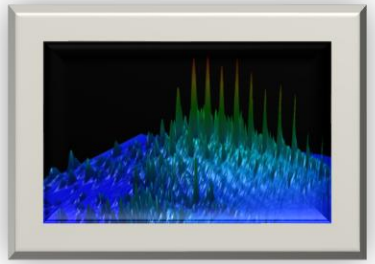
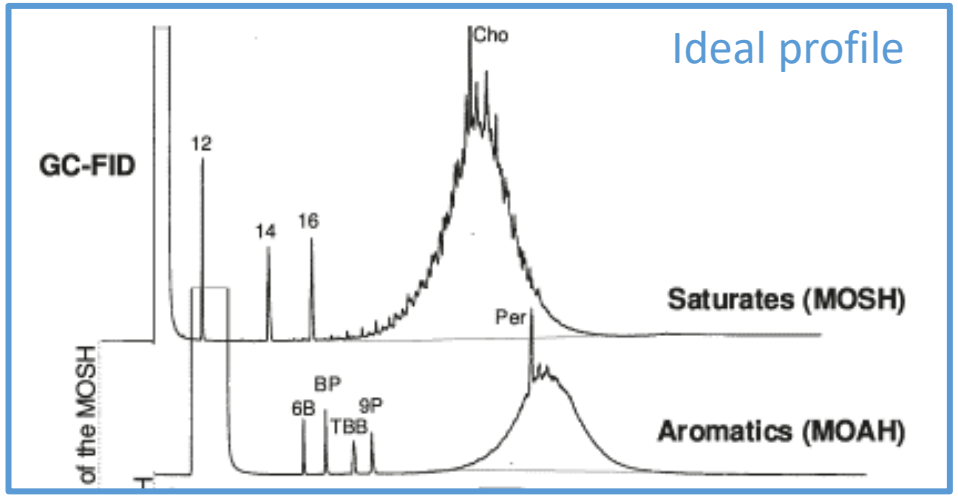
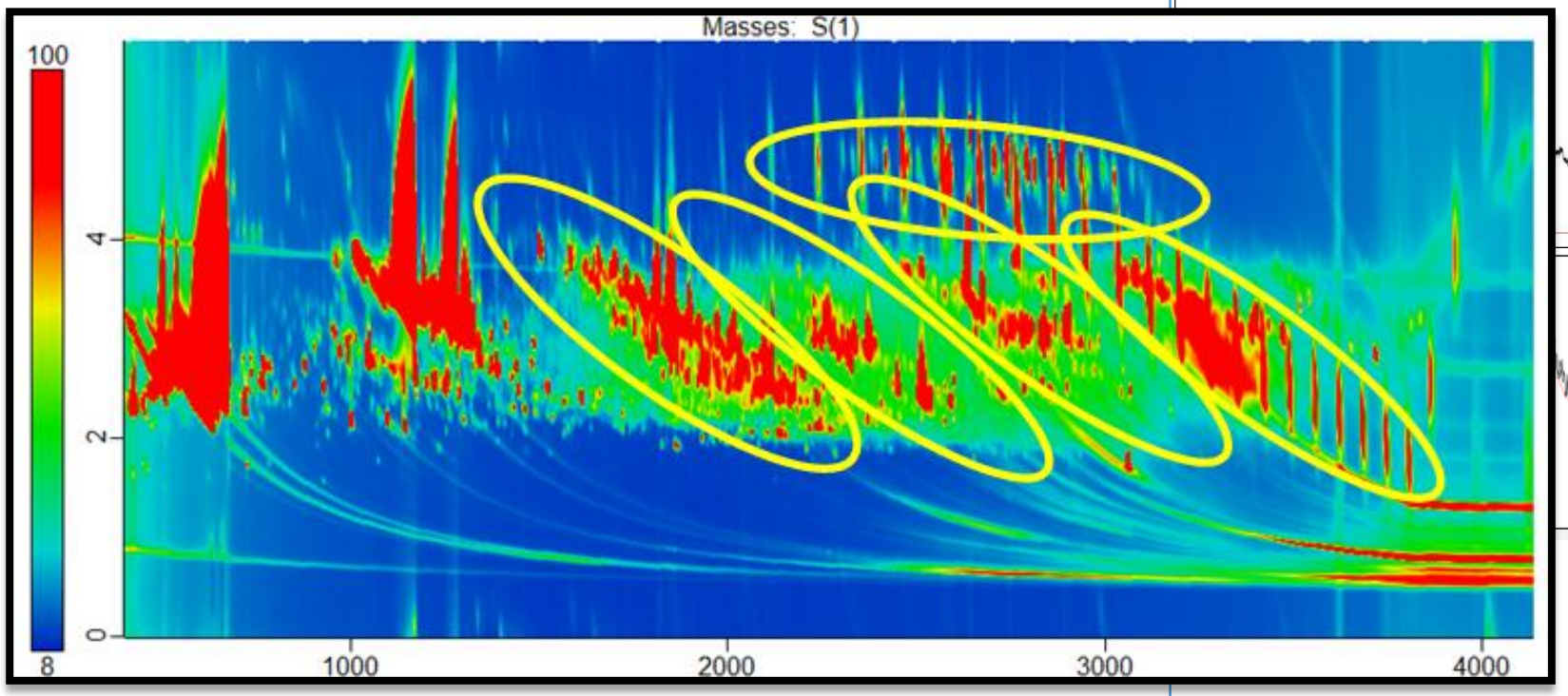
GC×GC benefits for MOSH and MOAH analysis



Easier interpretation



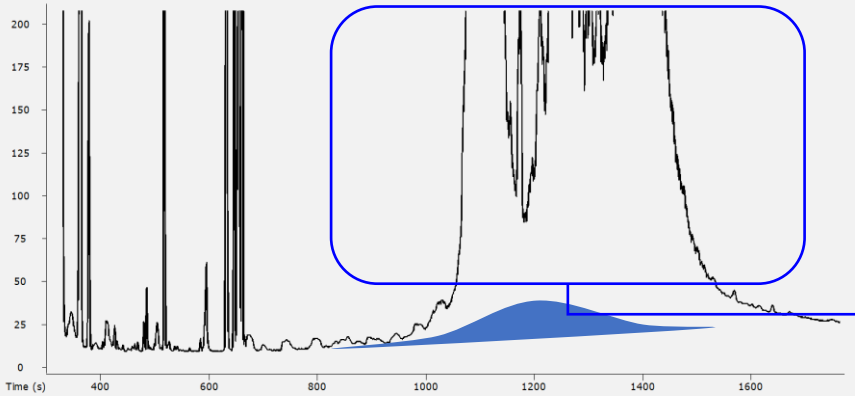


Coeluted interferences to be removed

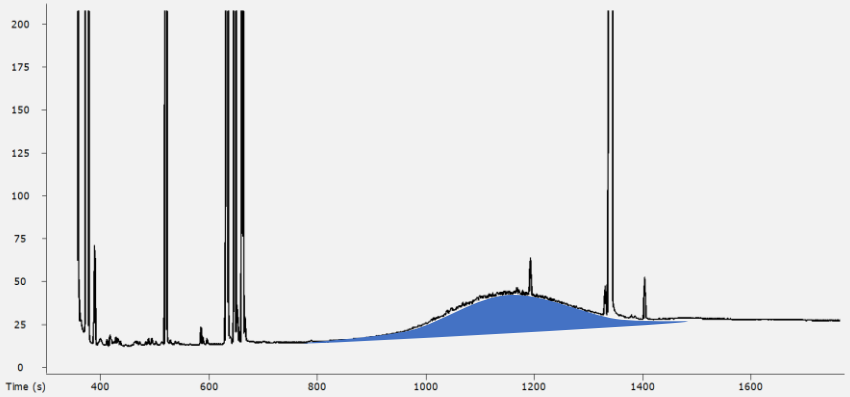
→ **High uncertainty of the result**

CHALLENGES in MOSH/MOAH analysis

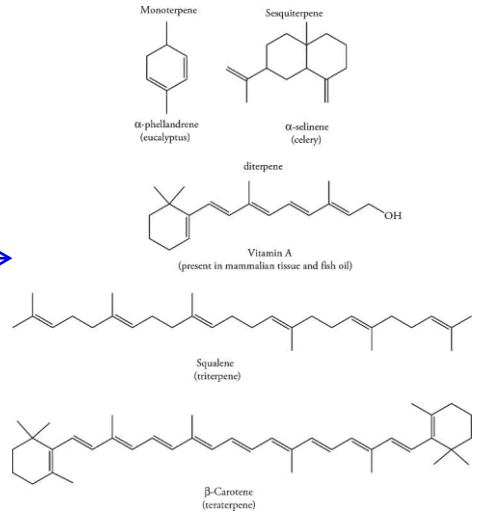
MOAH (20 mg/kg) – with matrix



MOAH (20 mg/kg) – without matrix



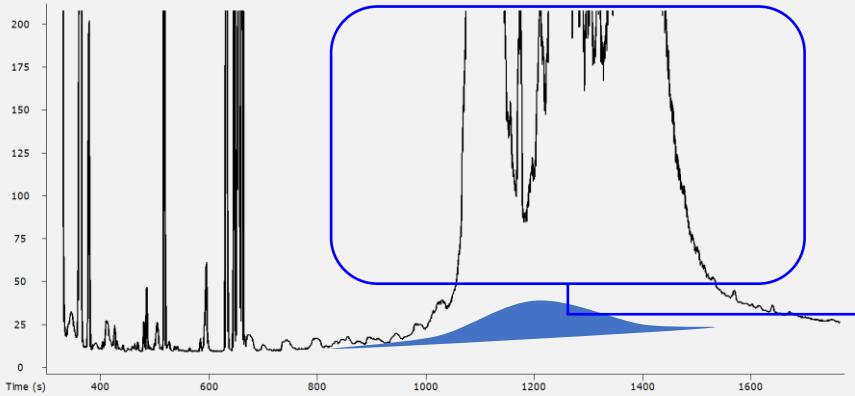
Food matrices can contain terpenes at concentrations that overload MOAH chromatograms



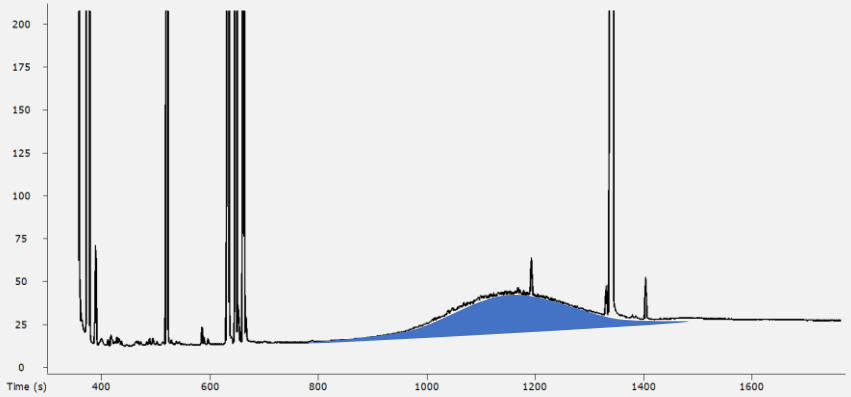
CHALLENGES in MOSH/MOAH analysis

Second very important sample preparation step for MOAH …

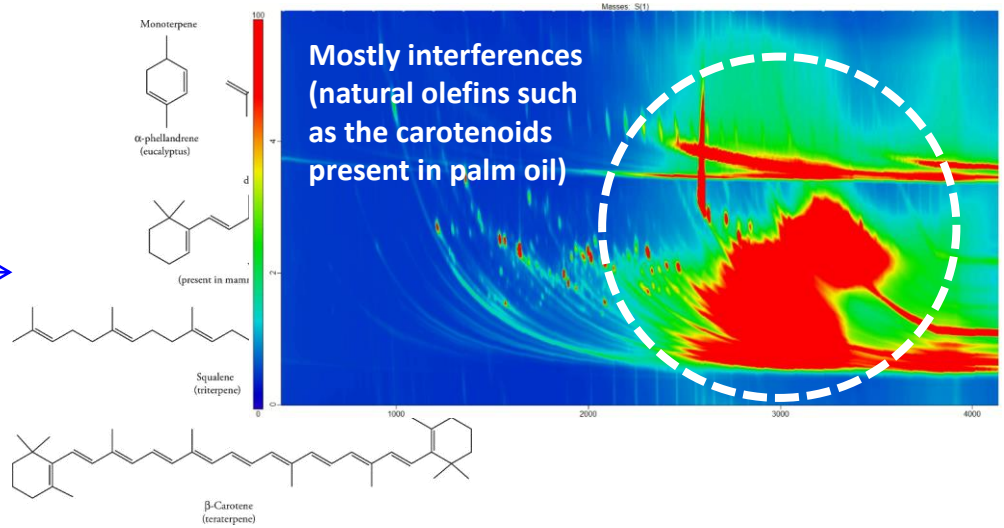
MOAH (20 mg/kg) – with matrix



MOAH (20 mg/kg) – without matrix



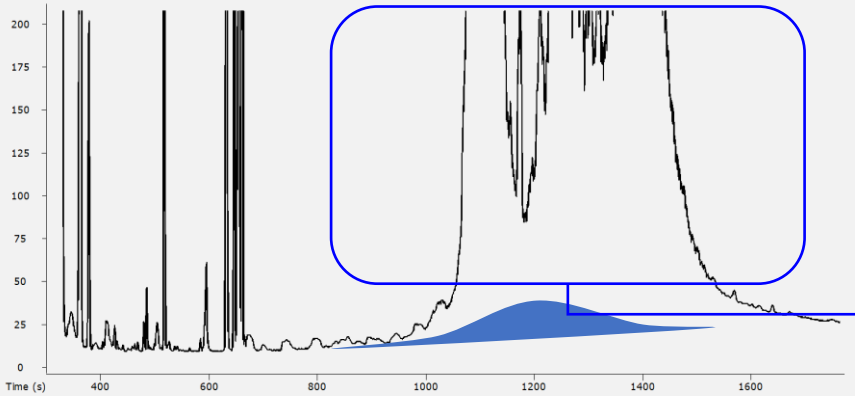
Food matrices can contain terpenes at concentrations that overload MOAH chromatograms



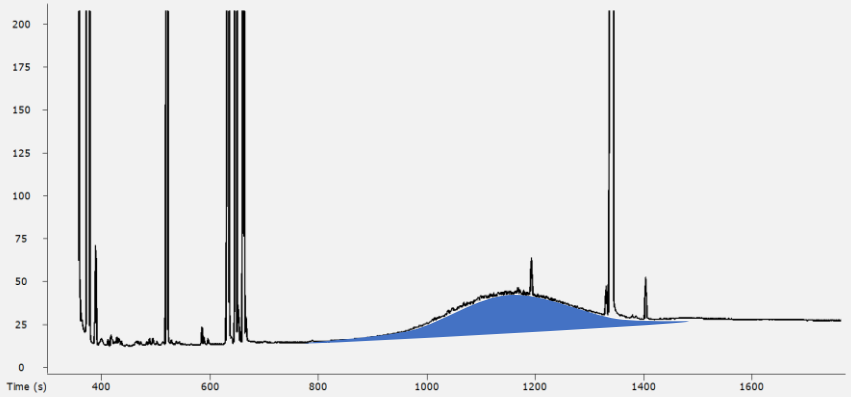
CHALLENGES in MOSH/MOAH analysis

Second very important sample preparation step for MOAH …

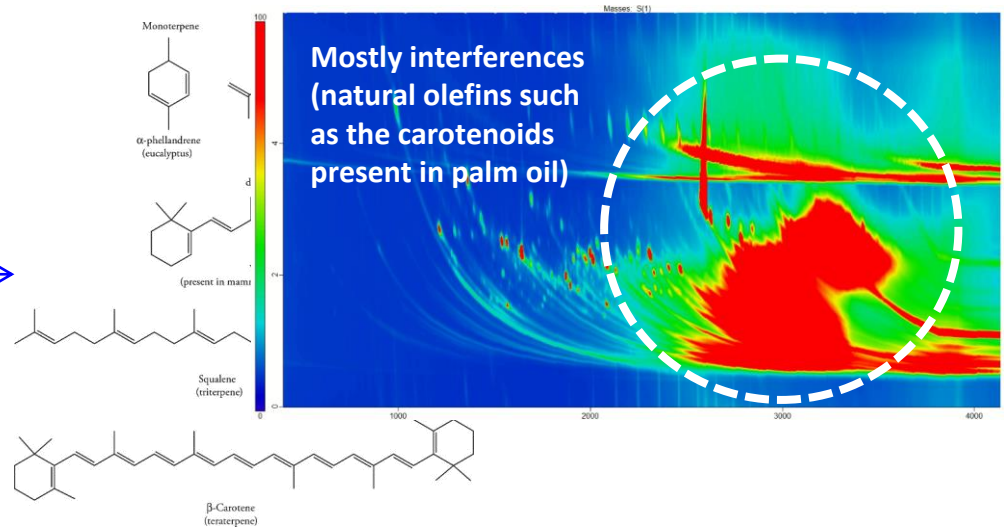
MOAH (20 mg/kg) – with matrix



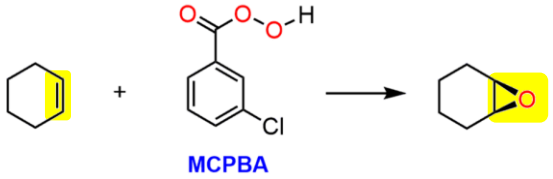
MOAH (20 mg/kg) – without matrix



Food matrices can contain terpenes at concentrations that overload MOAH chromatograms



Epoxidation is used to remove these terpenes



20-40% of MOAH can be also be lost (particularly those having many d.b.)

Another cause of uncertainty!

An alternative purification method

Journal of Chromatography A 1624 (2020) 461236

Contents lists available at ScienceDirect

Journal of Chromatography A

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

Epoxidation for the analysis of the mineral oil aromatic hydrocarbons in food. An update

Maurus Biedermann, Celine Munoz, Koni Grob*

Official Food Control Authority of the Canton of Zürich, PO Box, CH-8032 Zurich, Switzerland

Various attempts were made to remove the olefins from the MOAH in the liquid preseparation step. Zoccali et al. [25] added a second HPLC column with silver ions to improve the separation between the MOAH and the polyunsaturated olefins. Squalene was retained beyond the MOAH with up to three aromatic rings, but not beyond the larger aromatic ring structures, among which are the well-known potent carcinogenic species. Furthermore, a large part of the isomerized squalenes and most of the sterenes are eluted earlier [26]. From untreated silica gel, mono- and some di-enes fall into the MOSH fraction [27]. In fact, since the MOAH as well as these interferences are eluted in broad HPLC retention windows, the chromatographic separation does not seem promising.

I doubt,
therefore, I try

" and as I observed that this truth, I think, therefore I am (COGITO ERGO SUM), was so certain and of such evidence that no ground of doubt, however extravagant, could be alleged by the sceptics capable of shaking it, I concluded that I might, without scruple, accept it as the first principle of the philosophy of which I was in search. "

René Descartes (Discourse on the Method)

An alternative purification method

Saponification + L/L extraction (C6)

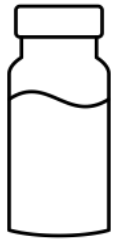
HPLC purification

HPLC-GC×GC-FID analysis

Allure Silica (250 mm × 2.1 mm i.d. × 5 µm d_p, 60 Å (Restek)

HPLC

C6/DCM gradient 1



Non-purified oil extract
(after saponification)

MOAH **MOSH**

GC(×GC)

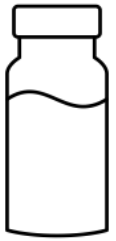
Gradient 1: 0 min 100% C6; 1.5–6 min 65% C6 35% DCM at 0.3 mL/min

An alternative purification method

Saponification + L/L extraction (C6)

HPLC purification

HPLC-GC×GC-FID analysis



Non-purified oil extract
(after saponification)

HPLC

C6/DCM gradient 1
C6/DCM gradient 2

MOAH MOSH

GC(×GC)

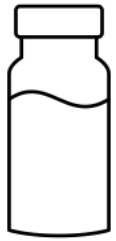
Gradient 1: 0 min 100% C6; 1.5–6 min 65% C6 35% DCM at 0.3 mL/min
Gradient 2: 0 min 100% C6; 1.5–14.5 min 98% C6 2% DCM; 14.5–23 min 95% C6 5% DCM at 0.3 mL/min

An alternative purification method

Saponification + L/L extraction (C6)

HPLC purification

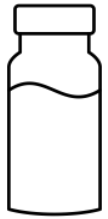
HPLC-GC×GC-FID analysis



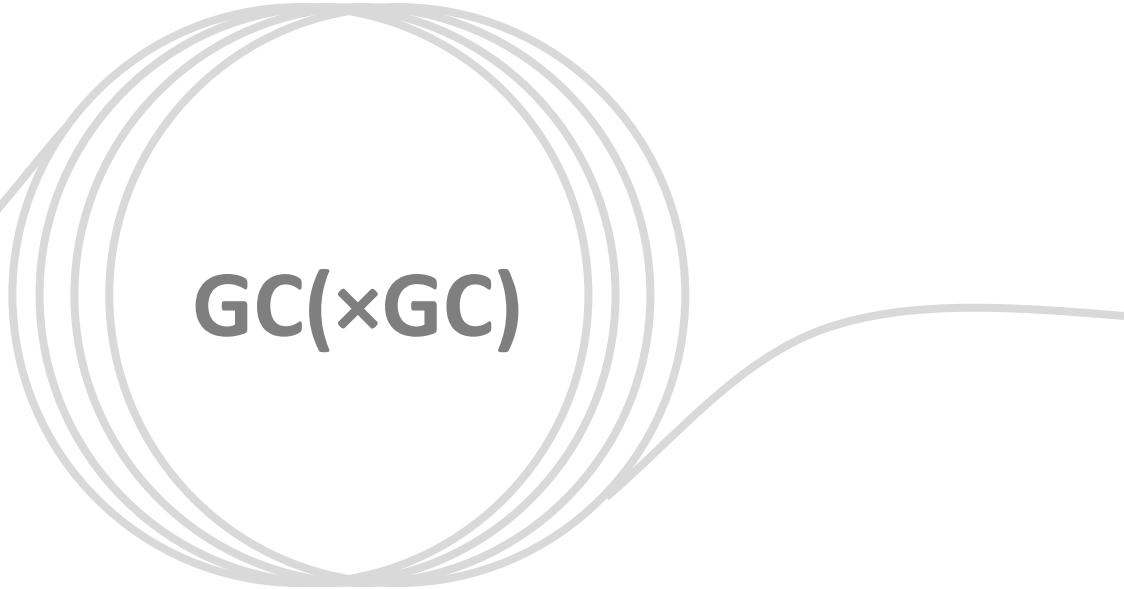
Non-purified oil extract
(after saponification)

HPLC

C6/DCM gradient 1
C6/DCM gradient 2



Purified MOAH



Gradient 1: 0 min 100% C6; 1.5–6 min 65% C6 35% DCM at 0.3 mL/min
Gradient 2: 0 min 100% C6; 1.5–14.5 min 98% C6 2% DCM; 14.5–23 min 95% C6 5% DCM at 0.3 mL/min

LC-GC×GC: 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

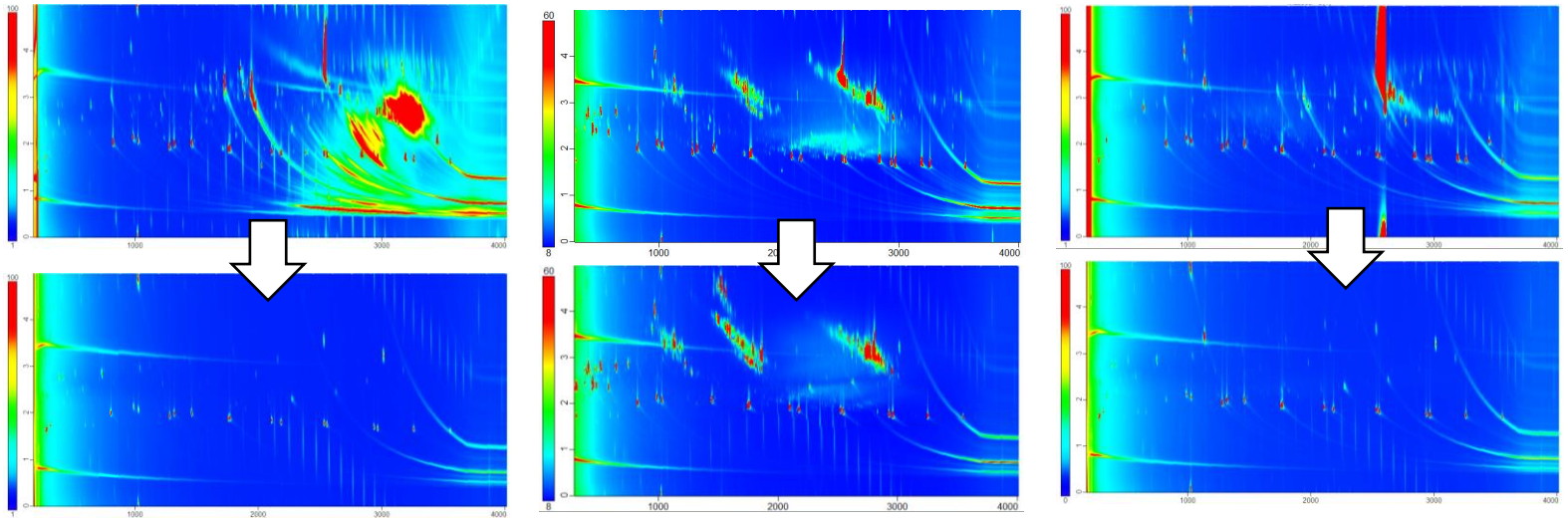
MOAH Recovery LC Purification:

94% ± 2%

Epoxidation

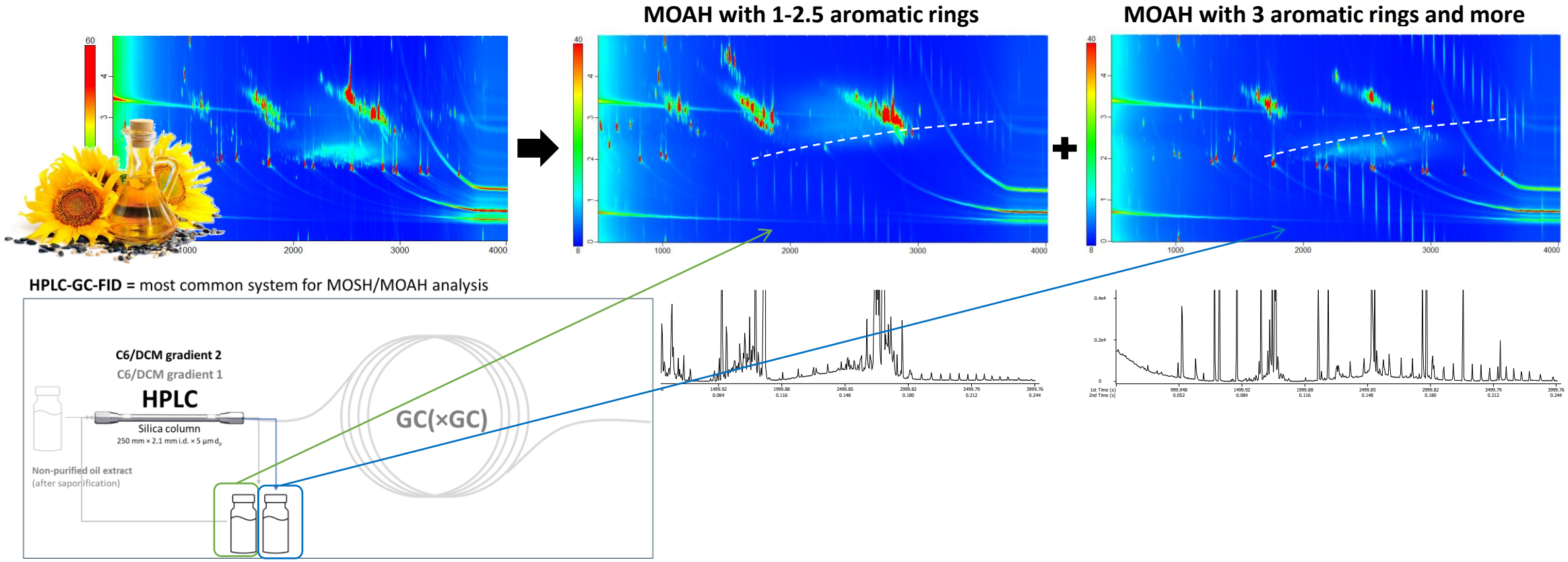
mCPBA 82%± 10%

performic acid 71 ± 16%



LC-GC×GC: An alternative purification method

The LC purification method also allows to separate and quantify MOAH based on their number of aromatic rings.

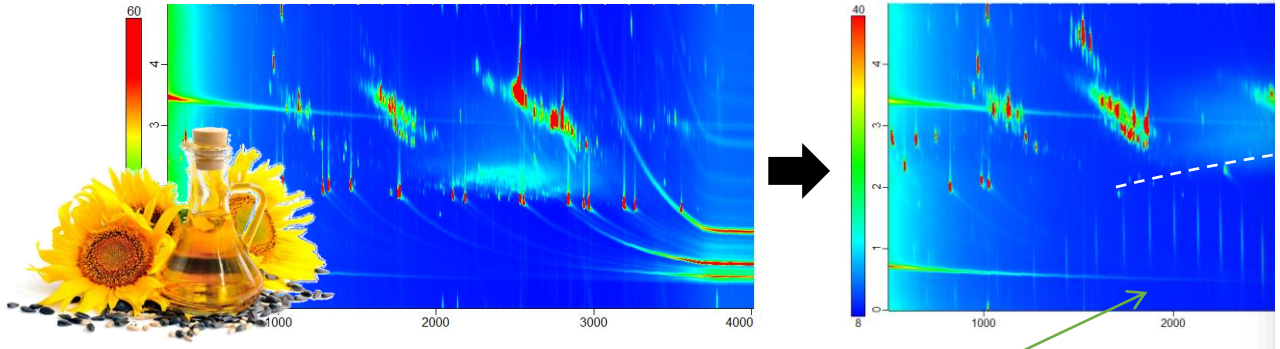


LC-GC×GC: An alternative purification method

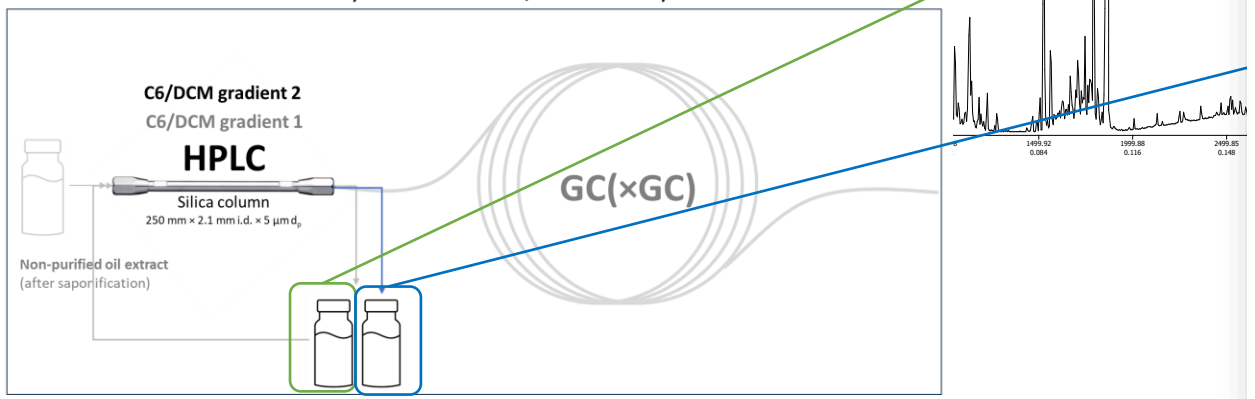
The LC purification method also allows to separate

aromatic r

MOAH with 1-2.5 arc



HPLC-GC-FID = most common system for MOSH/MOAH analysis



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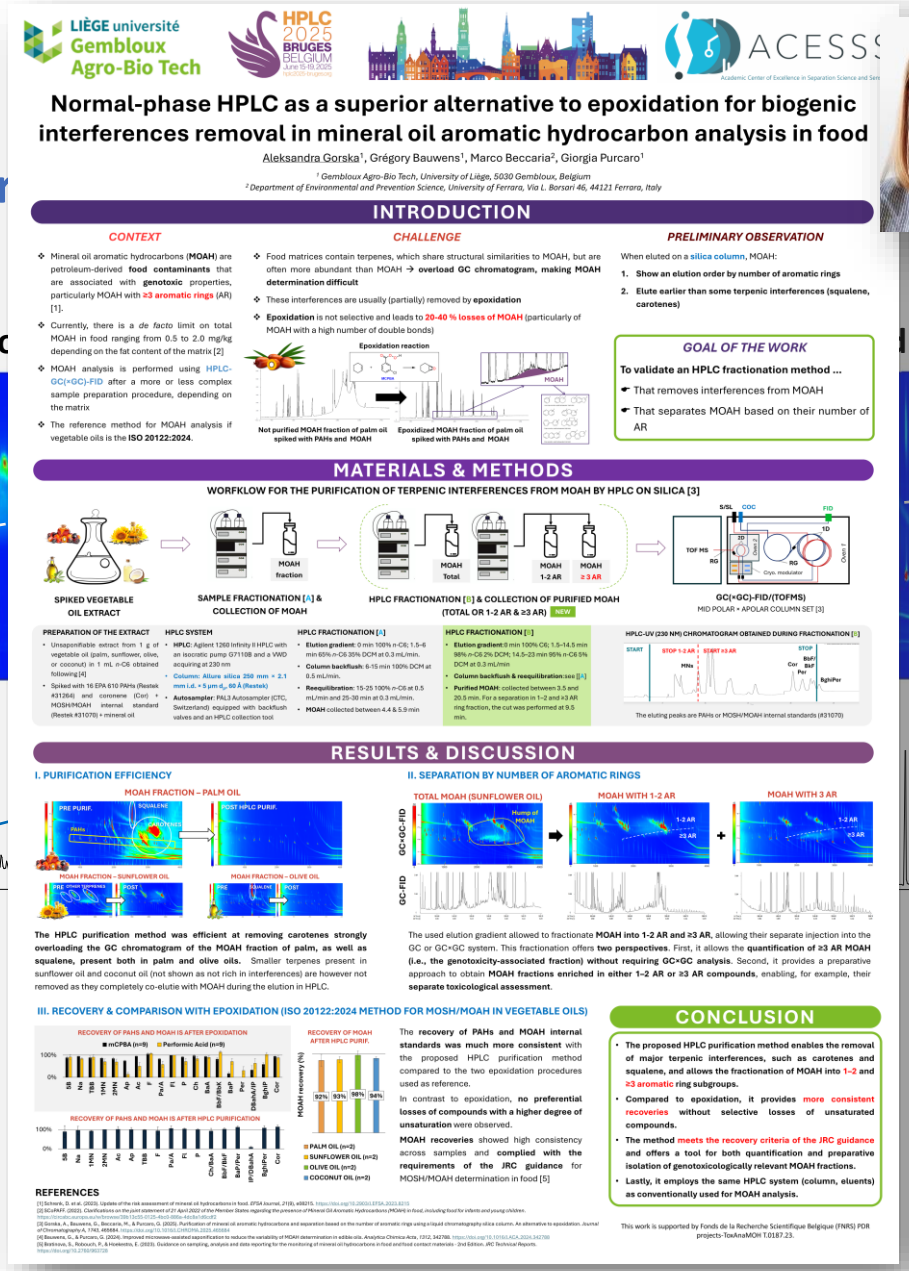


Normal-phase HPLC as a superior alternative to epoxidation for biogenic interferences removal in mineral oil aromatic hydrocarbon analysis in food

Aleksandra Gorska[1], Grégory Bauwens[1], Marco Beccaria[2], Giorgia Purcaro[1]

[1] Gemblioux Agro-Bio Tech, University of Liège, 5030 Gembloux, Belgium
[2] Department of Environmental and Prevention Science, University of Ferrara, Via L. Borsari 46, 44121 Ferrara, Italy

INTRODUCTION

CONTEXT

- Mineral oil aromatic hydrocarbons (**MOAH**) are petroleum-derived **food contaminants** that are associated with **genotoxic** properties, particularly MOAH with **≥3 aromatic rings** (AR) [1].
- Currently, there is a *de facto* limit on total MOAH in food ranging from 0.5 to 2.0 mg/kg depending on the fat content of the matrix [2]
- MOAH analysis is performed using HPLC-GC(×GC)-FID after a more or less complex sample preparation procedure, depending on the matrix
- The reference method for MOAH analysis if vegetable oils is the **ISO 20122:2024**.

CHALLENGE

- Food matrices contain terpenes, which share structural similarities to MOAH, but are often more abundant than MOAH → **overload GC chromatogram, making MOAH determination difficult**
- These interferences are usually (partially) removed by **epoxidation**
- **Epoxidation** is not selective and leads to **20-40 % losses of MOAH** (particularly of MOAH with a high number of double bonds)

Epoxidation reaction

Not purified MOAH fraction of palm oil spiked with PAHs and MOAH

Epoxidized MOAH fraction of palm oil spiked with PAHs and MOAH

PRELIMINARY OBSERVATION

When eluted on a **silica column**, MOAH:

1. **Show an elution order by number of aromatic rings**
2. **Elute earlier than some terpenic interferences (squalene, carotenes)**

GOAL OF THE WORK

To validate an HPLC fractionation method ...

- That removes interferences from MOAH
- That separates MOAH based on their number of AR

MATERIALS & METHODS

WORKFLOW FOR THE PURIFICATION OF TERPENIC INTERFERENCES FROM MOAH BY HPLC ON SILICA [3]

SPIKED VEGETABLE OIL EXTRACT

SAMPLE FRACTIONATION [A] & COLLECTION OF MOAH

HPLC FRACTIONATION [B] & COLLECTION OF PURIFIED MOAH (TOTAL OR 1-2 AR & ≥3 AR) NEW

GC(×GC)-FID/(TOFMS)
MID POLAR × APOLAR COLUMN SET [3]

PREPARATION OF THE EXTRACT

- Unsaponifiable extract from 1 g of vegetable oil (palm, sunflower, olive, or coconut) in 1 mL *n*-C6 obtained following [4]
- Spiked with 16 EPA 610 PAHs (Restek #31264) and coronene (Cor) + MOSH/MOAH internal standard (Restek #31070) + mineral oil

HPLC SYSTEM

- **HPLC:** Agilent 1260 Infinity II HPLC with an isocratic pump G7110B and a VWD acquiring at 230 nm
- **Column: Allure silica 250 mm × 2.1 mm i.d. × 5 µm d_p, 60 Å (Restek)**
- **Autosampler:** PAL3 Autosampler (CTC, Switzerland) equipped with backflush valves and an HPLC collection tool

HPLC FRACTIONATION [A]

- **Elution gradient:** 0 min 100% *n*-C6; 1.5–6 min 60%-*n*-C6:35% DCM at 0.3 mL/min.
- **Column backflush:** 6-15 min 100% DCM at 0.5 mL/min.
- **Reequilibration:** 15-25 100% *n*-C6 at 0.5 mL/min and 25-36 min at 0.3 mL/min.
- **MOAH** collected between 4.4 & 5.9 min

HPLC FRACTIONATION [B]

- **Elution gradient:** 0 min 100% C6; 1.5–14.5 min 98% *n*-C6 2% DCM; 14.5–23 min 95% *n*-C6 5% DCM at 0.3 mL/min
- **Column backflush & reequilibration:** see [A]
- **Purified MOAH:** collected between 3.5 and 20.5 min. For a separation in 1–2 and ≥3 AR ring fraction, the cut was performed at 9.5 min.

HPLC-UV (230 NM) CHROMATOGRAM OBTAINED DURING FRACTIONATION [B]

The eluting peaks are PAHs or MOSH/MOAH internal standards (#31070)

RESULTS & DISCUSSION

I. PURIFICATION EFFICIENCY

MOAH FRACTION – PALM OIL

MOAH FRACTION – SUNFLOWER OIL

MOAH FRACTION – OLIVE OIL

The HPLC purification method was efficient at removing carotenes strongly overloading the GC chromatogram of the MOAH fraction of palm, as well as squalene, present both in palm and olive oils. Smaller terpenes present in sunflower oil and coconut oil (not shown as not rich in interferences) are however not removed as they completely co-elutie with MOAH during the elution in HPLC.

II. SEPARATION BY NUMBER OF AROMATIC RINGS

TOTAL MOAH (SUNFLOWER OIL)

MOAH WITH 1-2 AR

MOAH WITH 3 AR

GC×GC-FID

GC-FID

The used elution gradient allowed to fractionate **MOAH into 1-2 AR and ≥3 AR**, allowing their separate injection into the GC or GC×GC system. This fractionation offers **two perspectives**. First, it allows the **quantification of ≥3 AR MOAH (i.e., the genotoxicity-associated fraction) without requiring GC×GC analysis**. Second, it provides a preparative approach to obtain **MOAH fractions enriched in either 1–2 AR or ≥3 AR compounds**, enabling, for example, their **separate toxicological assessment**.

III. RECOVERY & COMPARISON WITH EPOXIDATION (ISO 20122:2024 METHOD FOR MOSH/MOAH IN VEGETABLE OILS)

RECOVERY OF PAHS AND MOAH IS AFTER EPOXIDATION

RECOVERY OF PAHS AND MOAH IS AFTER HPLC PURIFICATION

RECOVERY OF MOAH AFTER HPLC PURIF.

MOAH recovery (%)

92% 93% 98% 94%

PALM OIL (n=2)
SUNFLOWER OIL (n=2)
OLIVE OIL (n=2)
COCONUT OIL (n=2)

The **recovery of PAHs and MOAH internal standards was much more consistent** with the proposed HPLC purification method compared to the two epoxidation procedures used as reference.

In contrast to epoxidation, **no preferential losses of compounds with a higher degree of unsaturation** were observed.

MOAH recoveries showed high consistency across samples and **complied with the requirements of the JRC guidance** for MOSH/MOAH determination in food [5]

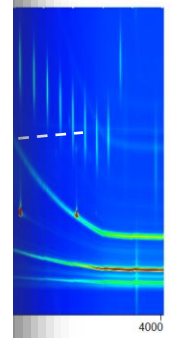
CONCLUSION

- **The proposed HPLC purification method enables the removal of major terpenic interferences, such as carotenes and squalene, and allows the fractionation of MOAH into 1–2 and ≥3 aromatic ring subgroups.**
- **Compared to epoxidation, it provides more consistent recoveries without selective losses of unsaturated compounds.**
- **The method meets the recovery criteria of the JRC guidance and offers a tool for both quantification and preparative isolation of genotoxicologically relevant MOAH fractions.**
- **Lastly, it employs the same HPLC system (column, eluents) as conventionally used for MOAH analysis.**

REFERENCES

This work is supported by Fonds de la Recherche Scientifique Belgique (FNRS) PDR projects-ToxAnaMOH T.0187.23.

more



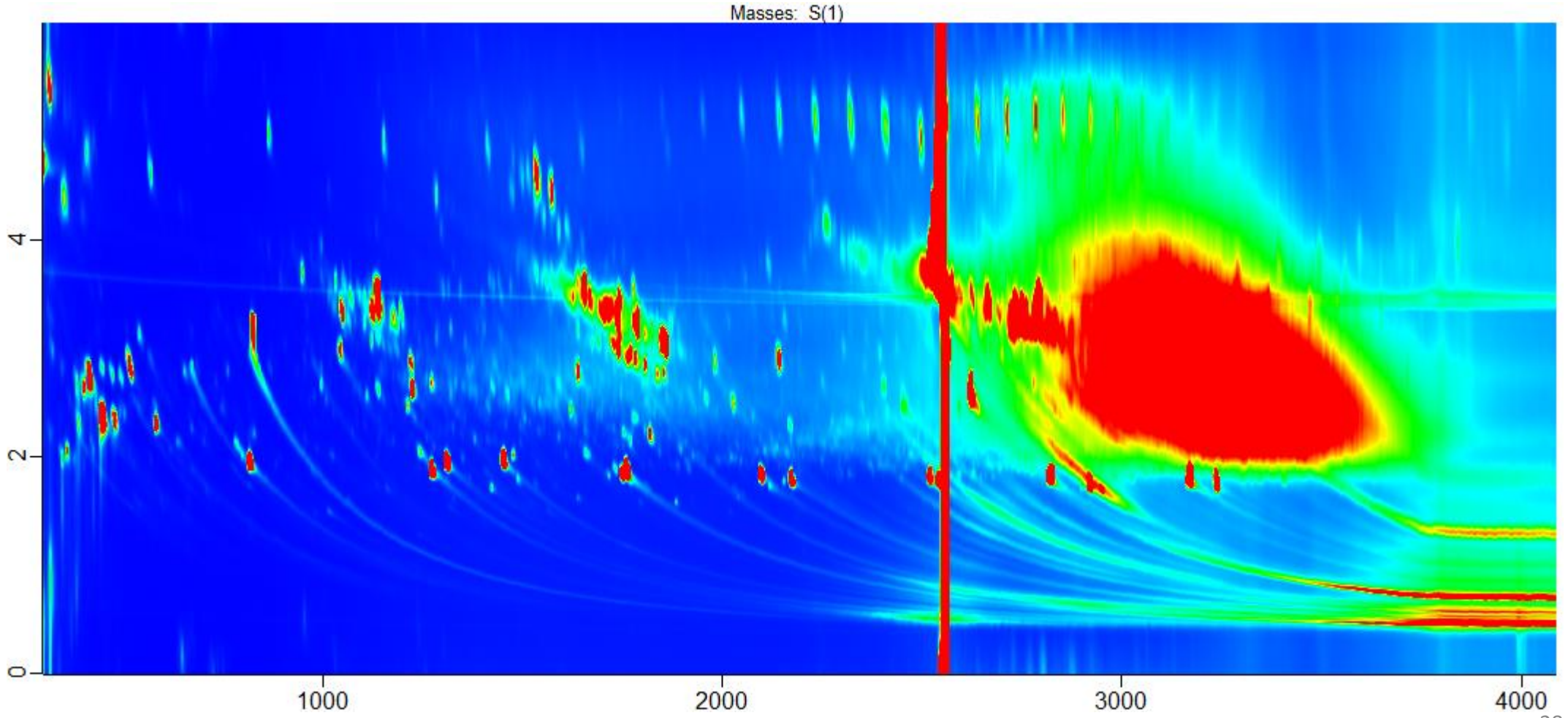
LIÈGE université Gembloux Agro-Bio Tech

Work in progress

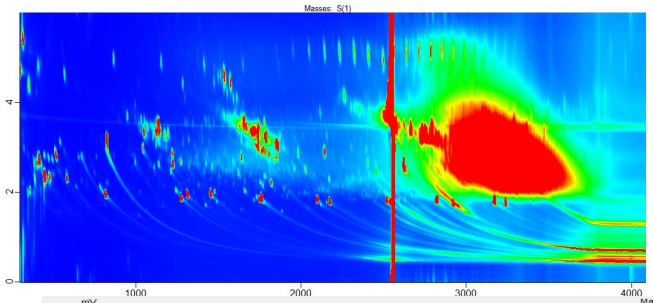
ACESSS Academic Center of Excellence in Separation Science and Sensing

Exploring different column selectivity

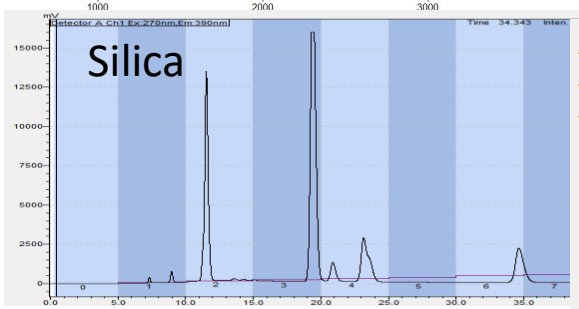
Olive oil + Refined Palm oil + Unrefined palm oil + Sunflower oil

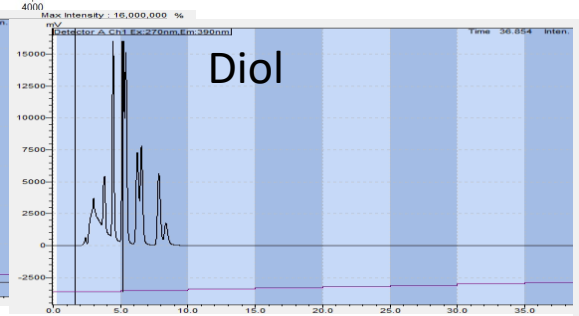


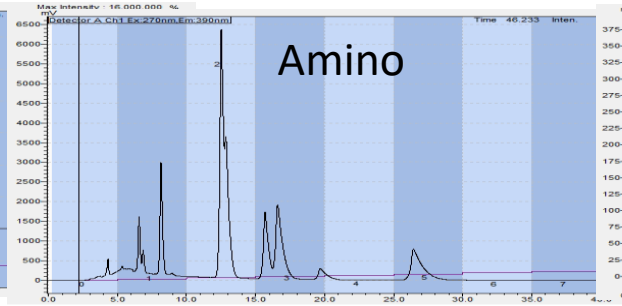
Work in progress

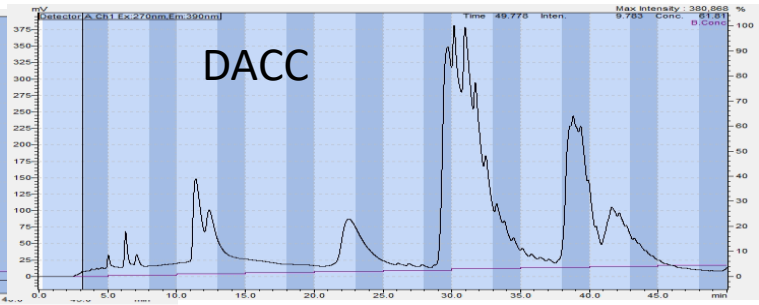


NP-LC

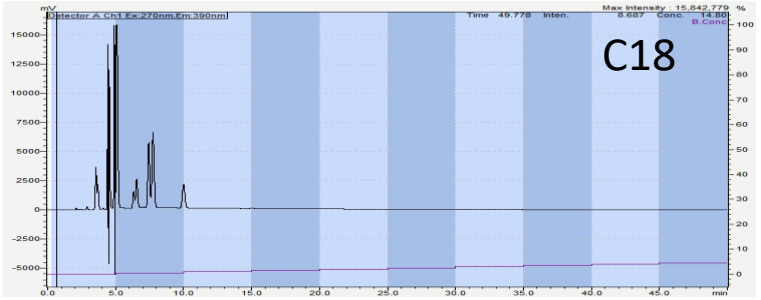


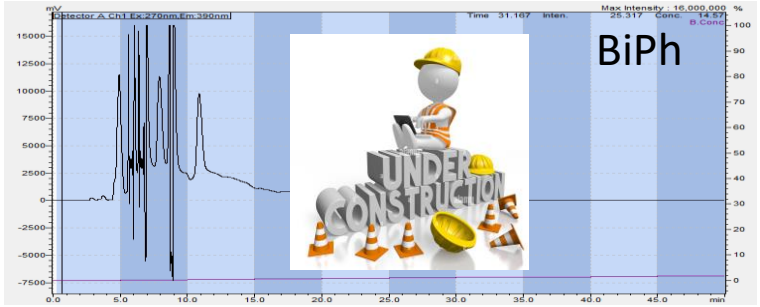






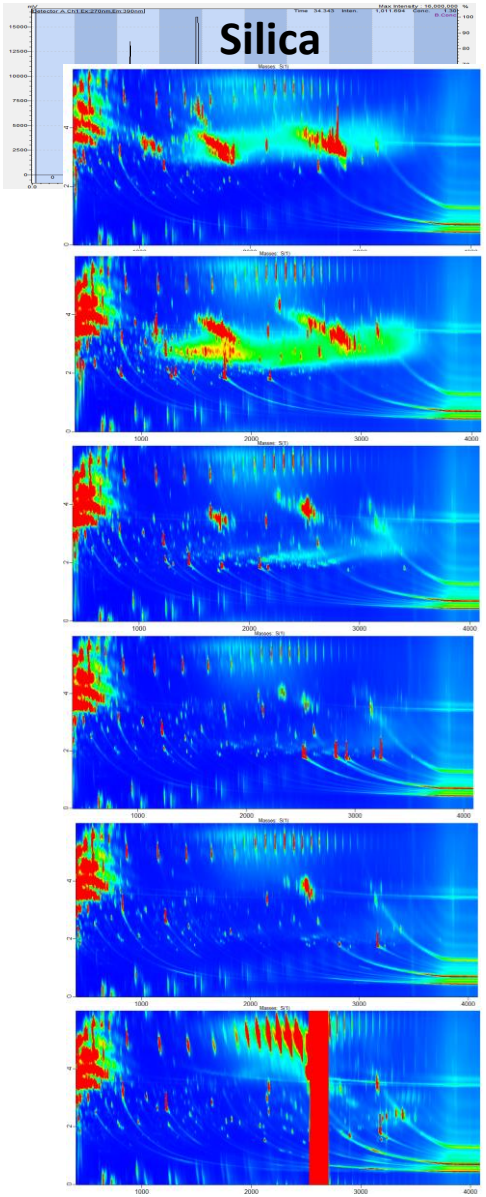
RP-LC

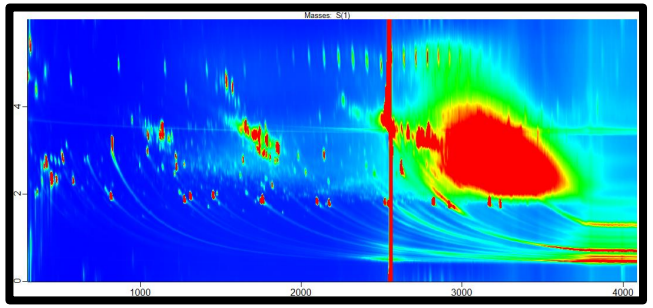




Gradient: 0 min 100% C6 to 95/5 C6/DCM at 0.2%/ 5in at 0.3 mL/min

Work in progress





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Work in progress

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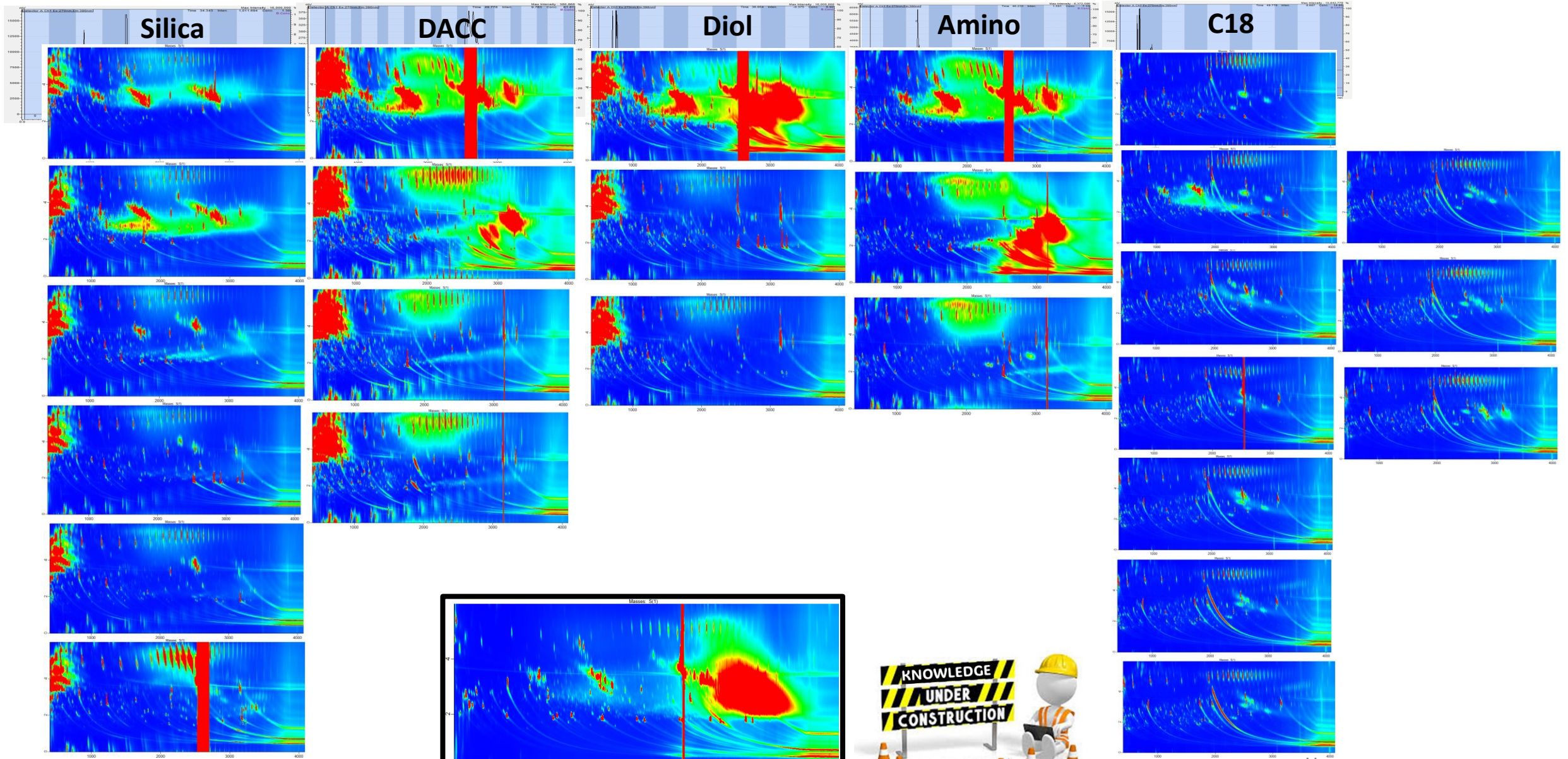

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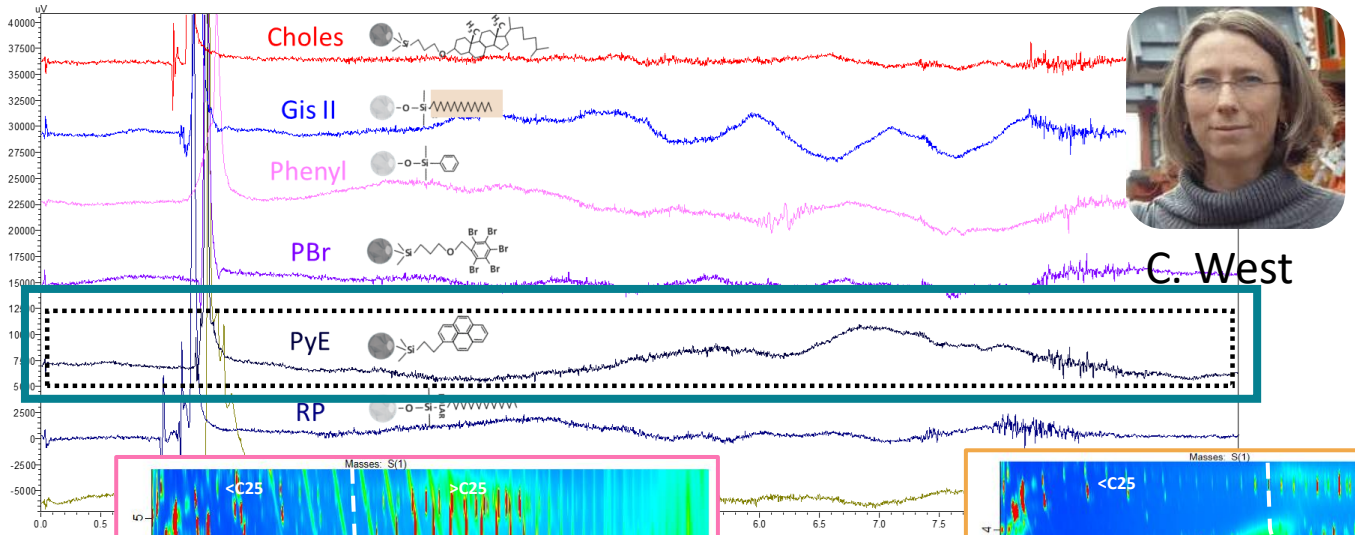
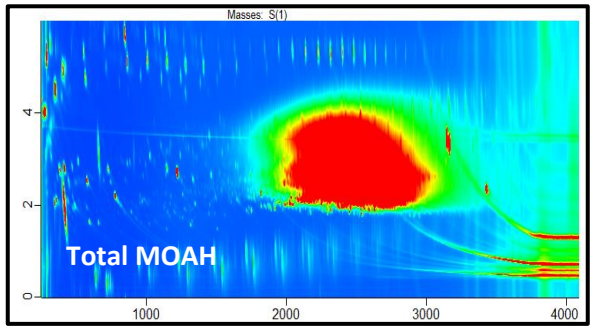
SFC/GC×GC for MOSH and MOAH analysis

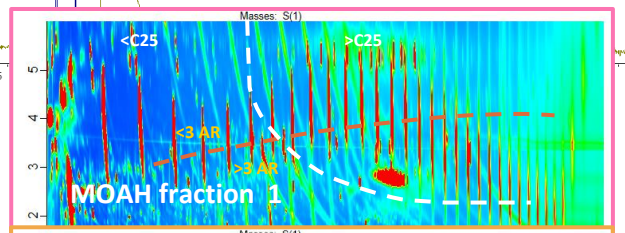
SFC-PDA

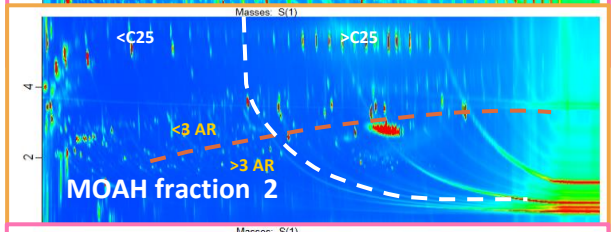


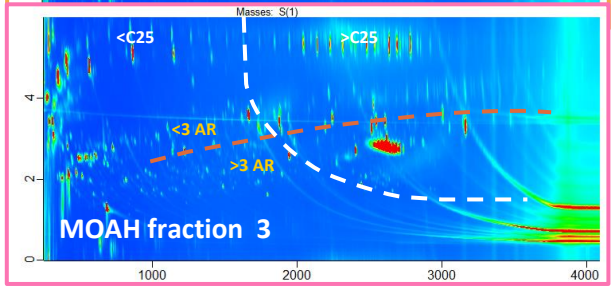
C. West

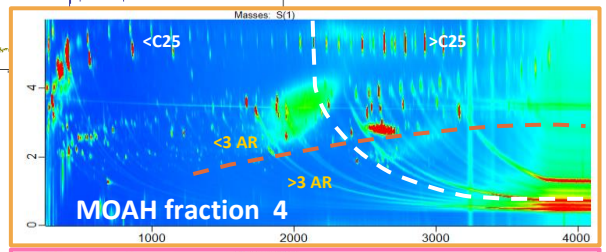
MOAH

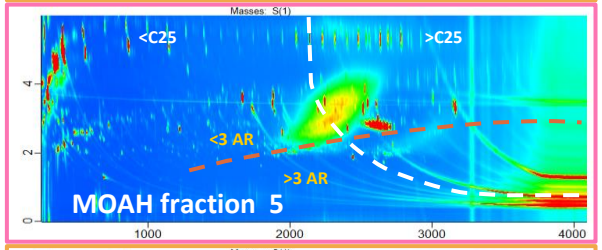


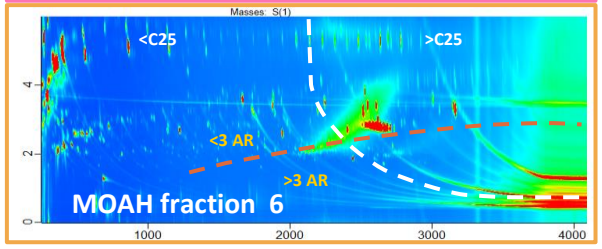


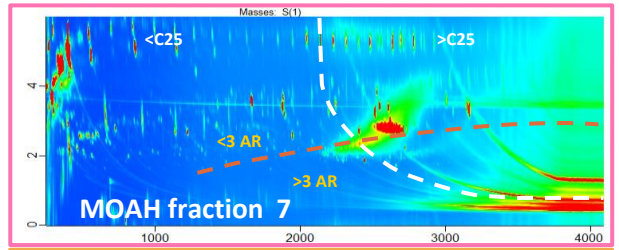


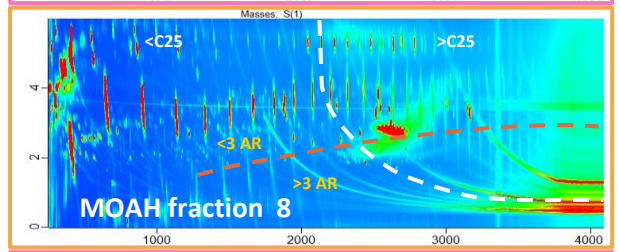


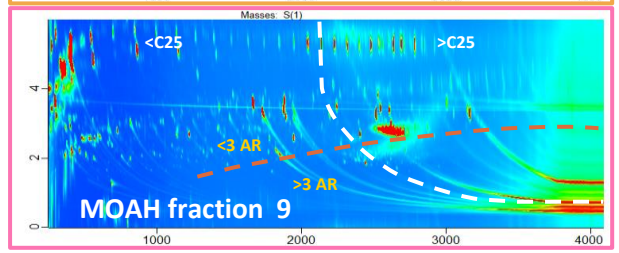






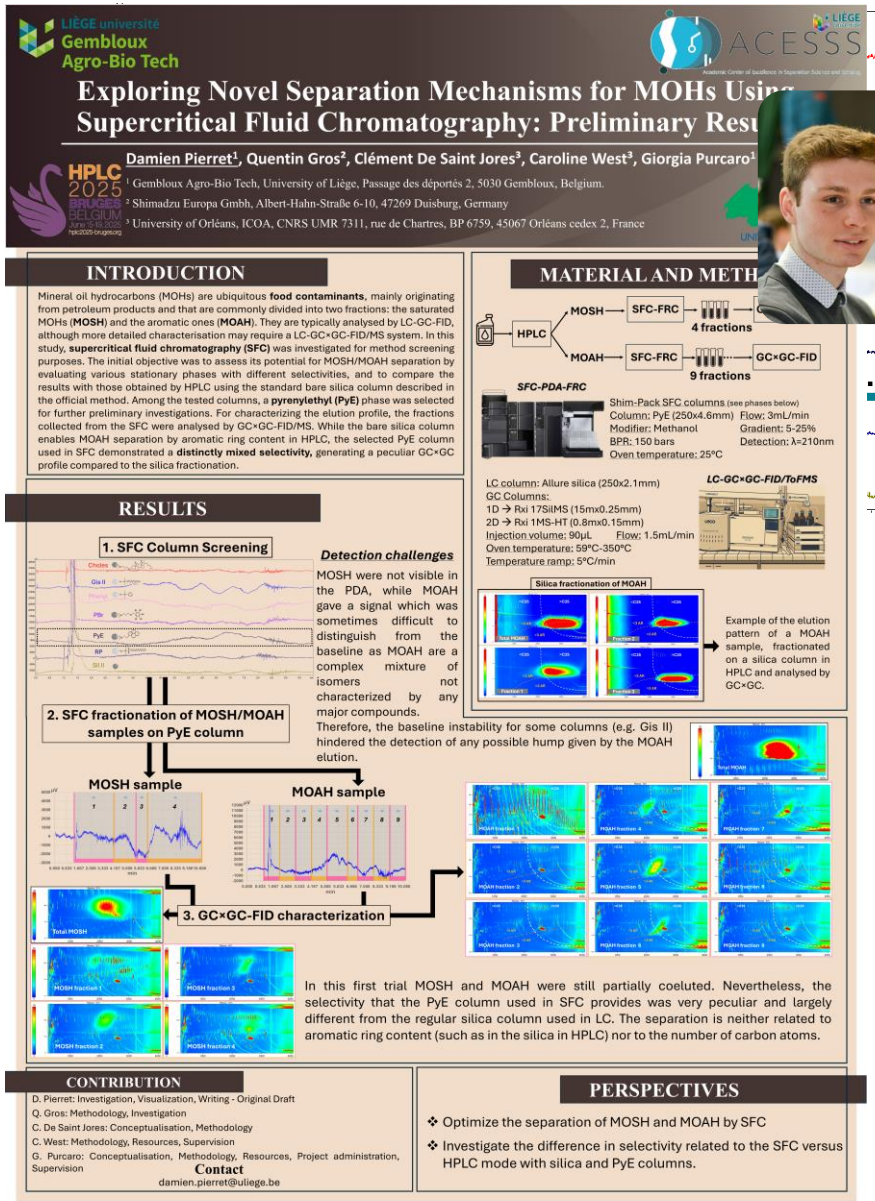


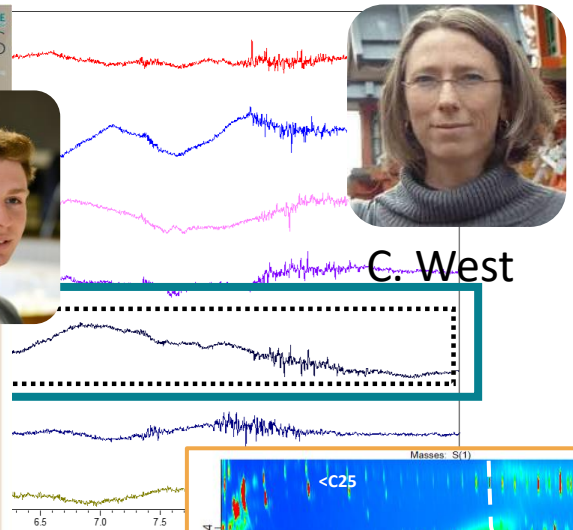




SFC/GC×GC for MOSH and MOAH analysis

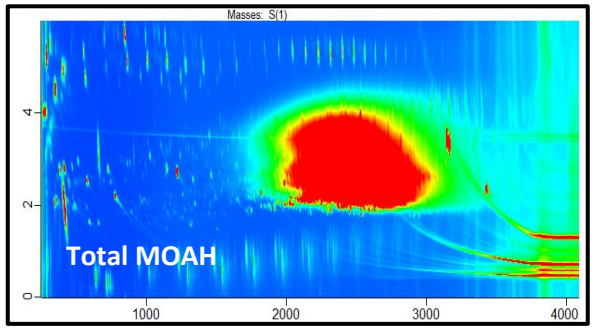
SFC-PDA

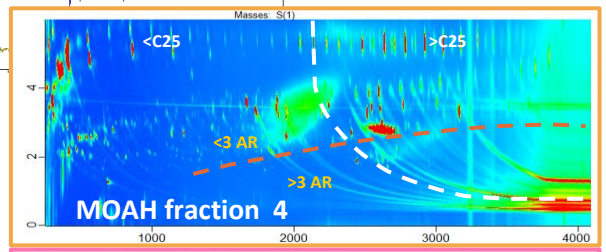


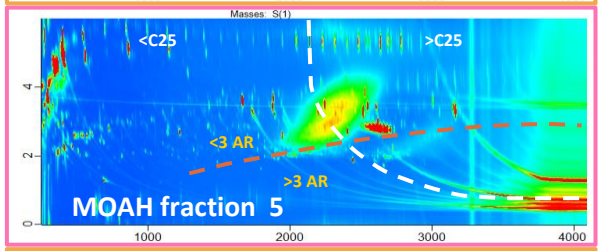


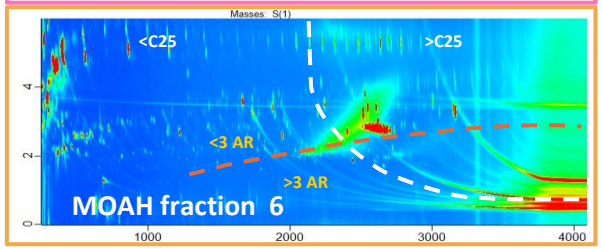
C. West

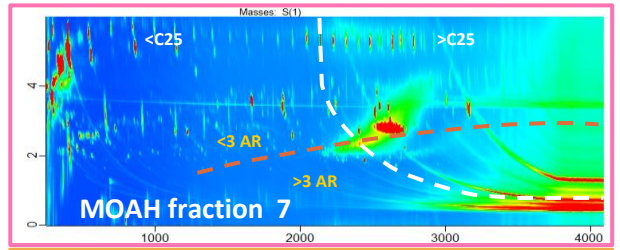
MOAH

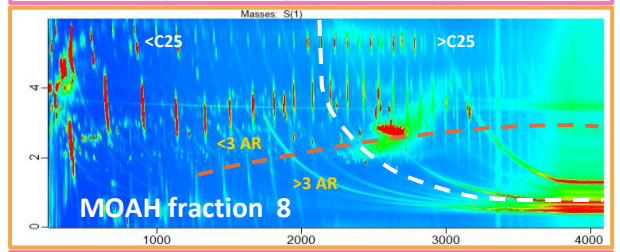


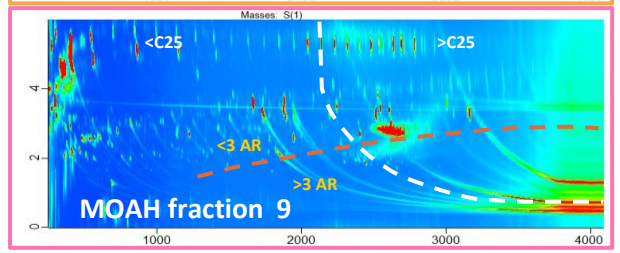




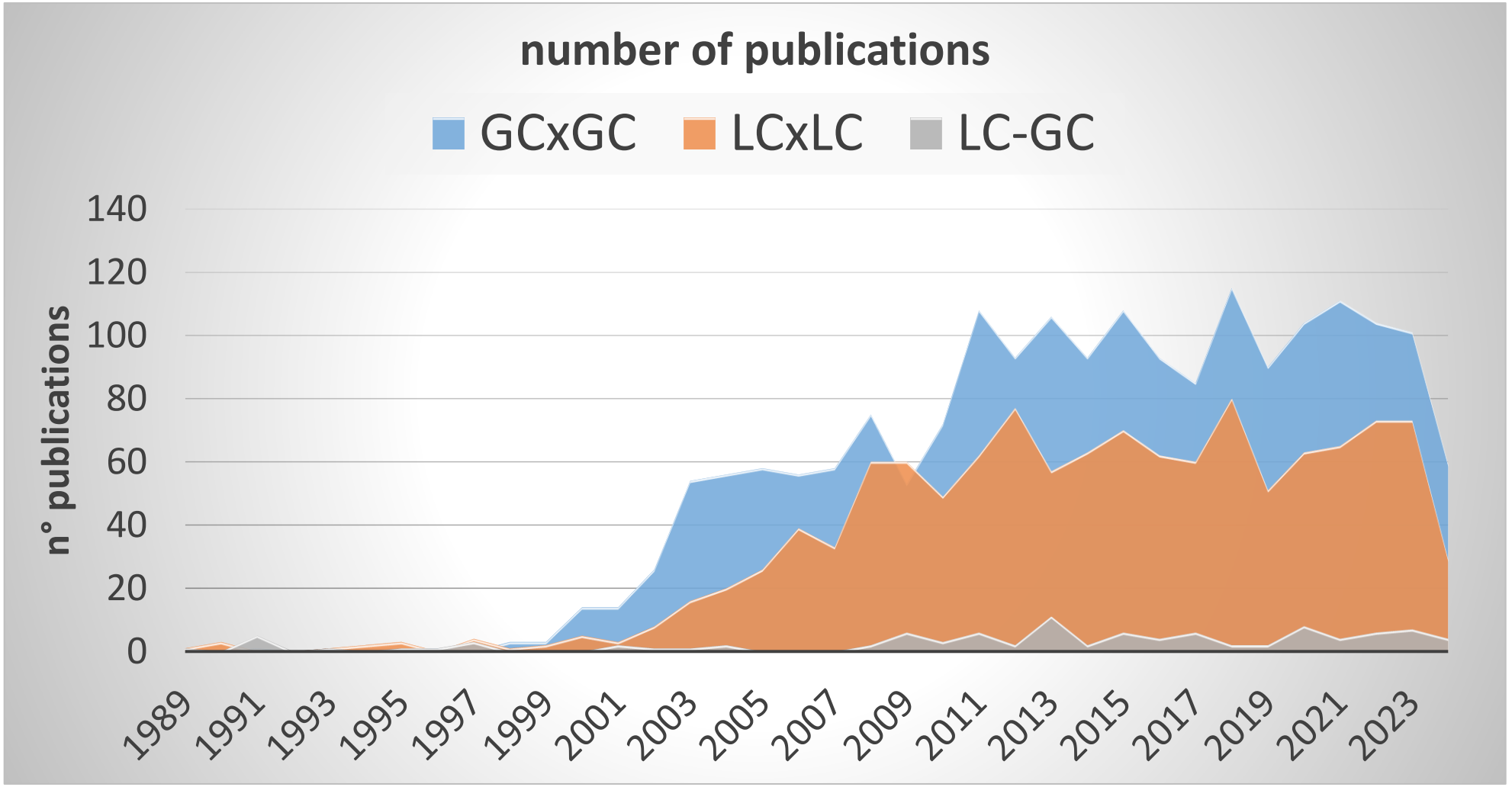








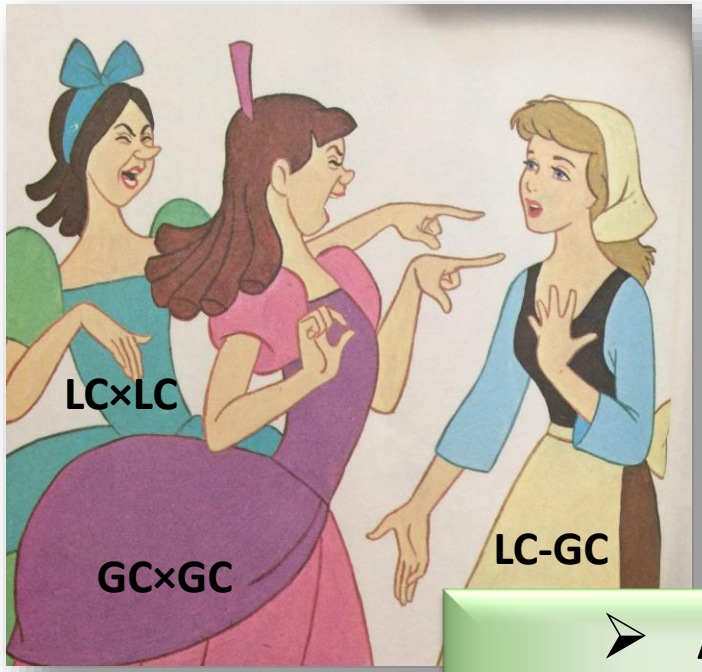
Conclusion and Perspective



Conclusion and Perspective

Underestimated Hyphenated technique

LC-GC



Redeemed Hyphenated technique

LC-GC(×GC)

- Alternative coupling: e.g., SFC-GC
- Miniaturization of LC-GC
- New column selectivity
- Advances deactivation processes

ExTech 2026

5-8 JULY 2026

SAVE the DATE

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Gembloux Agro-Bio Tech

University of Liège, Belgium

LIÈGE université
Gembloux
Agro-Bio Tech

ACESSS
Academic Center of Excellence in Separation Science and Sensing

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Nicola Ruin
Bryan Verbist
Jérôme Grandjean

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