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LC-GC×GC: A POWERFUL HYPHENATED TECHNIQUE TO ENHANCE CHROMATOGRAPHIC SEPARATION AND SIMPLIFY SAMPLE PREPARATION FOR ROUTINE APPLICATIONS

Aleksandra Gorska, Grégory Bauwens, **Giorgia Purcaro**,

Gembloux Agro Bio-Tech, University of Liège, Belgium

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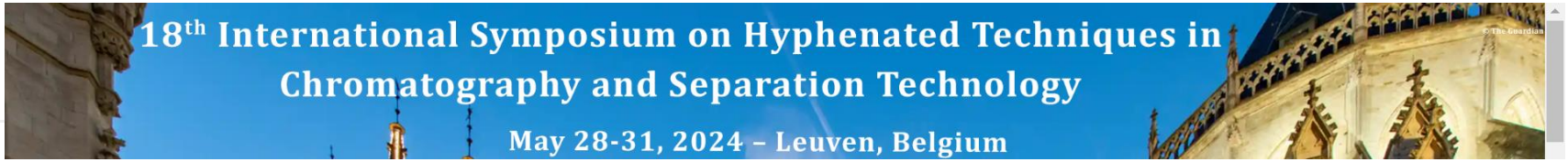

Session

10B: Unconventional hyphenated separation modes

Time: **Friday, 31/May/2024: 11:15am - 1:00pm**

Unconventional: not conventional ; not bound by or in accordance with convention ; being out of the ordinary

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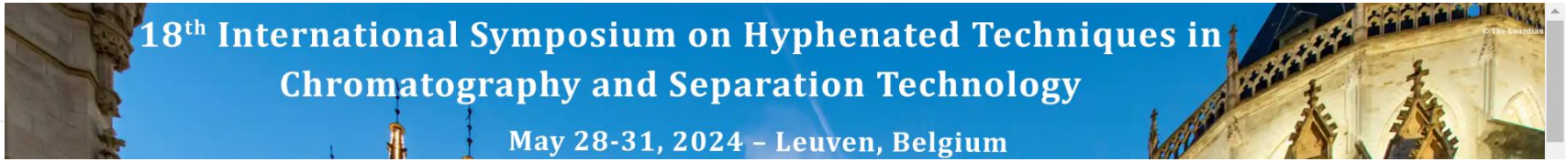

Session

10B: Unconventional hyphenated separation modes

Time: **Friday, 31/May/2024: 11:15am - 1:00pm**

LC-LC/LC×LC

GC-GC/GC×GC

Conventional Hyphenated Separation mode

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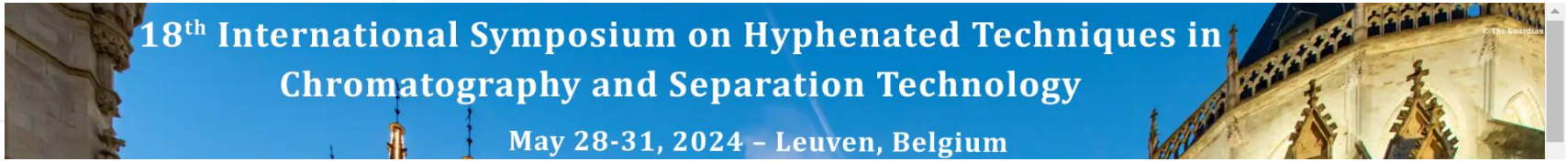
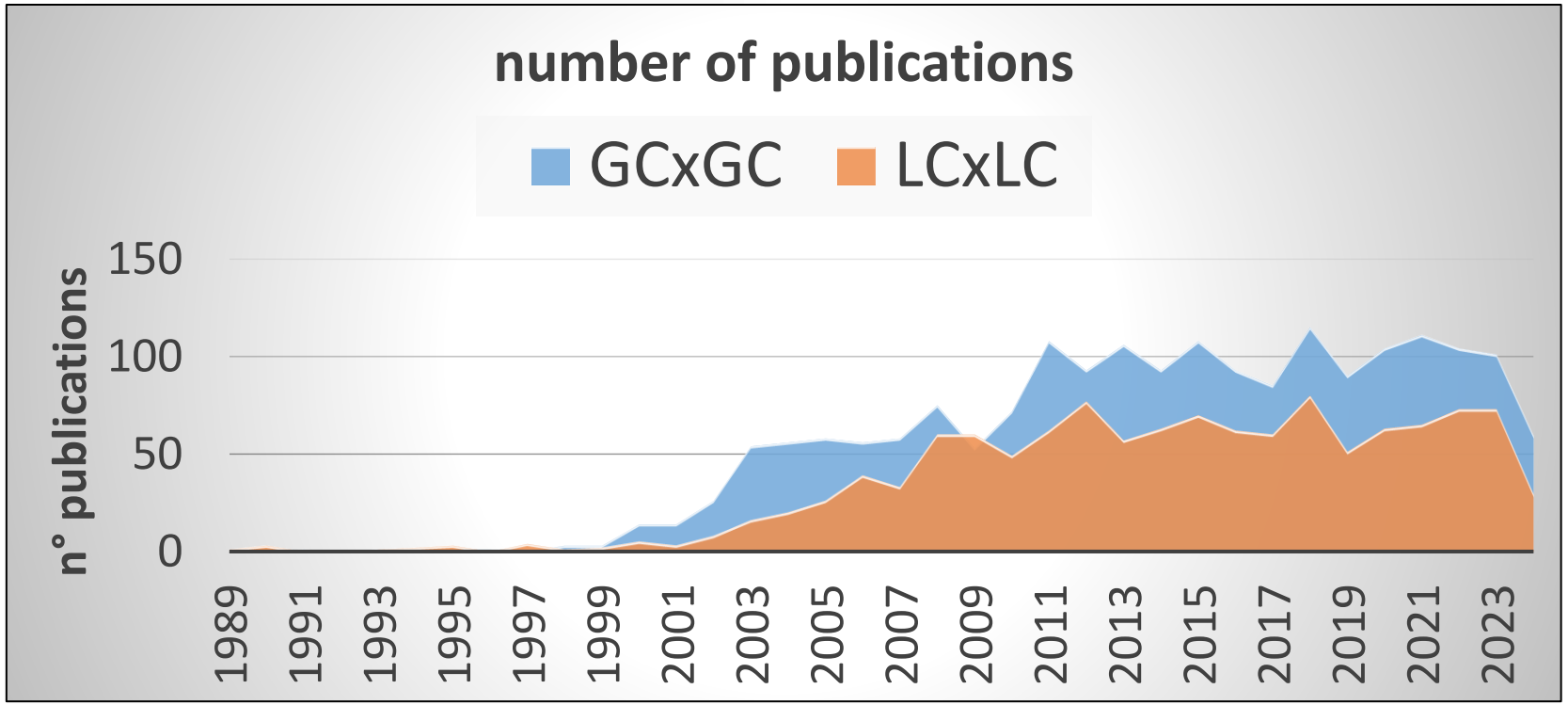


Session

10B: Unconventional hyphenated separation modes

Time: **Friday, 31/May/2024: 11:15am - 1:00pm**

LC×LC **GC×GC**



R&D

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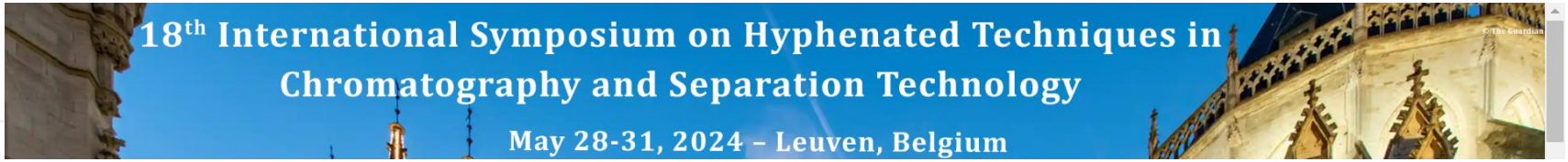

Session

10B: Unconventional hyphenated separation modes

Time: **Friday, 31/May/2024: 11:15am - 1:00pm**

LC-LC/LC×LC

GC-GC/GC×GC

Conventional Hyphenated Separation mode

Unconventional Hyphenated Separation mode

LC-GC

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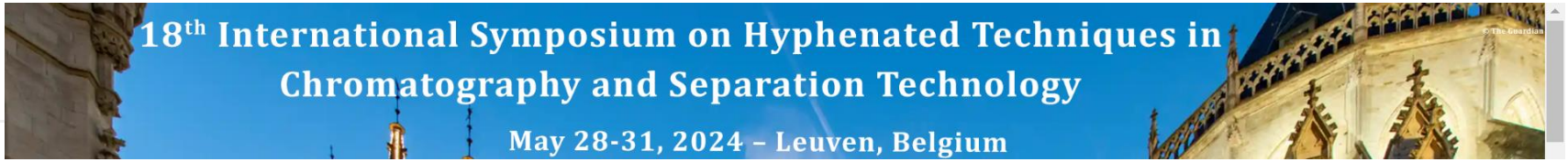
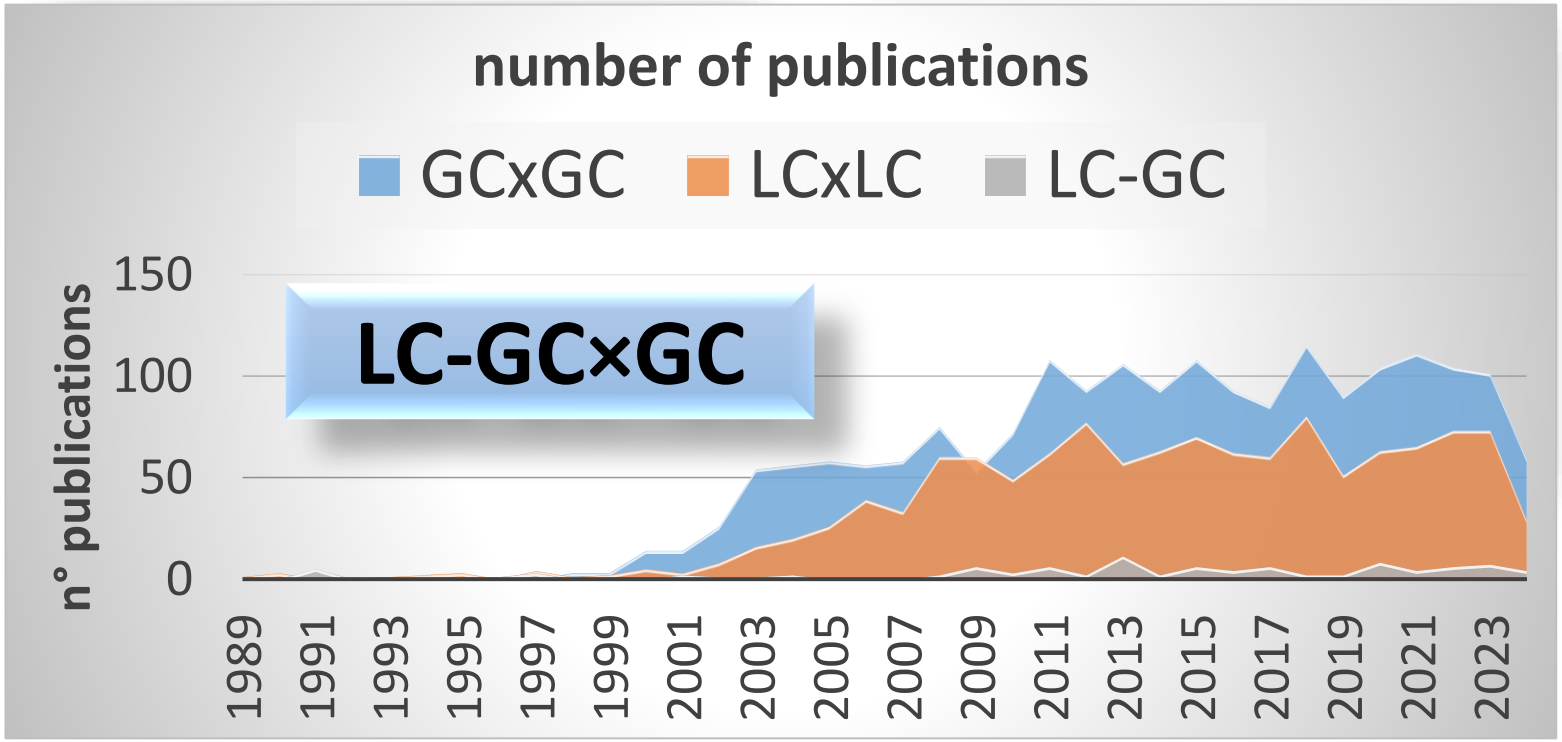


Session

10B: Neglected hyphenated separation modes

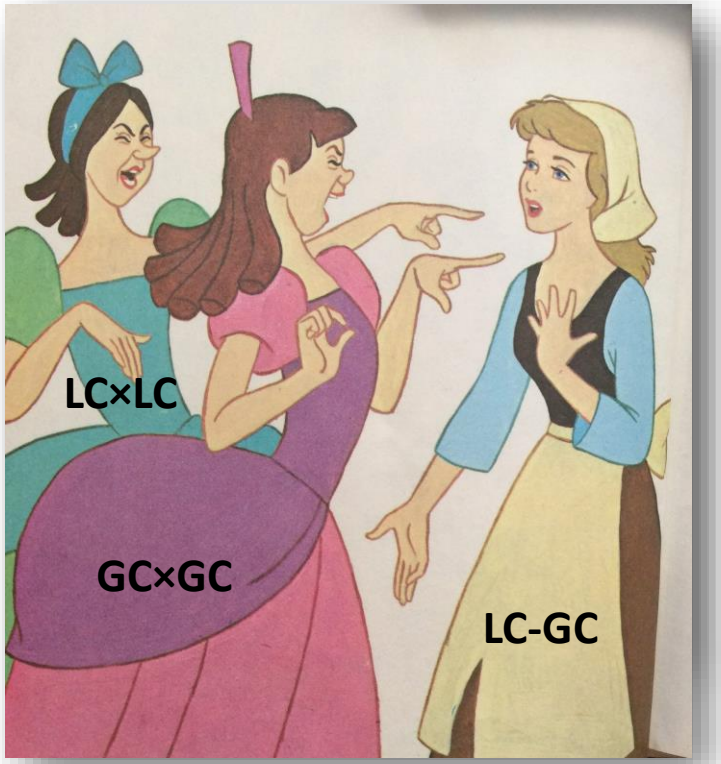
Time: **Friday, 31/May/2024: 11:15am - 1:00pm**

LC-LC/LC×LC **GC-GC/GC×GC**



Unconventional Hyphenated Separation mode

LC-GC



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ROUTINE APPLICATIONS ↔ HYPHENATED TECHNIQUE

Advanced Routine ↔ Complex applications

Mineral oil Hydrocarbons (MOH)

LC-GC(×GC)

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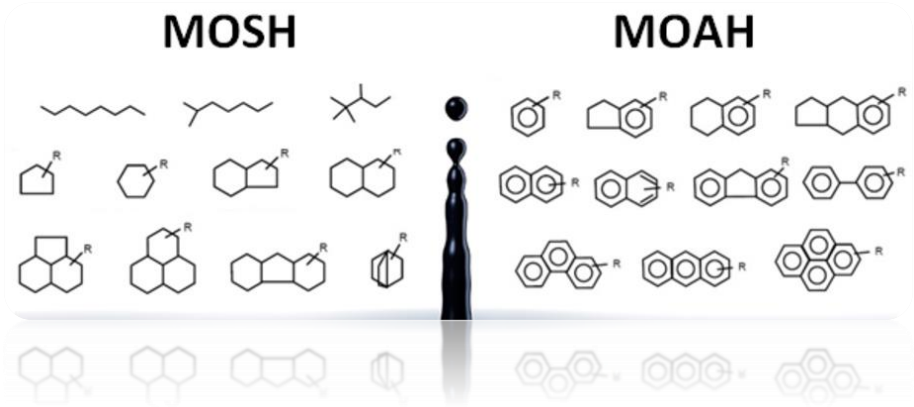
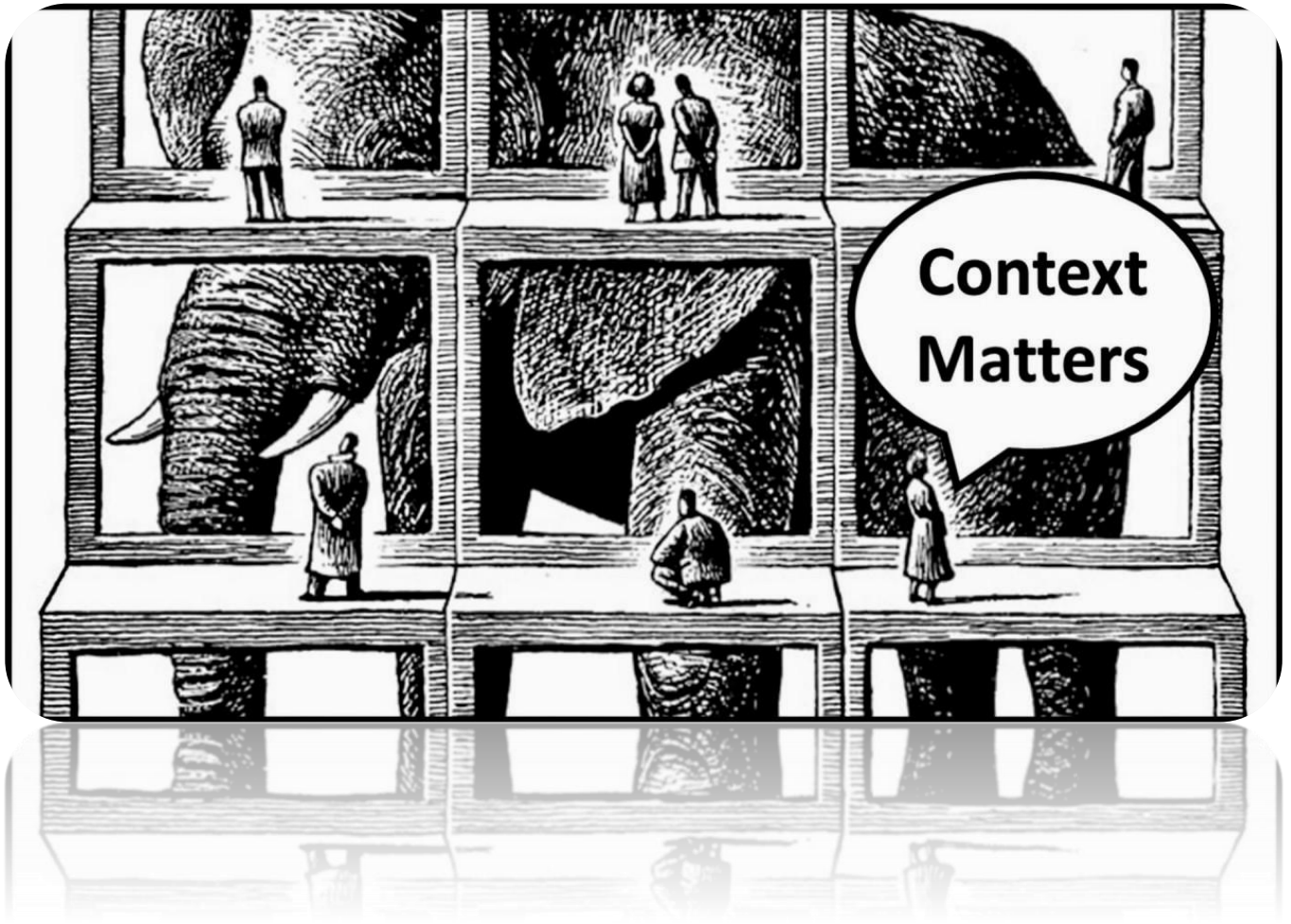
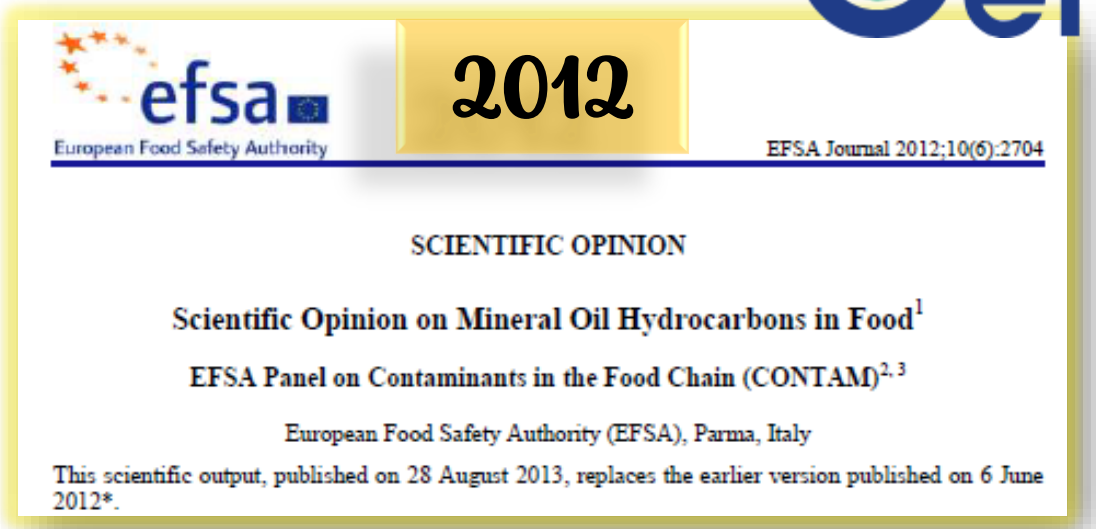
The complexity of MOH analysis





MOSH & MOAH: STATE-OF-THE ART


efsa European Food Safety Authority

2012

EFSA Journal 2012;10(6):2704

SCIENTIFIC OPINION

Scientific Opinion on Mineral Oil Hydrocarbons in Food[1]

EFSA Panel on Contaminants in the Food Chain (CONTAM)[2, 3]

European Food Safety Authority (EFSA), Parma, Italy

This scientific output, published on 28 August 2013, replaces the earlier version published on 6 June 2012*.

MOSH


SCIENTIFIC OPINION

2023

efsa JOURNAL

ADOPTED: 12 July 2023

doi: 10.2903/j.efsa.2023.8215

Update of the risk assessment of mineral oil hydrocarbons in food

EFSA Panel on Contaminants in the Food Chain (CONTAM),

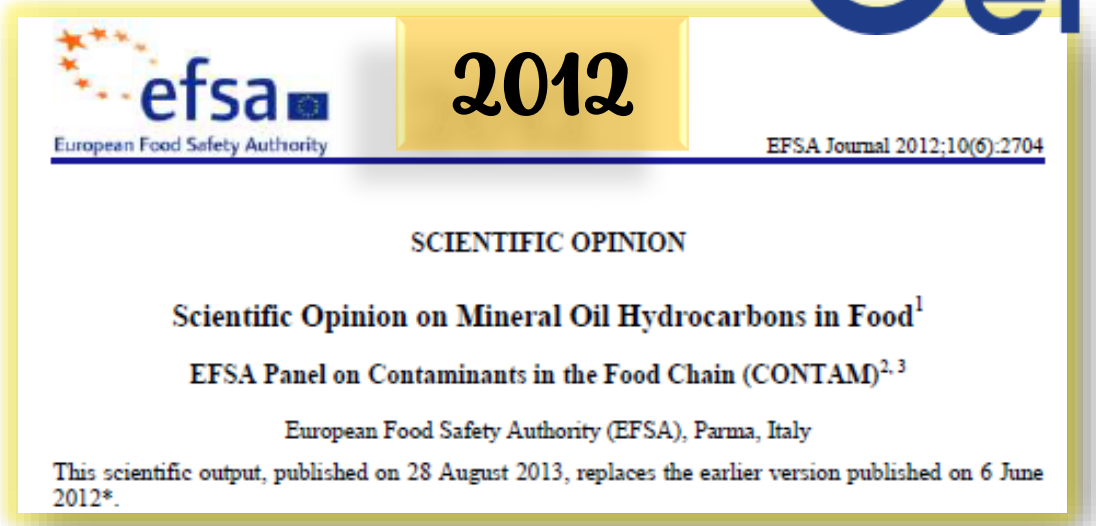
MOAH

Generation of further data for the refinement of the risk assessment is needed

- Generally considered of **no concern** at the concentration found.

- **genotoxicity** of **MOAH with ≥3 aromatic rings**
- in the absence of reliable toxicity data, the dietary exposure to **1–2 ring MOAH might raise a concern**

MOSH & MOAH: STATE-OF-THE ART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MOSH


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MOAH

Generation of further data for the refinement of the risk assessment is needed

➢**Improvement of analytical methodology**

for better characterisation of MOSH&MOAH and **consistency in reporting**

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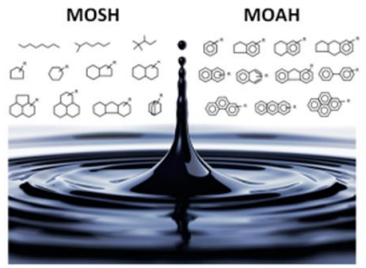
ROUTINE METHOD FOR MOH DETERMINATION



LC-GC

LC

Sample preparation step

✓ Purification form TAGs

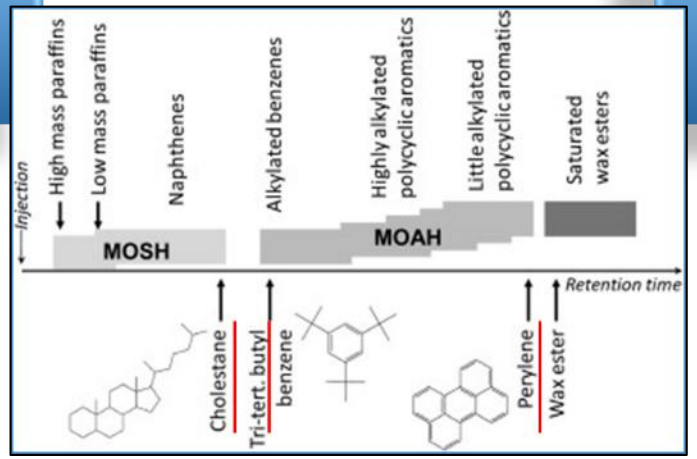
✓ Pre-fractionation

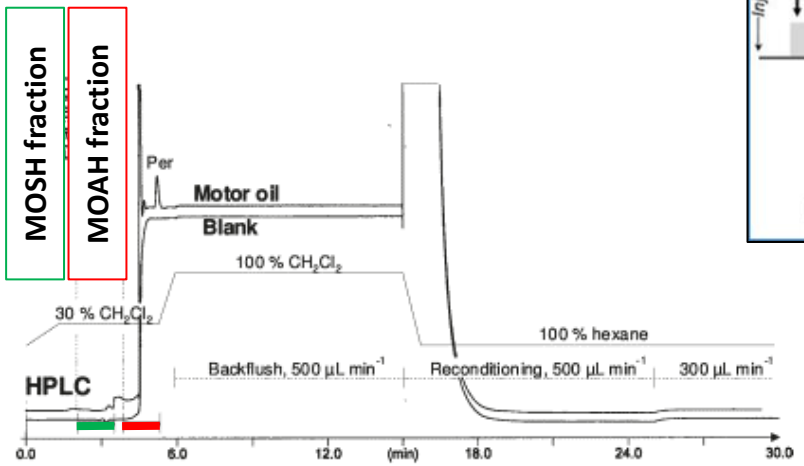
GC

Analytical Determination

✓ Analytical Separation

✓ Quantification





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

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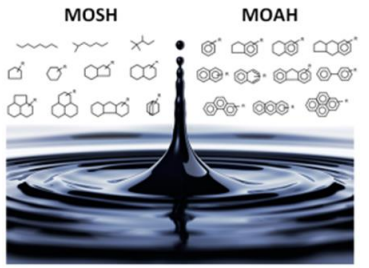
ROUTINE METHOD FOR MOH DETERMINATION



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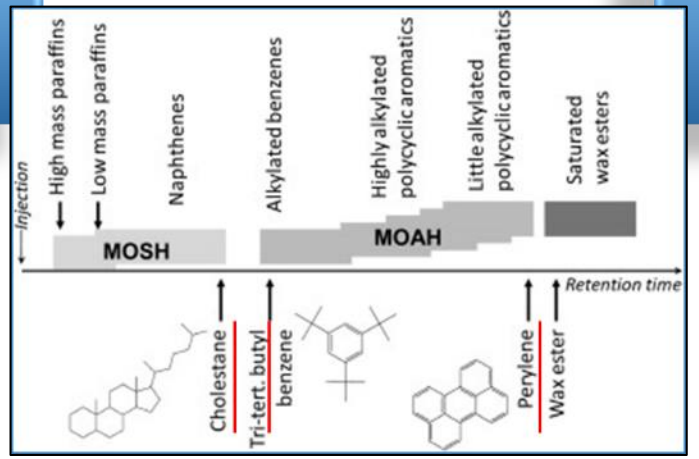

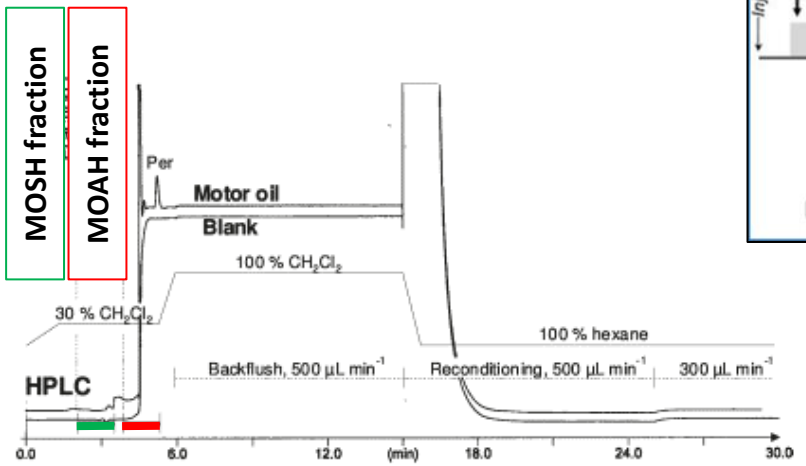

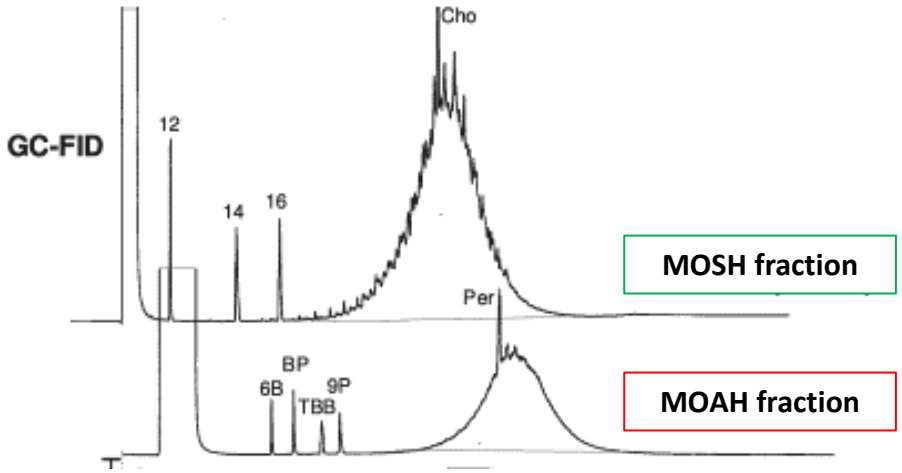

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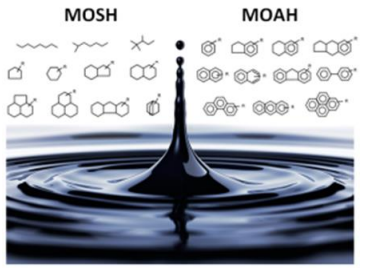
ROUTINE METHOD FOR MOH DETERMINATION



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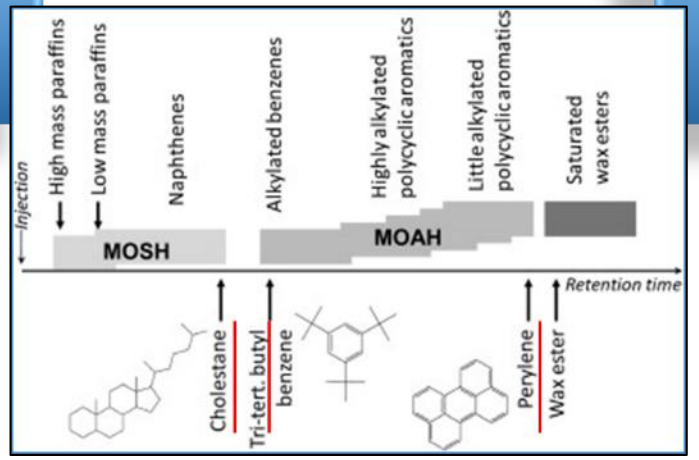

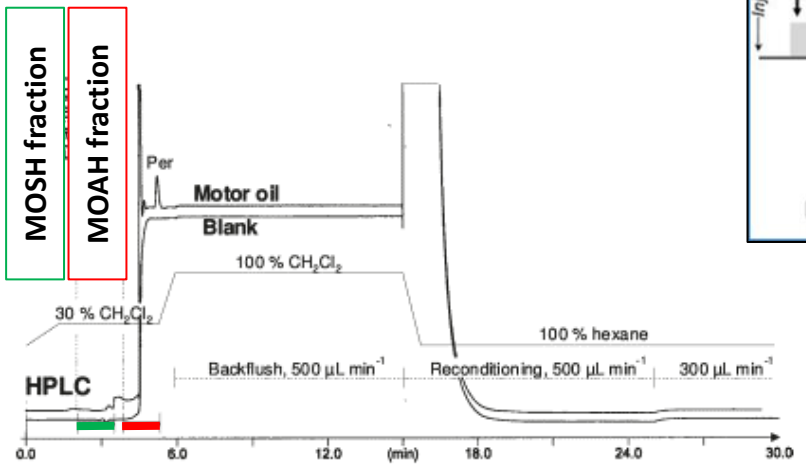

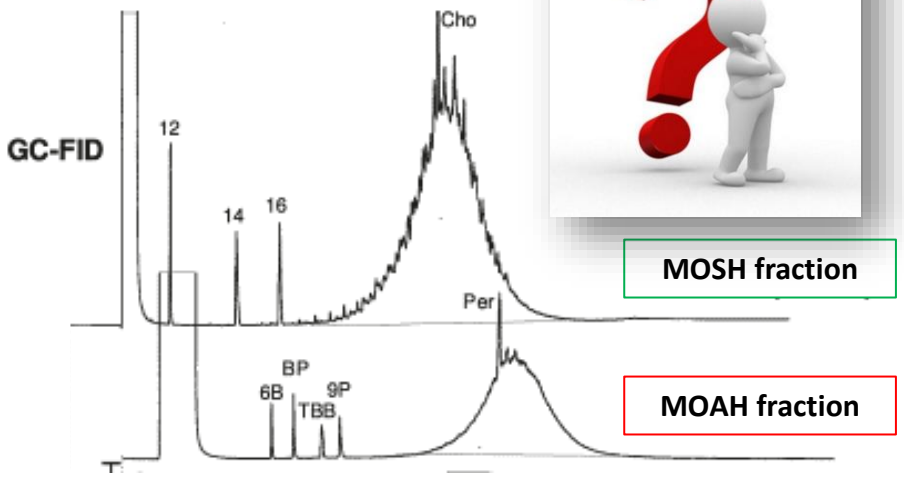

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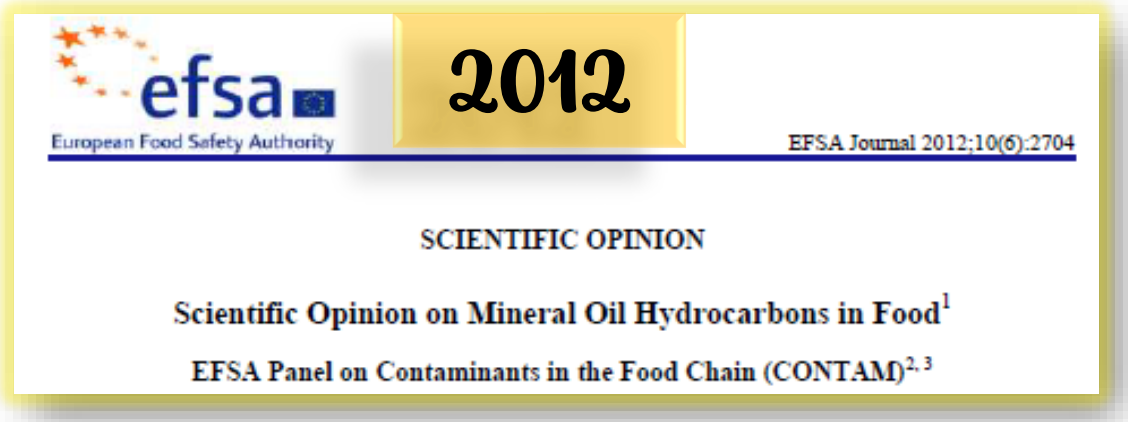
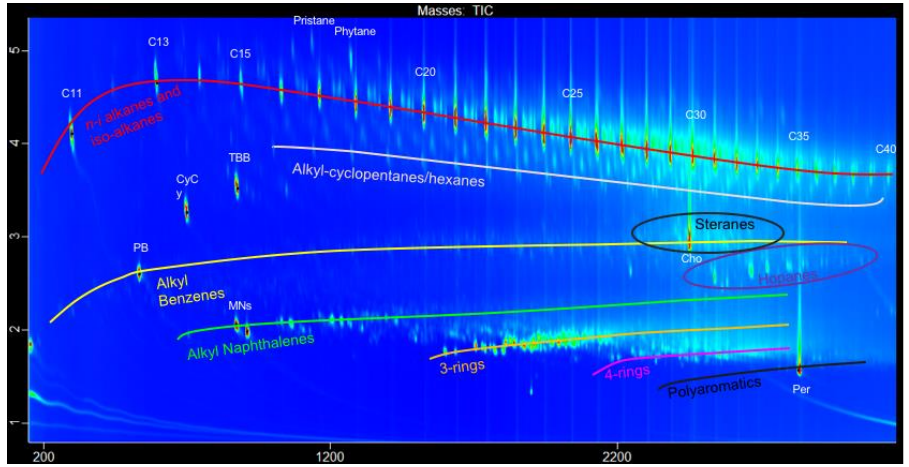
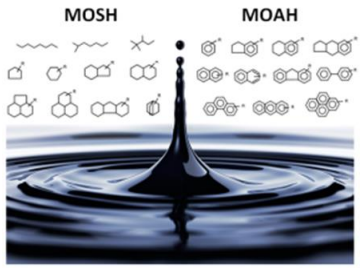
MOSH & MOAH: STATE-OF-THE ART





Currently, the most efficient methods for analysis of MOSH and MOAH in food and feed comprise extraction followed by pre-separation by **high performance liquid chromatography (HPLC) on-line coupled to GC with flame ionisation detection (FID)**. Detection limits depend on the mass distribution, the sample matrix and any prior enrichment, and can be as low as 0.1 mg/kg. **Comprehensive GC×GC-FID** enables a rough separation and quantification of paraffins and naphthenes in the MOSH fraction, but it is of limited practicality for routine analysis. Contamination with polyolefin oligomeric saturated hydrocarbons (POSH), e.g. from plastic bags, heat sealable layers or adhesives, may interfere with MOSH analysis. Analytical capacity to distinguish the different MOAH subclasses in food is limited. For this purpose, **GC×GC appears to be the most effective method**. Due to the complexity and the variable composition of MOH mixtures, it is not possible to define certified standards of general applicability.

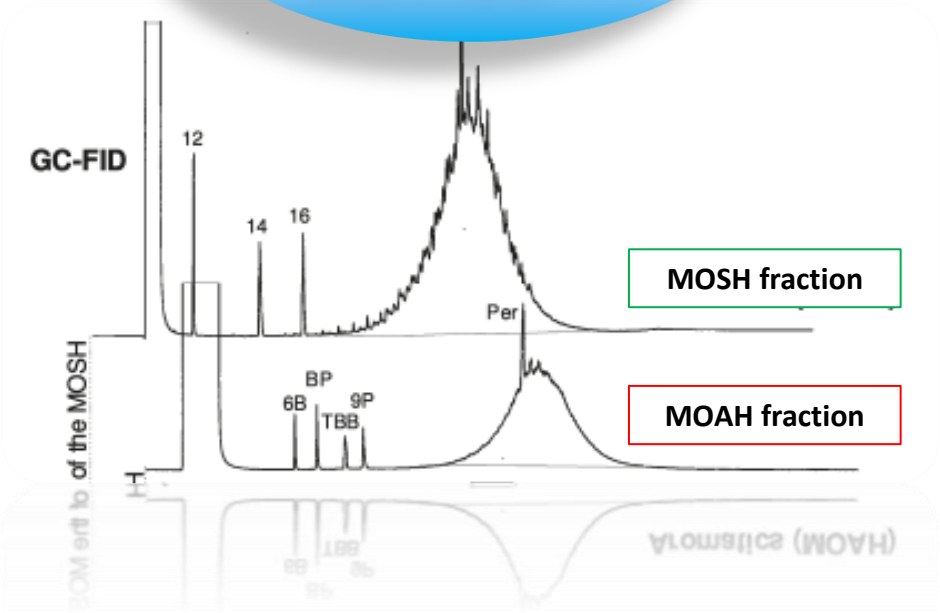


MOSH & MOAH: STATE-OF-THE ART

MOH

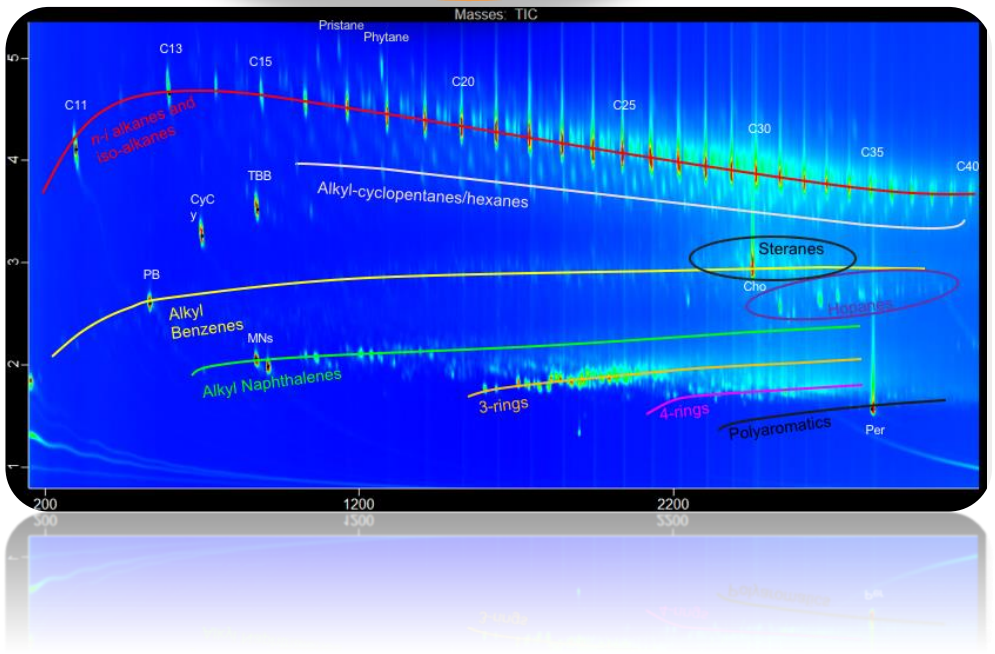
LC-GC

ROUTINE METHOD



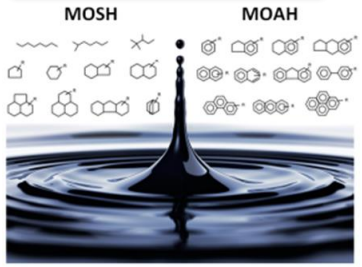
GC×GC

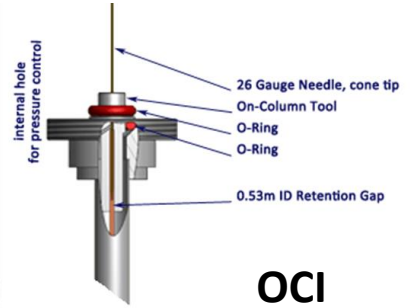
CONFIRMATORY METHOD



2021

MOH AND MULTIDIMENSIONAL TECHNIQUEs





OCI

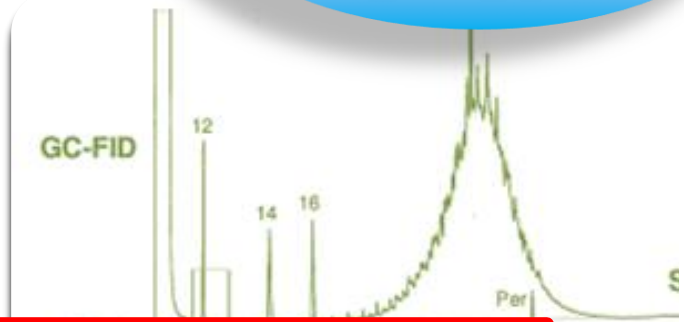
MOH

LC-GC

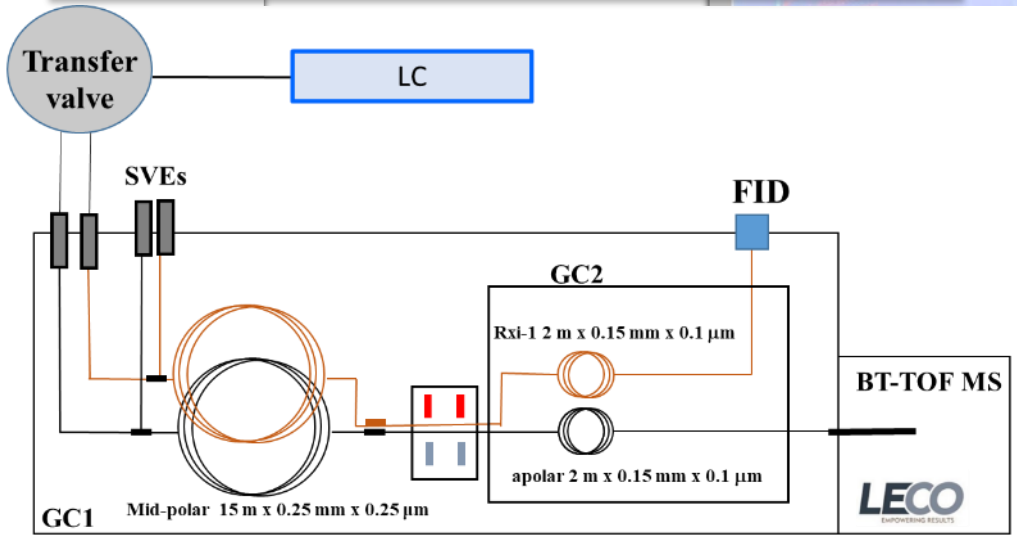
ROUTINE METHOD

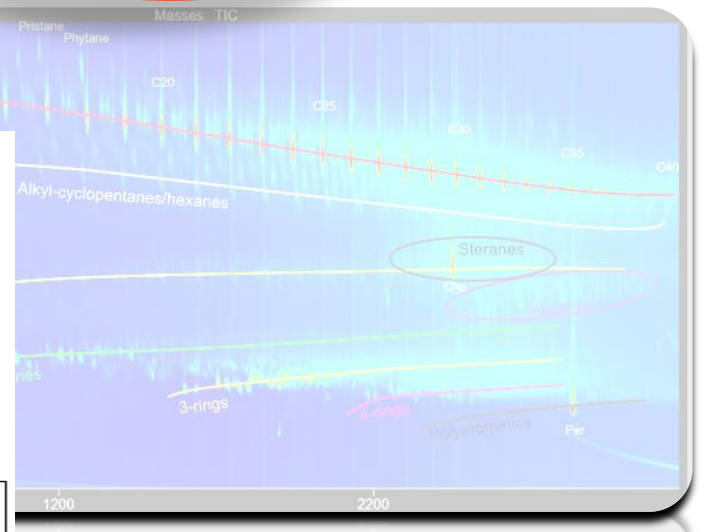
GC×GC

CONFIRMATORY METHOD



LC-2GC×GC-FID/MS





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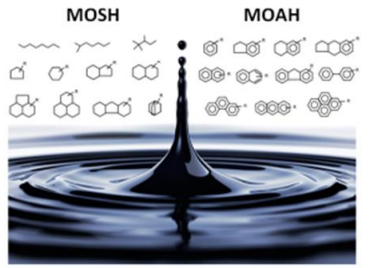
MOH & DATA INTEGRATION

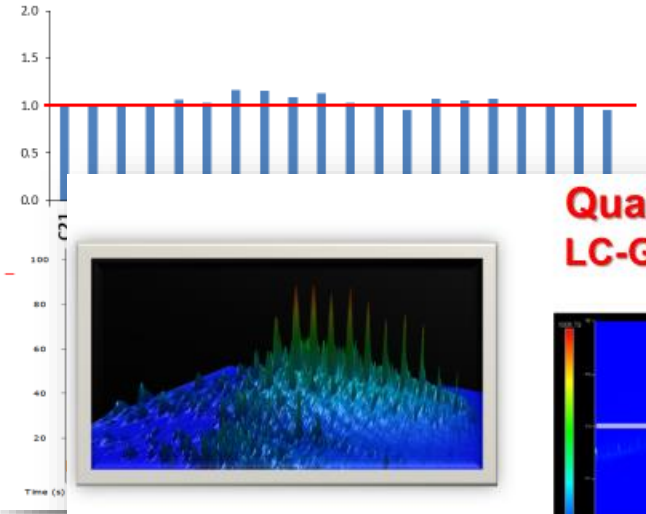


Quantification

Paraffin Mixture + n-Alkanes Mix

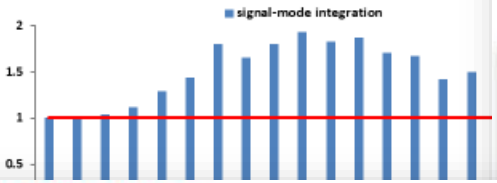
LC-GC-FID

1D



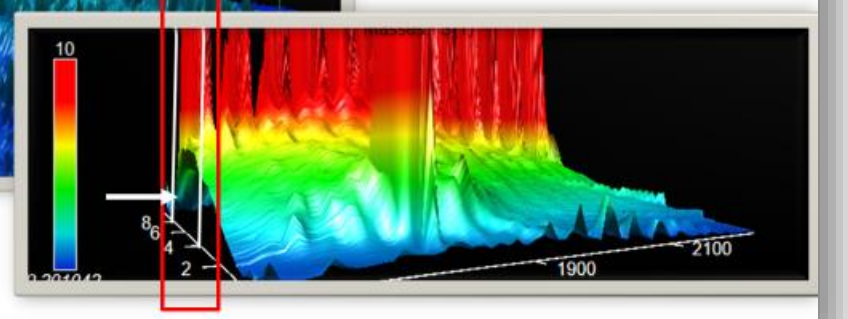
LC-GC×GC-FID

2D

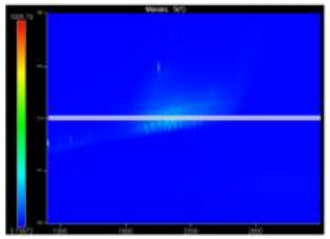


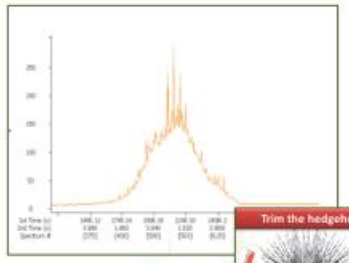
Quantification
LC-GC×GC-FID

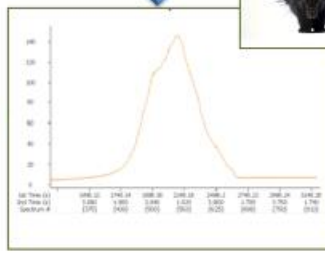
Integration of part of the hump below the alkane as alkane

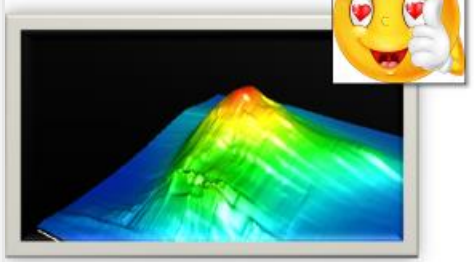


Quantification
LC-GC×GC-FID*









*patent pending

Quantitative advantages of the GC×GC over the GC

- "…confirmatory measurements can help to verify whether the compounds in the sample are of MO origin, but they do not verify the quantitative data themselves. Presently, the most powerful method for characterization of the MOSH and MOAH humps is **GC × GC**."

GC×GC's higher separation power is useful for **quantitatively determining MOSH and MOAH** and **sub-class determination**

Journal of Chromatography A 1643 (2021) 462044

Contents lists available at ScienceDirect

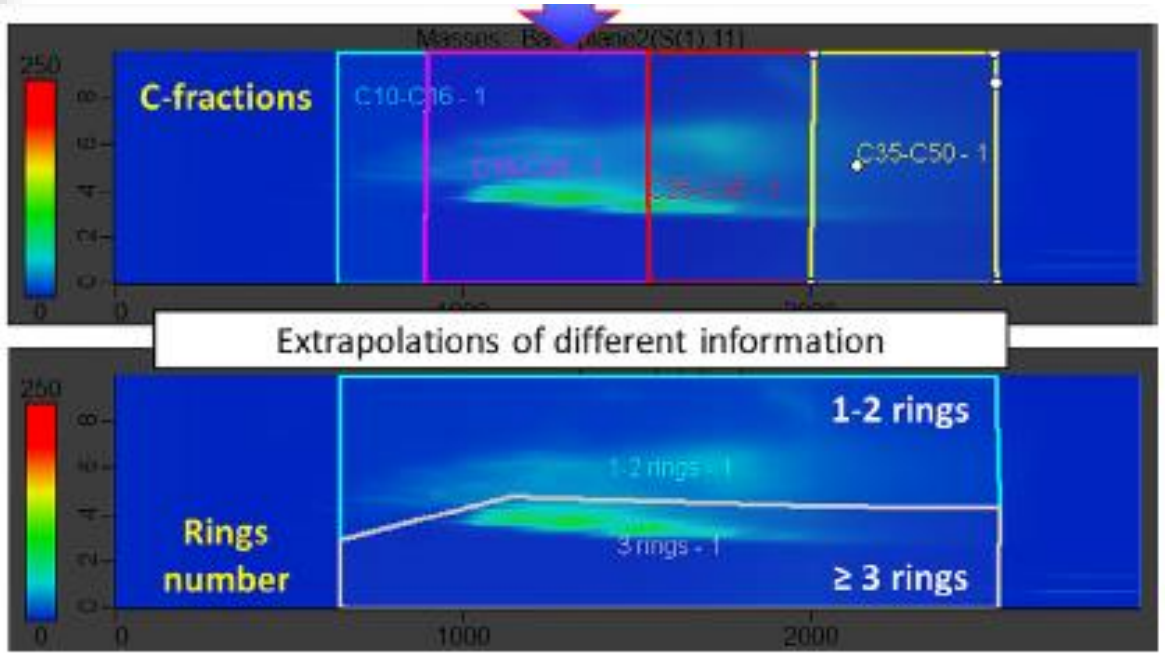
Journal of Chromatography A

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

Mineral oil saturated and aromatic hydrocarbons quantification: Mono- and two-dimensional approaches

Grégory Bauwens[a], Sebastiano Pantó[b], Giorgia Purcaro[a,*]

[a] Analytical Chemistry Lab, Gembloux Agro-Bio Tech, University of Liège, Gembloux, 5030, Belgium
[b] LECO European Application and Technology Center (EATC), Berlin, Germany



MOSH & MOAH sub-classes quantification

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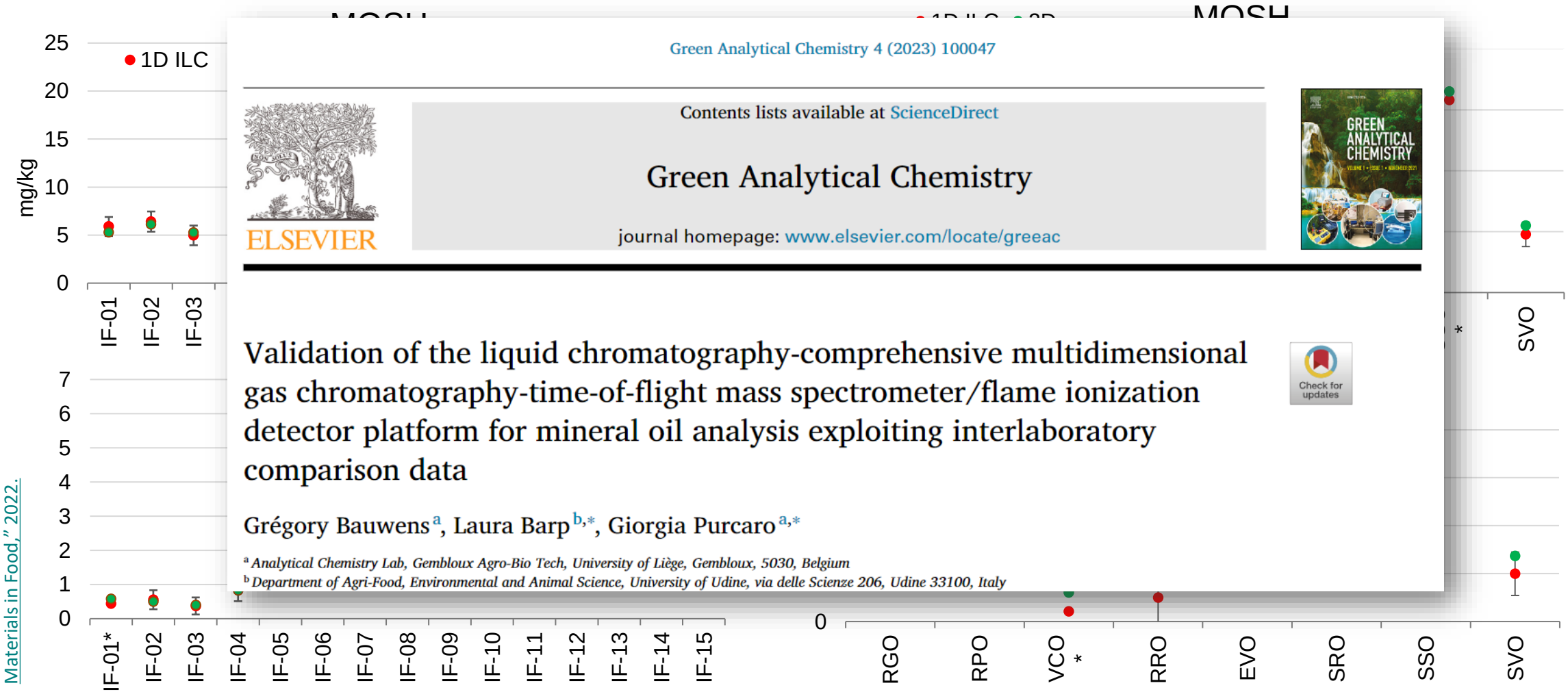
LC-GC×GC-FID/MS

Data Integration

Data Interpretation

MOAH in infant formula (IF)
"JRC-IF-2022/05"

revision of DGF-EN 16995:2017



Green Analytical Chemistry 4 (2023) 100047

Contents lists available at ScienceDirect

Green Analytical Chemistry

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

Validation of the liquid chromatography-comprehensive multidimensional gas chromatography-time-of-flight mass spectrometer/flame ionization detector platform for mineral oil analysis exploiting interlaboratory comparison data

Grégory Bauwens [a], Laura Barp [b,*], Giorgia Purcaro [a,*]

[a] Analytical Chemistry Lab, Gembloux Agro-Bio Tech, University of Liège, Gembloux, 5030, Belgium
[b] Department of Agri-Food, Environmental and Animal Science, University of Udine, via delle Scienze 206, Udine 33100, Italy

Check for updates

S. Bratinova, Outcome from the collaborative trial for method validation "MOAH in IF," in: S. Bratinova (Ed.), 11th International Akademie Fresenius Conference "Residues of Food Contact Materials in Food," 2022.

Vegetable oils - Determination of mineral oil saturated hydrocarbons (MOSH) and aromatic hydrocarbons (MOAH) with online coupled HPLC-GC-FID analysis -Method for low limit of quantification. Draft version for updating EN-16995:2017, October 2022-V5.

G. Bauwens, L. Barp, G. Purcaro, *Validation of the liquid chromatography-comprehensive multidimensional gas chromatography-time-of-flight mass spectrometer/flame ionization detector platform for mineral oil analysis exploiting interlaboratory comparison data*, **GreenAC (2023) 4 100047**

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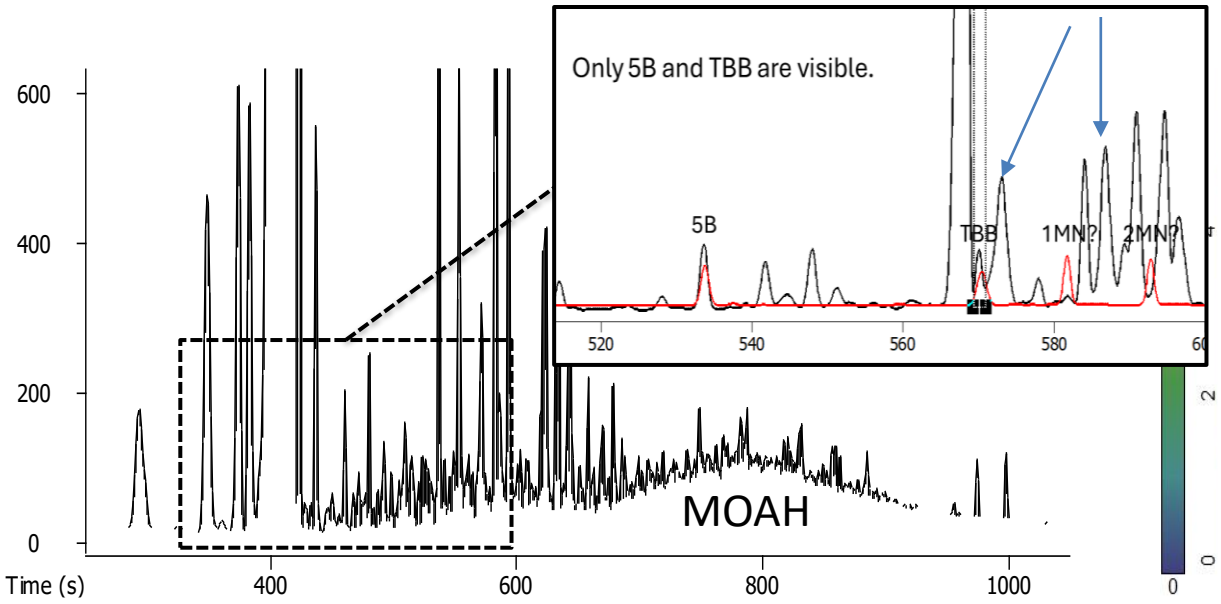
Quantitative advantages of the GC×GC over the GC

- "…confirmatory measurements can help to verify whether the compounds in the sample are of MO origin, but they do not verify the quantitative data themselves. Presently, the most powerful method for characterization of the MOSH and MOAH humps is **GC × GC**."

GC×GC's higher separation power is useful for **more accurately determining MOSH and MOAH** and **sub-class determination**

MOAH analysis by GC-FID

IS for quantification



Internal standards coeluted with interferences

→ impossible MOAH quantification

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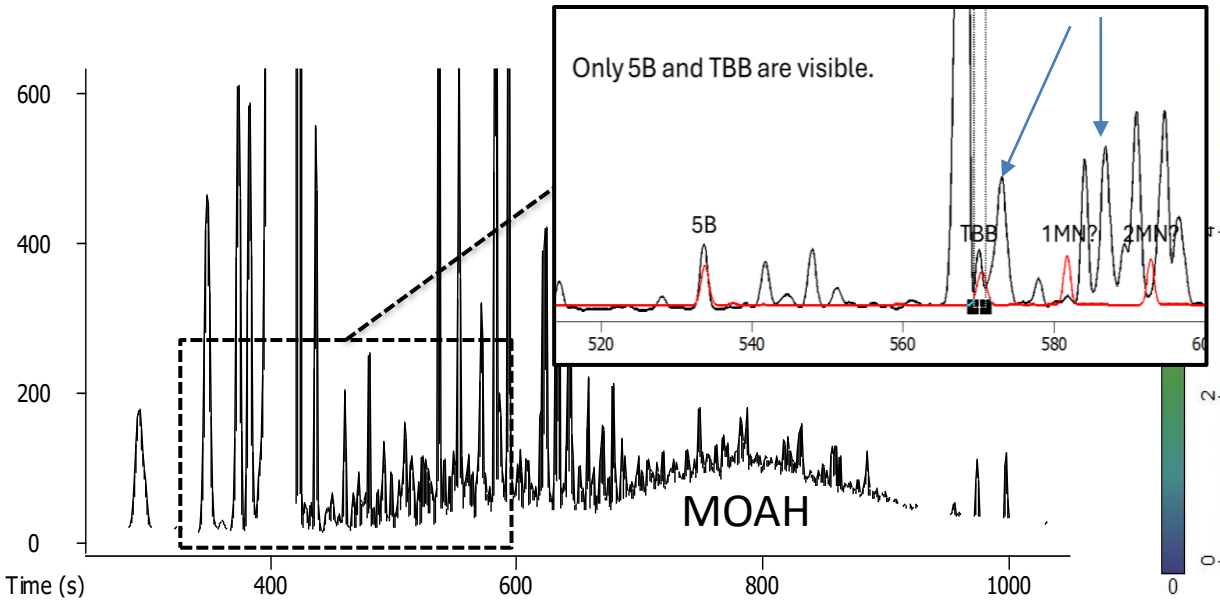
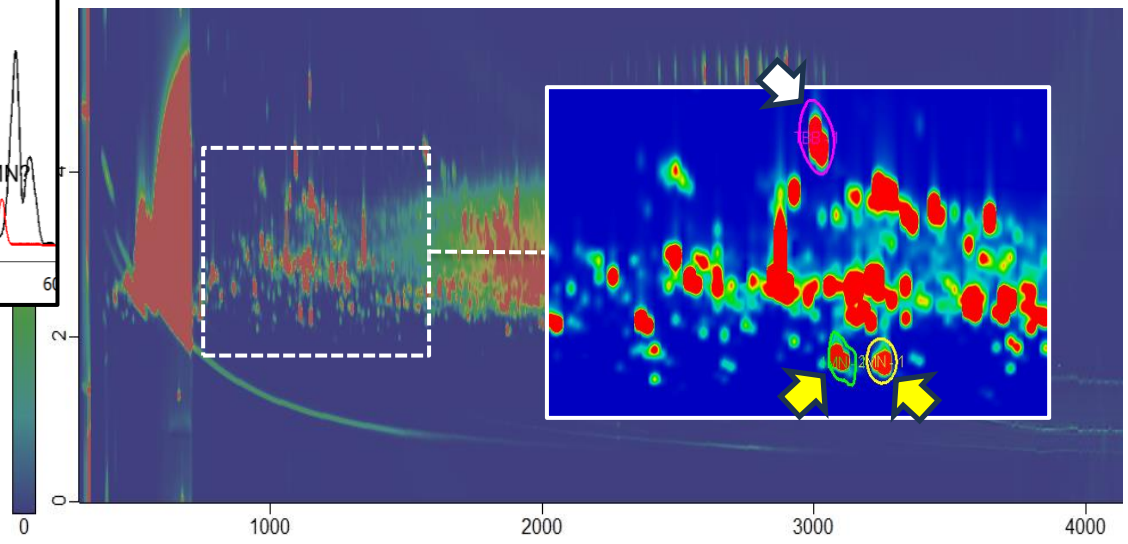


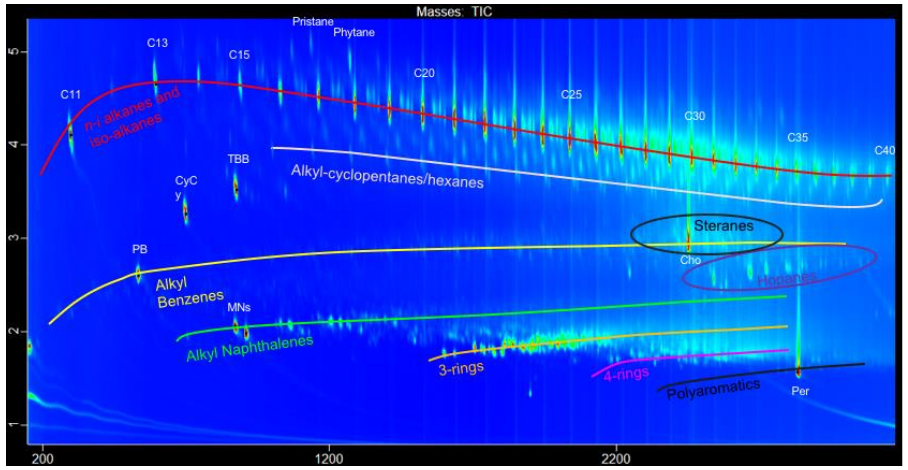
MOAH analysis by GC×GC-FID



Internal standards coeluted with interferences
→ impossible MOAH quantification

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

MOSH & MOAH: STATE-OF-THE ART



2023

SCIENTIFIC OPINION

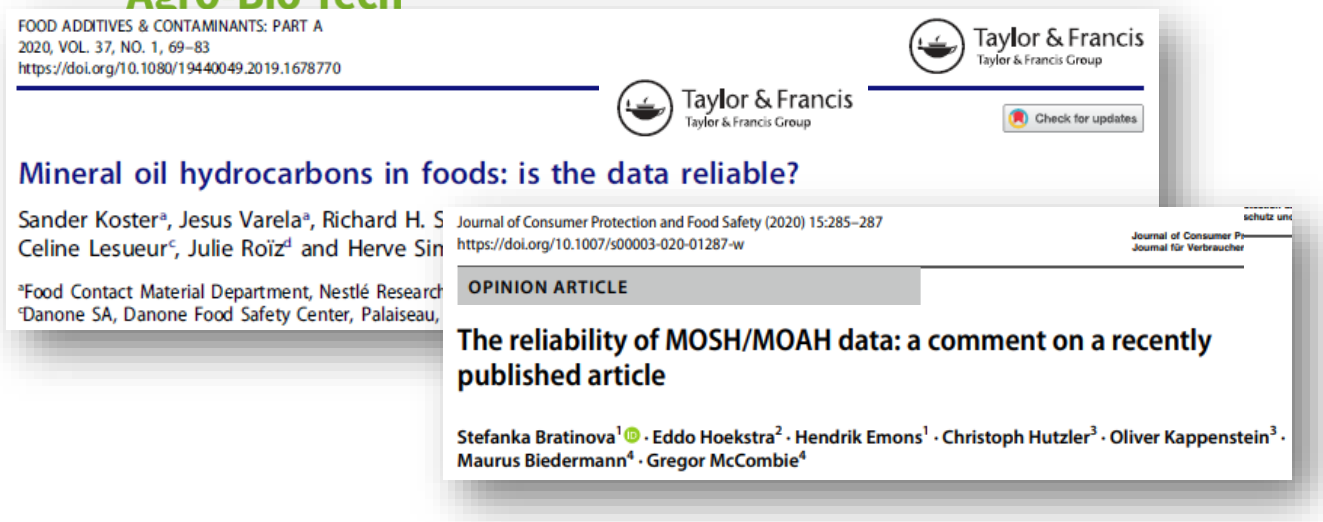
efsa JOURNAL

Update of the risk assessment of mineral oil hydrocarbons in food

EFSA Panel on Contaminants in the Food Chain (CONTAM),

"…Over the last 10 years, **progress in compositional analysis** has been achieved through comprehensive two-dimensional GC (GC × GC) with FID and MS. The main features of **GC × GC** are not only significantly **better separation** and **lower detection limits**, but also placing structurally related compounds in an order, e.g. MOAH according to the number of aromatic rings. In this way, GC × GC may **provide structural information** if just a single compound, or even no compound of the series could be identified owing to lacking standards or reference mass spectra. …"

MOSH & MOAH: STATE-OF-THE ART



"…There have been, and still are, **discussions about the reliability of the results,** particularly for measurements at low concentrations. The LC-GC-FID method can be considered as standard and reliable, validated by collaborative tests. Nonetheless, sometimes there were large differences in the results from different laboratories. There were several reasons for this."

(i) Blank and cross-contamination due to the **ubiquitous presence** of MOH.

(ii) Interference removal is a critical step ↔ at lower **limit of quantitation**

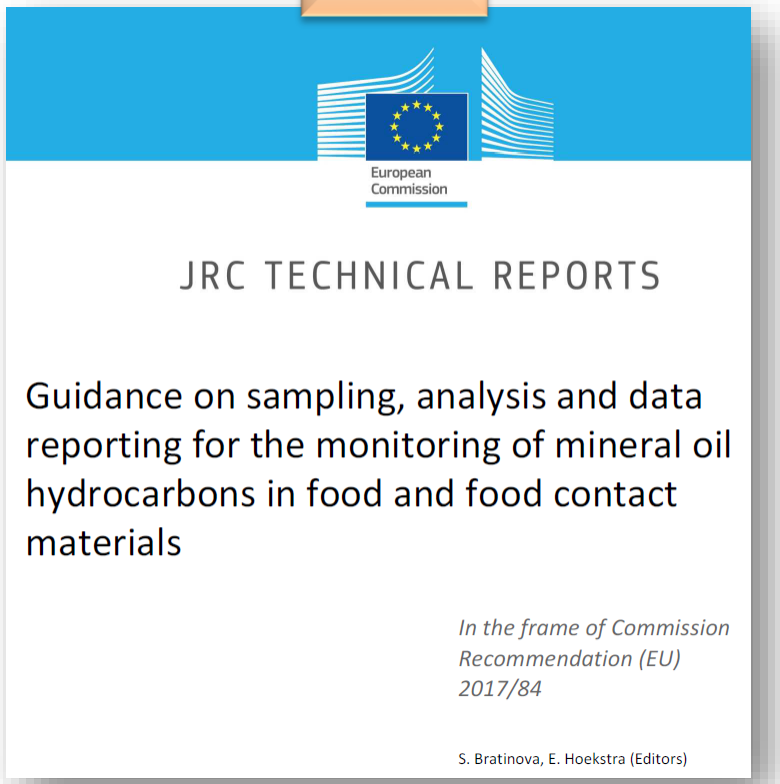
(iii) Chromatogram **interpretation** → needs experience in the interpretation

Consistency in reporting and data reliability

JRC guidance: **harmonised procedures**

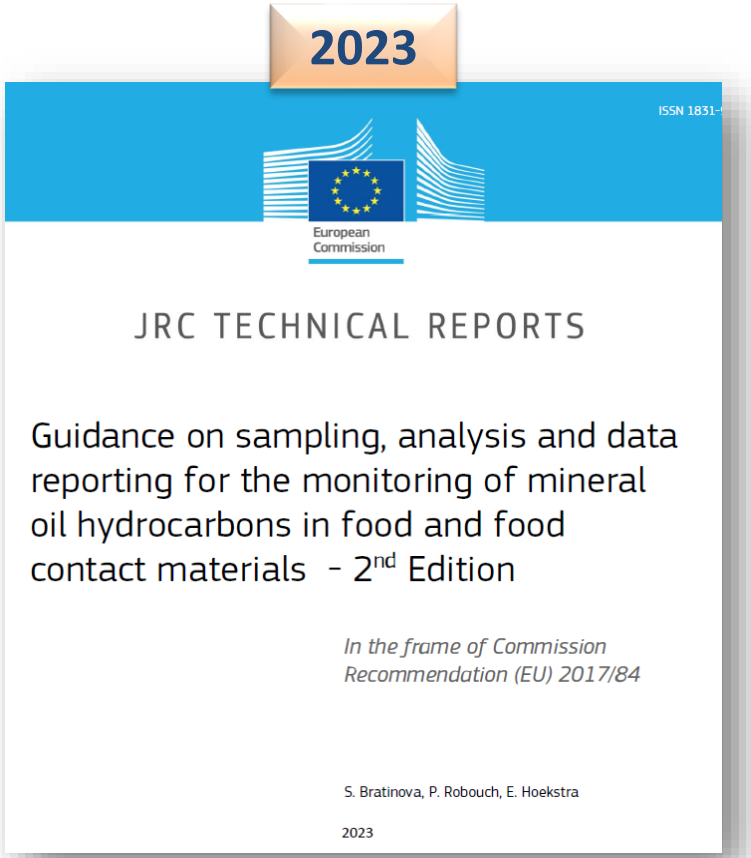
- ✓ for sample prep (decision tree)/standardization
- ✓ C-fraction reported (extended to C50)

2019



2022

2023



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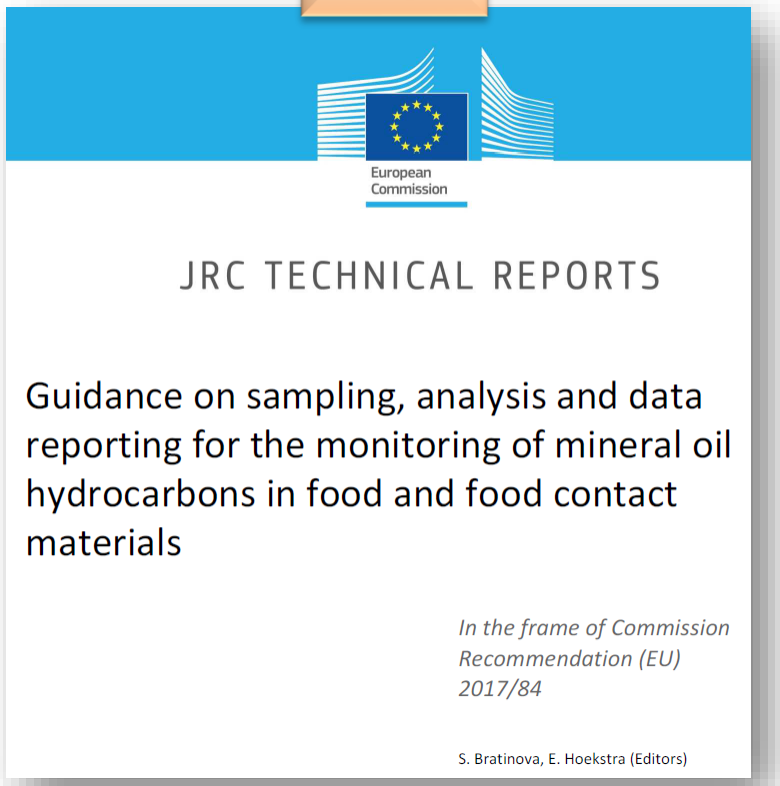
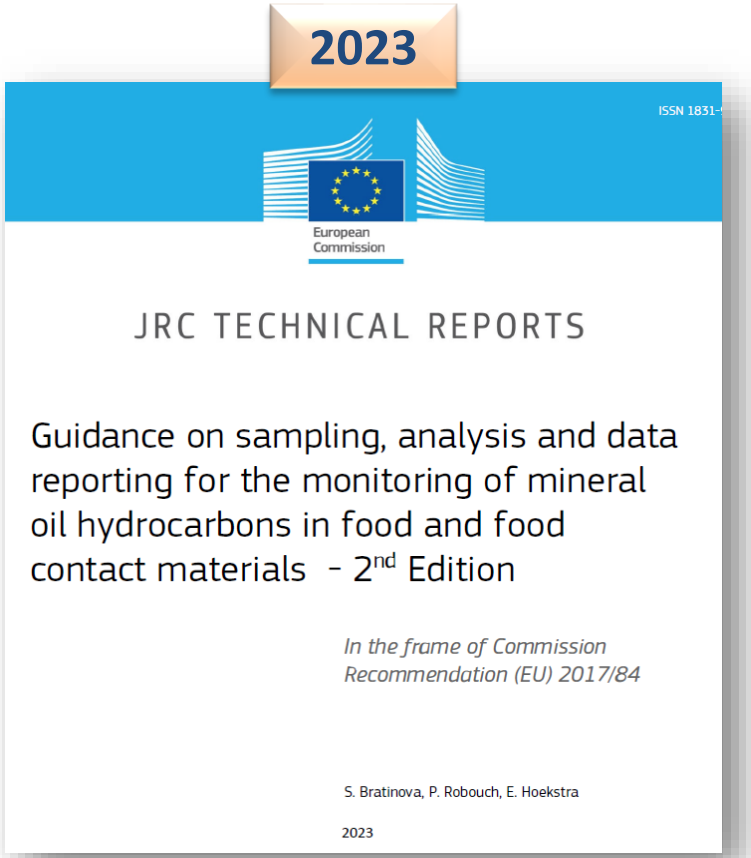


2022

2023



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Outline of the Analytical approach

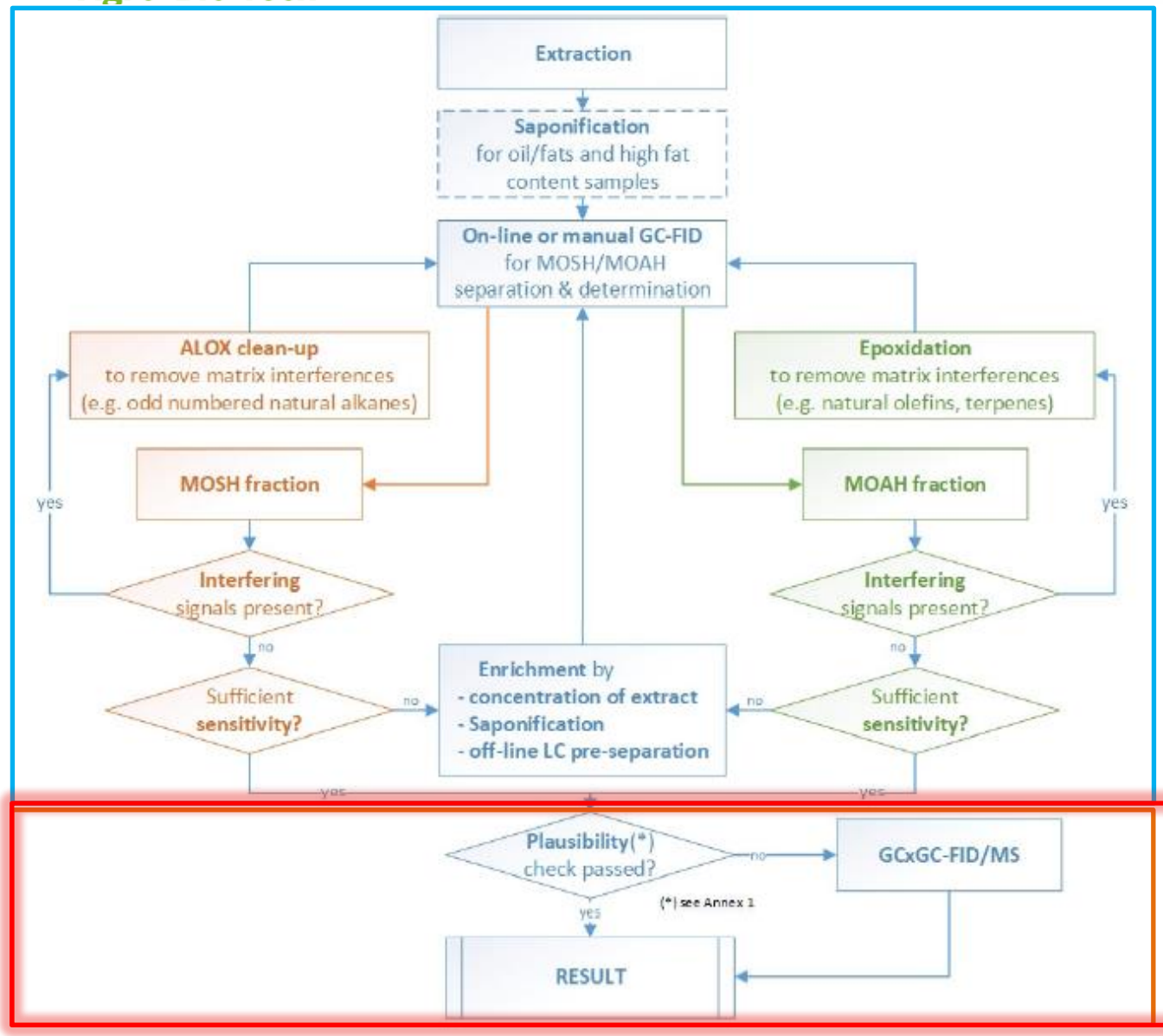
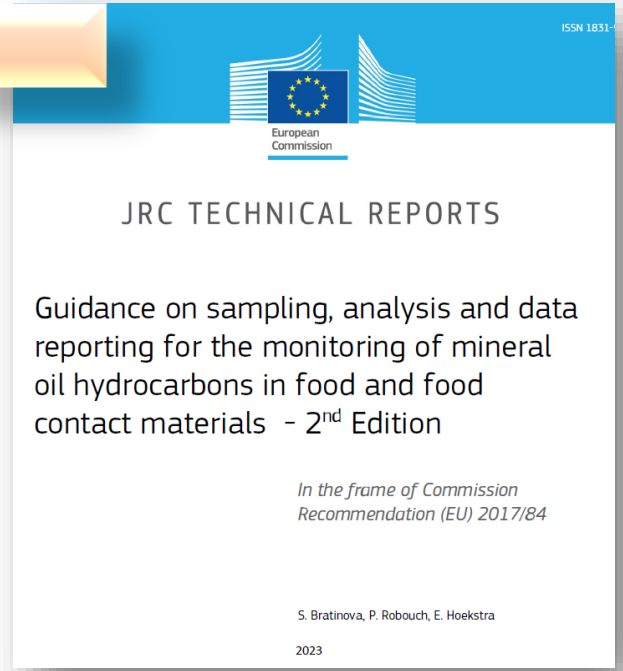


Figure 5 Decision tree on the use of auxiliary methods.

Sample Preparation



✓*Need for matrix-tailored sample prep protocols*

> 20% of uncertainty!

Data Interpretation

Data Integration

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Consistency in reporting and data reliability

> 20% of uncertainty!

Data Interpretation

Data Integration

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

Consistency in reporting and data reliability

> 20% of uncertainty!

Data Interpretation

Data Integration

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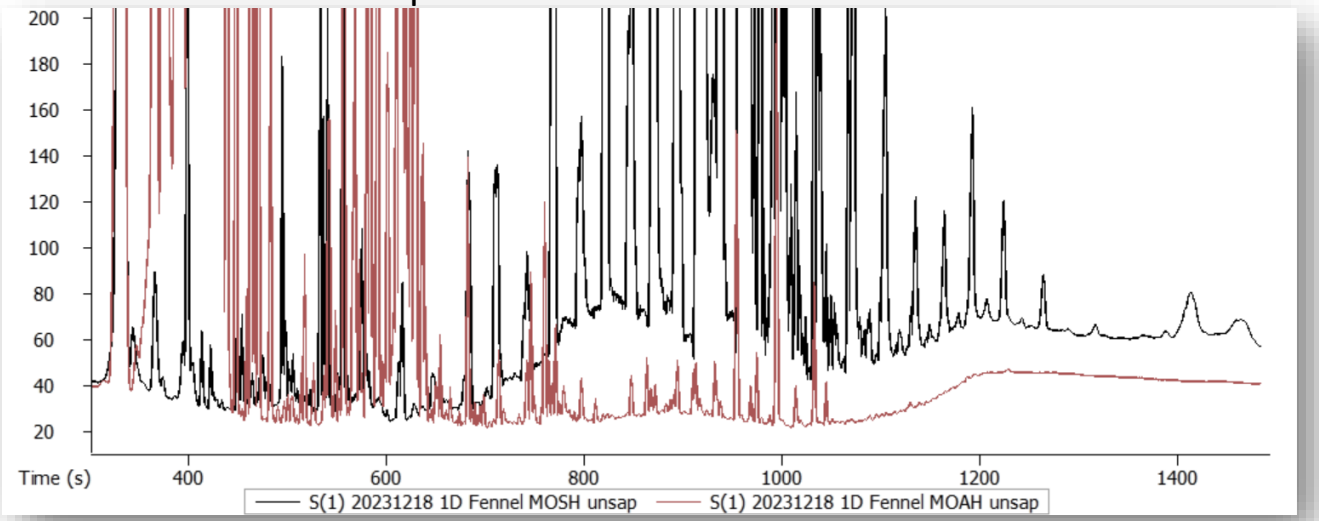
MOSH vs MOAH – No purification



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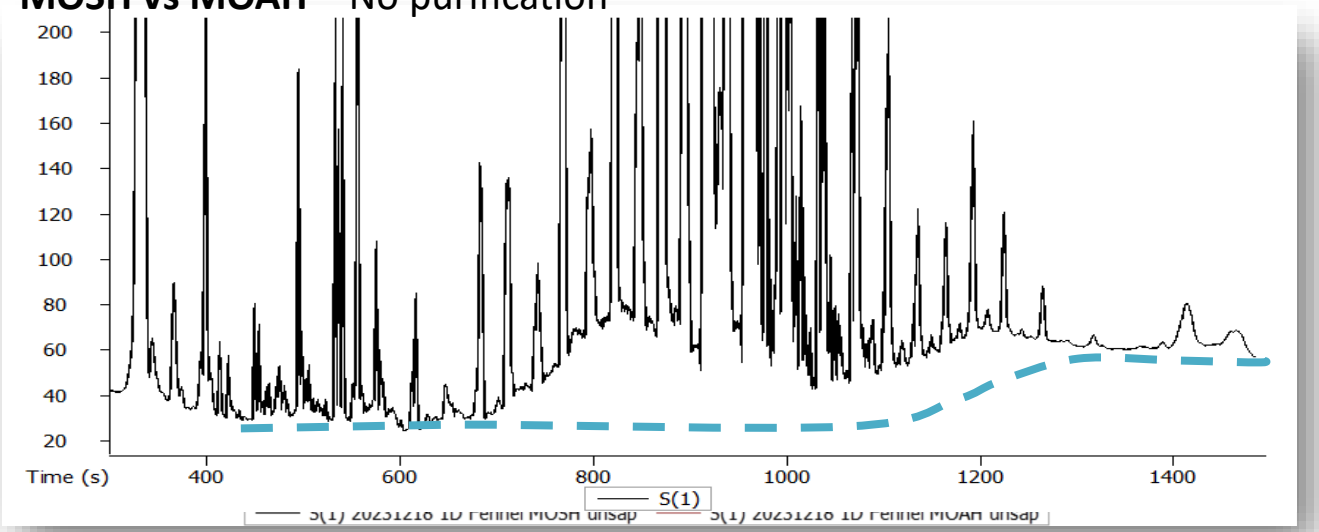

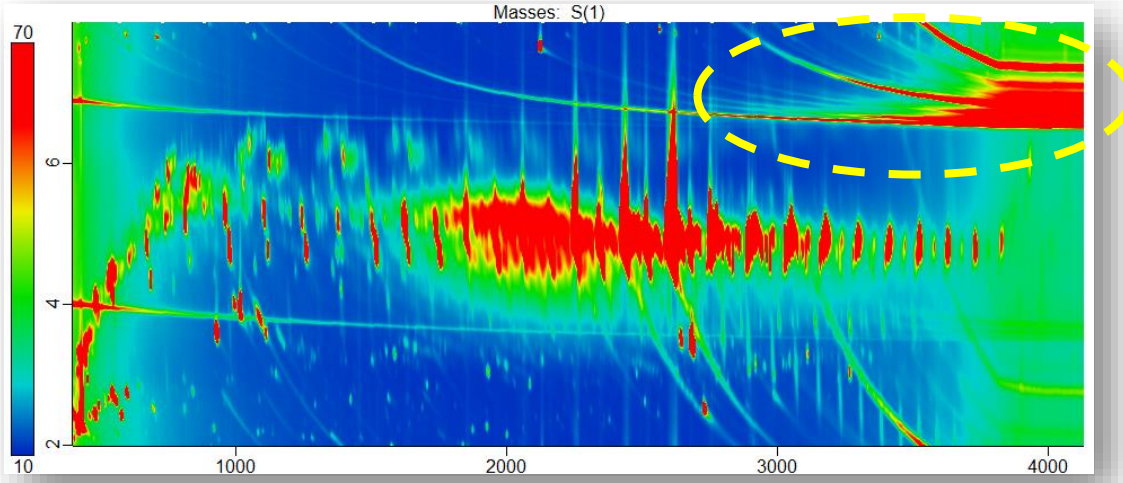

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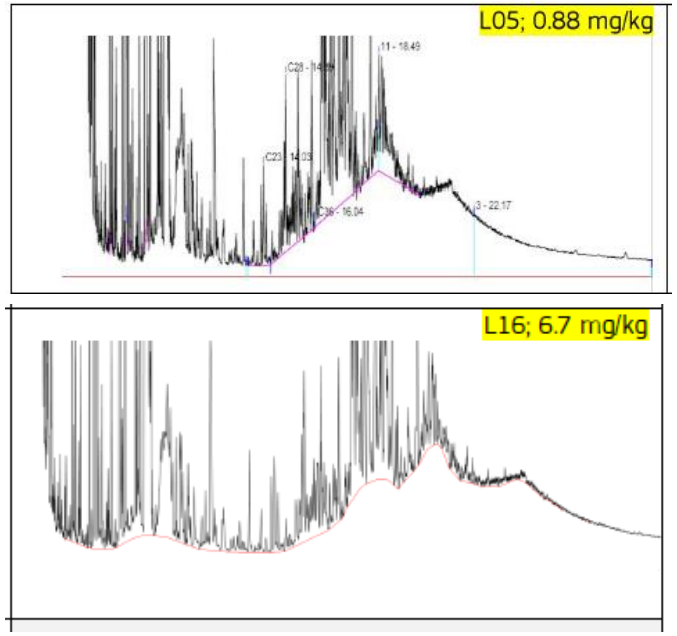
- **Riding peaks subtraction**



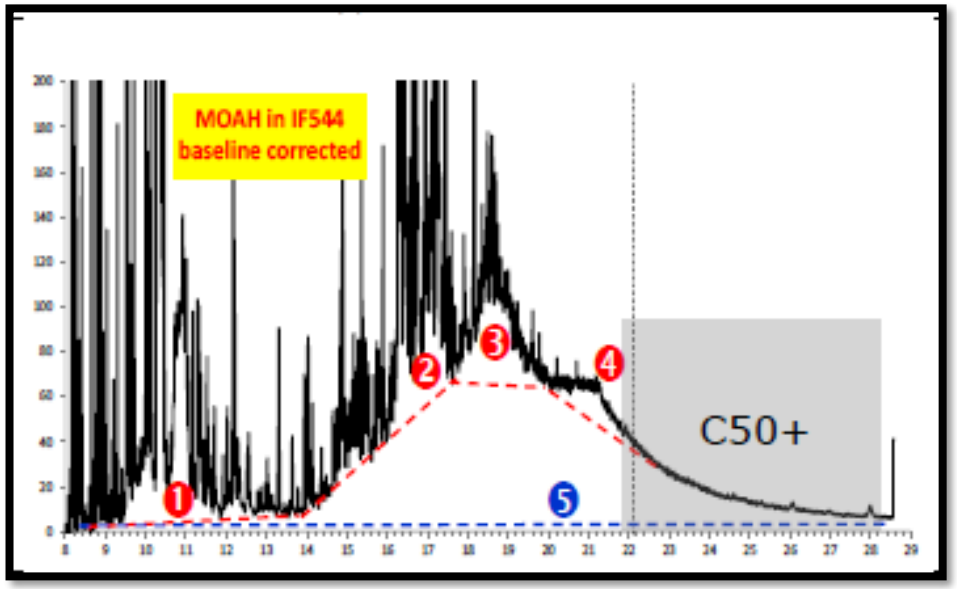


Figure 3: MOAH in IF 544 chromatograms - Comparison of different integration approaches presented by laboratories having reported total mass fractions of MOAH (C10-C50) ranging from 0.88 to 6.7 mg/kg.

← The last chromatogram (on the left) highlights various RT regions to be considered (1 to 5), while the table below summarises the riding peaks/humps included (yes, no, or partially) by the laboratories.

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Consistency in reporting and data reliability

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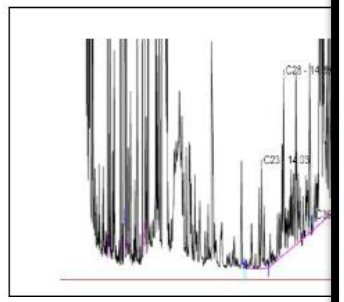
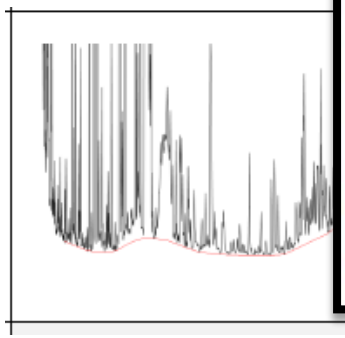
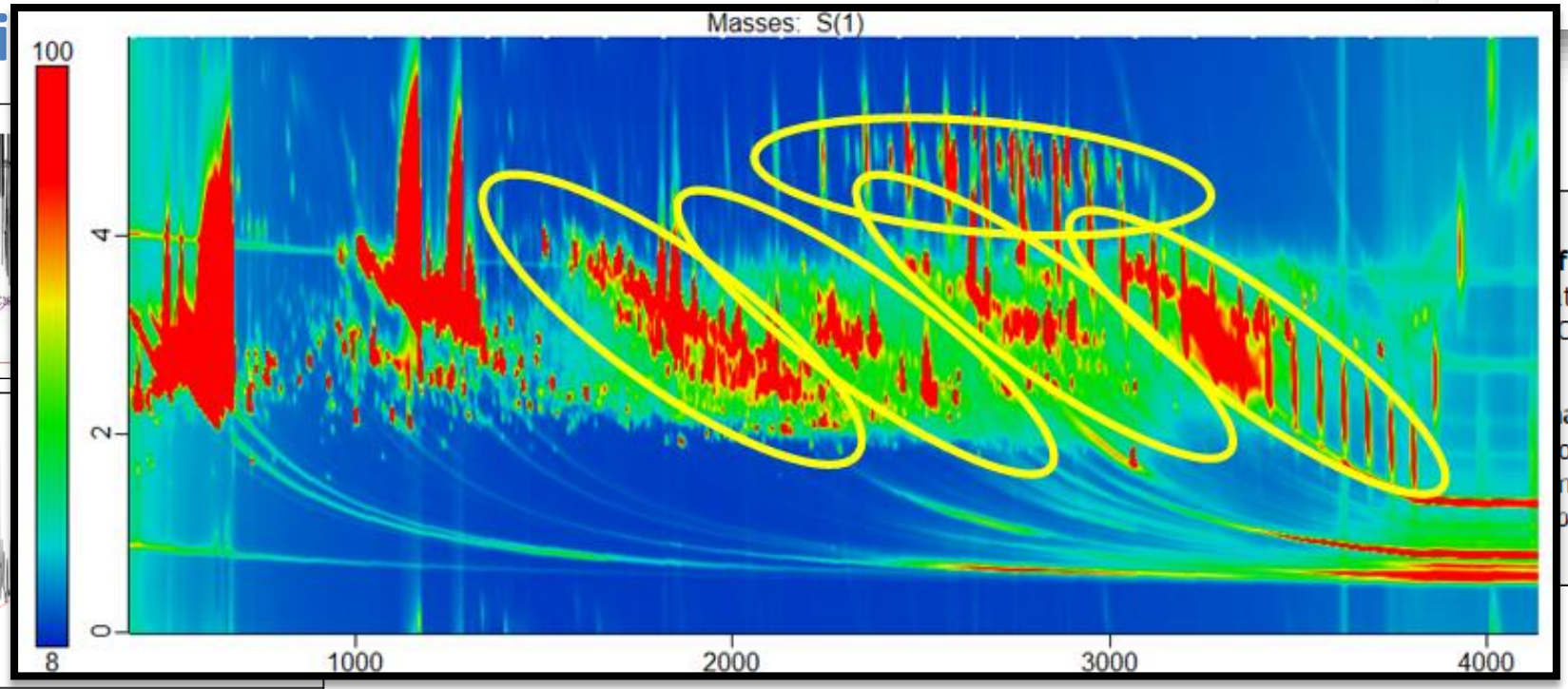
Data Interpretation

Data Integration

- Baseline
- Ridi







2022

European Commission

JRC TECHNICAL REPORT

Mineral oil in infant formulas
- guidelines for integrating chromatograms

JRC IF 2021-04: a virtual inter-laboratory comparison

Robouch P., Bratinova S., Goncalves C., Karasek L., Beldi G., Senaldi C., Valzacchi S. and Hoekstra E.

in IF 544 chromatograms - fferent integration approaches
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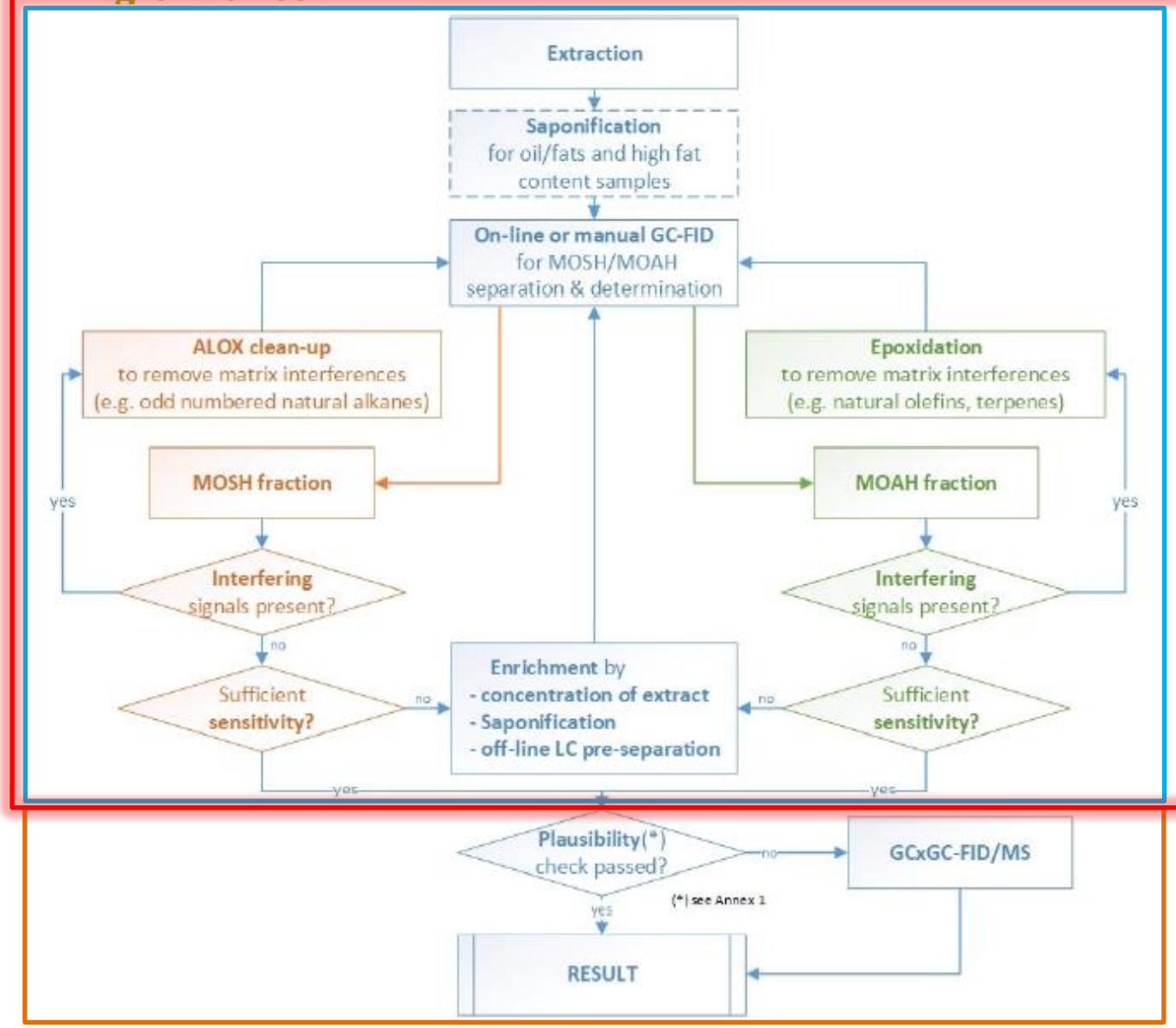
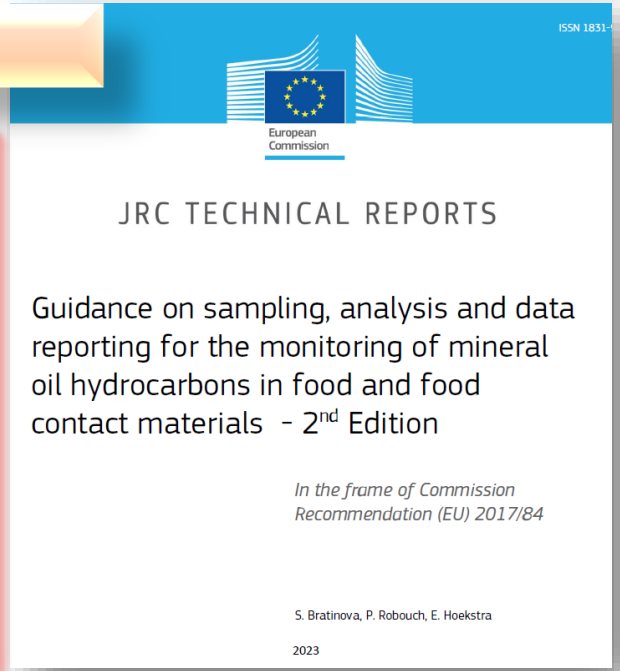


Figure 5 Decision tree on the use of auxiliary methods.

Sample Preparation



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Data Integration

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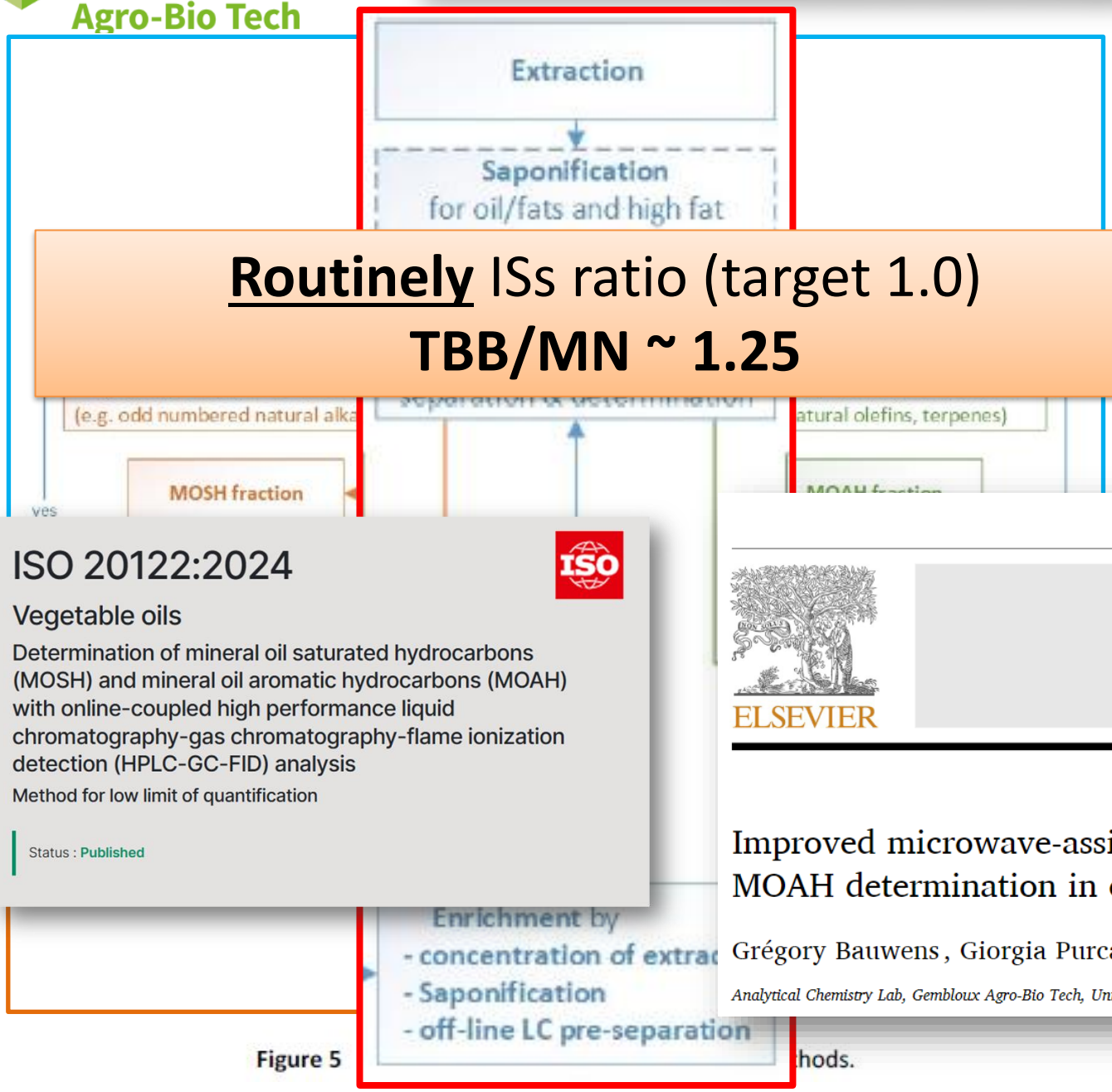

<u>Routinely</u> ISs ratio (target 1.0)

TBB/MN ~ 1.25

Sample Preparation

European Commission

JRC TECHNICAL REPORTS

Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials - 2nd Edition

In the frame of Commission Recommendation (EU) 2017/84

S. Bratinova, P. Robouch, E. Hoekstra

2023

ISO 20122:2024

Vegetable oils

Determination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with online-coupled high performance liquid chromatography-gas chromatography-flame ionization detection (HPLC-GC-FID) analysis

Method for low limit of quantification

Status : **Published**

Analytica Chimica Acta 1312 (2024) 342788

Contents lists available at ScienceDirect

Analytica Chimica Acta

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

ELSEVIER

Improved microwave-assisted saponification to reduce the variability of MOAH determination in edible oils

Grégory Bauwens, Giorgia Purcaro *

Analytical Chemistry Lab, Gembloux Agro-Bio Tech, University of Liège, Gembloux, 5030, Belgium

1.05±0.02

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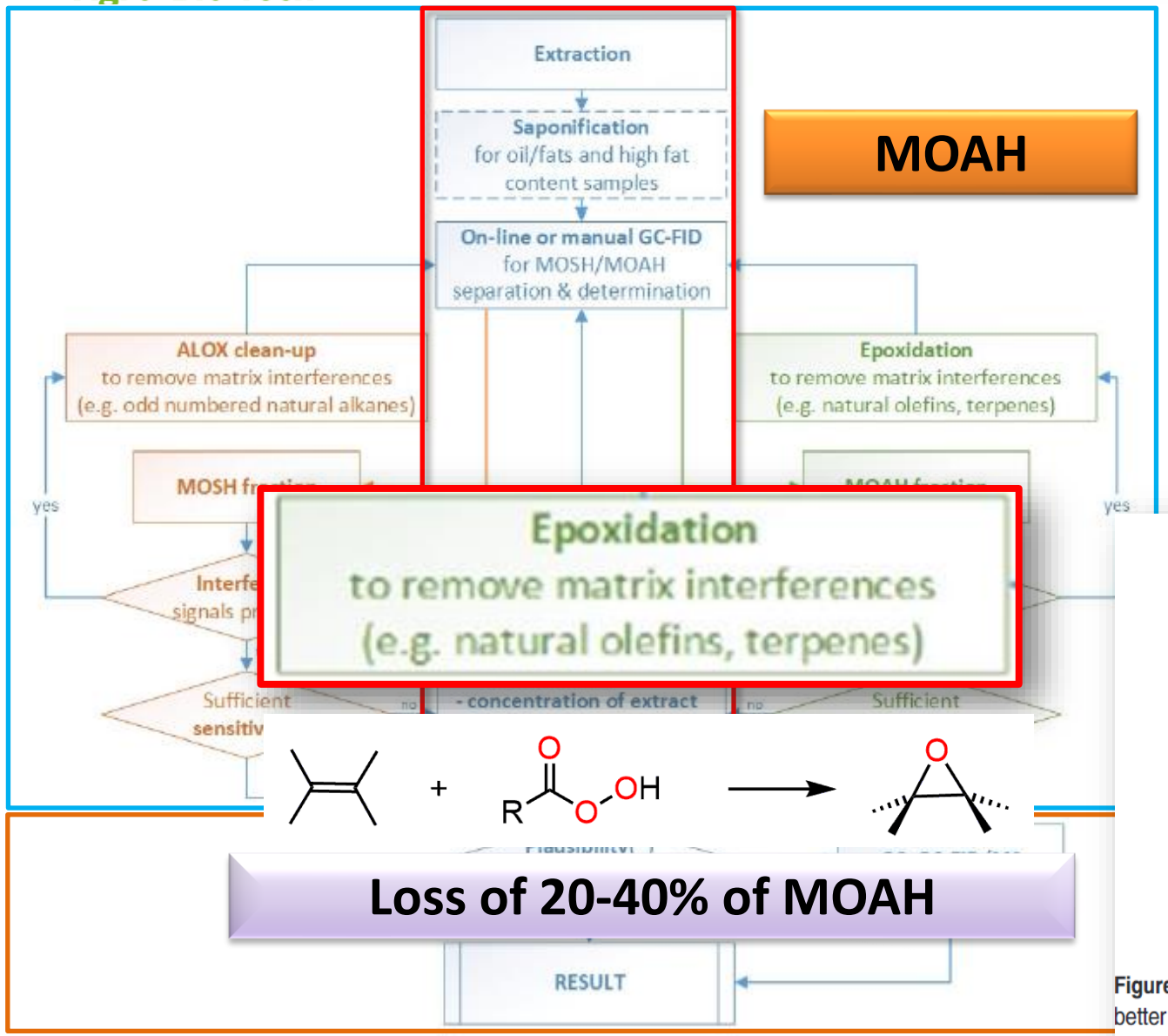


MOAH

Sample Preparation

Epoxidation
to remove matrix interferences
(e.g. natural olefins, terpenes)

Loss of 20-40% of MOAH

Figure 5 Decision tree on the use of auxiliary methods.

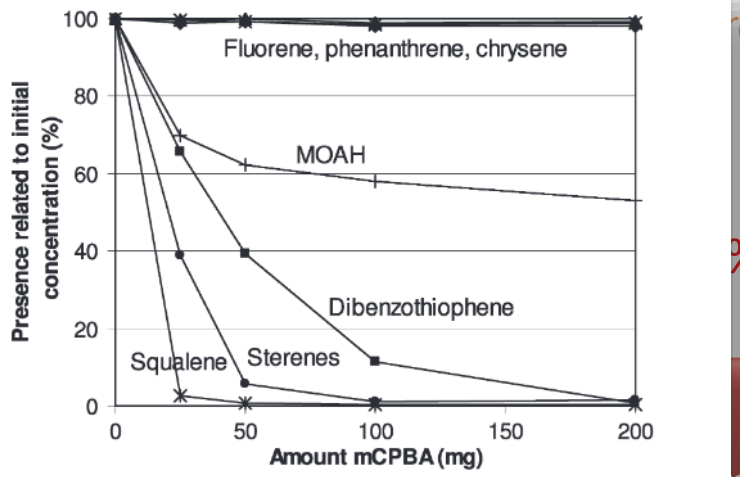
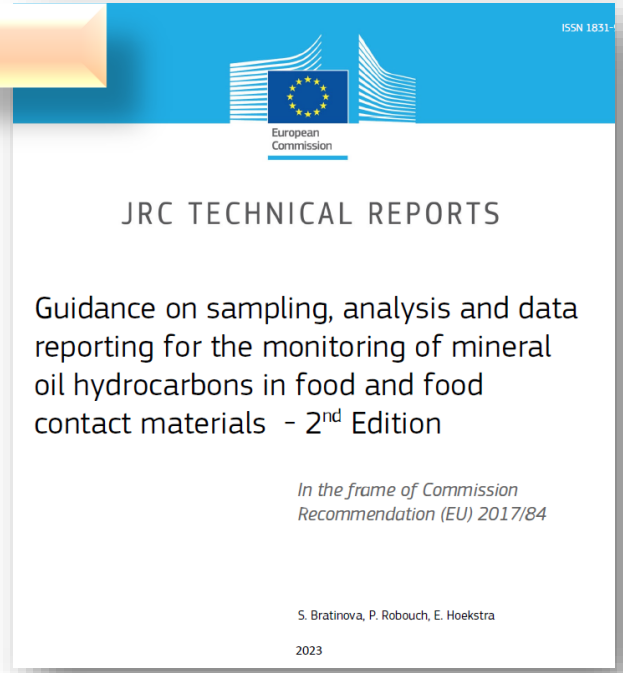


Figure 3. Epoxidation with different amounts of peracid. More peracid better removes squalene and the sterenes, but also the loss of certain aromatics increases.



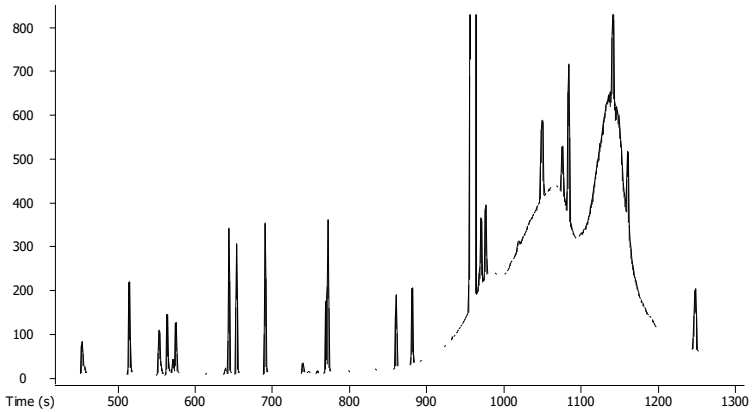
✓*Need for matrix-tailored*
ep protocols

% of uncertainty!

Data
ntegration

MOAH or not MOAH?

MOAH fraction of a palm oil extract



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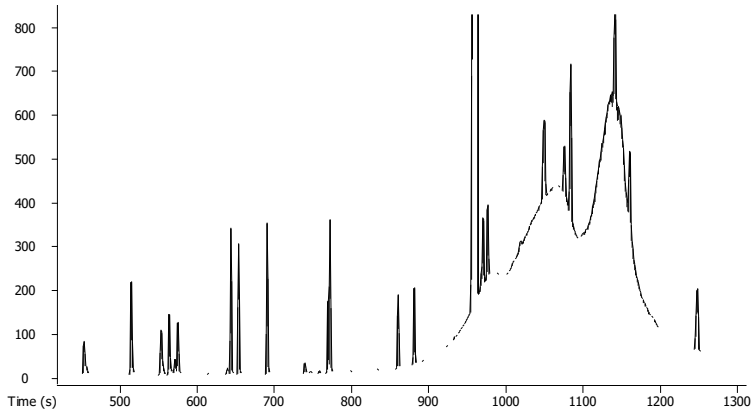

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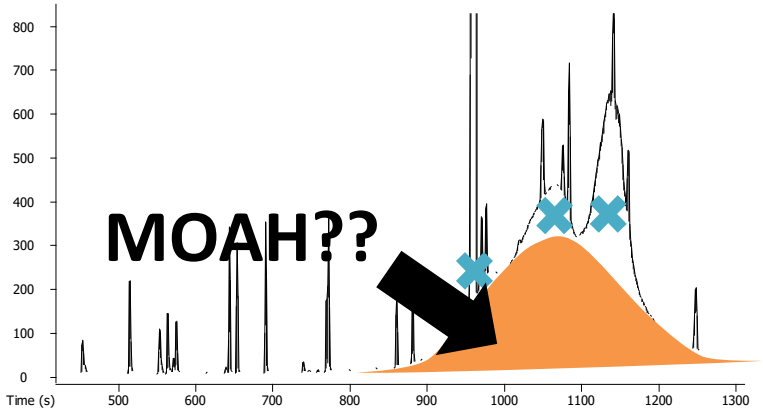

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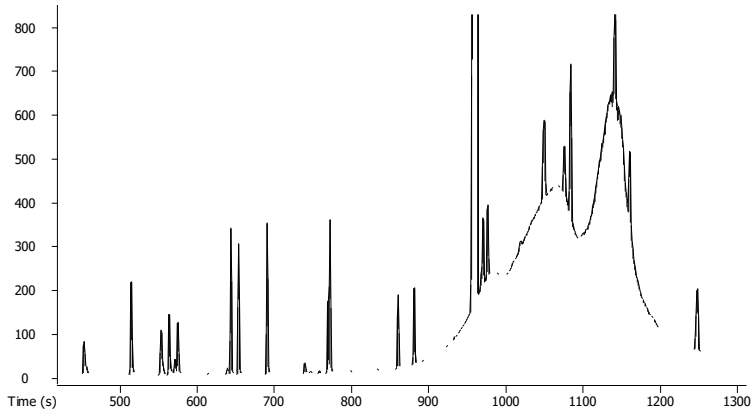

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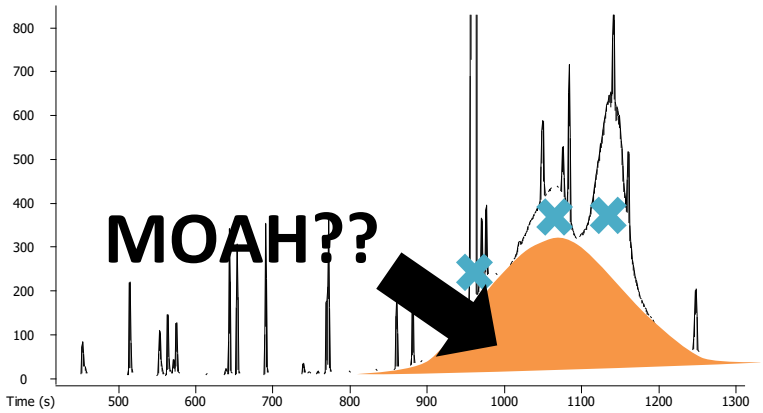
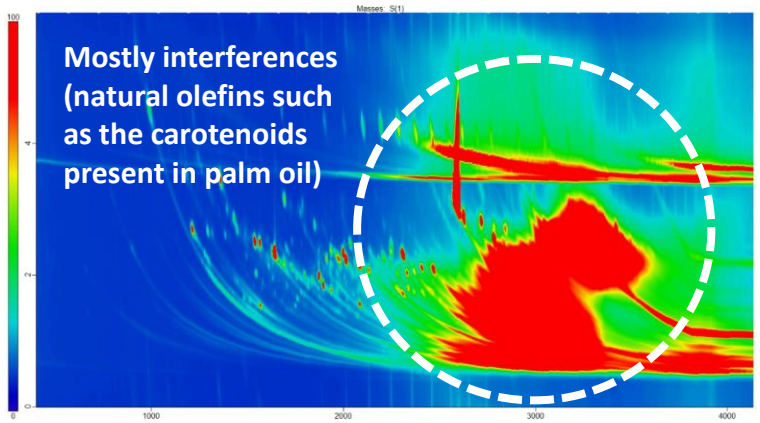


GC×GC



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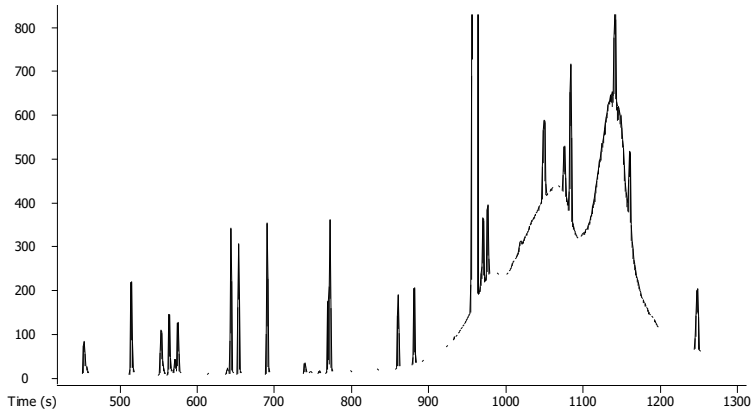

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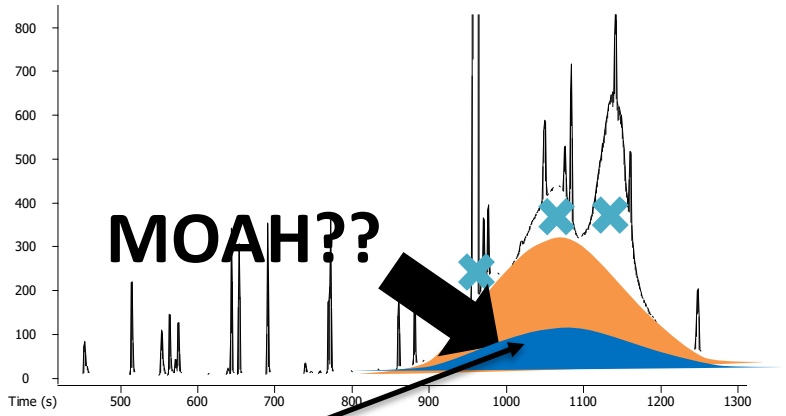

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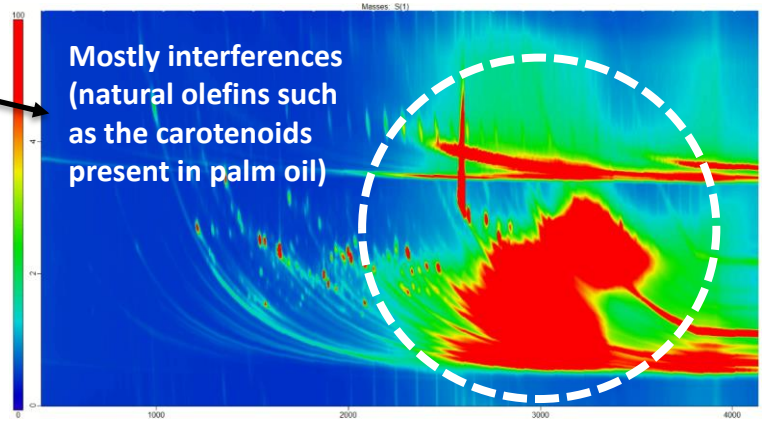

Epoxidation

is needed to determine MOAH

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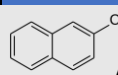
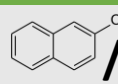
ROUTINE PURIFICATION METHOD

Purification

Recovery of MOAH after epoxidation

Epoxidation performed manually

MOAH RECOVERY (%)

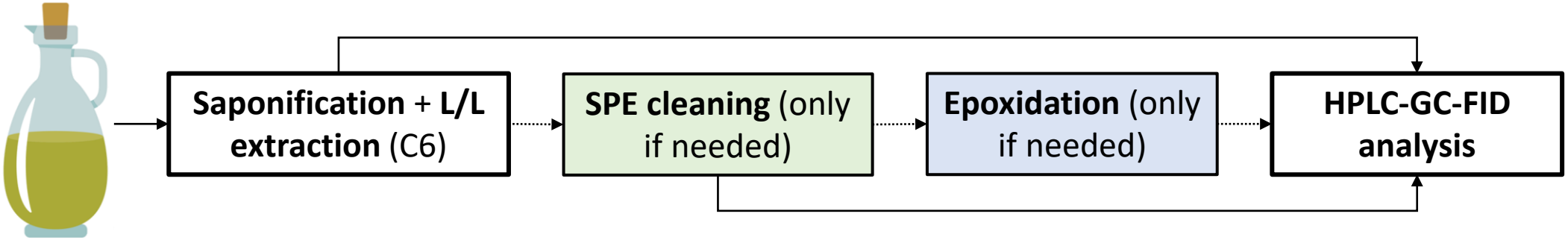
	PALM OIL		SUNFLOWER OIL	
	 /2MN	 /PYR	 /2MN	 /PYR
MCPBA (n_{PO}=2, n_{SFO}=1)	*65% (± 21%)	*38% (± 7%)	98%	56%
PER AC C6 (n=3)	177% (± 36%)	105% (± 36%)	146 (± 12%)	124 (± 58%)
PER AC $CHCl_3$ (n=3)	79% (± 29%)	61% (± 29%)	61% (± 19%)	47% (± 19%)

Oils spiked with **15 mg/kg** (*7.5 mg/kg) of a mixture of of **Gravex** (very volatile MOAH), **SN100 Aromatic Extract** (mid volatility), **SN500 Aromatic Extract** (heavy fraction)

Low and Variable Recoveries!!!

An alternative purification method

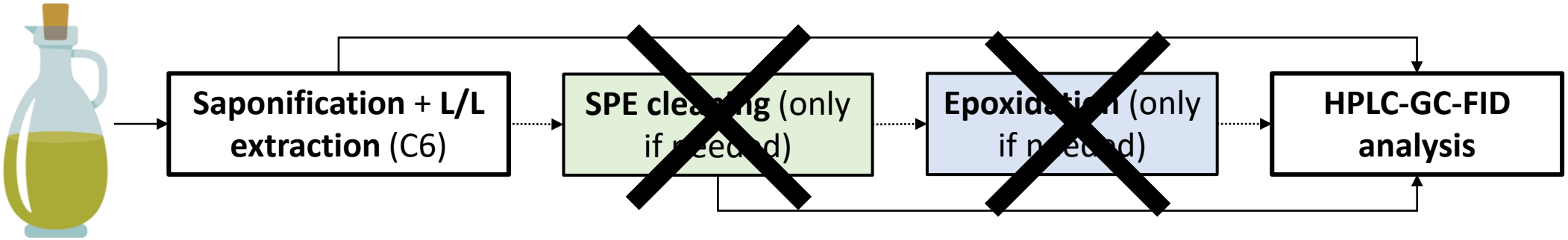
ISO 20122:2024 Vegetable oils — **Determination of mineral oil saturated hydrocarbons (MOSH) and aromatic hydrocarbons (MOAH) with online coupled HPLC-GC-FID analysis** — Method for low limit of quantification



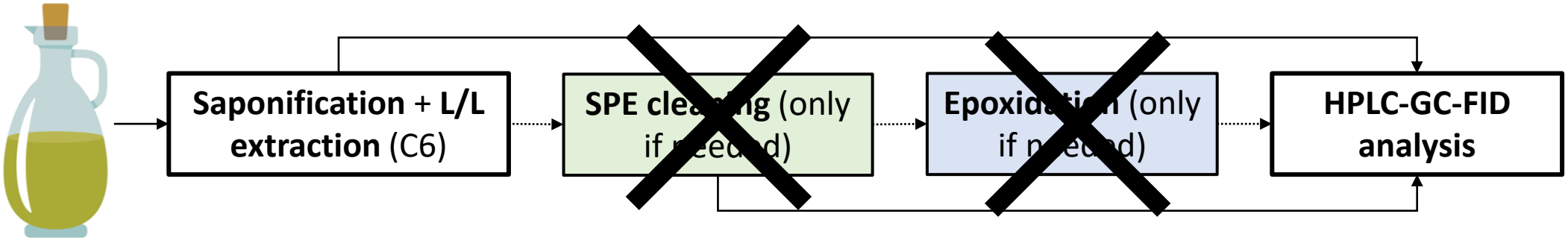
The issue with the current purification method for MOAH (i.e., epoxidation) is that it relies on a **non-selective chemical reaction which also attacks MOAH.**

It would therefore be relevant to develop a purification method which is not based on a chemical reaction anymore.

An alternative purification method



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

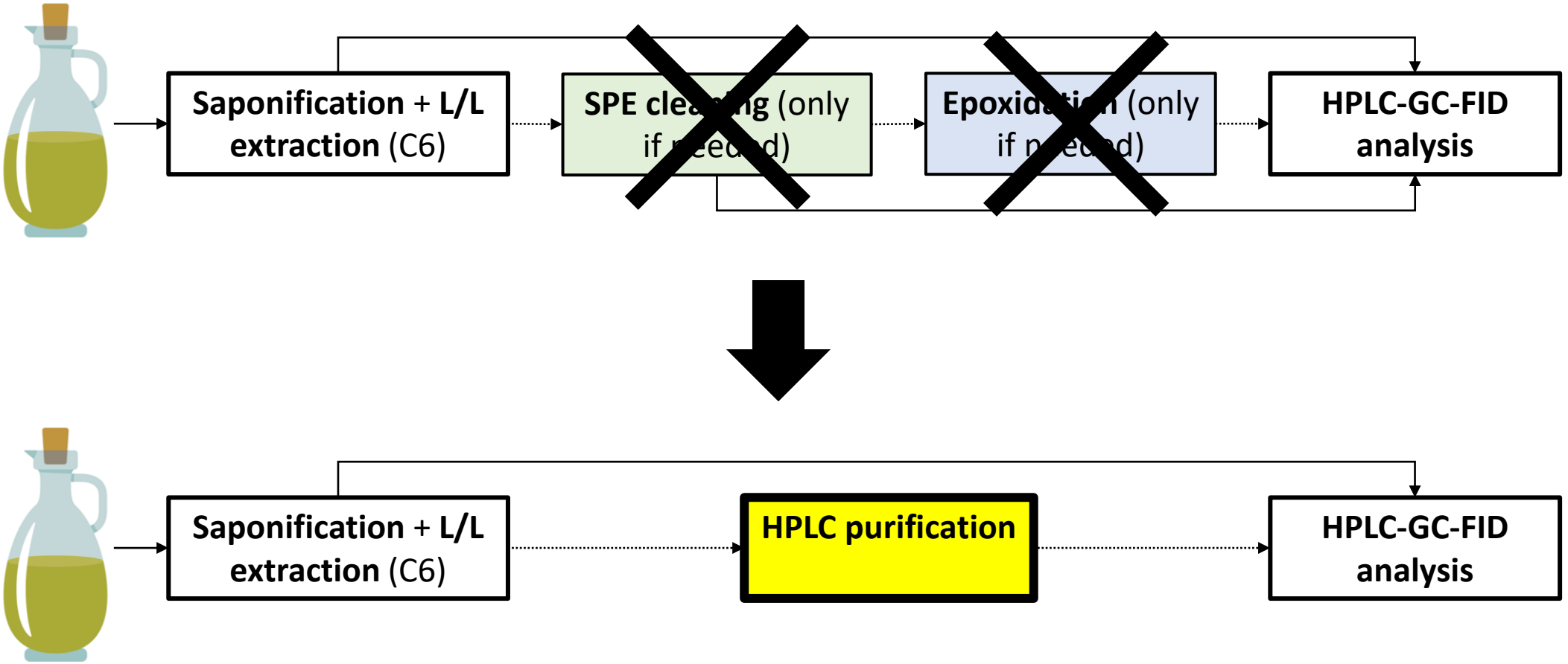
Epoxidation for the analysis of the mineral oil aromatic hydrocarbons in food. An update

Maurus Biedermann, Celine Munoz, Koni Grob*

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

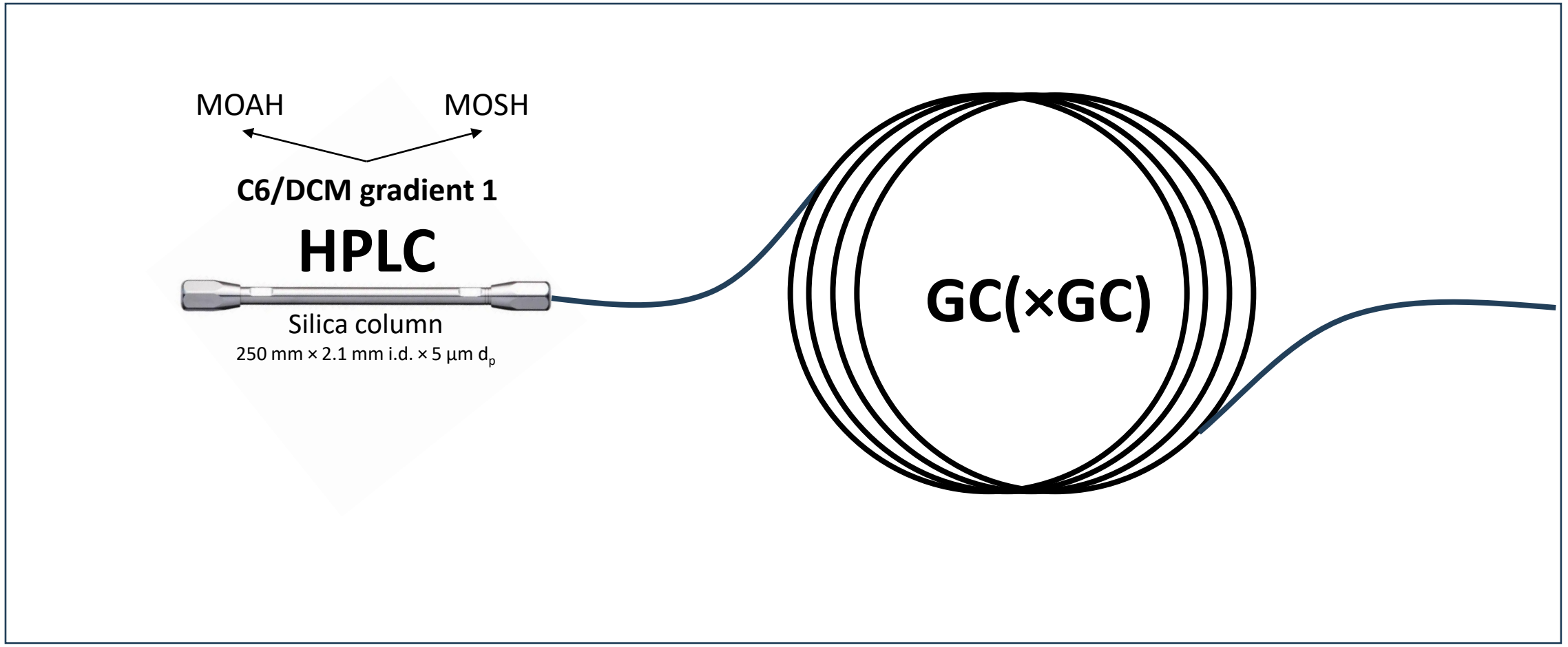
Various attempts were made to remove the olefins from the MOAH in the liquid preseparation step. Zoccali et al. [25] added a second HPLC column with silver ions to improve the separation between the MOAH and the polyunsaturated olefins. Squalene was retained beyond the MOAH with up to three aromatic 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.

An alternative purification method



Purification

An alternative purification method



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Purification

An alternative purification method

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

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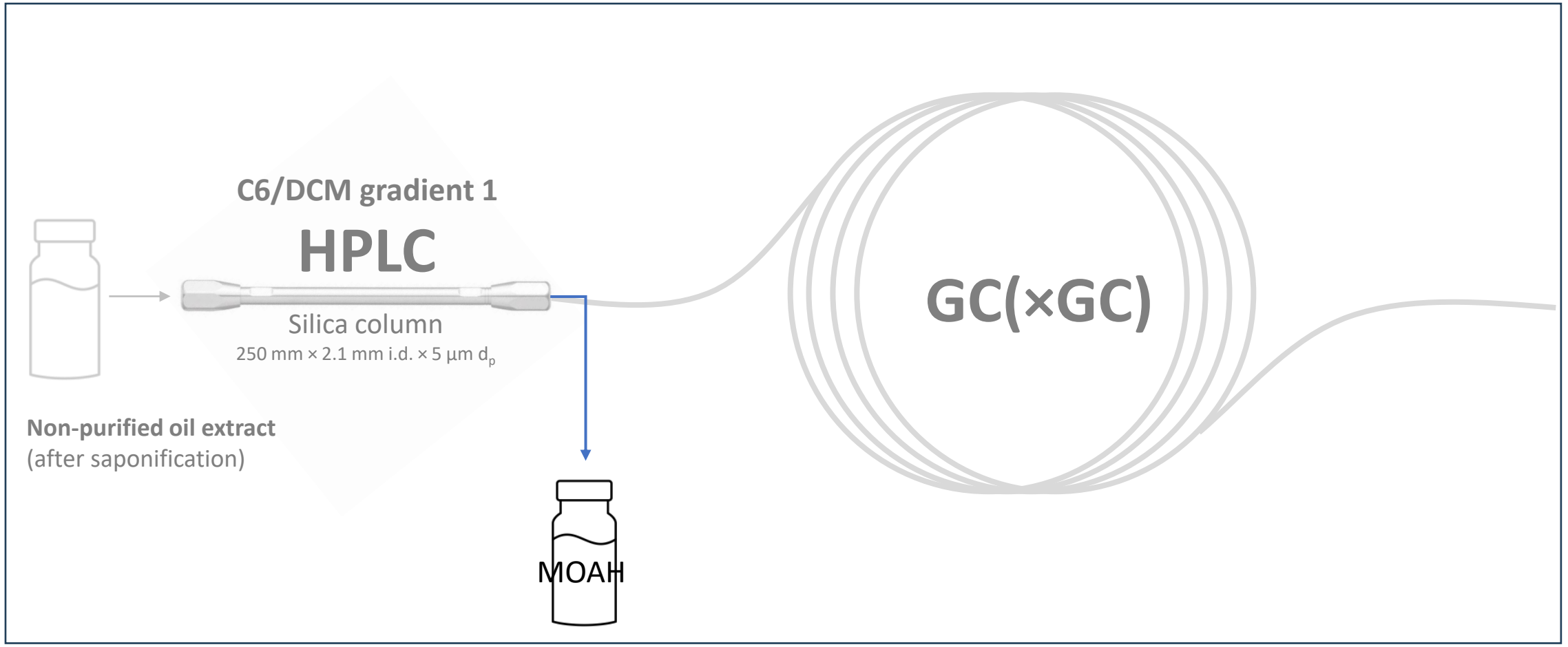

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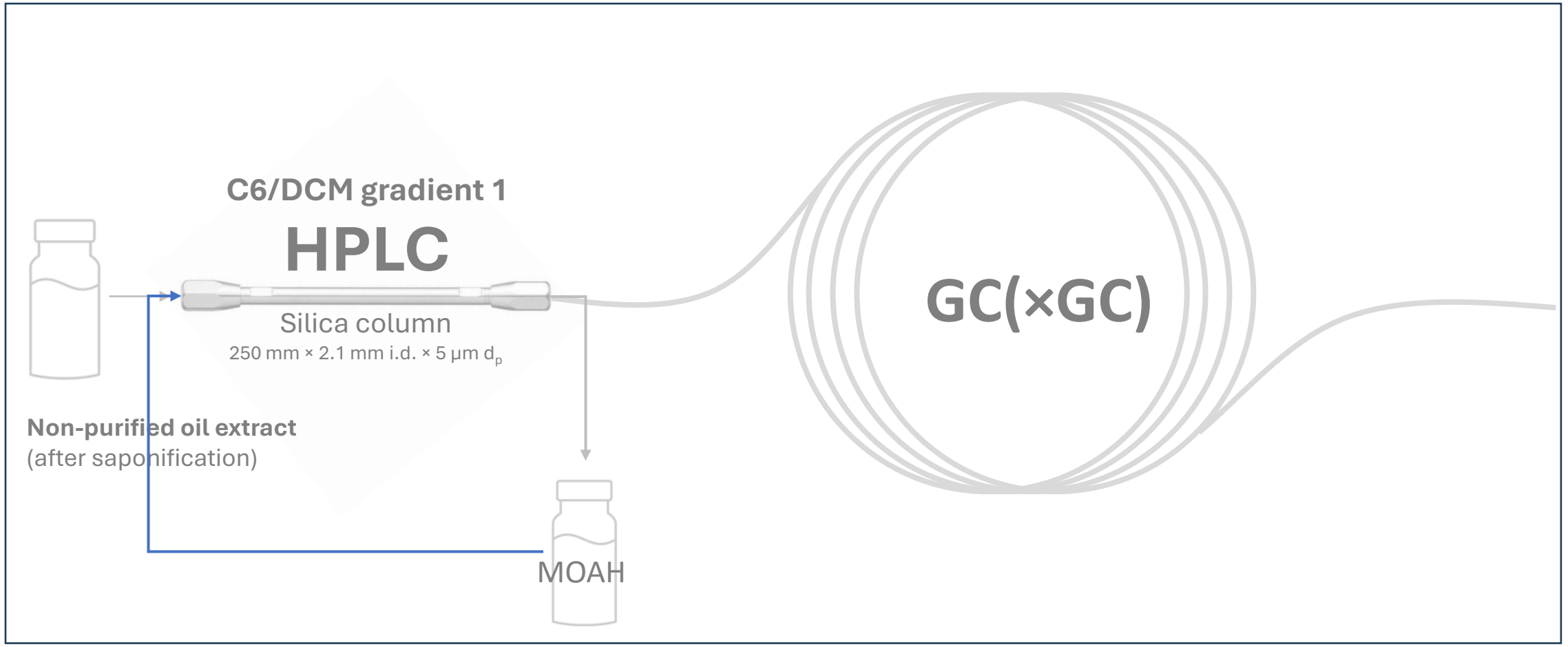

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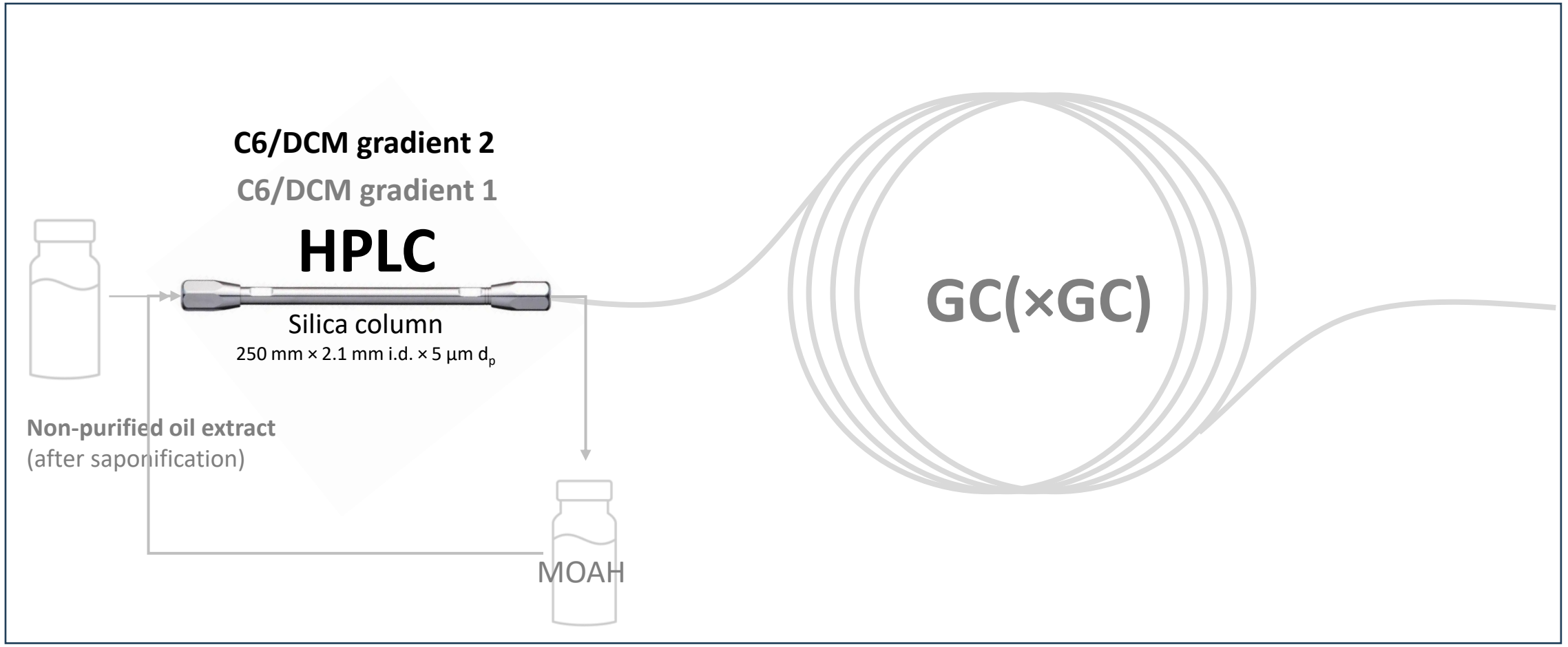

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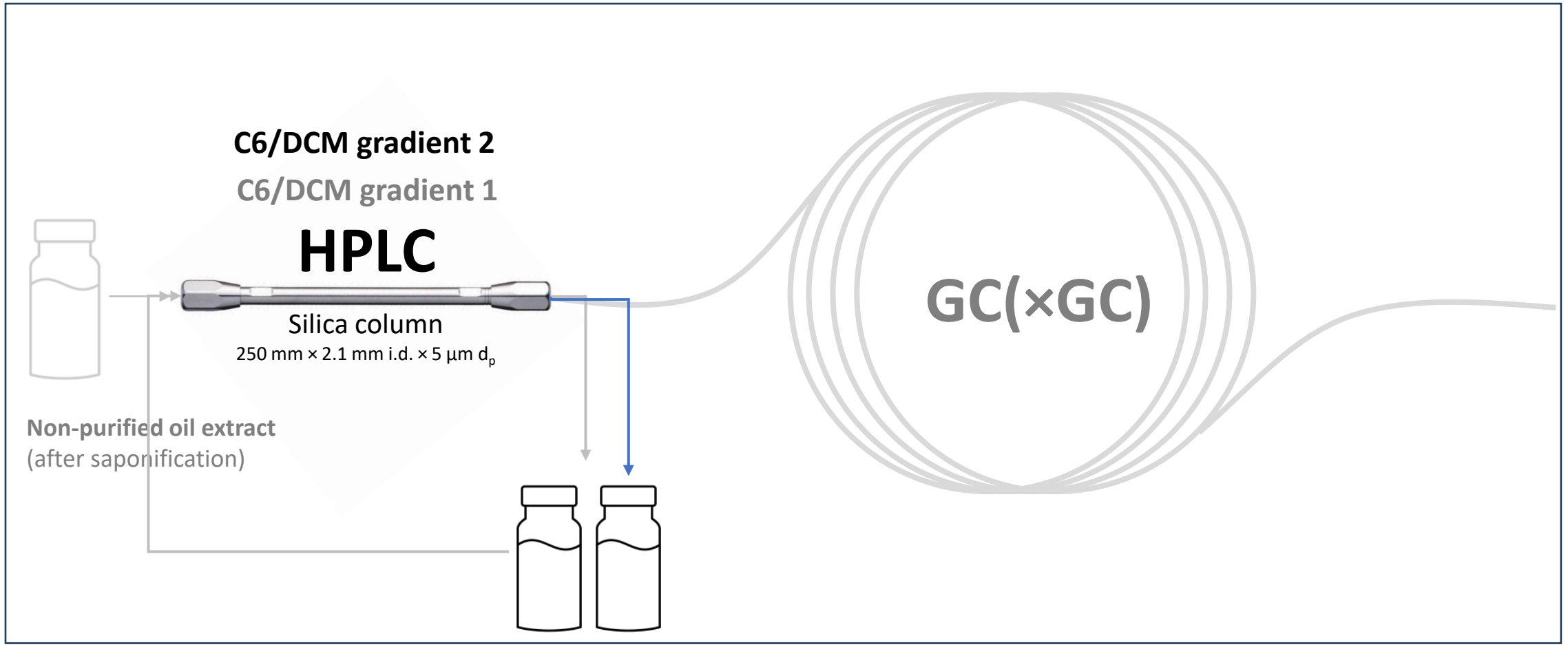

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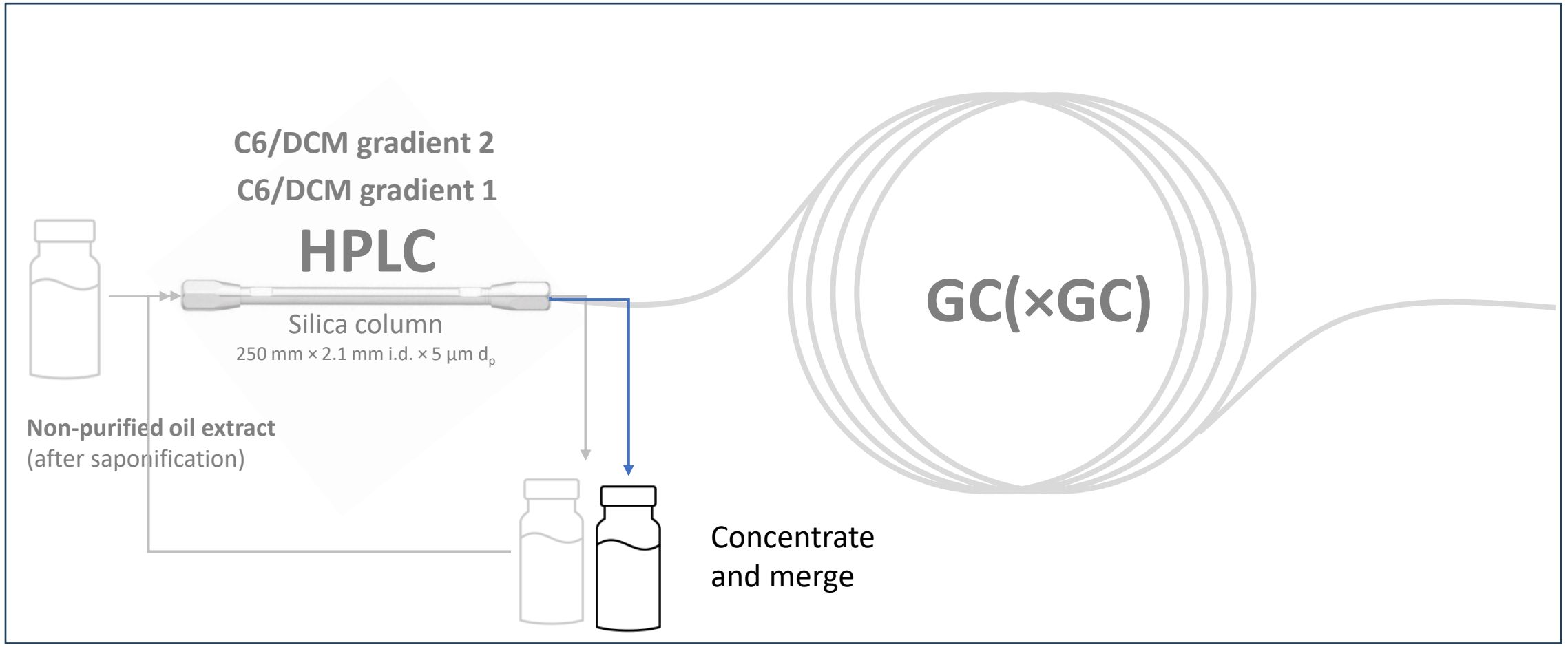

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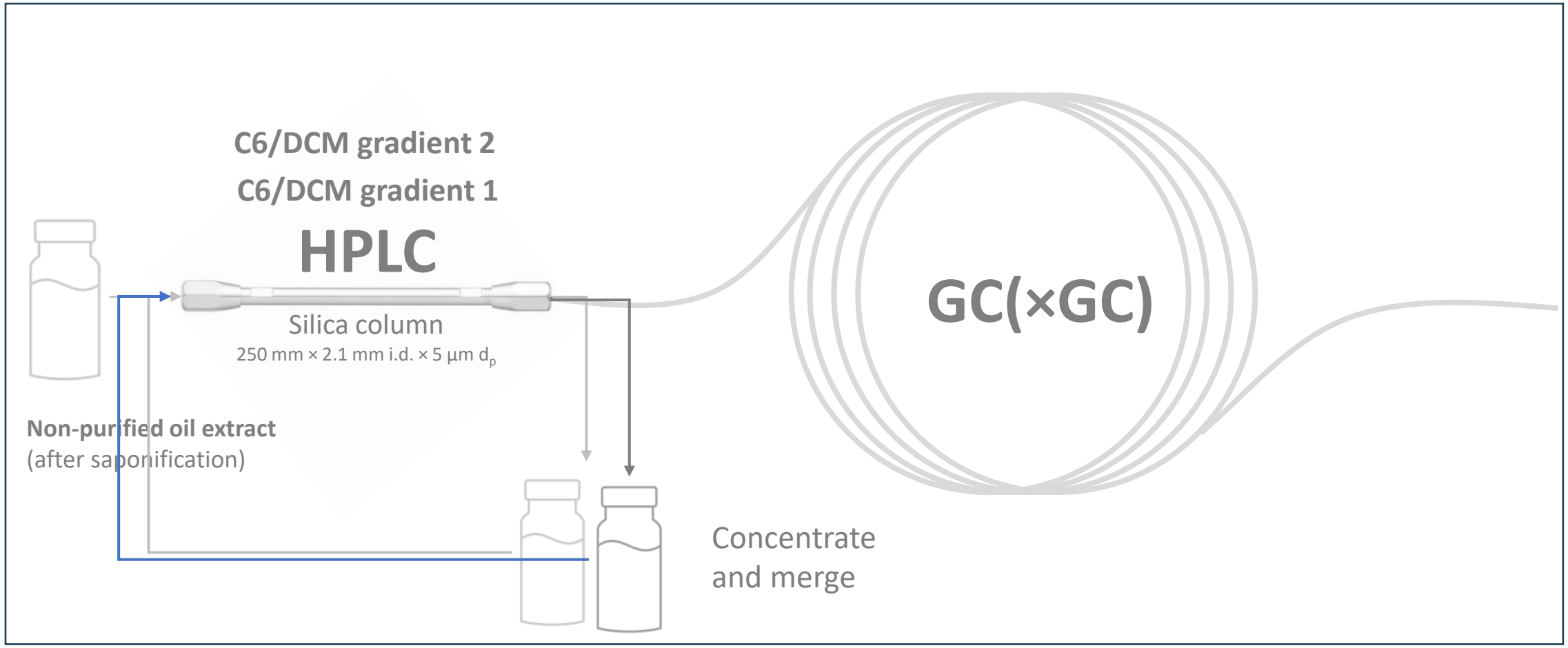

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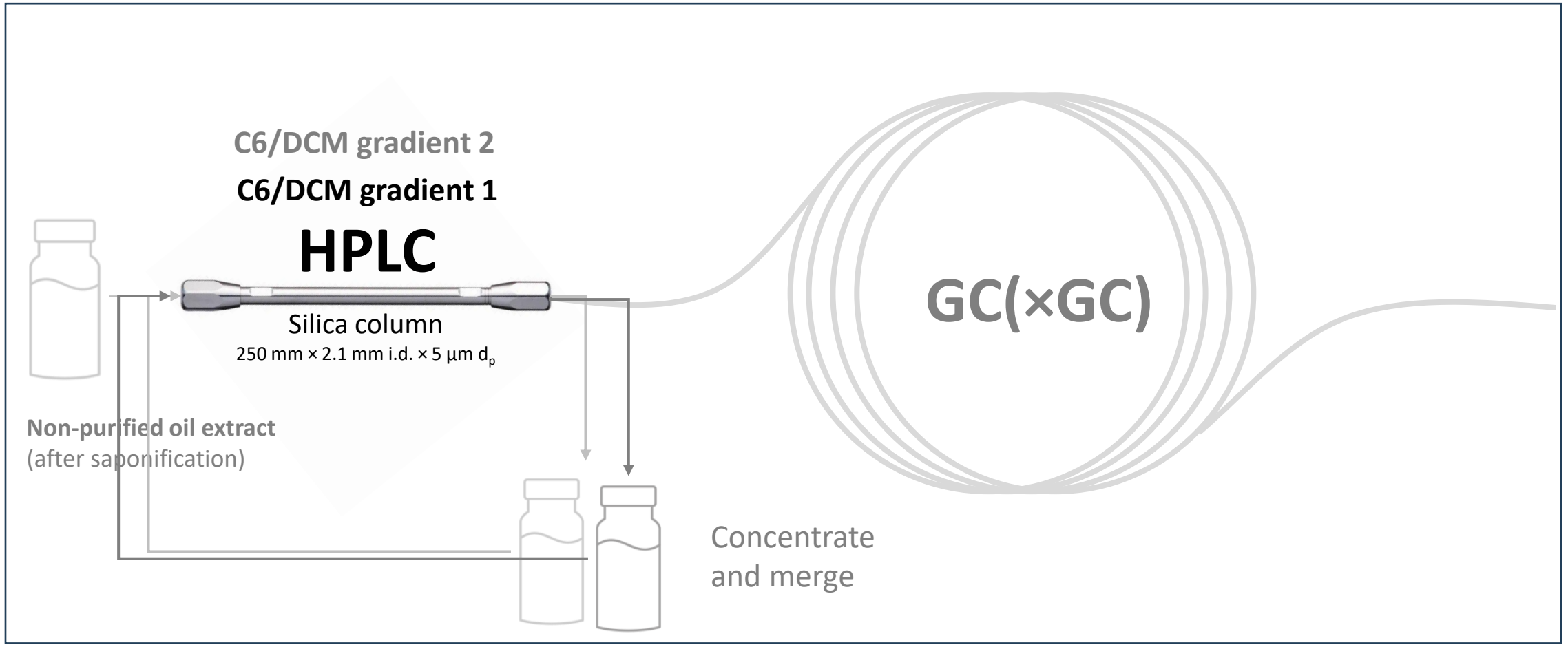

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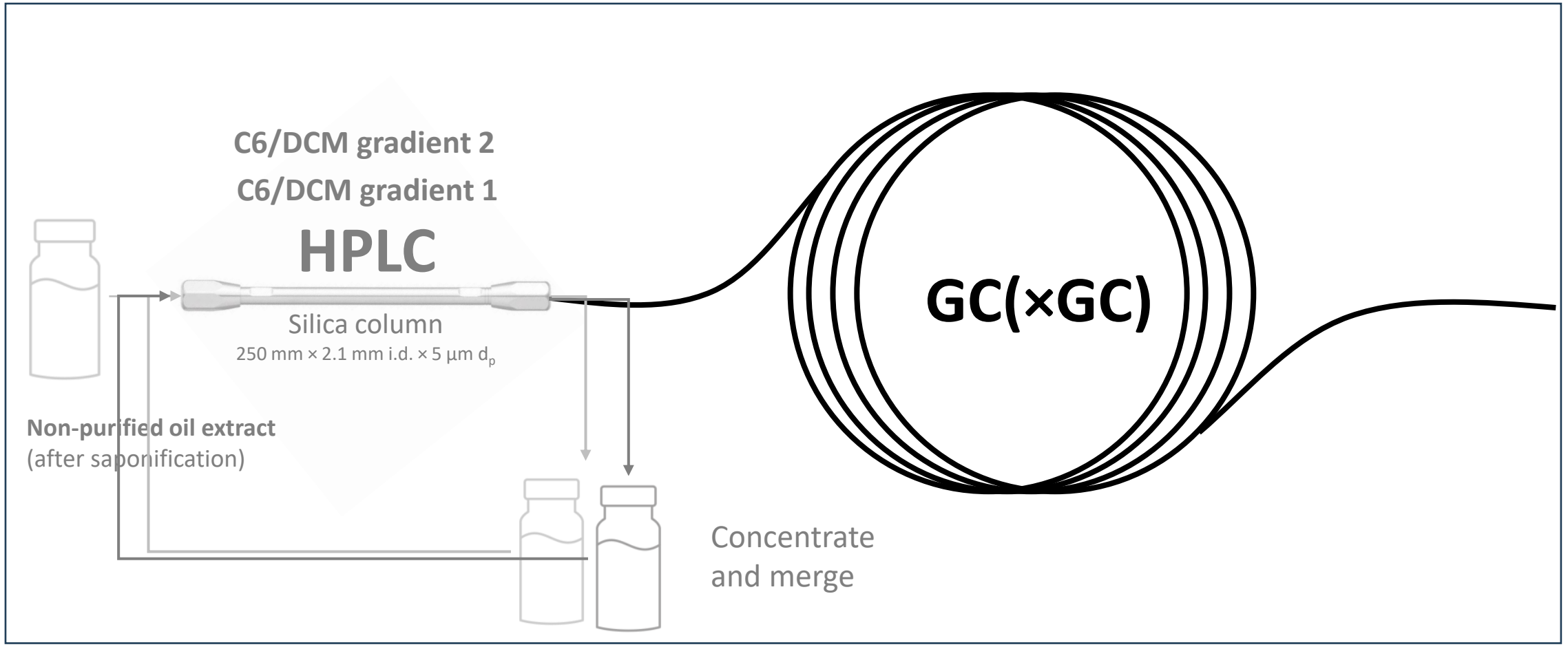

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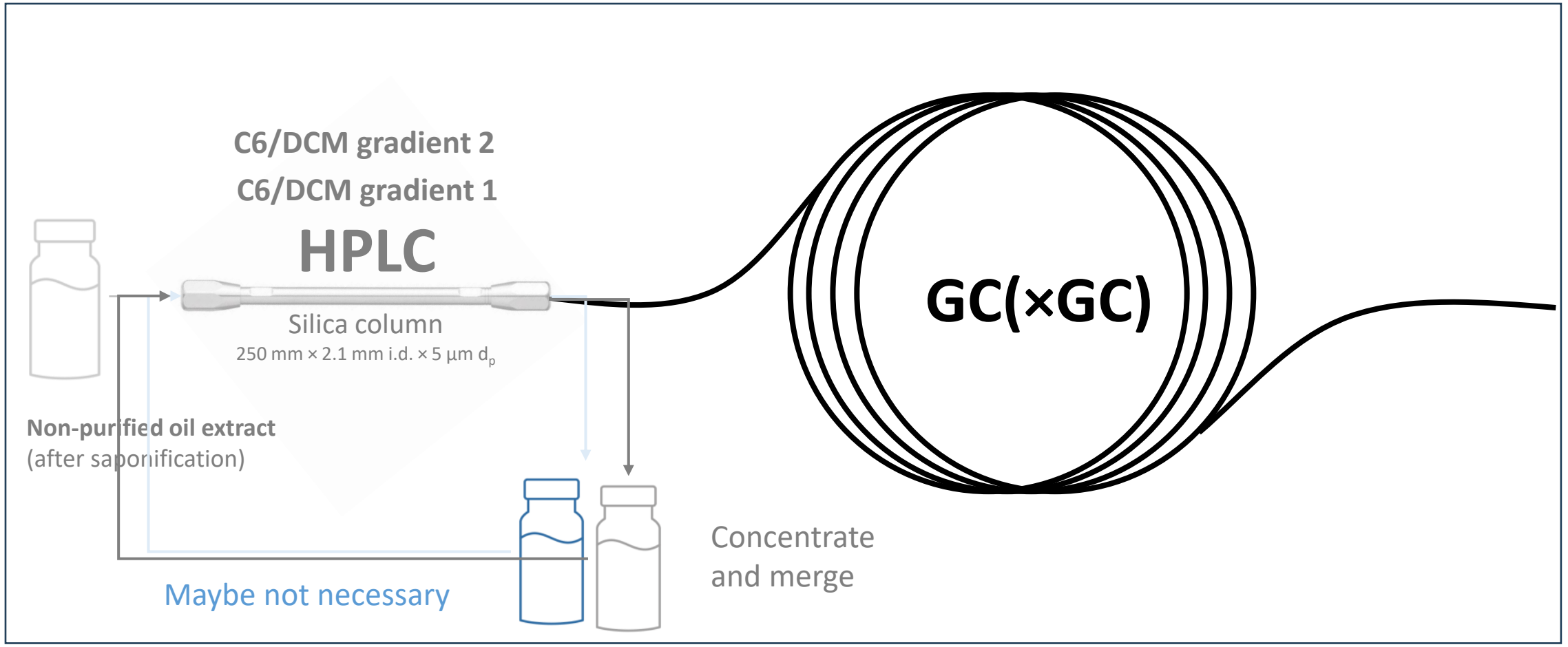

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Evaluation

Recovery after LC purification

- Evaluation of the recovery of **MOAH** in ***n*-hexane**
- Evaluation of the recovery of **MOAH and PAHs in different edible oils**

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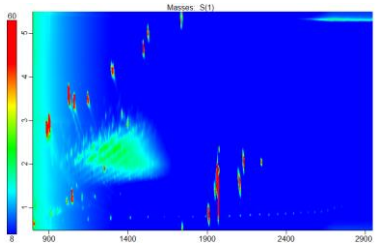
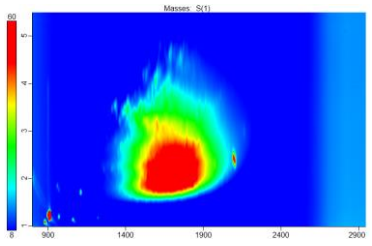
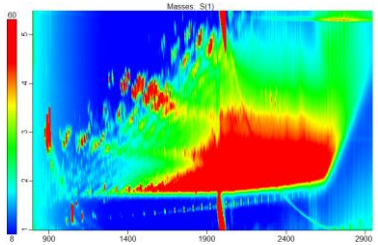


Gravex



Sternel motor oil



Heavy gas oil (HVGO)

Cover different volatility ranges

Assessment of the recovery (/2MN)

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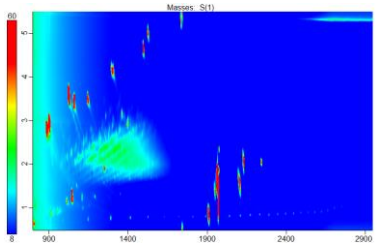

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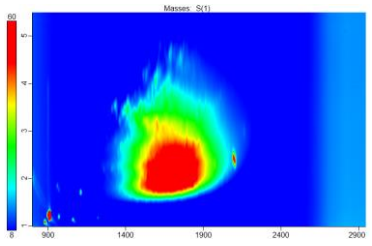

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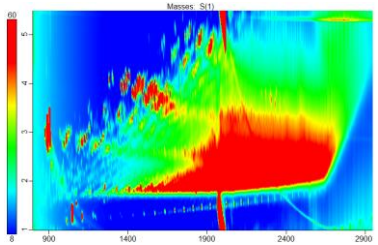

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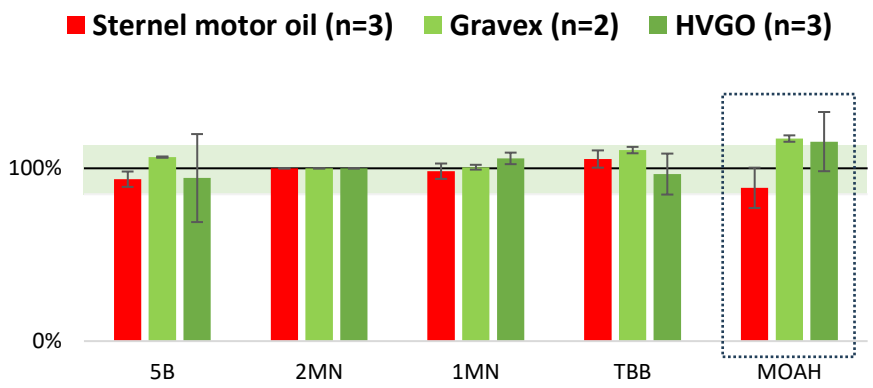

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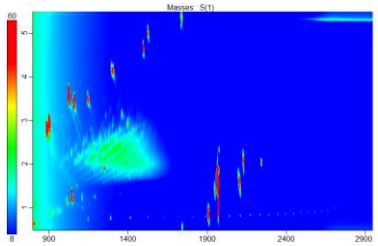

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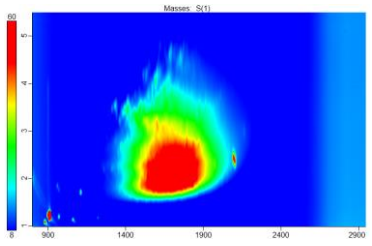

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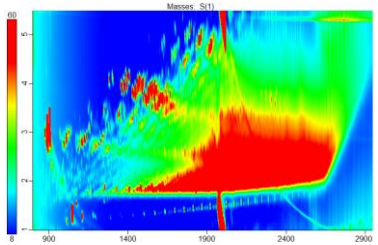

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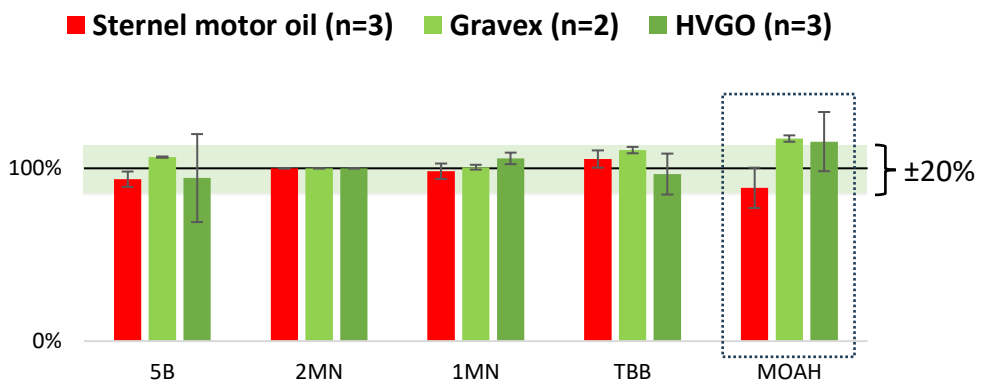

On average 107% ± 16%
(Different operators, different days)

Spiking concentration: 5 ug MOAH/ml solution

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Good recovery of MOAH ✓

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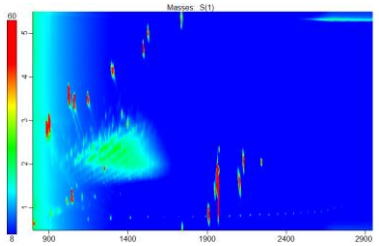

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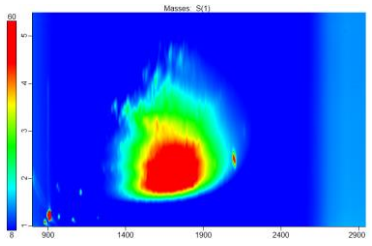

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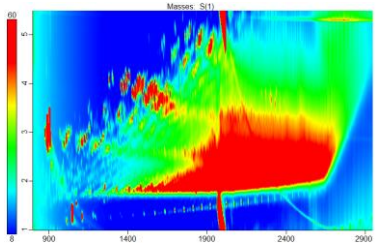

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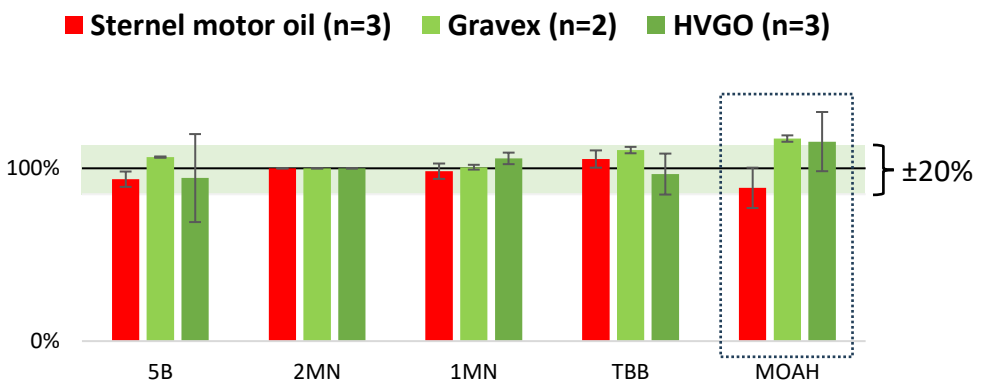

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PALM

SUNFLOWER

OLIVE

COCONUT

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Gravex + SN100 Aromatic Extract + SN500 Aromatic Extract (7.5 ppm MOAH)

SUNFLOWER

Sternel motor oil
(42 ppm MOAH)

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Shell Gravex 912
(37.5-43.5 ppm MOAH)

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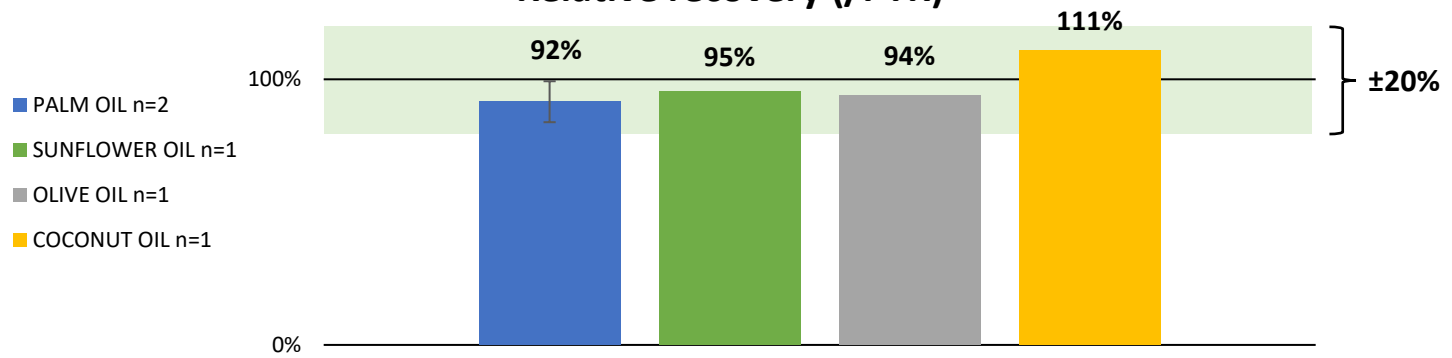

On average (n=5), 97%±9%

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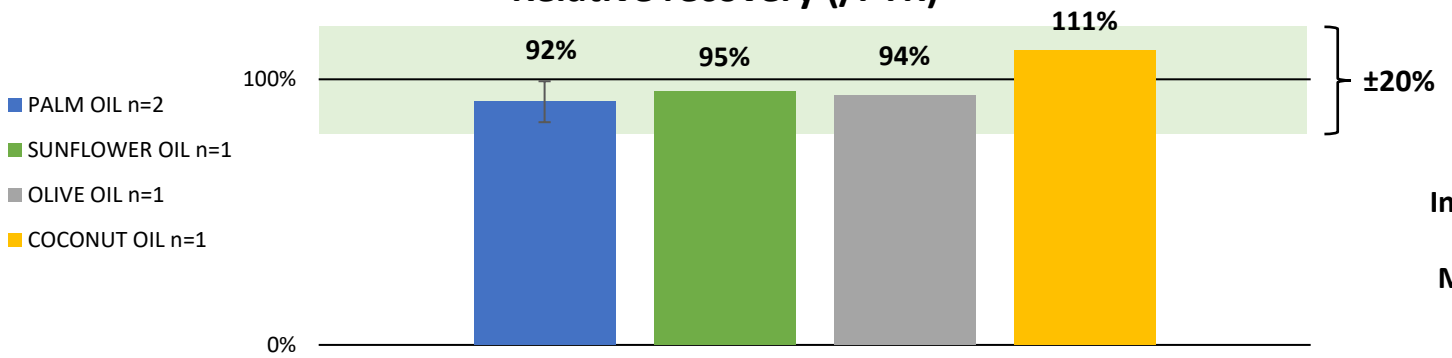

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In line with the requirements of the JRC updated guidance for MOSH/MOAH analysis in food and FCM (2023)

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Purification

Purification efficiency

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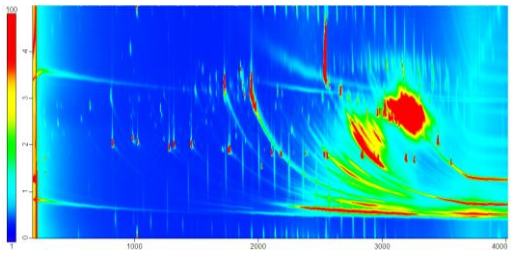

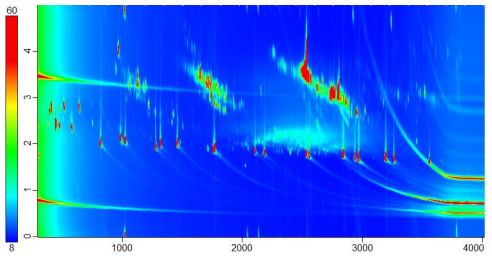

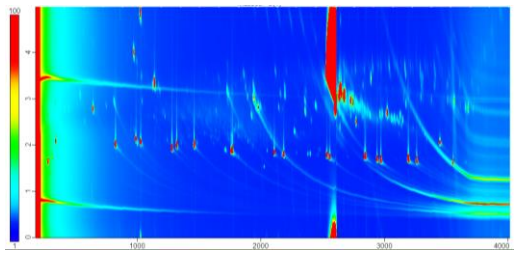

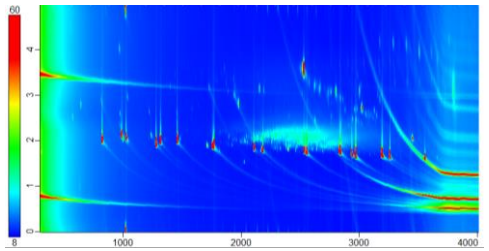

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Purification

Purification efficiency


✓


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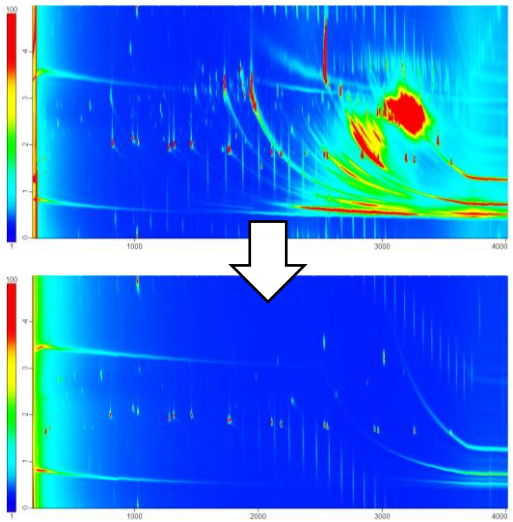

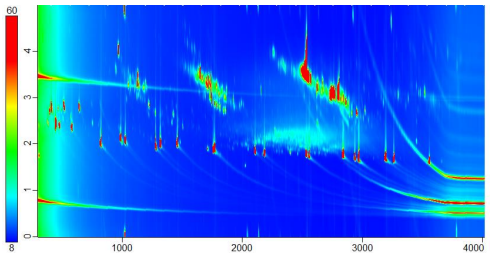

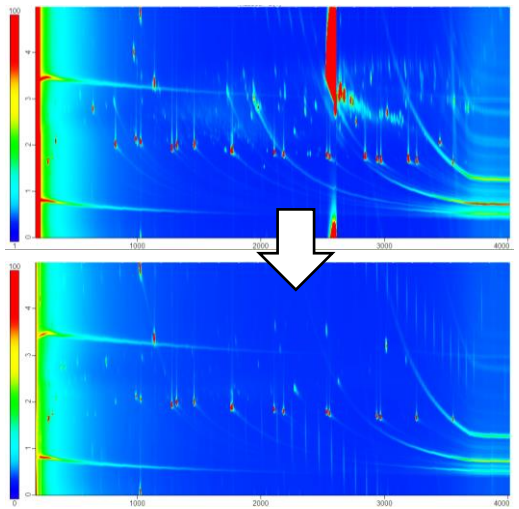

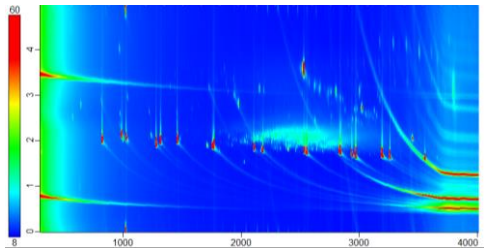

Very good removal of carotenoids and squalene

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Purification

Purification efficiency


✓


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✓


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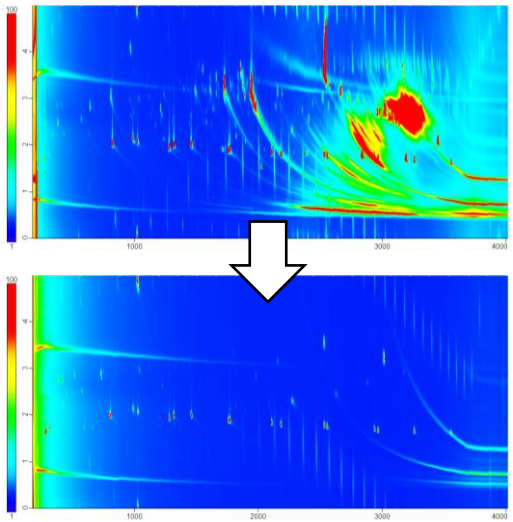

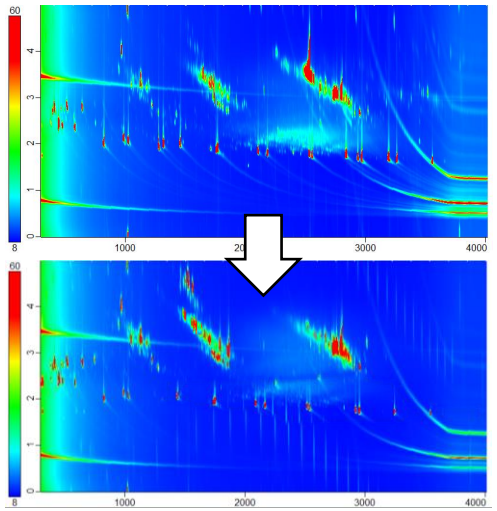

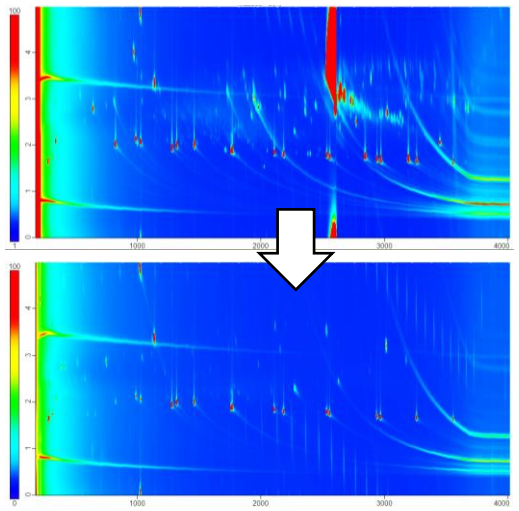

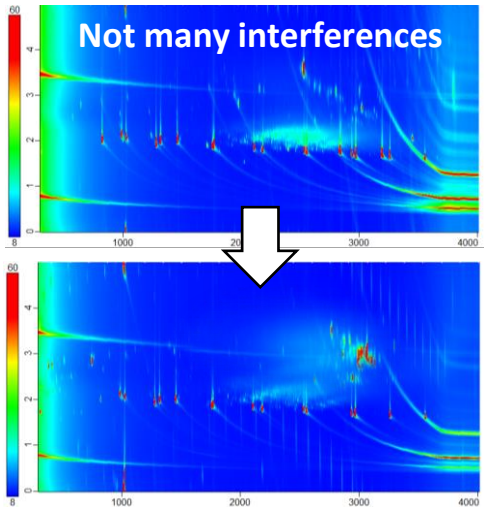

Very good removal of carotenoids and squalene

Other terpenoids are less well removed

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Purification

Purification efficiency


✓


~


✓


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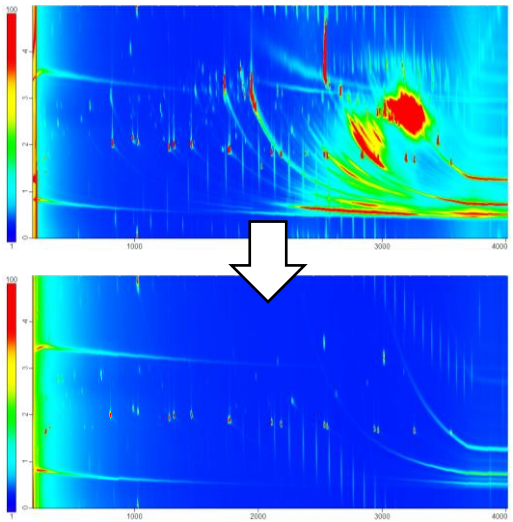

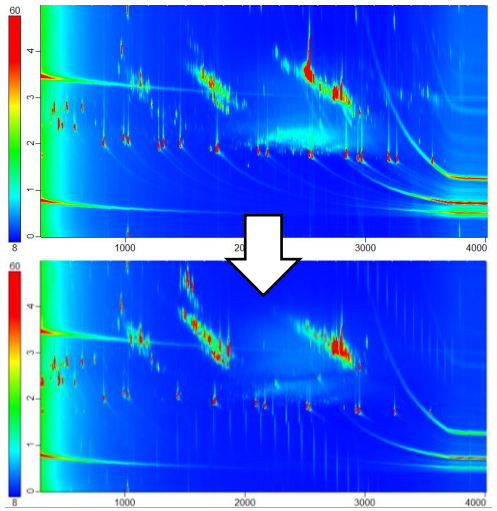

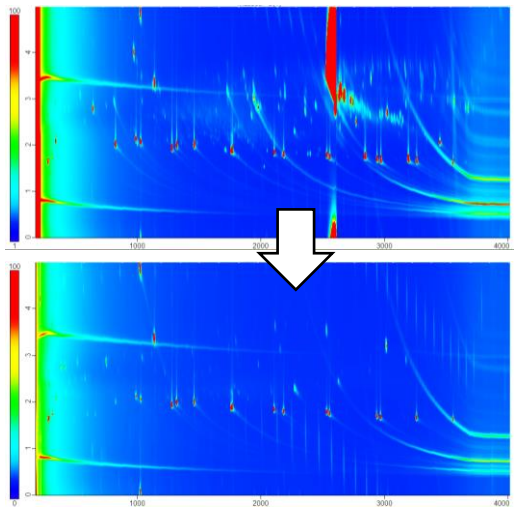

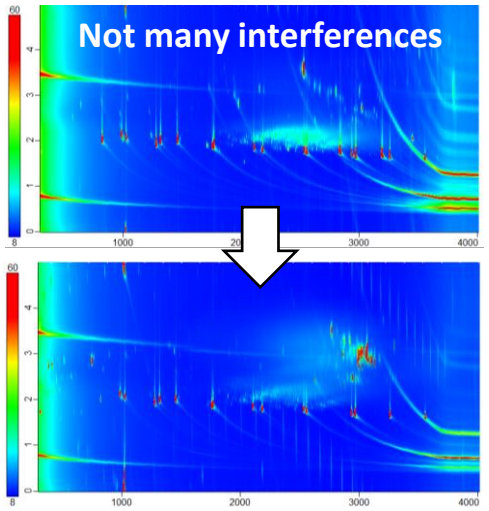

Very good removal of carotenoids and squalene

Other terpenoids are less well removed

BUT the method has another big advantage

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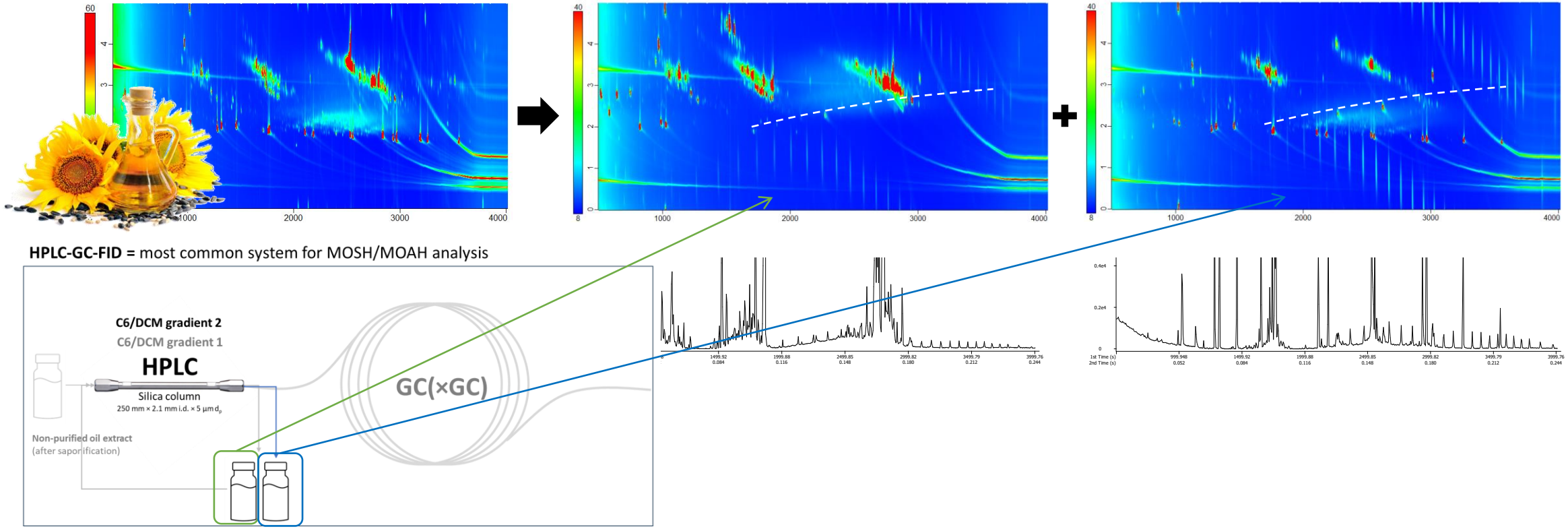
LC purification

Quantification by number of aromatic rings

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



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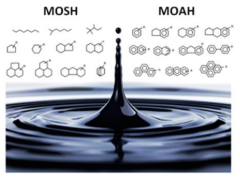
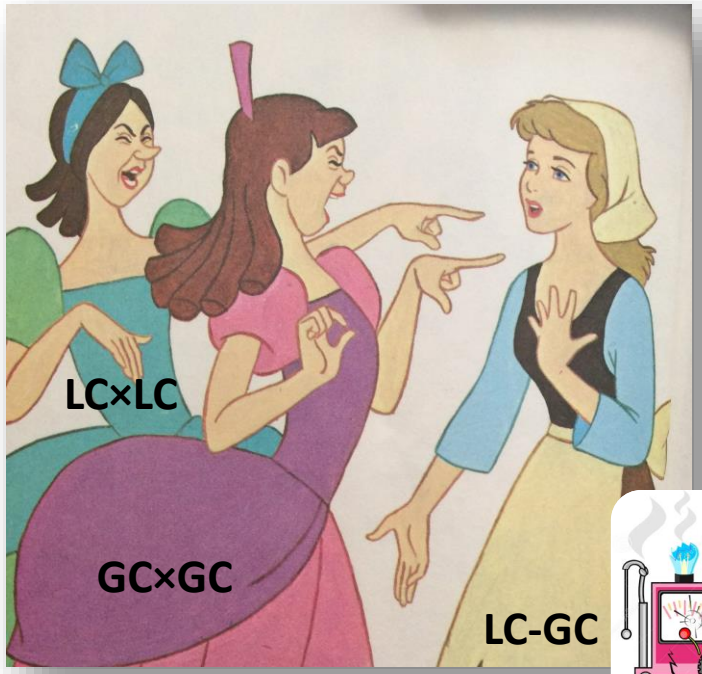
CONCLUSION AND PERSPECTIVE



Neglected Hyphenated Separation mode

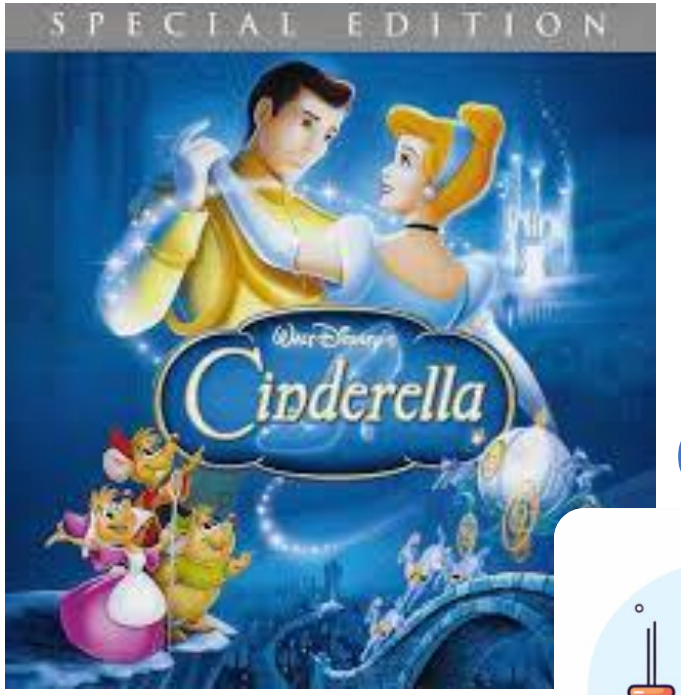
LC-GC



R&D

Redeemed Hyphenated Separation mode

LC-GC(×GC)



Q&A

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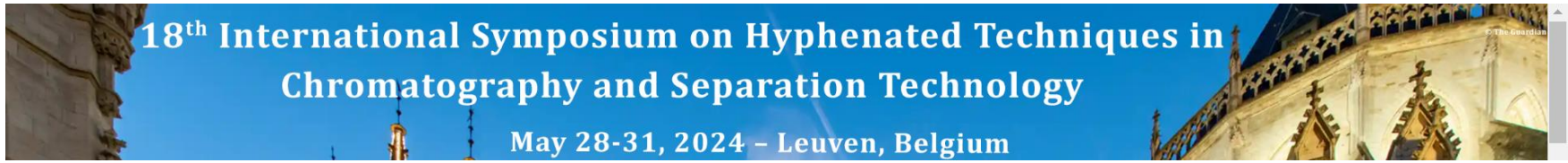

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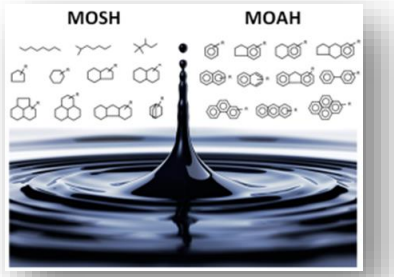

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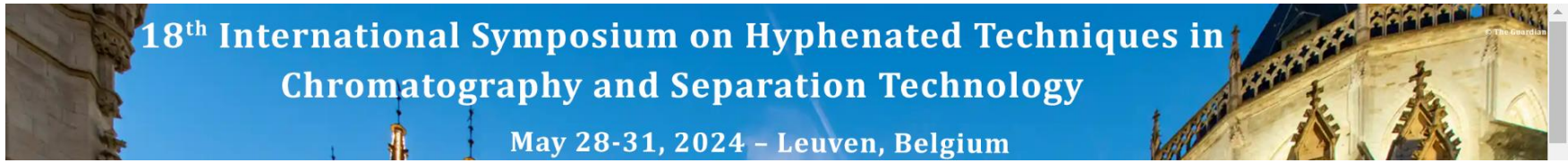

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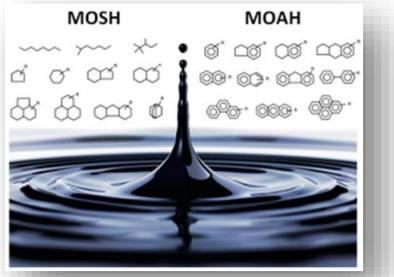

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