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19th Euro Fed Lipid Congress and Expo
17 – 20 September 2023
Poznań · Poland
Hosted by the
OILS & FATS SECTION

TACKLING THE CHALLENGE OF MOSH AND MOAH ANALYSIS IN FOOD USING ADVANCED CHROMATOGRAPHIC TECHNIQUE

Giorgia Purcaro, , Aleksandra Gorska, Grégory Bauwens

Gembloux Agro Bio-Tech, University of Liège, Belgium

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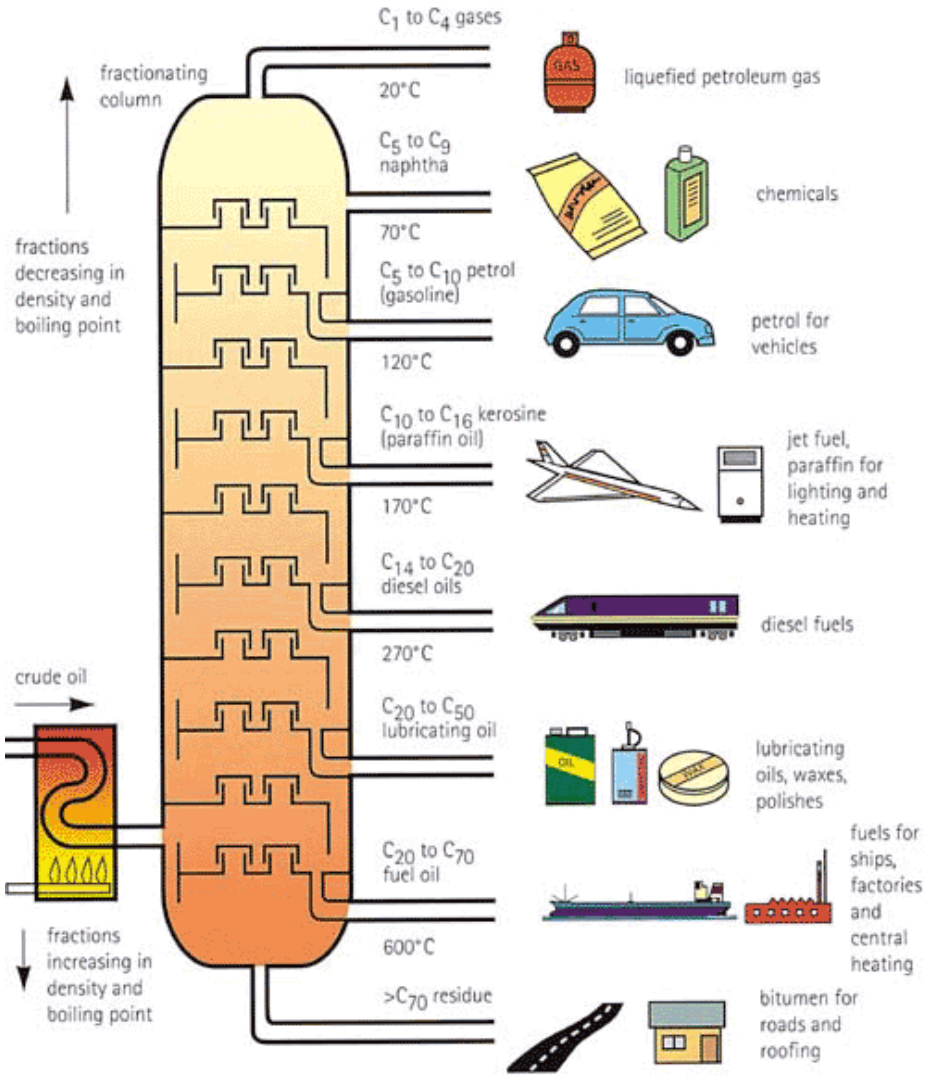
19th Euro Fed Lipid Congress and Expo

Fats, Oils and Lipids: Fats, Oils and Lipids: from Raw Materials to Consumer Expectations

17-20 September 2023, Poznan, Poland

MINERAL OIL HYDROCARBONS (MOH): DEFINITION*

a wide range of products deriving from petroleum distillation fractions

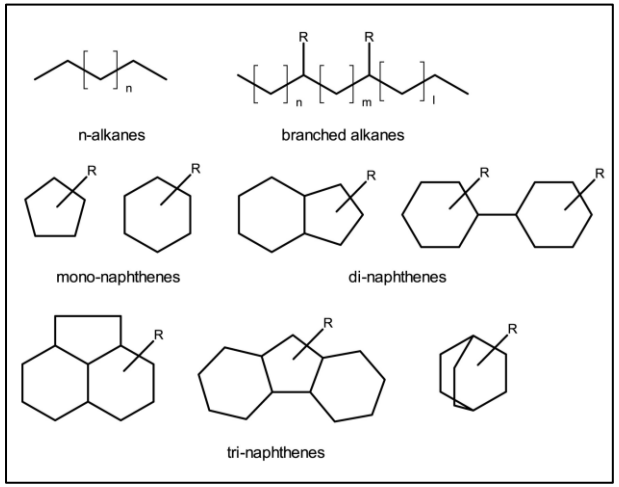


Containing mainly:

MOSH

Mineral oil saturated hydrocarbons

-n-alkane
- isoalkane
- cycloalkane

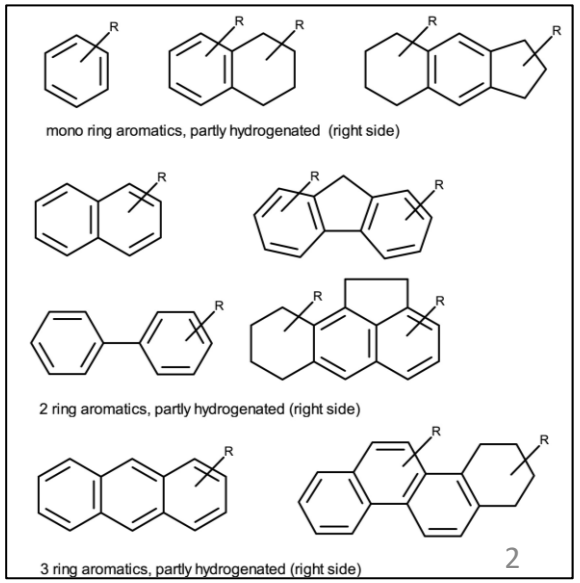


MOAH

Mineral oil aromatic hydrocarbons

May contains also:

-Aromatic hydrocarbons, mainly alkylated



*Biedermann M, Fiselier K, Grob K Aromatic hydrocarbons of mineral oil origin in foods: Method for determining the total concentration and first result. J Agric Food Chem. 2009;57:8711- 21.

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- 1989

LC-GC&MOH

Original Research Papers

Partially Concurrent Eluent Evaporation with an Early Vapor Exit; Detection of Food Irradiation through Coupled LC-GC Analysis of the Fat

Maurus Biedermann, Konrad Grob*, and **Werner Meier**
Kantonales Labor, P. O. Box, CH-8030 Zürich, Switzerland

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1989

2008/2009

LC-GC&MOH

Original Researcl

Partially Concurrent Eluent Evaporati
Exit; Detection
Ana

efsa
European Food Safety Authority

ABOUT NEWSROOM TOPICS RESOURC

Home / Newsroom / Sunflower oil: contamination with mineral oil from Ukrain...

Sunflower oil: contamination with mineral oil from Ukraine - Update

Published: 28 April 2008

BfR
Risiken erkennen – Gesundheit schützen

Migration of mineral oil from packaging materials to foodstuffs

BfR Opinion No. 008/2010, 09 December 2009

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1989

2008/2009

2012

BfR
Risiken erkennen – Gesundheit schützen

➢ **<u>Improvement of the analytical methods is recommended</u>**.

➢ Future monitoring should **distinguish between MOAH and MOSH**, and between **subclasses of MOSH** <u>based on carbon numbers and chemical structures</u>.

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Original Researcl

Partially Concurrent Eluent Evaporati
Exit; Detection
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efsa
European Food Safety Authority

EFSA Journal 201

NEWS | 24.10.2019

foodwatch Laboratory Tests: Suspected Carcinogenic Mineral Oil Residues in Baby Milk

2019

MINERAL OIL IN FOODS

High number of calendars made out of virgin fibre board

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1989

2008/2009

2012

2019

LC-GC&MOH

Original Researcl

Partially Concurrent Eluent Evaporati
Exit; Detection
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efsa
European Food Safety Authority
Supporting Publications

TECHNICAL REPORT

APPROVED: 15 November 2019

doi:10.2903/sp.efsa.2019.EN-1741

Rapid risk assessment on the possible risk for public health due to the contamination of infant formula and follow-on formula by mineral oil aromatic hydrocarbons (MOAH)

ests: Suspected Carcinogenic
Baby Milk

This scientific output, published on 28 August 2013,
2012*.

Recommendations

- Analytical methods to **identify 3-7 PAC should be routinely applied when MOAH are detected in food**.

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- 1989 → LC-GC&MOH
- 2008/2009 → Original Researcl
  Partially Concurrent Eluent Evaporati
  Exit; Detection
  Ana
- 2012 → TECHNICAL REPORT
  APPROVED: 15 November 2019
  doi:10.2903/sp.efsa.2019.EN-1741
- 2019 → EUROPEAN COMMISSION
  Health and Food Safety Directorate General
- 2022 → sante.g.3(2022)8681453

Standing Committee on Plants, Animals, Food and Feed
Section *Novel Food and Toxicological Safety of the Food Chain*
19 October 2022

EU set a *de facto* limits of **for Total MOAH,** which is **2 mg/kg** for fats an oils

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2008/2009

2012

2019

2022

2023

LC-GC&MOH

Original Researcl

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Exit; Detection
Ana

TECHNICAL REPORT

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Risiken erkennen – Gesundheit schützen

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European Food Safety Authority
Supporting Publications

l 201

e

ected Carcinogenic

SCIENTIFIC OPINION

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),
Dieter Schrenk, Margherita Bignami, Laurent Bodin, Jesús del Mazo, Bettina Grasl-Kraupp,
Christer Hogstrand, Laurentius (Ron) Hoogenboom, Jean-Charles Leblanc,
Carlo Stefano Nebbia, Elsa Nielsen, Evangelia Ntzani, Annette Petersen, Salomon Sand,
Tanja Schwerdtle, Christiane Vleminckx, Heather Wallace, Jan Alexander,
Christophe Goldbeck, Konrad Grob, Jose Ángel Gómez Ruiz, Olaf Mosbach-Schulz,
Marco Binaglia and James Kevin Chipman

EFSA OPINION-ANALYTICAL FOCUS

2023

SCIENTIFIC OPINION

ADOPTED: 12 July 2023

doi: 10.2903/j.efsa.2023.8215

Update of the risk assessment of mineral oil hydrocarbons in food

Generation of further data for the refinement of the risk assessment was recommended:

MOSH **MOAH**

➢ **Improvement of analytical methodology**

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

- **Generally considered of no concern at the concentration found**.
- **More data on human MOSH tissue concentrations** or **development and use of biomarkers of exposure** are needed, particularly from individuals born after 1995.
- More data on MOAH composition **by aromatic ring number in food are needed, in particular with respect to the levels of 3- or more ring MOAH**
- Sources of food contamination should be investigated when MOAH are detected. To this end, **more selective and sensitive analytical methods should be implemented**.
- The **contribution from the environment needs further investigation**.

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1989

2008/2009

2012

2019

2022

2023

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Guidance on sampling, analysis and data reporting for the mo
hydrocarbons in food
materials

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

Outline of the Analytical approach

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✓*Need for matrix-tailored sample prep protocols*

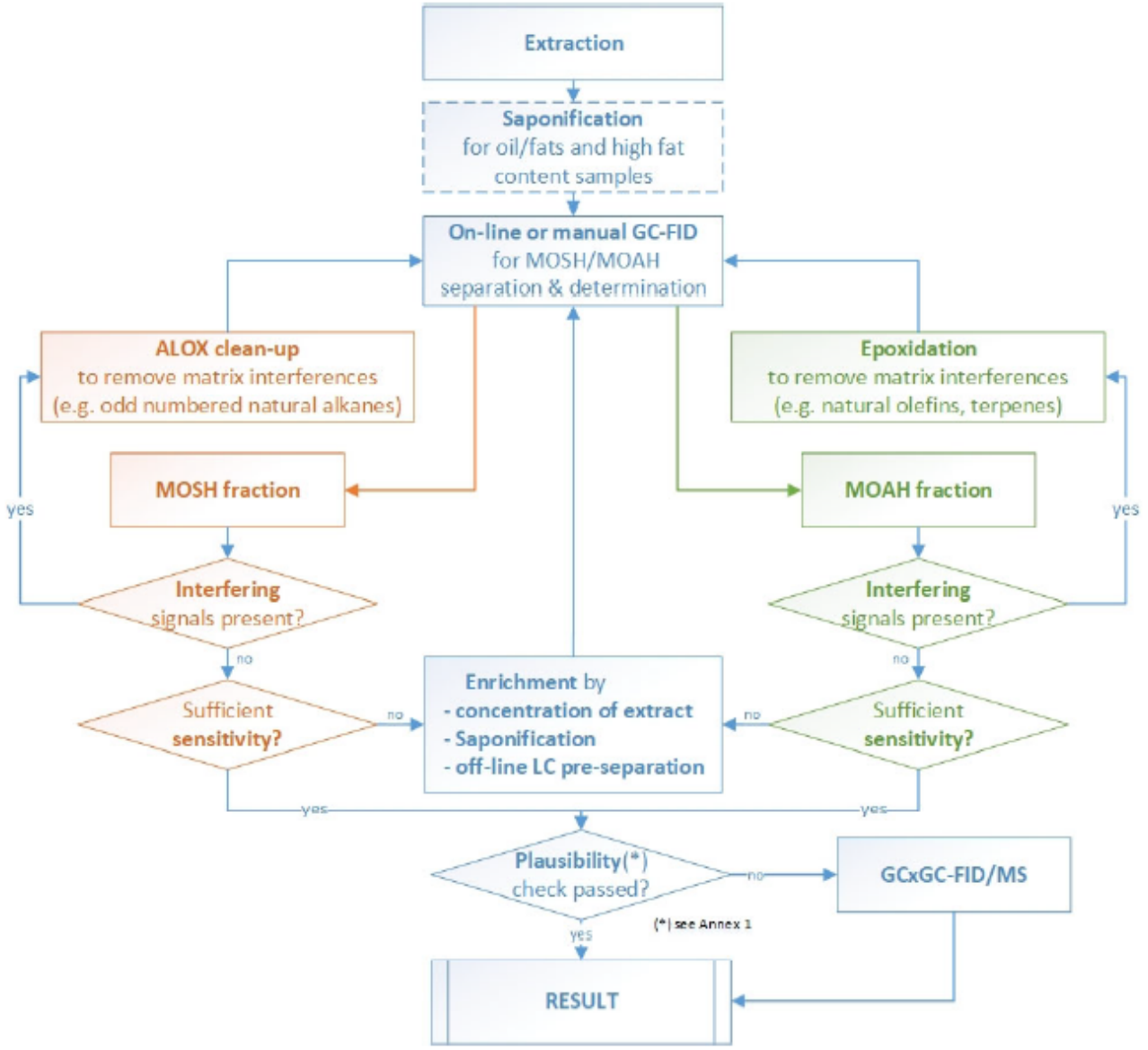

Figure 5 Decision tree on the use of auxiliary methods.

ISSN 1831-

European Commission

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2023

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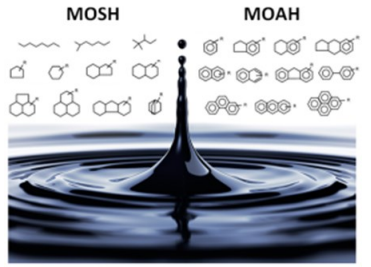
Is the data reliable?

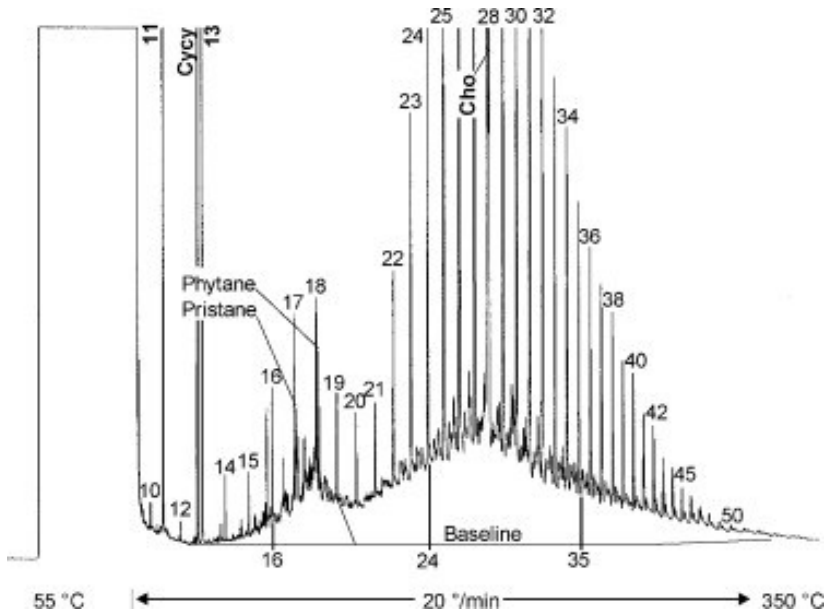


Sample Preparation

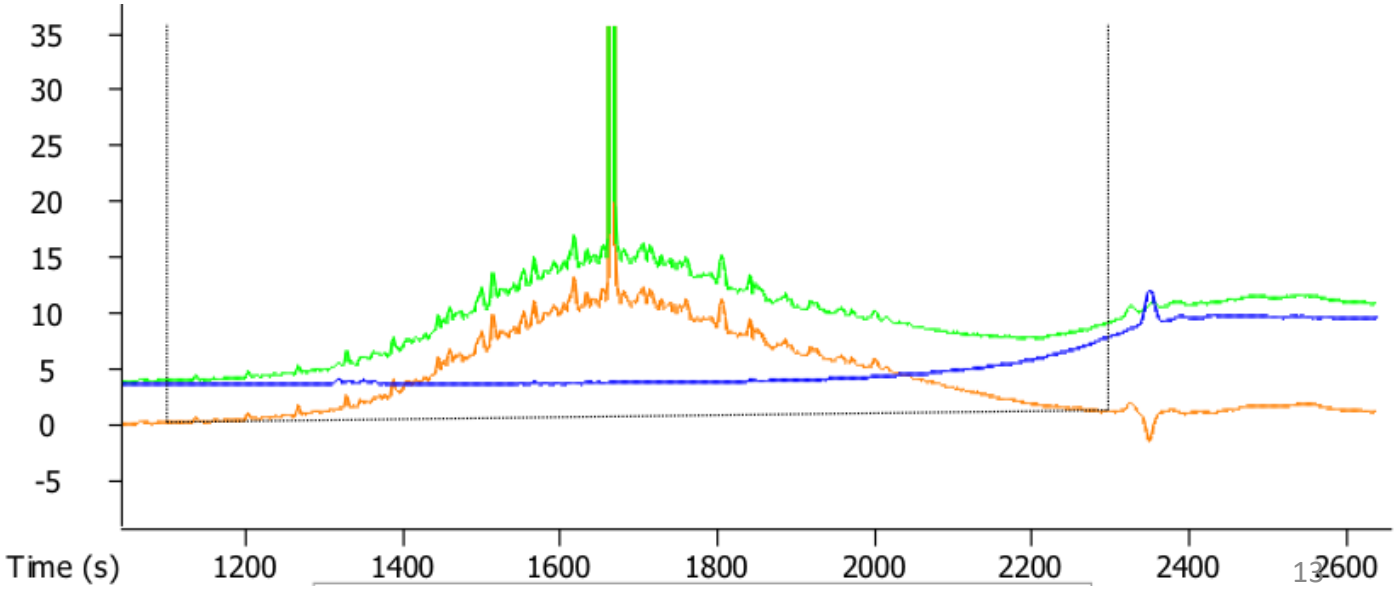
Data Interpretation

Data Integration

20% of total variability



Baseline



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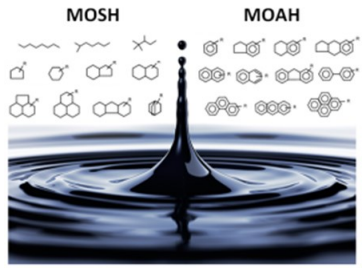
Is the data reliable?



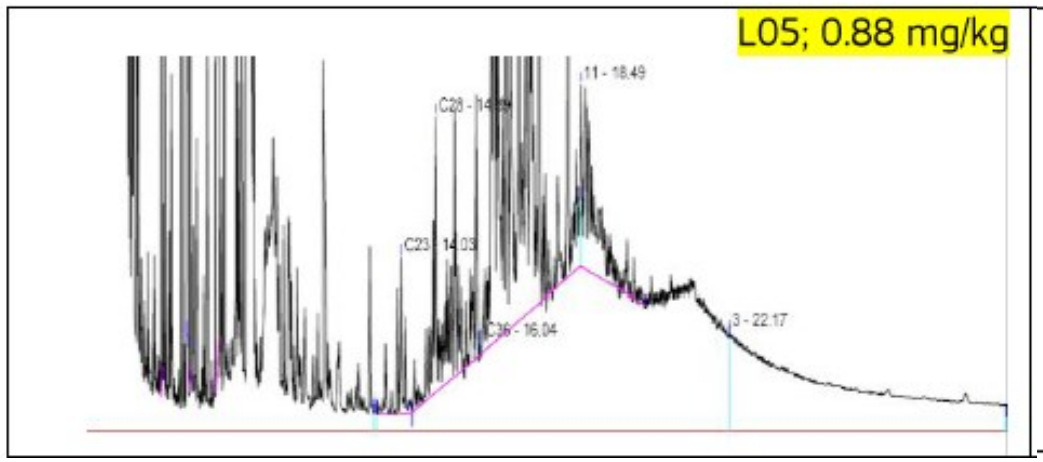
Sample Preparation

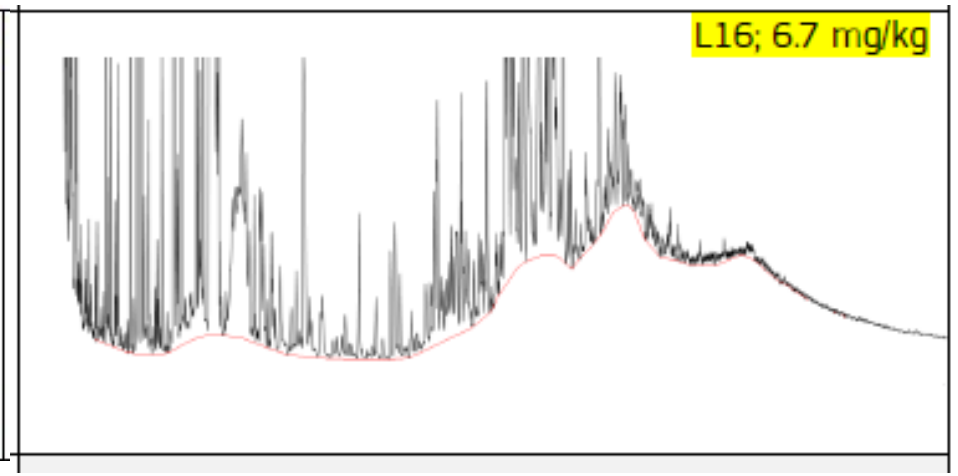
Data Interpretation

Data Integration

20% of total variability

Riding peaks subtraction





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Is the data reliable?

MOSH MOAH

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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.

2022

Sample Preparation

Data Interpretation

20% of total

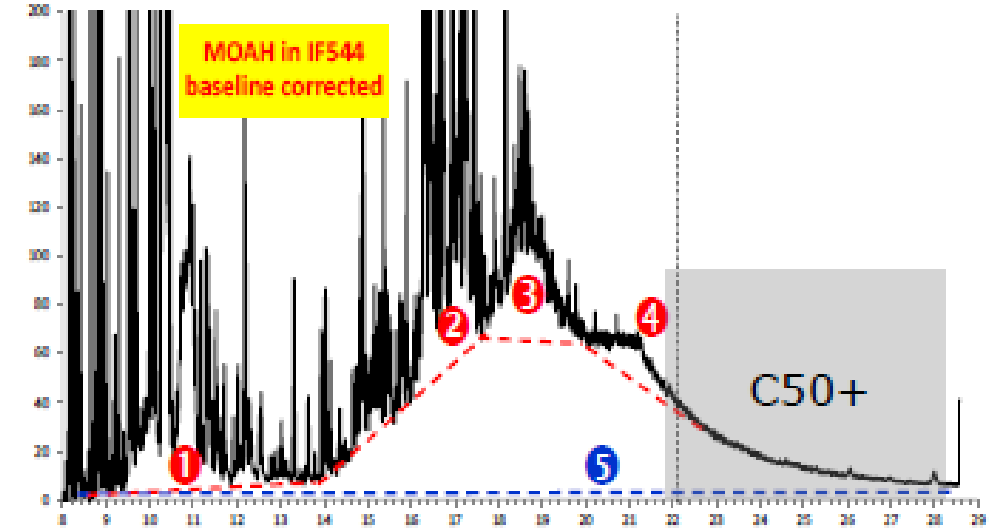

Figure 3: MOAH in IF 544 chromatograms - Comparison of different integration approaches presented by laboratories having reported total mass fractions of MOAH (C10-C50) ranging from 0.88 to 6.7 mg/kg.

← 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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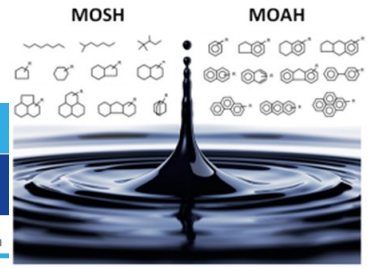
Is the data reliable?



JRC TECHNICAL REPORT

Sample Preparation

Data Interpretation

Data Integration

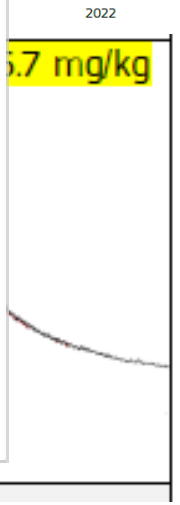
20% of total variability

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

- ✓ Improvement
- ✓ Standardization is needed

- ✓ Extra information
- ✓ Improvement sample prep

- ✓ Harmonization



Outline of the Analytical approach

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✓*Need for matrix-tailored sample prep protocols*

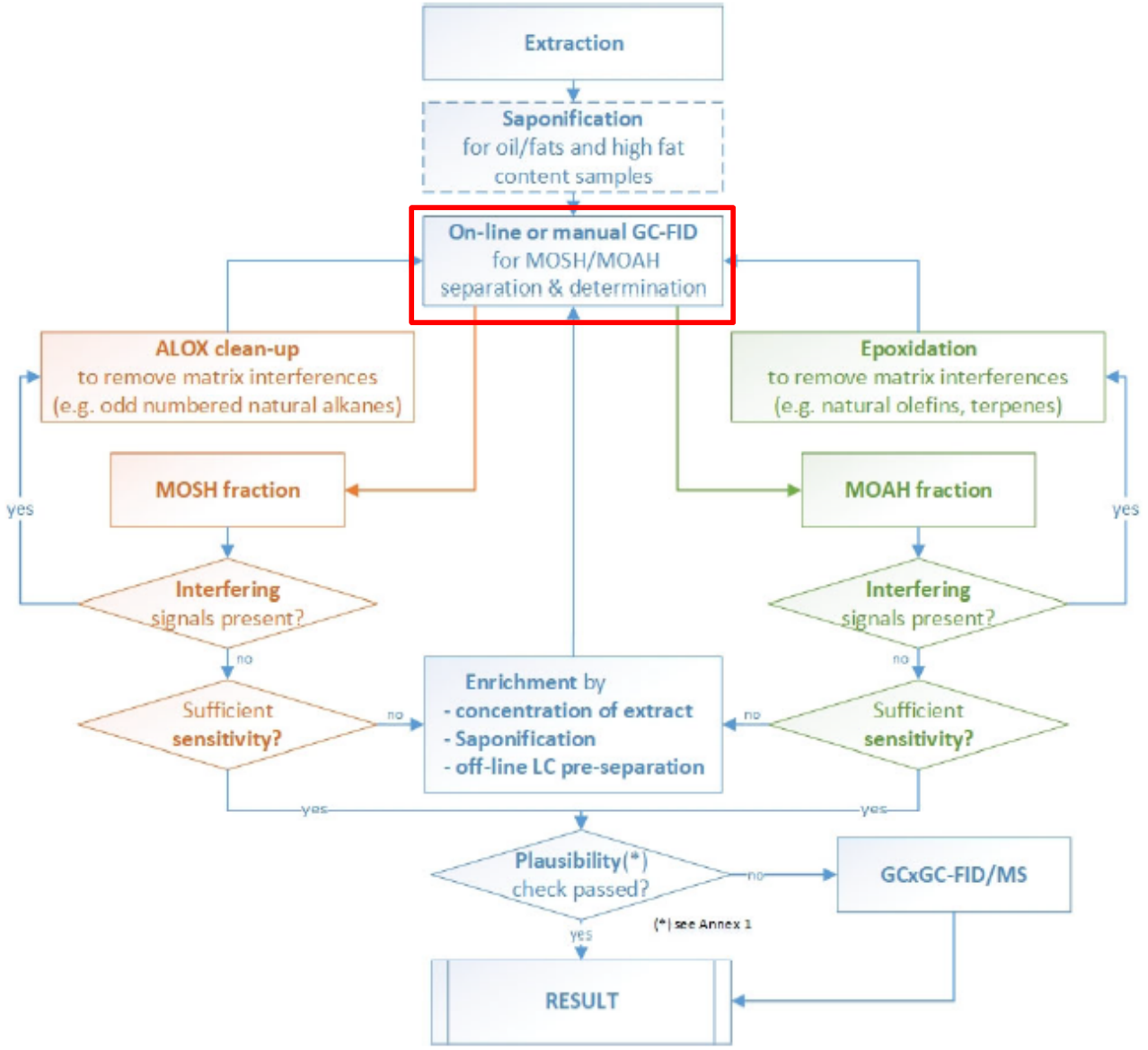

Figure 5 Decision tree on the use of auxiliary methods.

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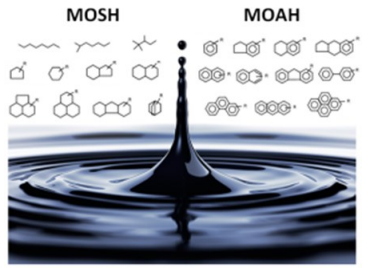
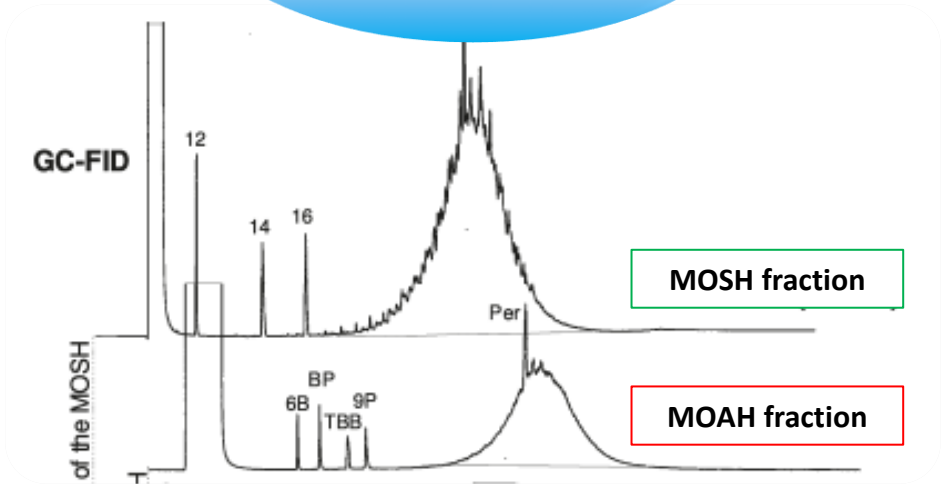
MOH AND MULTIDIMENSIONAL TECHNIQUEs

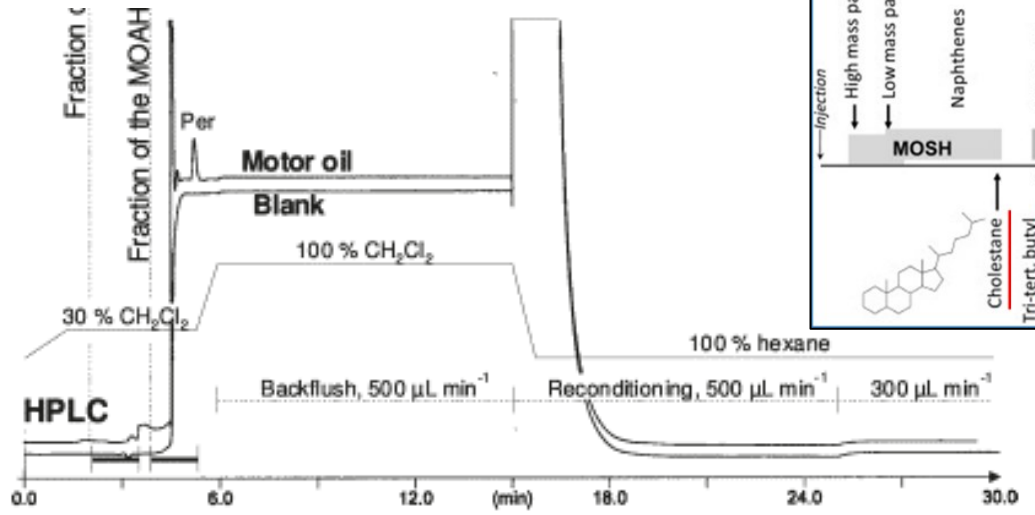


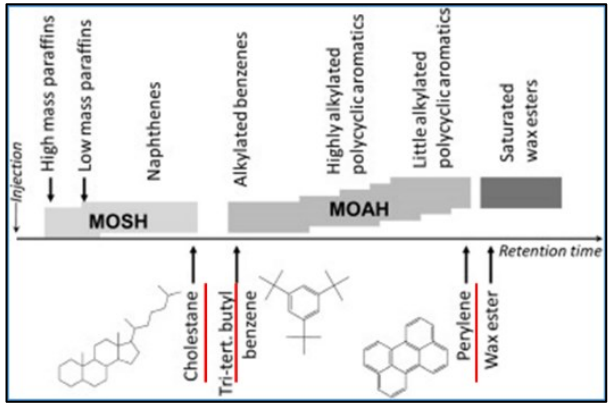
MOH

LC-GC

ROUTINE METHOD







+ C11, C13 and CyCy (cyclohexylcyclohexane)
  - C11/CyCy loss of volatiles
  - C13/CyCy possible coelution
  - CyCy **quantification**

+ 5B (pentyl benzene), 1MN (1-Me Na) and 2MN (2-Me Na)
  - 5B loss of volatiles
  - 1MN=2MN possible coelution/ **quantification**

C10-C50

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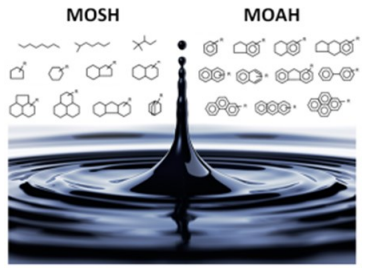
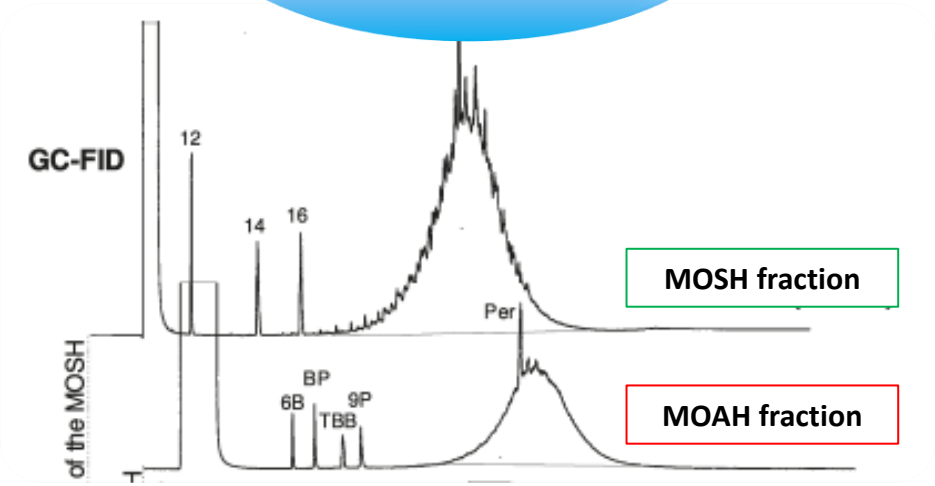
MOH AND MULTIDIMENSIONAL TECHNIQUEs



MOH

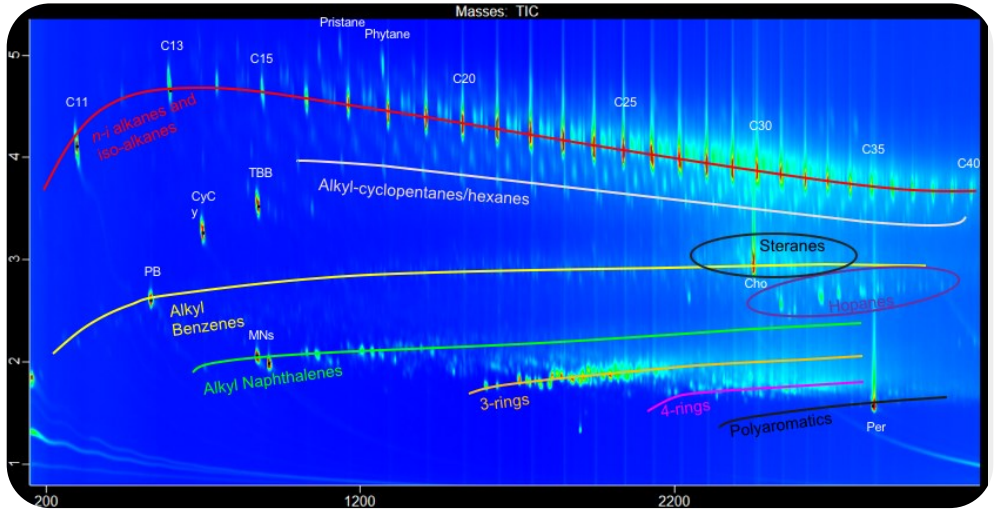
LC-GC

ROUTINE METHOD



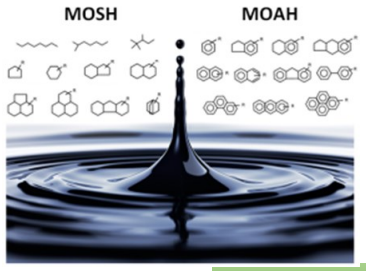
GC×GC-MS

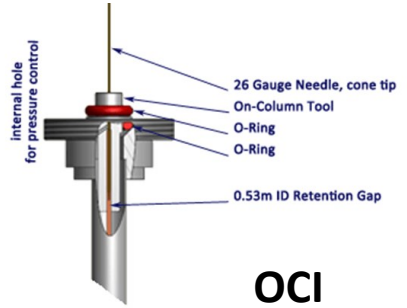
CONFIRMATORY METHOD



MOH AND MULTIDIMENSIONAL TECHNIQUEs

2021





OCI

MOH

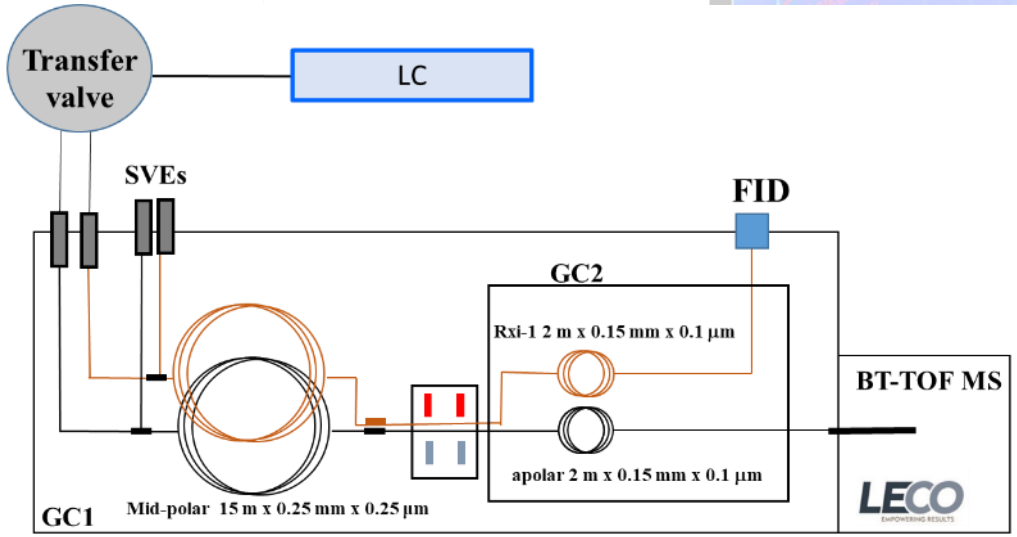
LC-GC

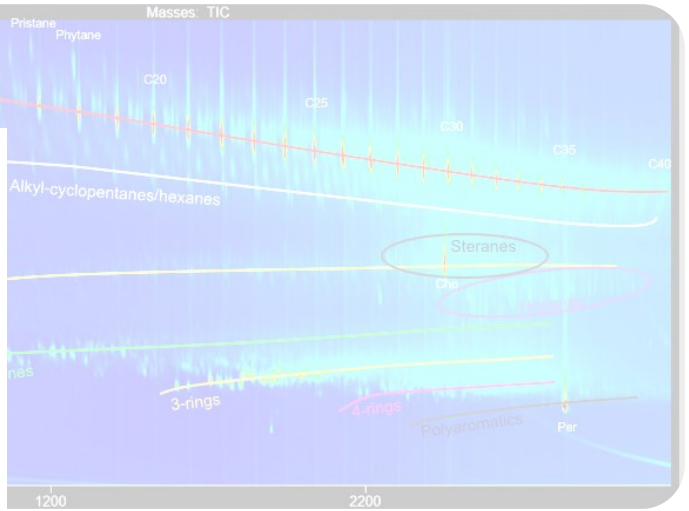
ROUTINE METHOD

GC×GC

CONFIRMATORY METHOD

LC-2GC×GC-FID/MS





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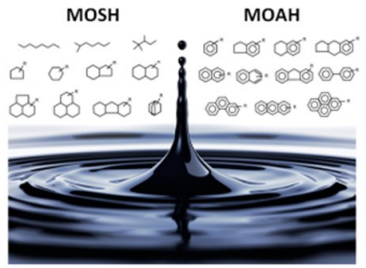
MOH & DATA INTEGRATION

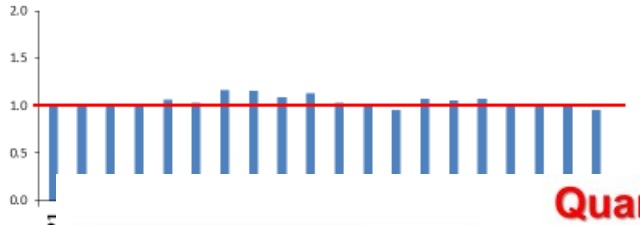


Quantification

Paraffin Mixture + n-Alkanes Mix

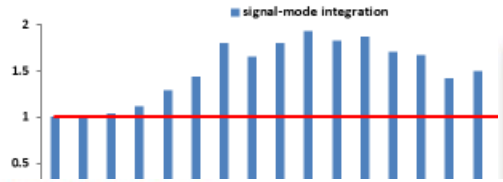
LC-GC-FID

1D



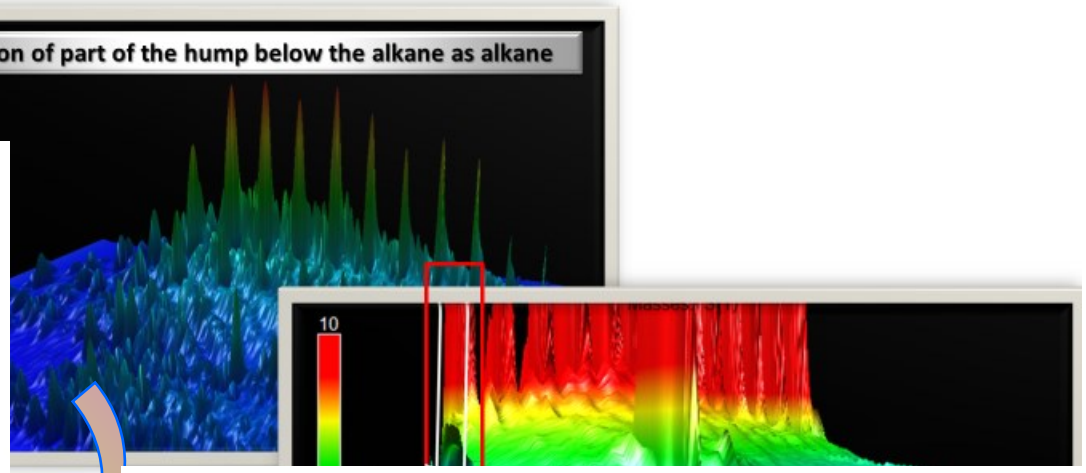
LC-GC×GC-FID

2D

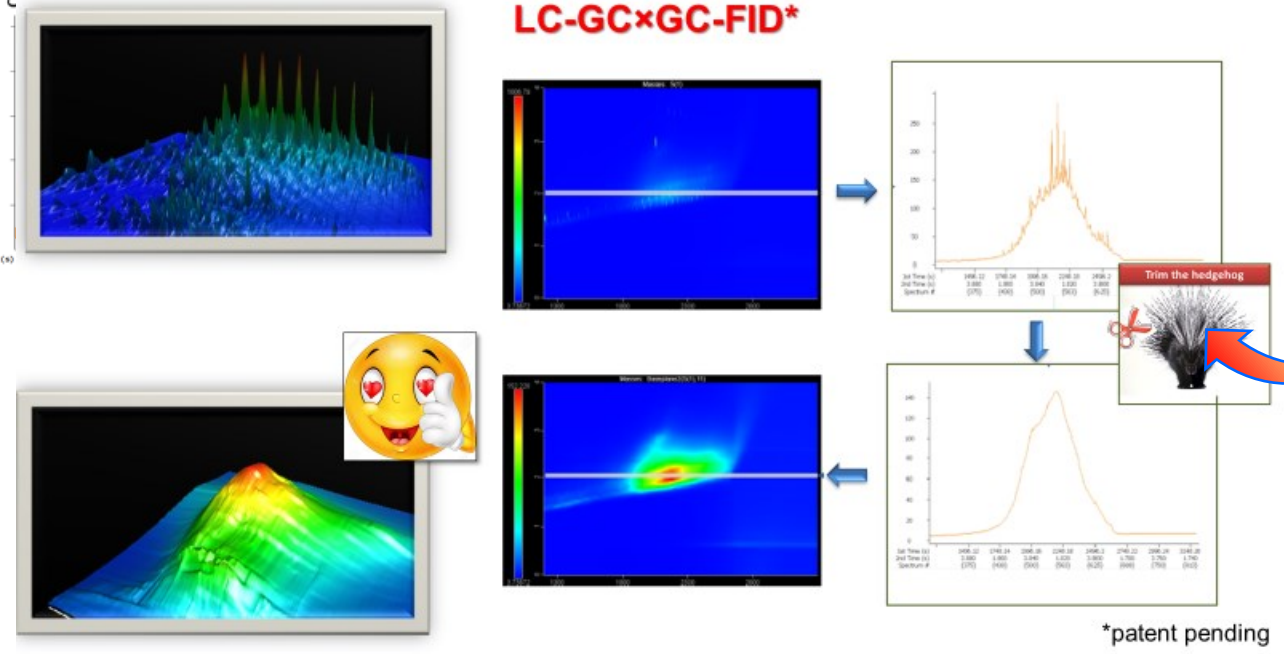


Quantification
LC-GC×GC-FID

Integration of part of the hump below the alkane as alkane



Quantification
LC-GC×GC-FID*



*patent pending

Journal of Chromatography A 1643 (2021) 462044

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

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LC-GC×GC-FID VALIDATION

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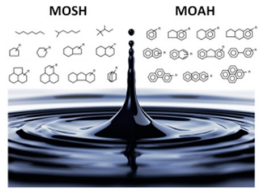
LC-GC×GC-FID VALIDATION



European Commission

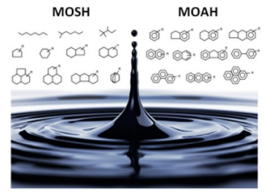
JRC TECHNICAL REPORT

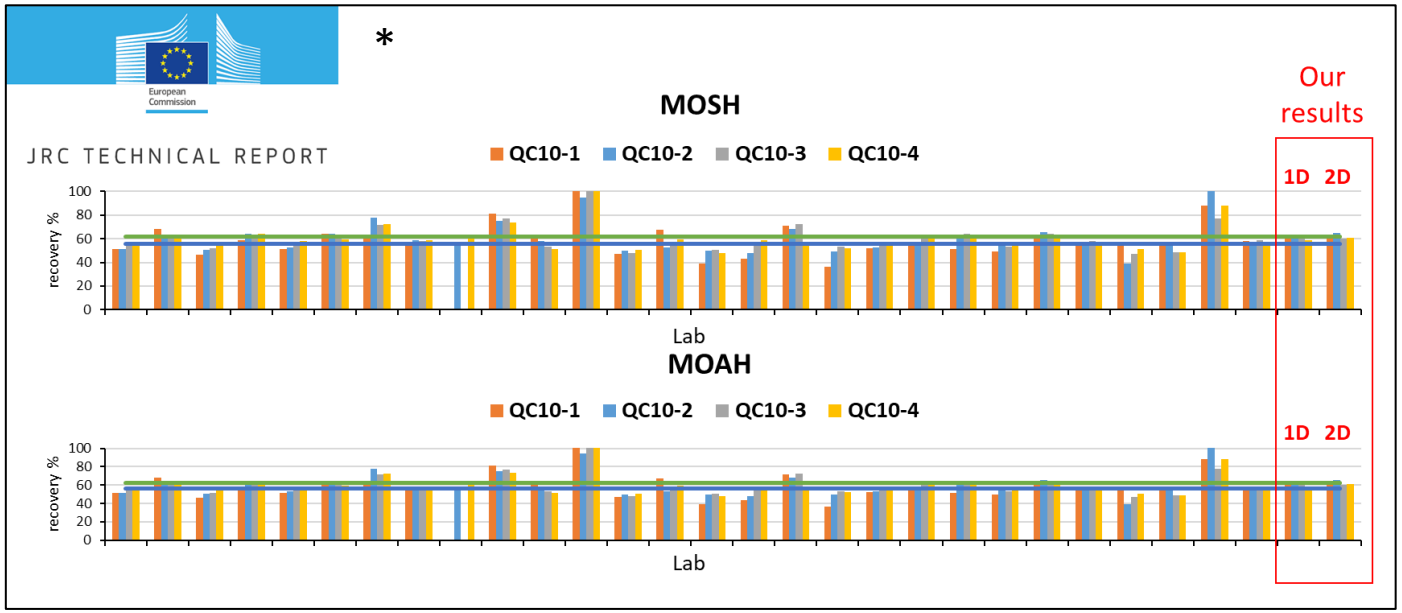
Determination of MOSH/MOAH in Shell SN500* mineral oil

JRC IF 2021-03 - The third interlaboratory comparison

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

LC-GC×GC-FID Quantification





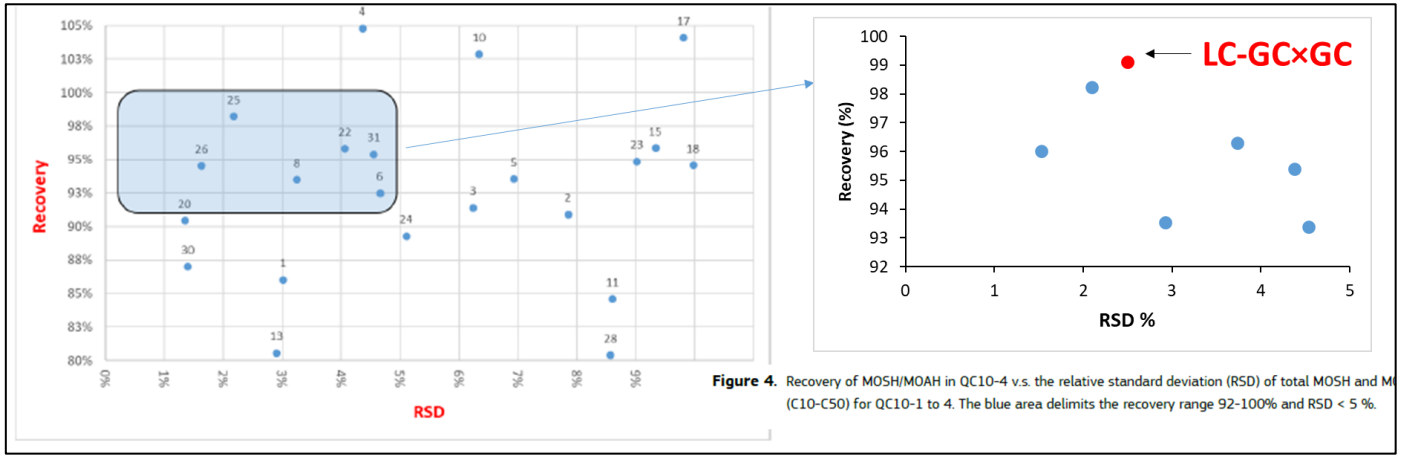


Figure 4. Recovery of MOSH/MOAH in QC10-4 v.s. the relative standard deviation (RSD) of total MOSH and M (C10-C50) for QC10-1 to 4. The blue area delimits the recovery range 92-100% and RSD < 5 %.

✓ **Less integration variability**

* C. Goncalves, L. Karasek, S. Bratinova, P. Robouch, G. Beldi, C. Senaldi, S. Valzacchi, E. Hoekstra, Determination of MOSH/MOAH in Shell SN500 mineral oil. JRC IF 2021-03: the third interlaboratory comparison., 2022.

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EXISTING STANDARD METHODS

- **ISO 17780:2015** - only for MOSH (GC-FID)

- **EN 16995:2017** - for MOSH/MOAH (on-line HPLC-GC/FID)
  - working range starting from 10 mg/kg –
  - currently reduce to 3mg/kg MOSH and 2 mg/kg MOAH (**EN 16995 rev)**

- **EN 17517:2021** – MOSH/MOAH in feed (on-line HPLCGC/FID) > 10 mg/kg

BS EN 17517. Animal feeding stuffs: Methods of sampling and analysis. Determination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with on-line HPLC-GC-FID analysis

- **JRC eILC on integration in 2021**
- **JRC SOP** for Infant formula

bsi. ...making excellence a habit.

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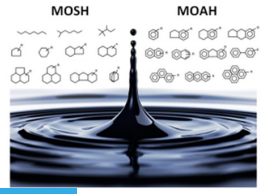
LC-GC×GC-FID VALIDATION



European Commission

JRC TECHNICAL REPORT

Determination of MOSH/MOAH in Shell SN500* mineral oil

JRC IF 2021-03 - The third interlaboratory comparison

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

European Commission

JRC TECHNICAL REPORT

Determination of MOAH in Infant Formula

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

CSN EN 16995

English Version

Foodstuffs - Vegetable oils and foodstuff on basis of vegetable oils - Determination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with on-line HPLC-GC-FID analysis

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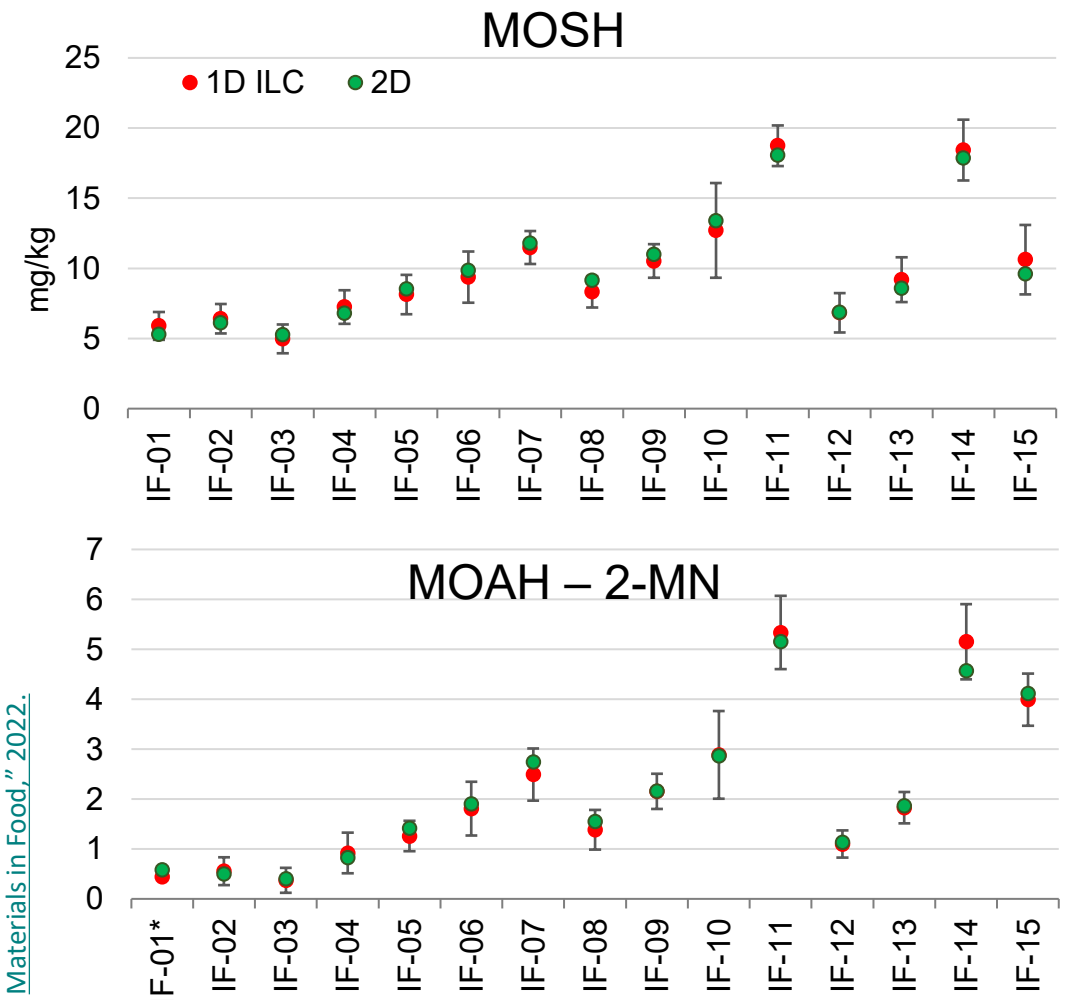
LC-GC×GC-FID/MS

Data Integration

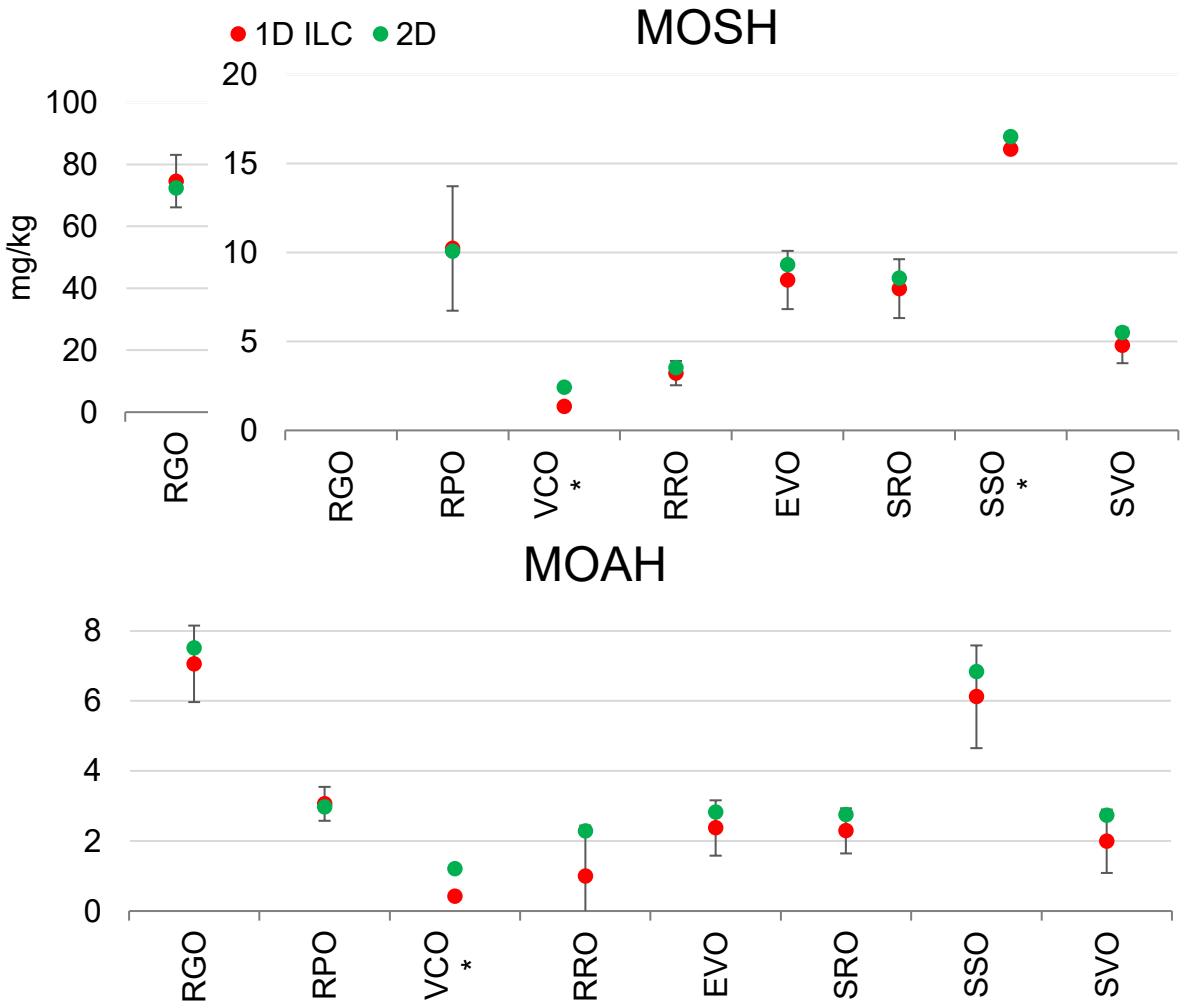
Data Interpretation

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



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.

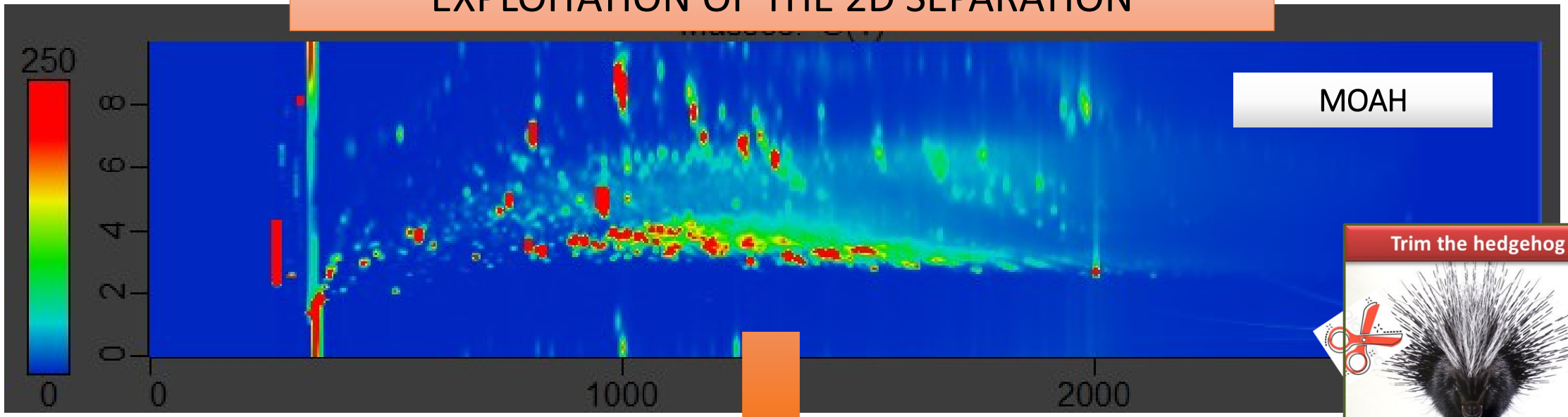
revision of DGF-EN 16995:2017

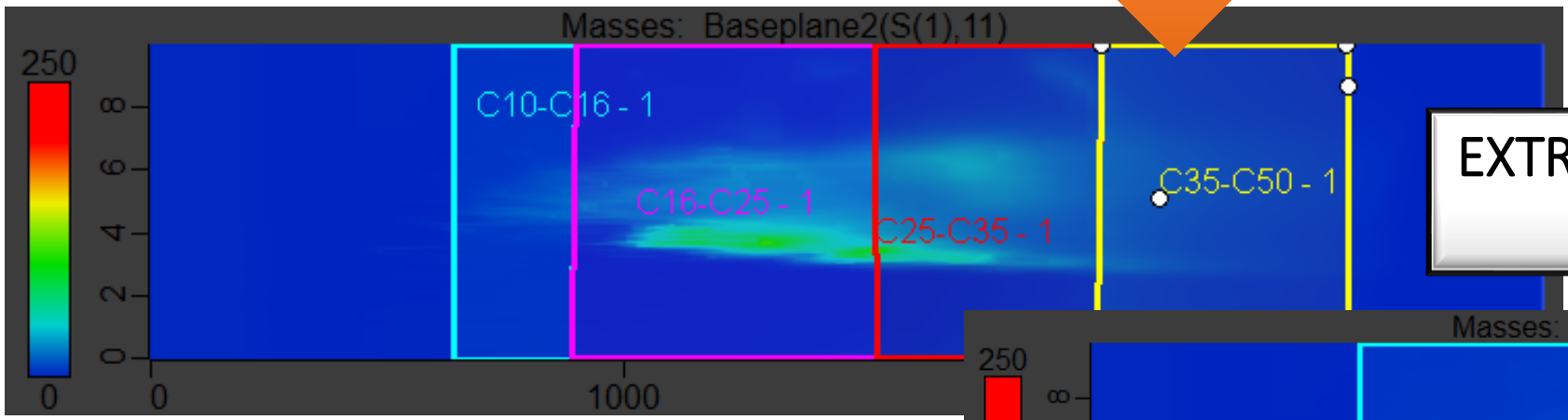


Vegetable oils - Determination of mineral oil saturated hydrocarbons (MOSH) and aromatic hydrocarbons (MOAH) with online coupled HPLC-GC-FID analysis -Method for low limit of quantification. Draft version for updating EN-16995:2017, October 2022-V5.

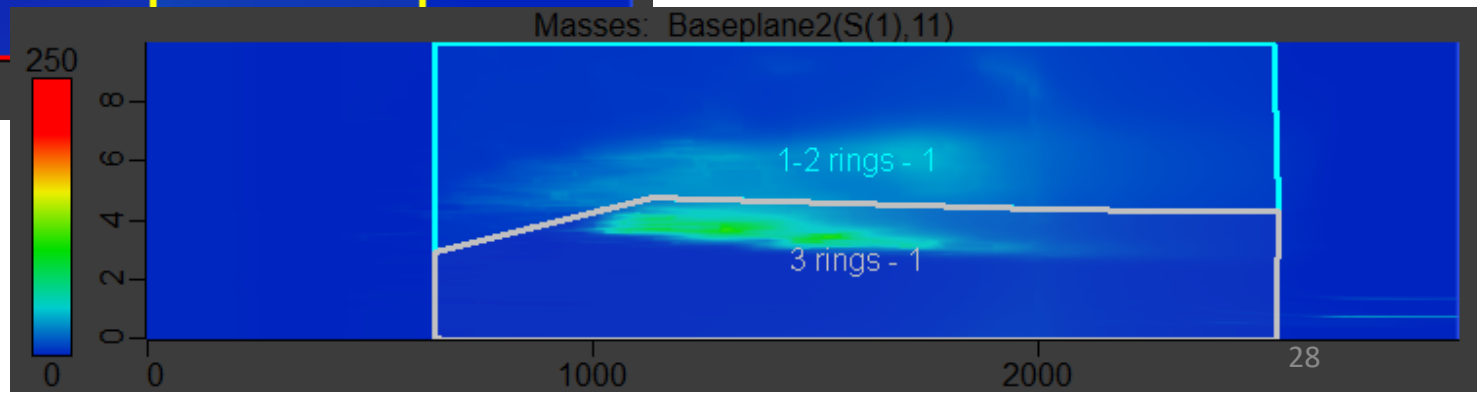
G. Bauwens, L. Barp, G. Purcaro, *Validation of the liquid chromatography-comprehensive multidimensional gas chromatography-time-of-flight mass spectrometer/flame ionization detector platform for mineral oil analysis exploiting interlaboratory comparison data*, **GreenAC (2023) 4 100047**

EXPLOITATION OF THE 2D SEPARATION





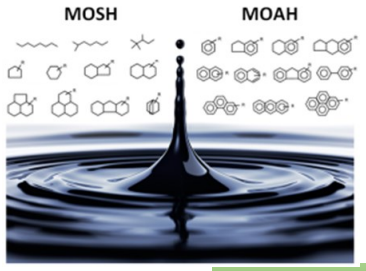
EXTRAPOLATIONS OF DIFFERENT INFORMATION

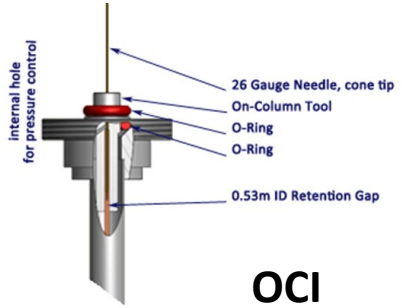


Toxicological relevance

MOH AND MULTIDIMENSIONAL TECHNIQUEs

2021





OCI

MOH

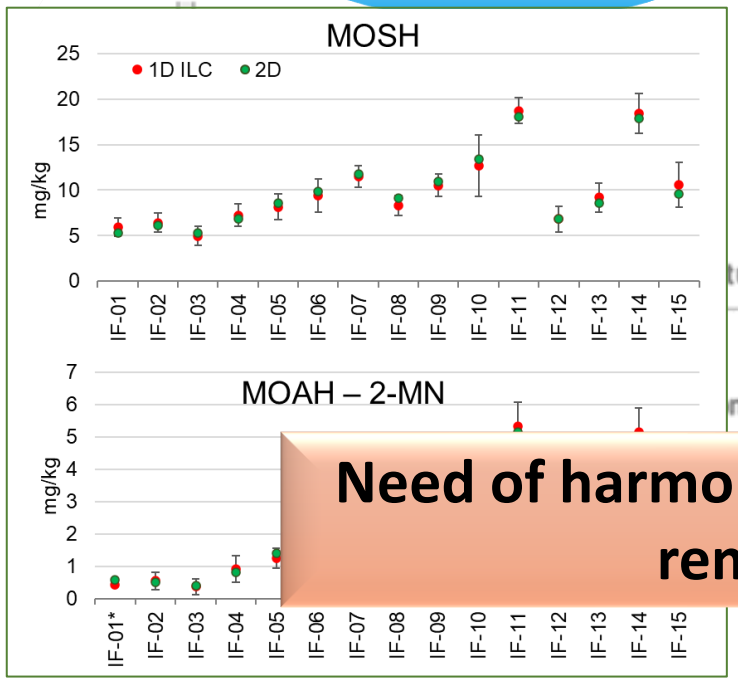
LC-GC

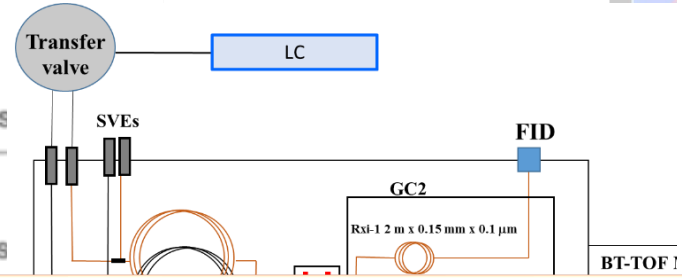
ROUTINE METHOD

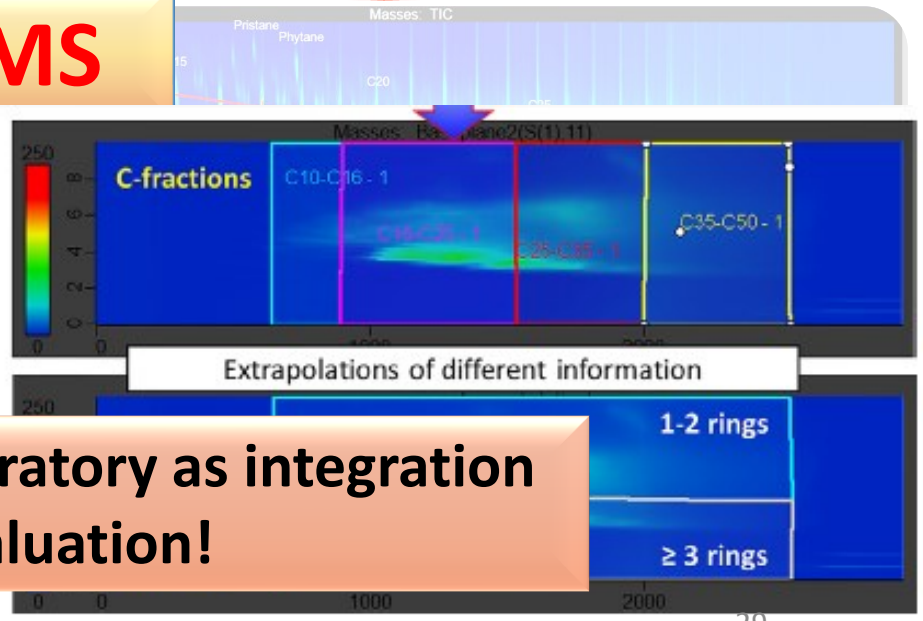
GC×GC

CONFIRMATORY METHOD

LC-2GC×GC-FID/MS







Need of harmonization among control laboratory as integration remains subject to operator evaluation!

Outline of the Analytical approach

✓*Need for matrix-tailored sample prep protocols*

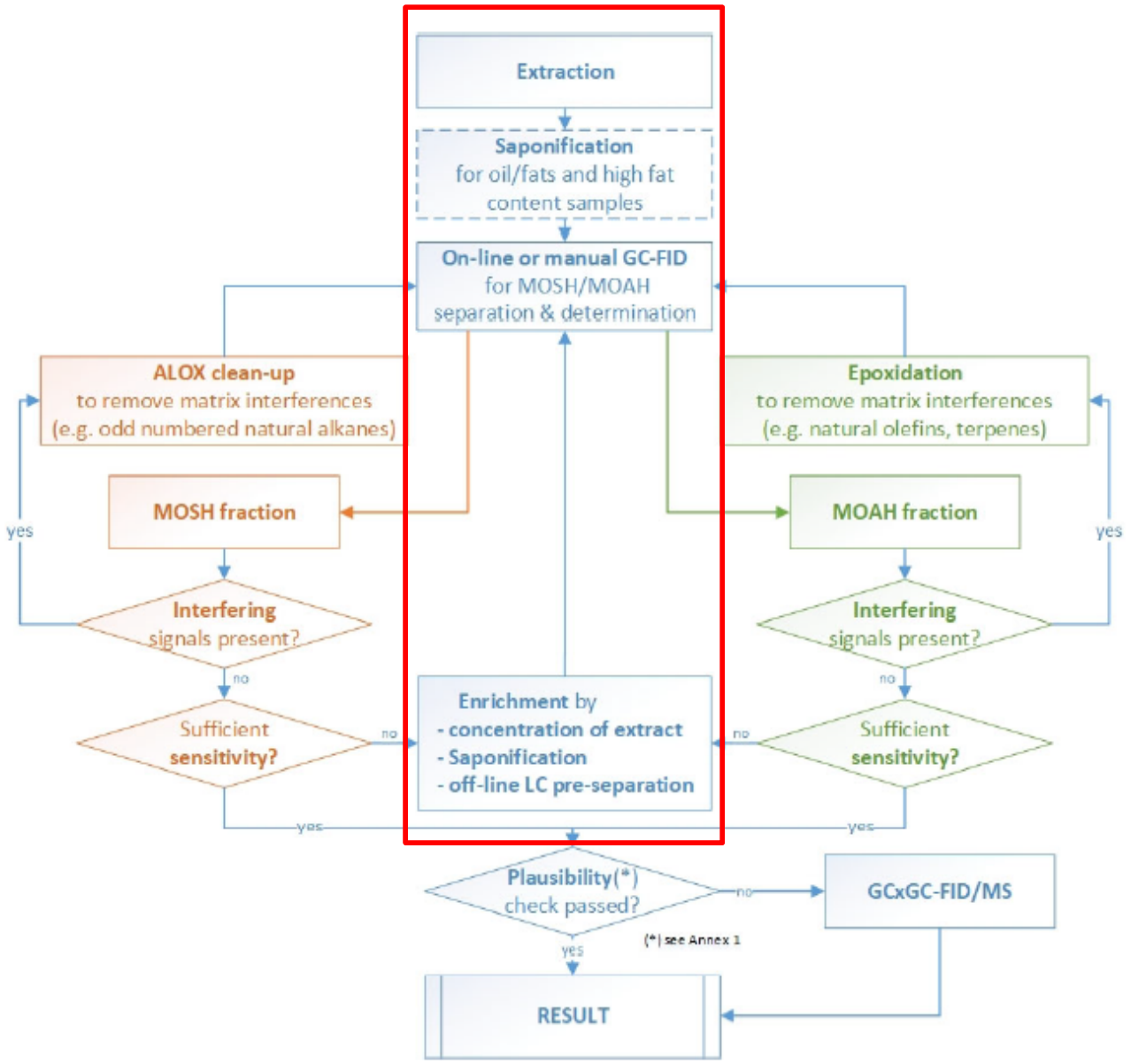


Figure 5 Decision tree on the use of auxiliary methods.

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

➢ Application of the auxiliary method for removing interferences

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MOSH

MOAH

Enrichment by **concentration** of extracts and/or **saponification** and/or off-line LC pre-separation

Saponification

➢ Traditional saponification

DGF C-VI 22 (2020)

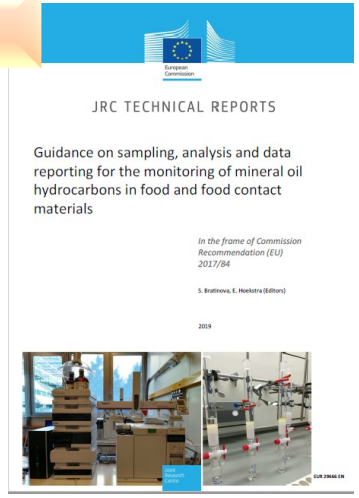
➢ Microwave assisted saponification

Moret et al. Food Chem 196 (2016) 50-57

G. Purcaro, S. Moret, L. S. Conte, **Meat Science**, 81 (2009) 275-280.

V. O. E. Akpambang, G. Purcaro, et al., **Food Addit. Contam.**, 26(7) (2009) 1096-1103.

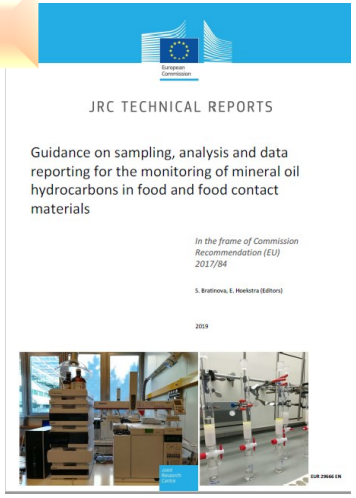
S. Moret*, G. Purcaro, L.S. Conte, **Food Chem**, 122(1) (2010) 333-338. (IF: 3.146)



✓ higher sensitivity, less frequent washing of the LC column.

➢ Application of the auxiliary method for removing interferences

Gembloux Agro-Bio Tech



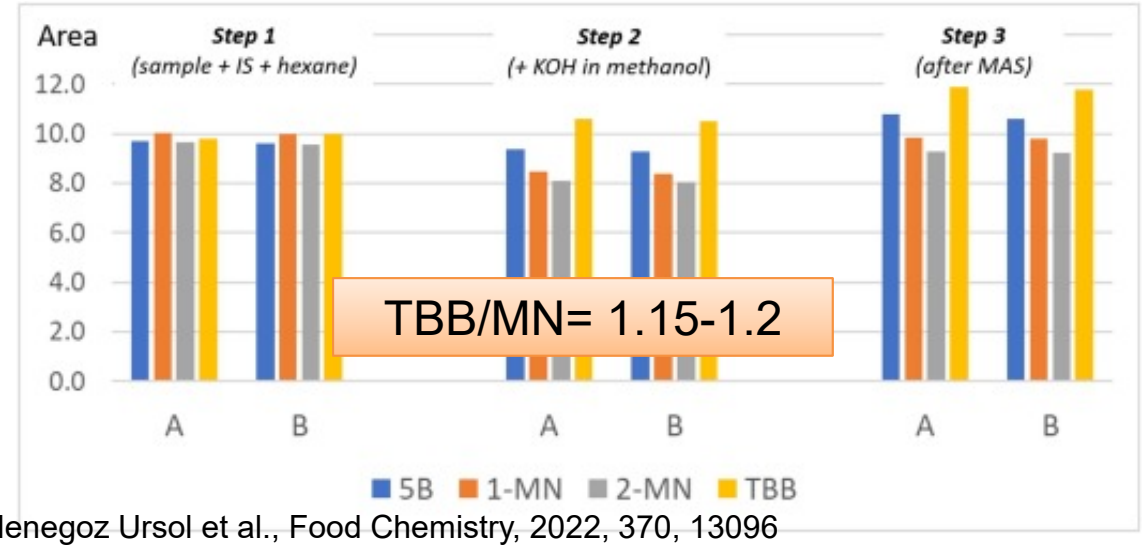
MOAH

Enrichment by **concentration** of extracts and/or **saponification** and/or off-line LC pre-separation

Saponification

- Traditional saponification
  DGF C-VI 22 (2020)
- Microwave assisted saponification-MAS

✓ High fat-content samples.

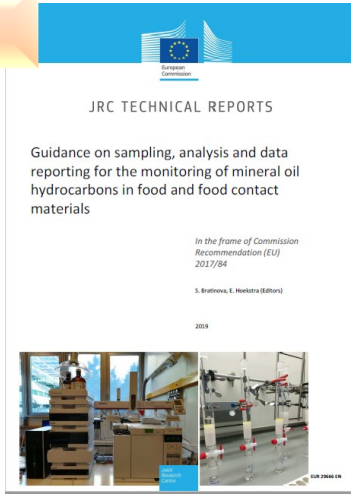


TBB/MN= 1.15-1.2

Menegoz Ursol et al., Food Chemistry, 2022, 370, 13096

➢ Application of the auxiliary method for removing interferences

Gembloux Agro-Bio Tech



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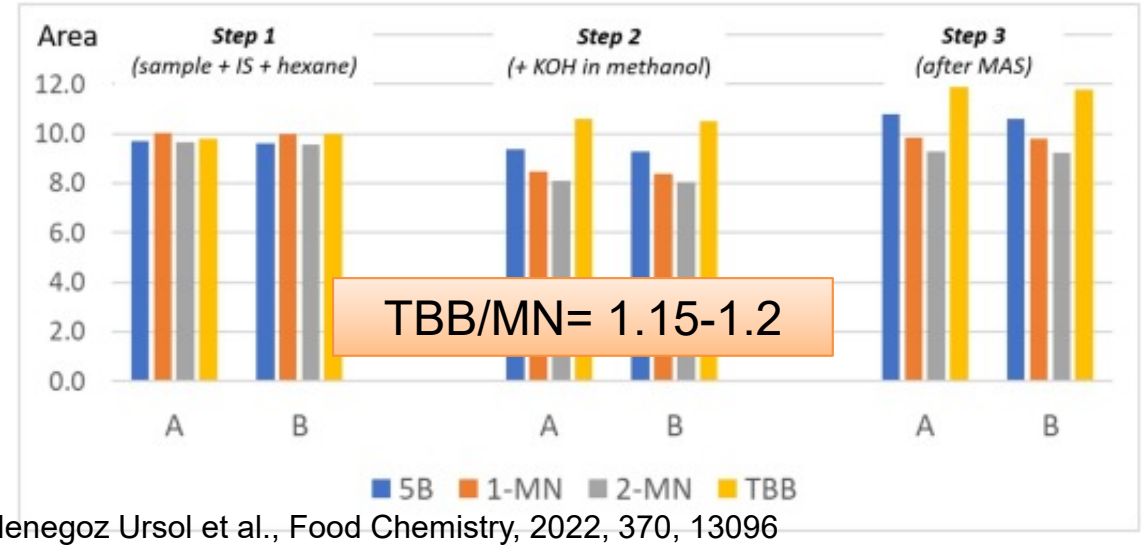

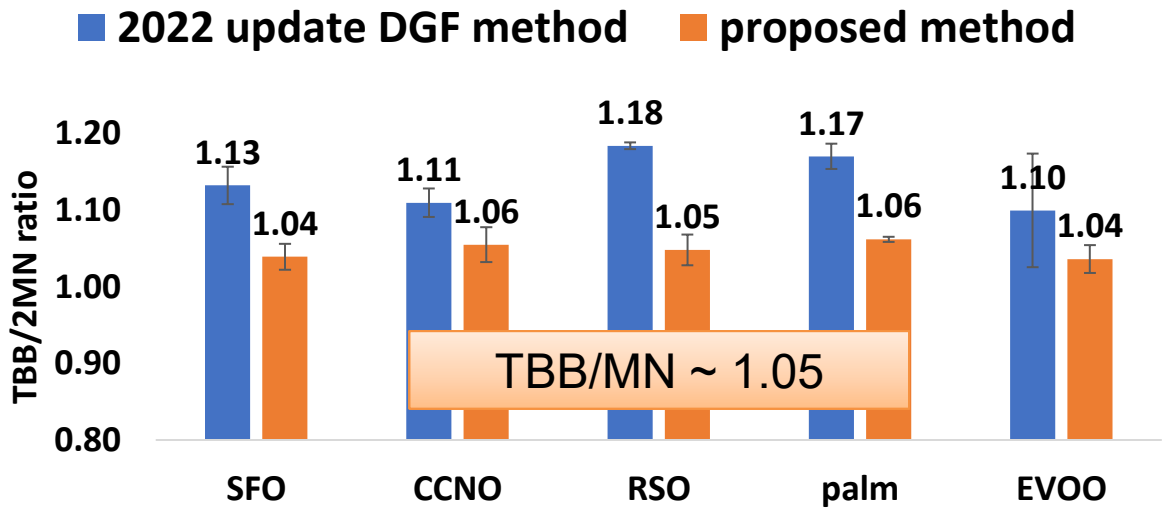

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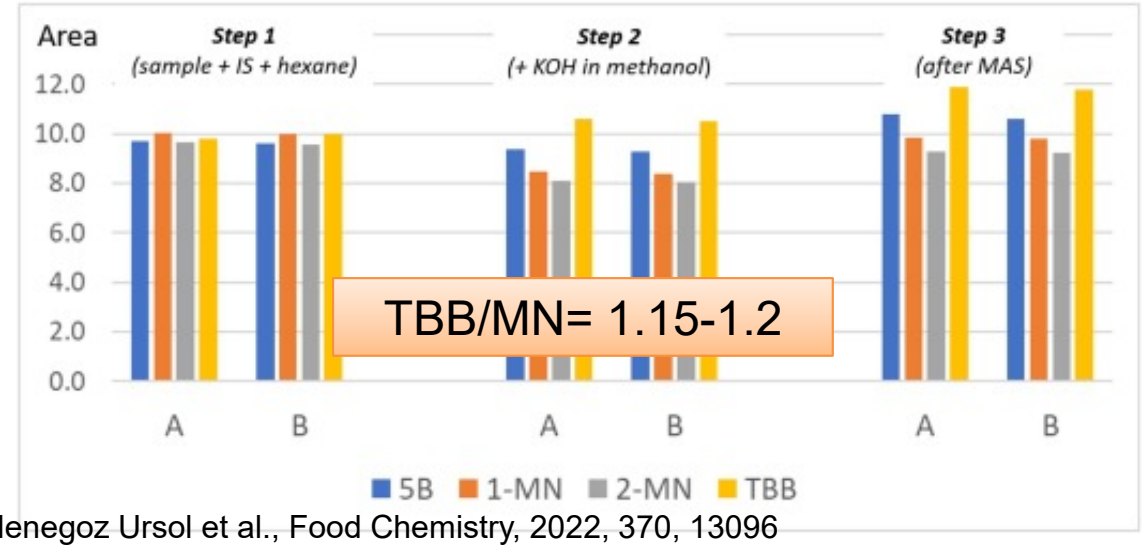

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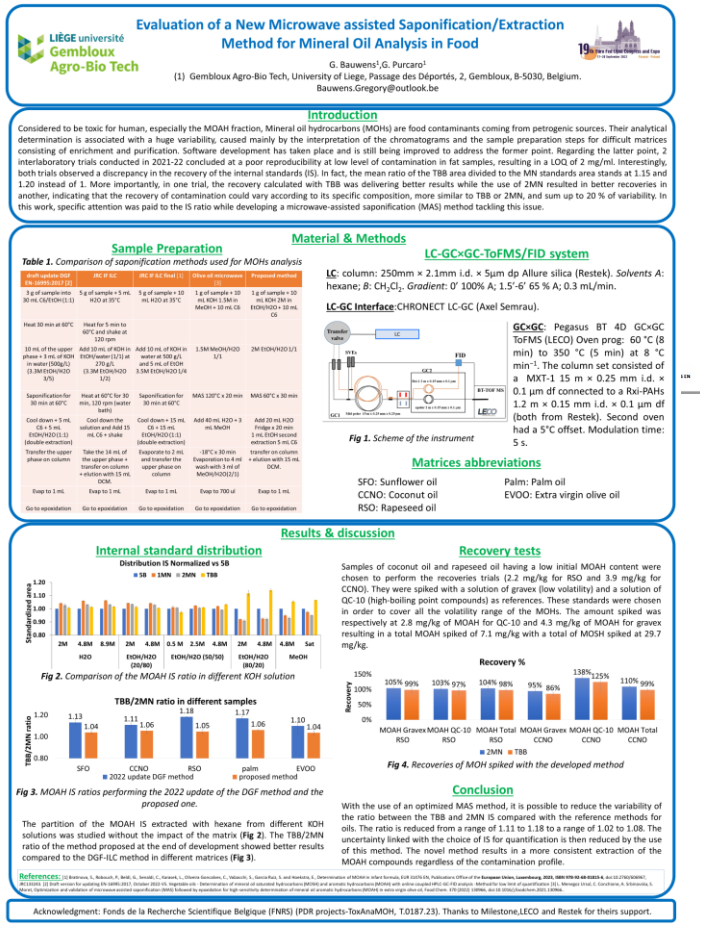
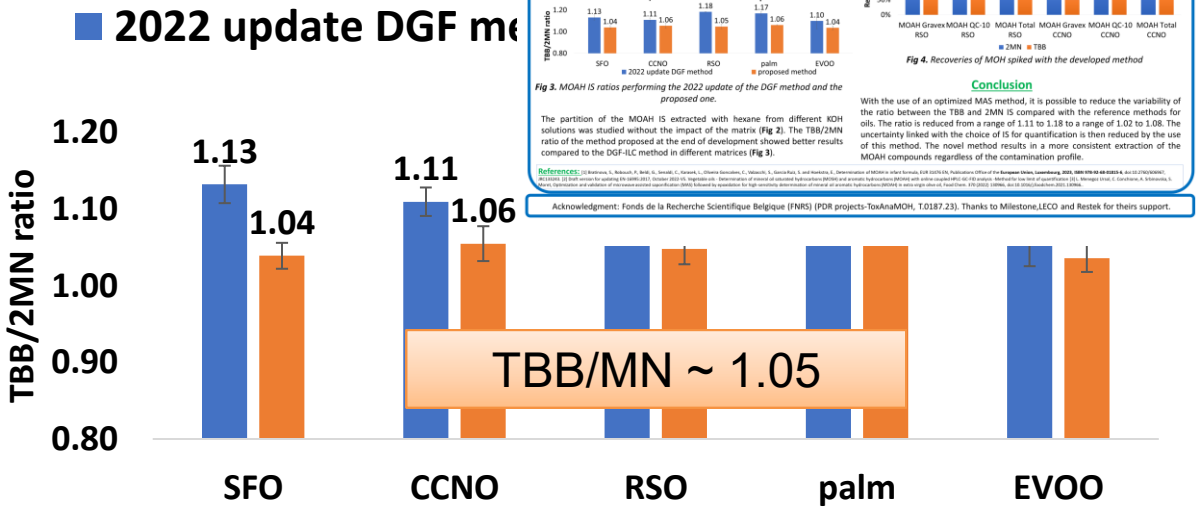
P-Cont_004
Bauwens and Purcaro





Outline of the Analytical approach

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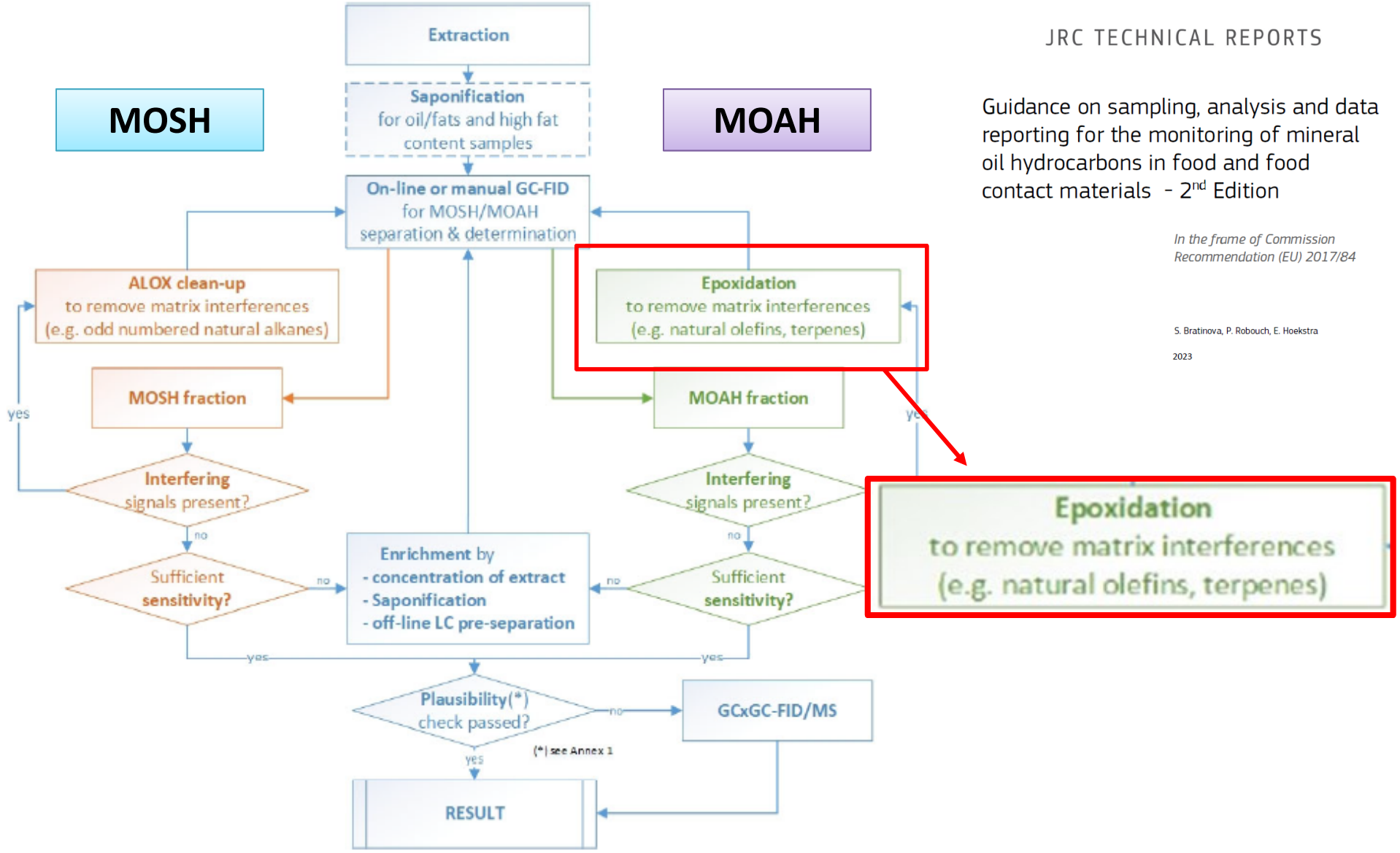
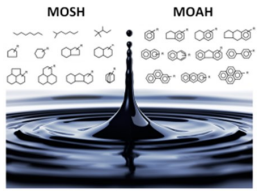


Figure 5 Decision tree on the use of auxiliary methods.

LC-GC×GC-FID/MS & EPOXIDATION

Sample Preparation



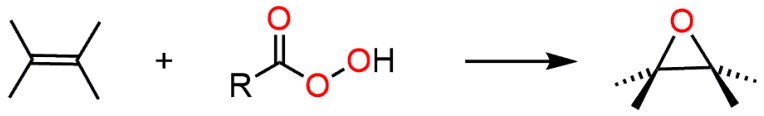
JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY
ARTICLE

J. Agric. Food Chem. **2009,** *57,* 8711–8721
DOI:10.1021/jf901375e

Aromatic Hydrocarbons of Mineral Oil Origin in Foods: Method for Determining the Total Concentration and First Results

MAURUS BIEDERMANN, KATELL FISELIER, AND KONI GROB*

Kantonales Labor (Official Food Control Authority of the Canton of Zurich), Fehrenstrasse 15, CH-8032 Zurich, Switzerland



Loss of 20-40% of MOAH

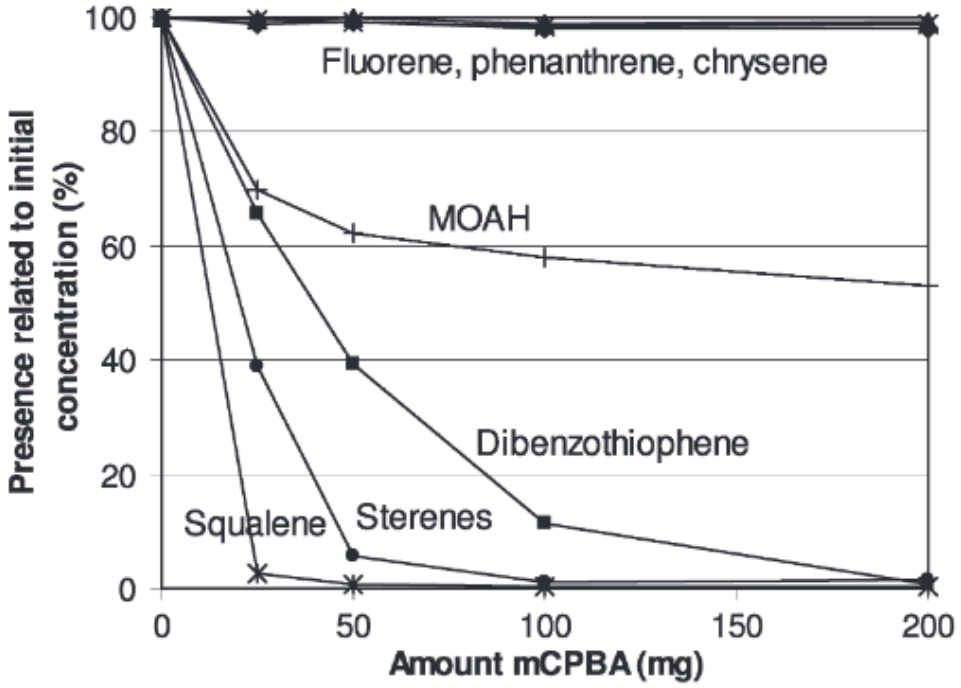


Figure 3. Epoxidation with different amounts of peracid. More peracid better removes squalene and the sterenes, but also the loss of certain aromatics increases.

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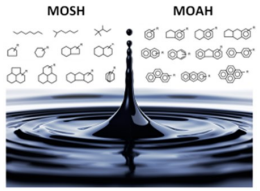
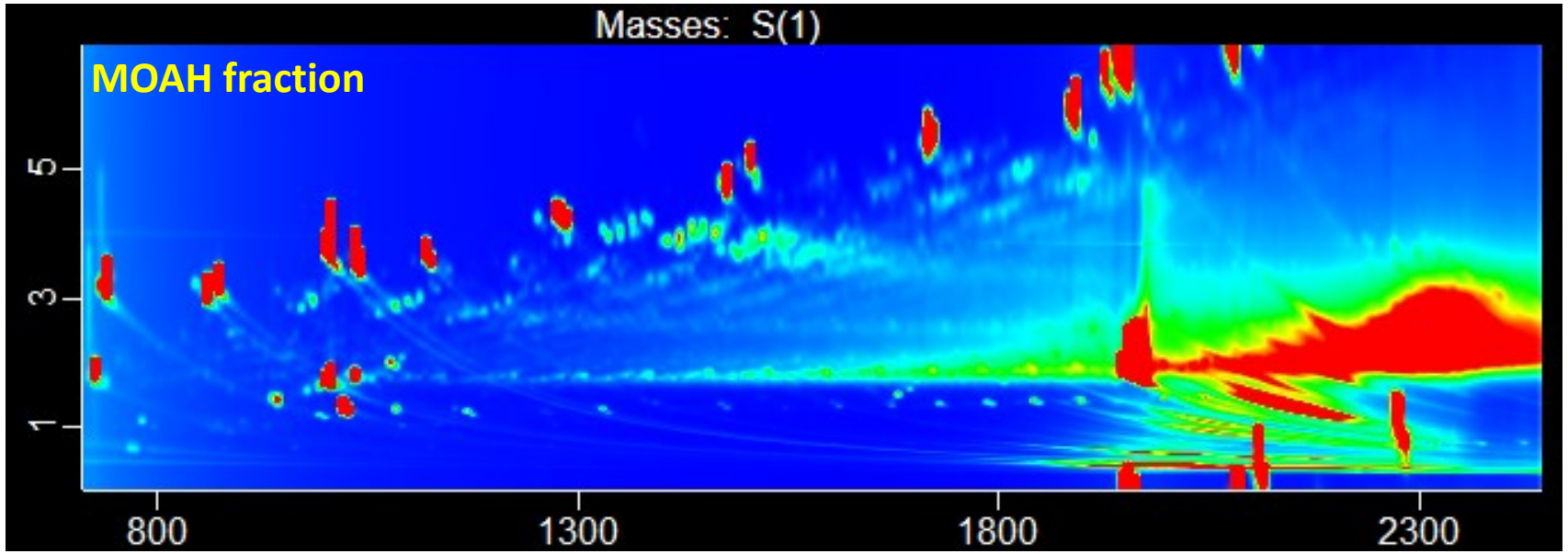
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



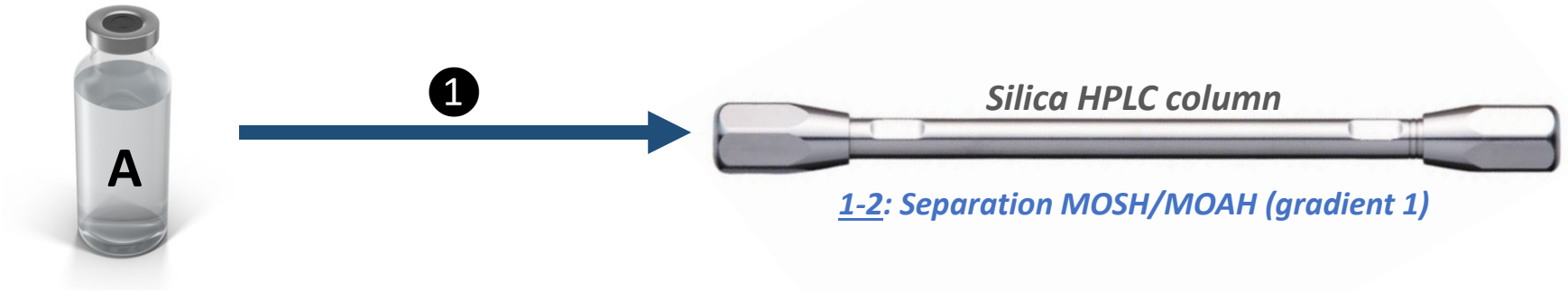


MOSH&MOAH IS+ EPA PAHs + Coronene + QC10 +HVGO

Interferences

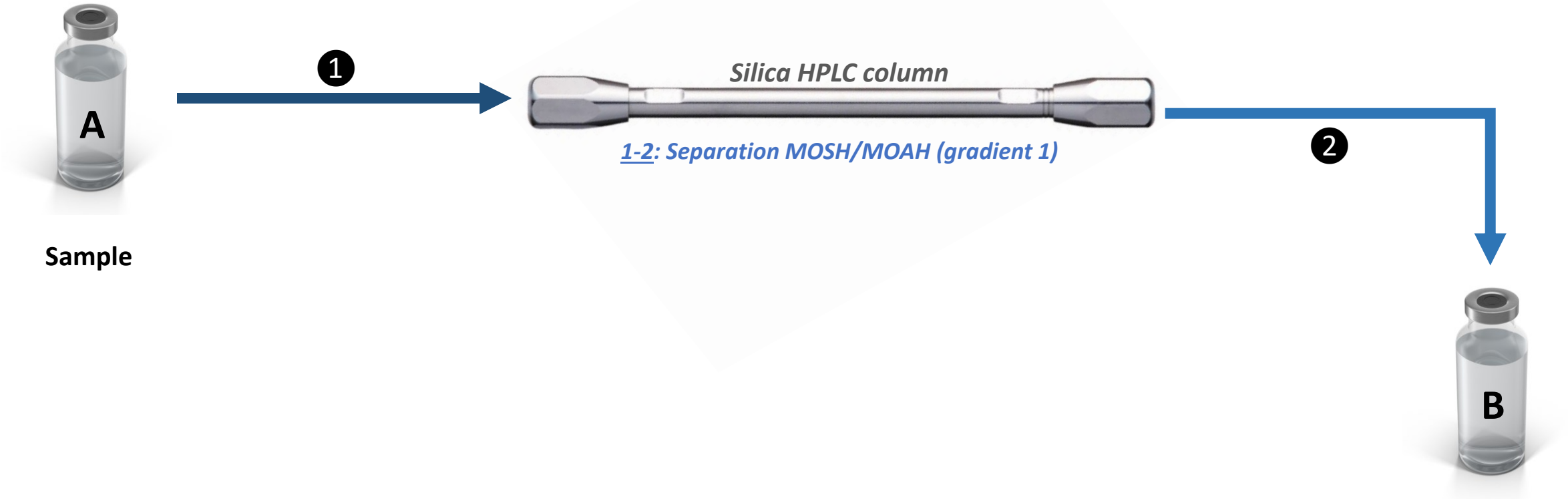
squalene, lycopene, β-carotene, trans β-carotene, β-caryophyllene

ALTERNATIVE TO EPOXIDATION: LC-GC×GC



Sample

ALTERNATIVE TO EPOXIDATION: LC-GC×GC



Sample

MOAH fraction
(450 µL)

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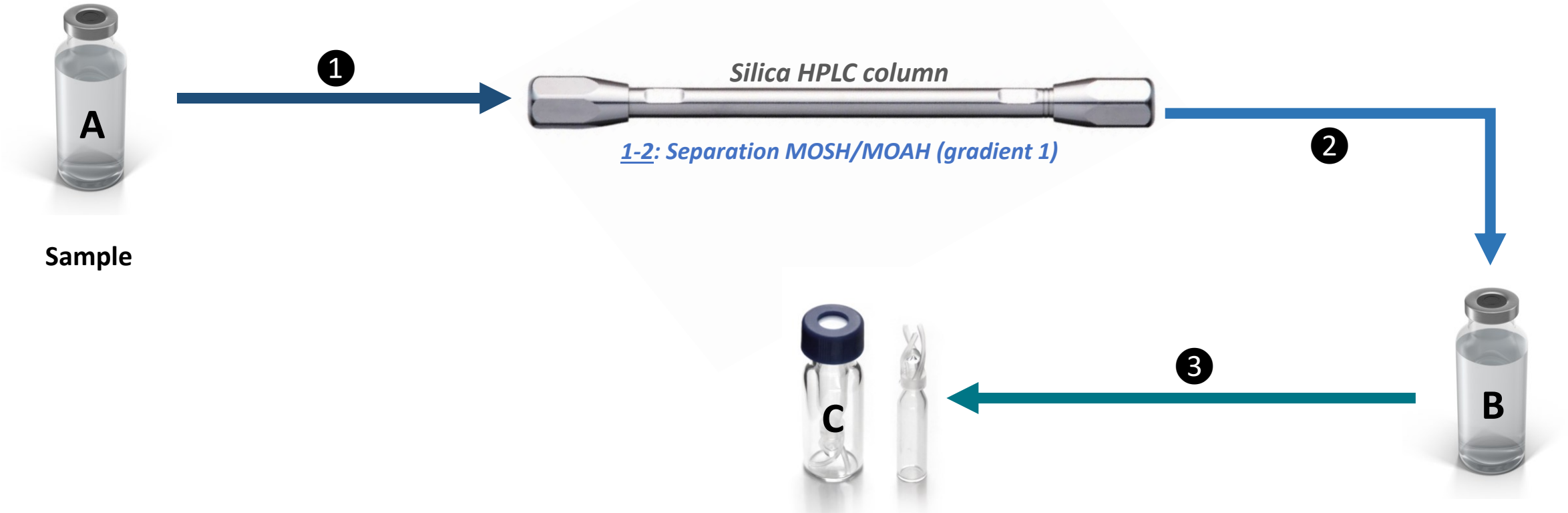
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



Sample

Concentrated MOAH fraction (~100 µL)

MOAH fraction (450 µL)

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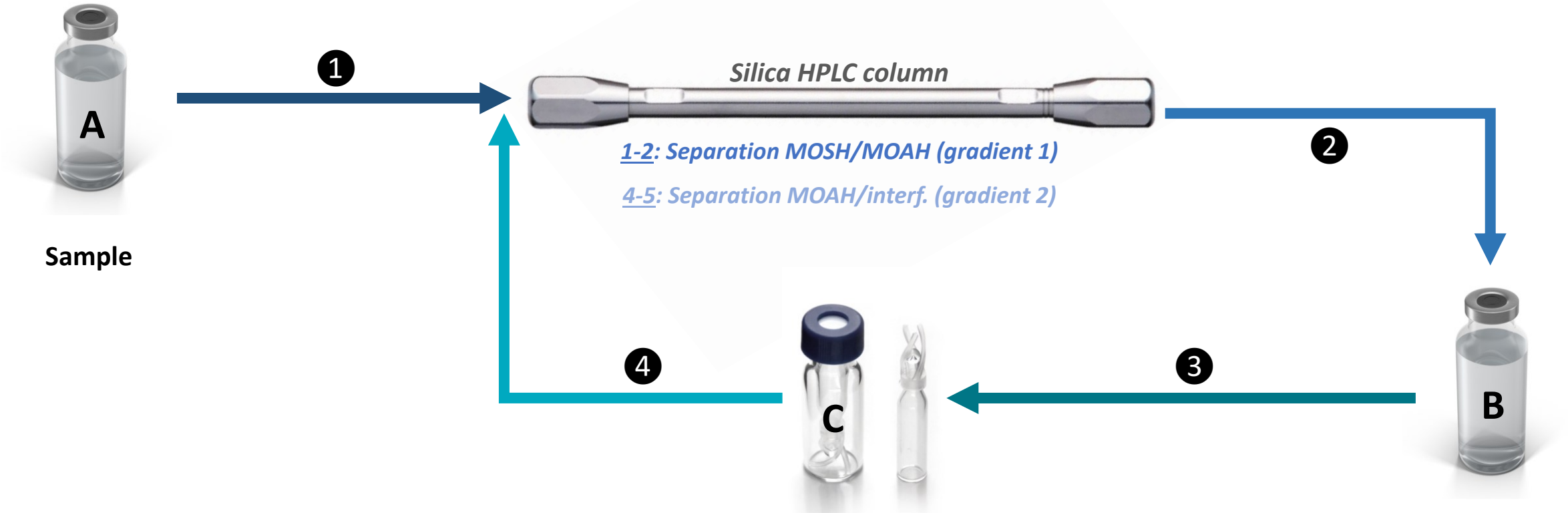
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



Sample

Concentrated MOAH fraction (~100 µL)

MOAH fraction (450 µL)

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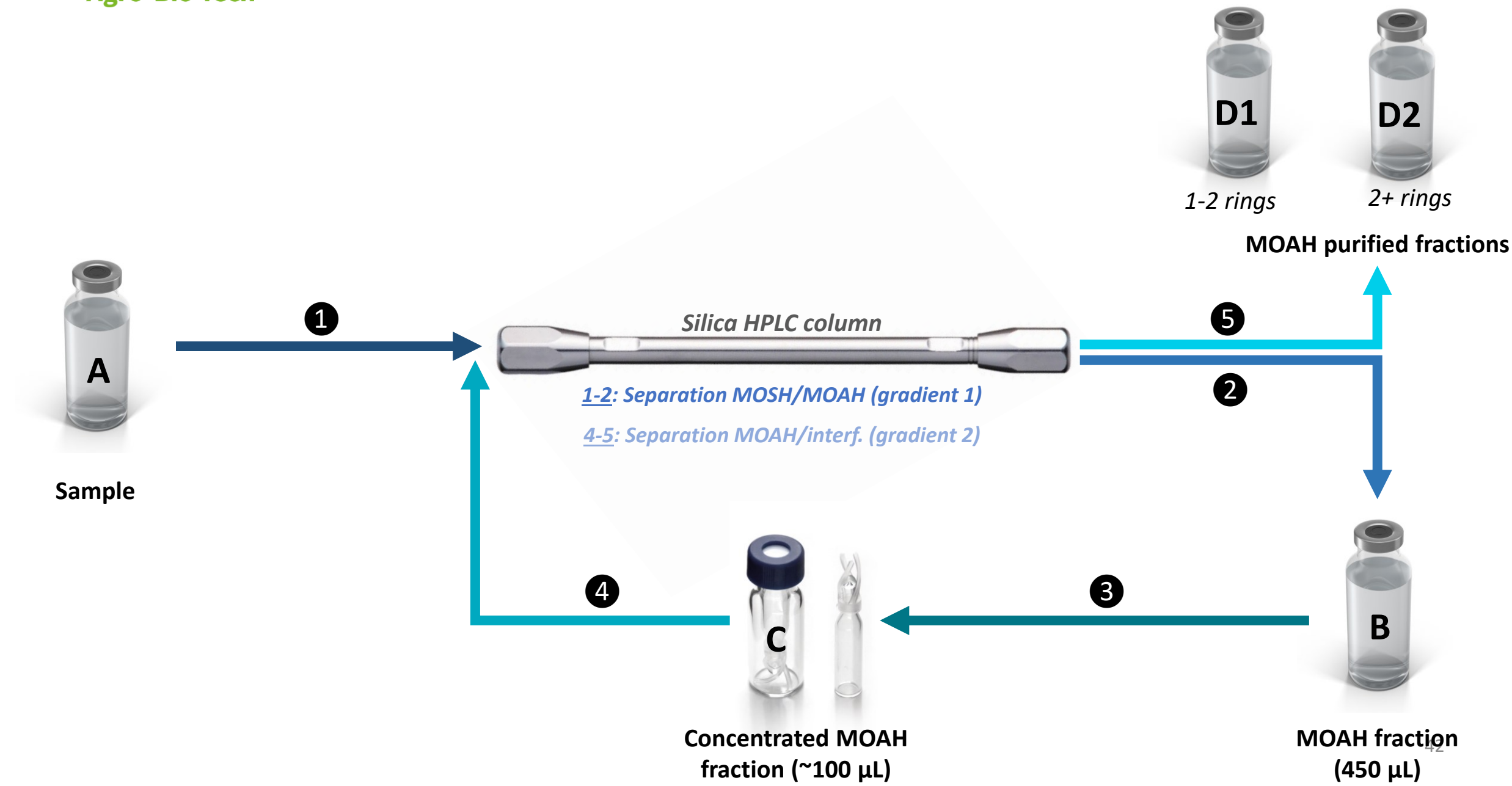
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



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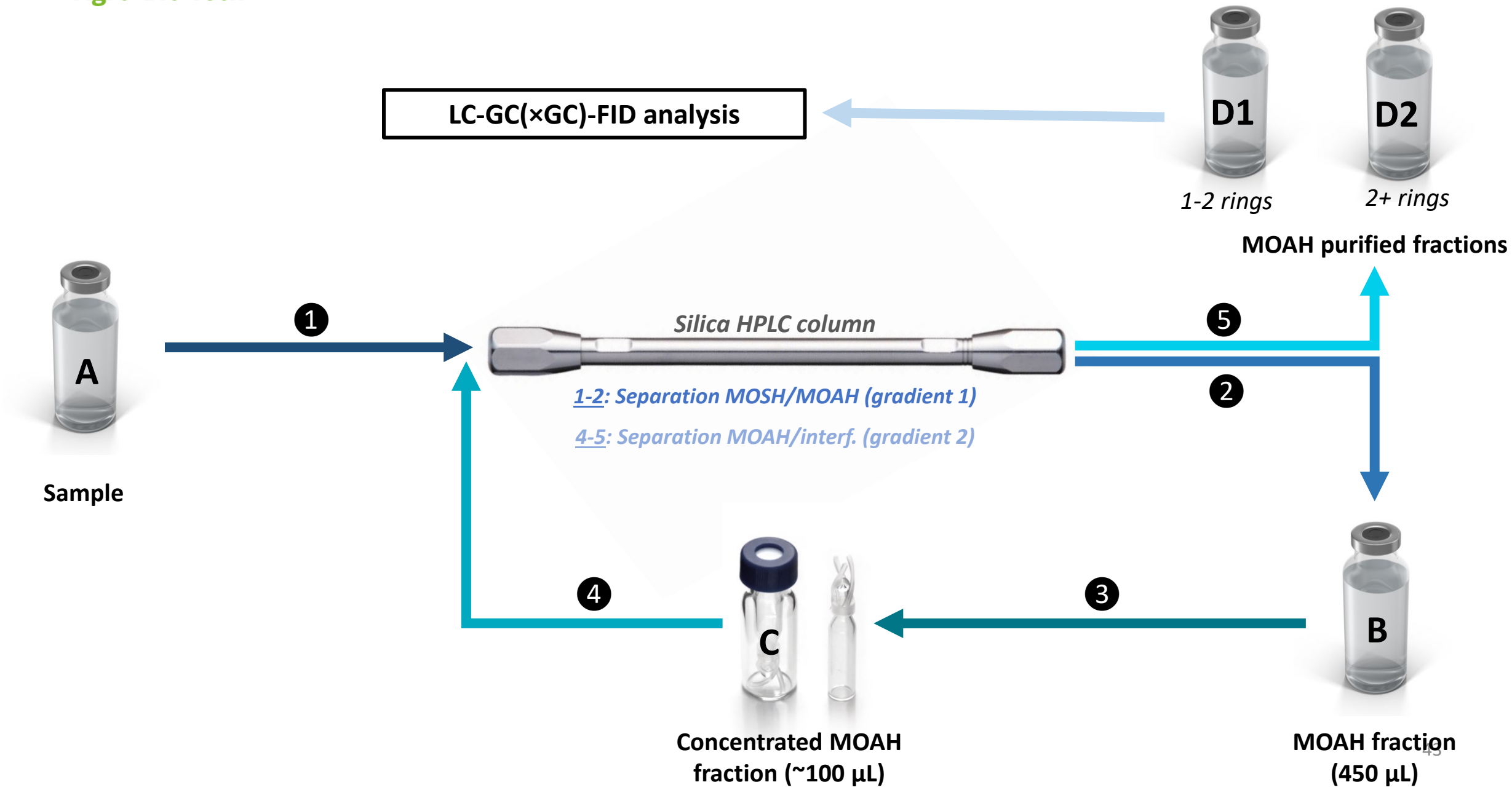
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



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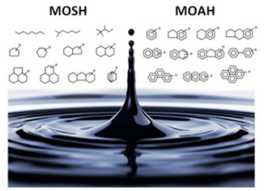
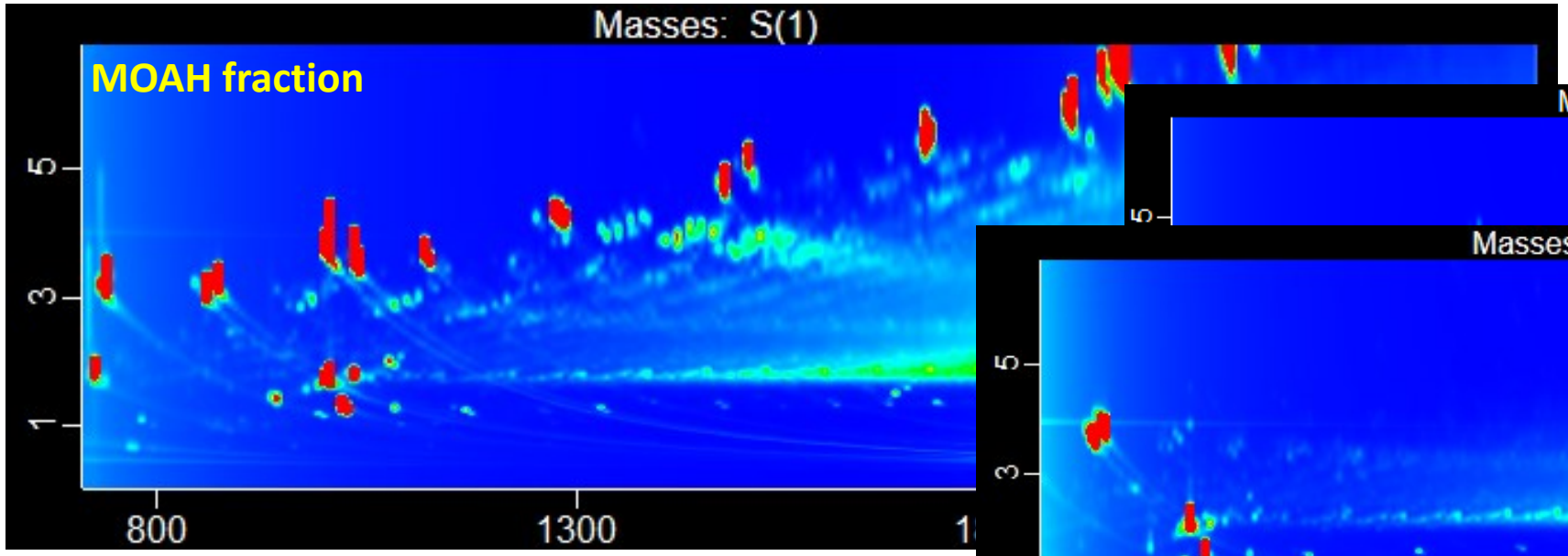
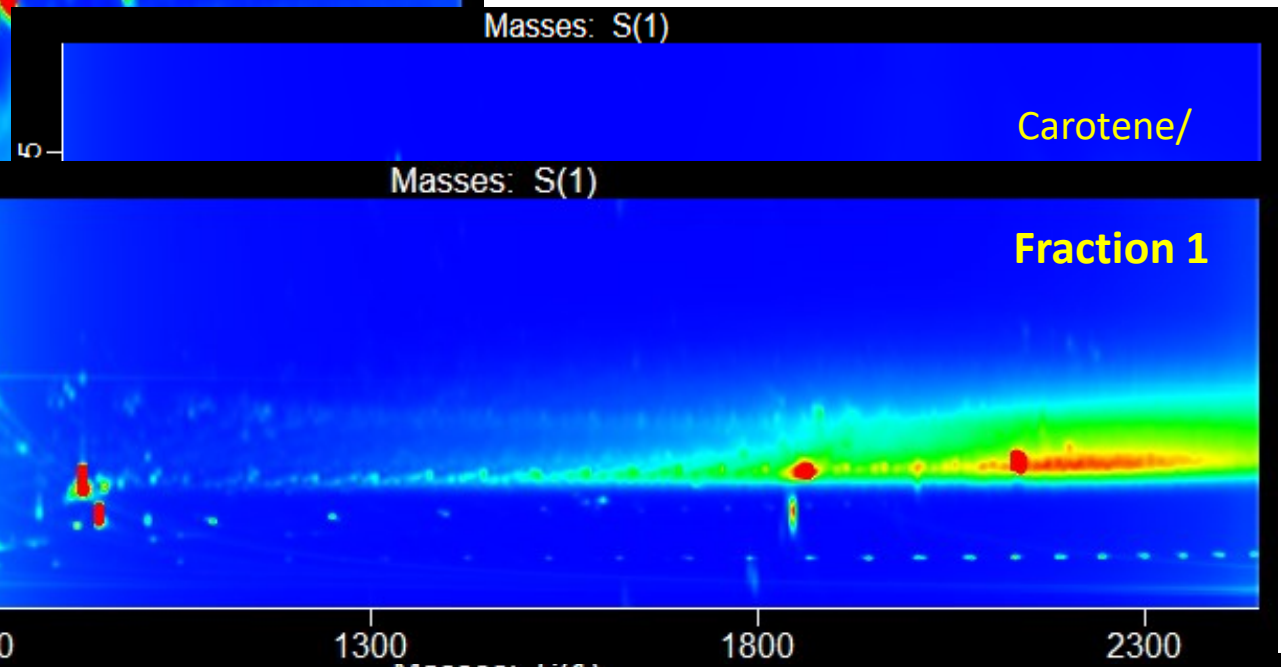
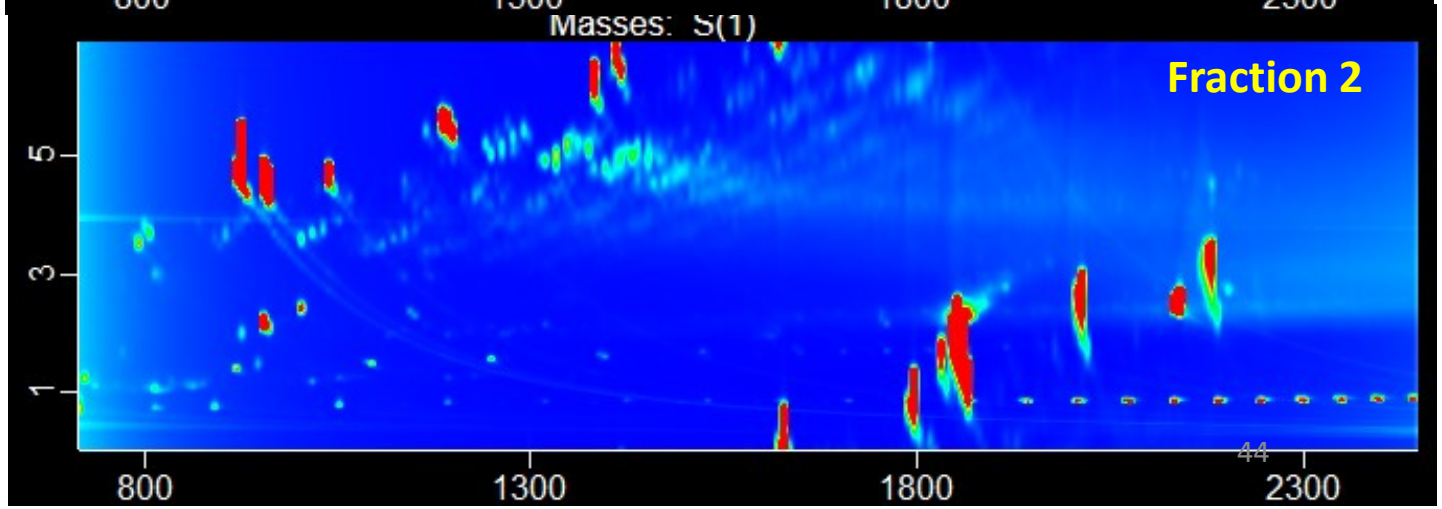
ALTERNATIVE TO EPOXIDATION: LC-GC×GC









MOSH&MOAH IS+ EPA PAHs + Coronene + QC10 +HVG

Interferences

squalene, lycopene, β-carotene, trans β-carotene, β-caryophyllene

Recovery of Fr1+Fr2

104±4%

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ALTERNATIVE TO EPOXIDATION: LC-GC×GC

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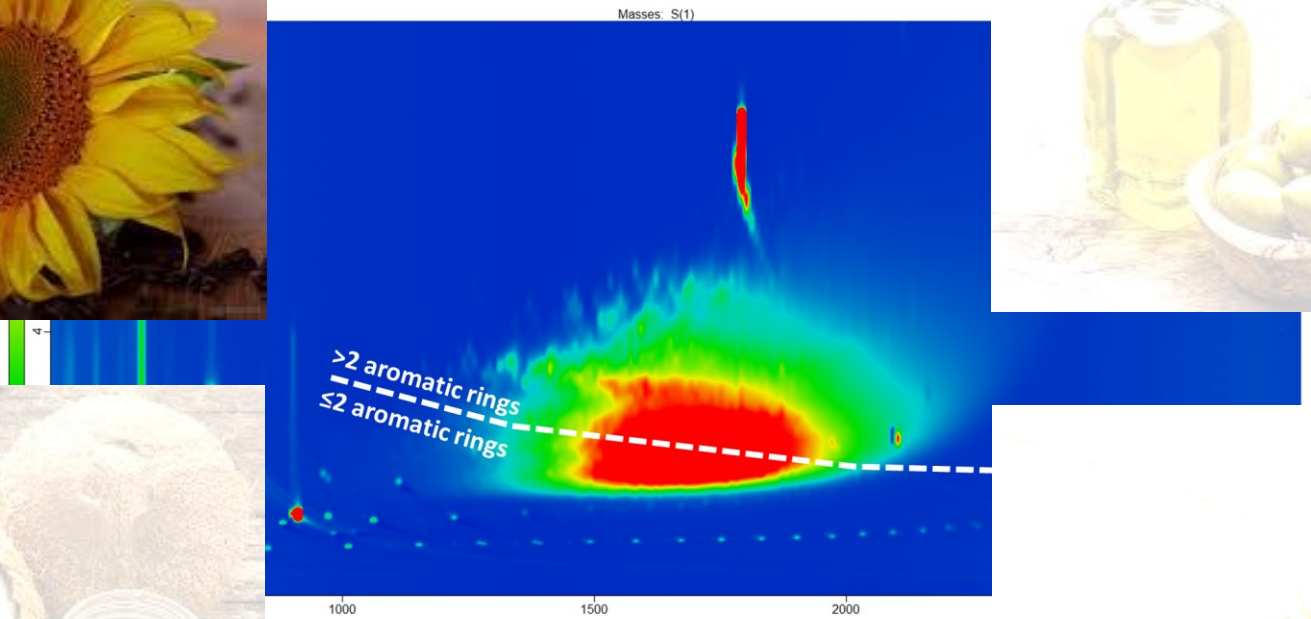
ALTERNATIVE TO EPOXIDATION: LC-GC×GC



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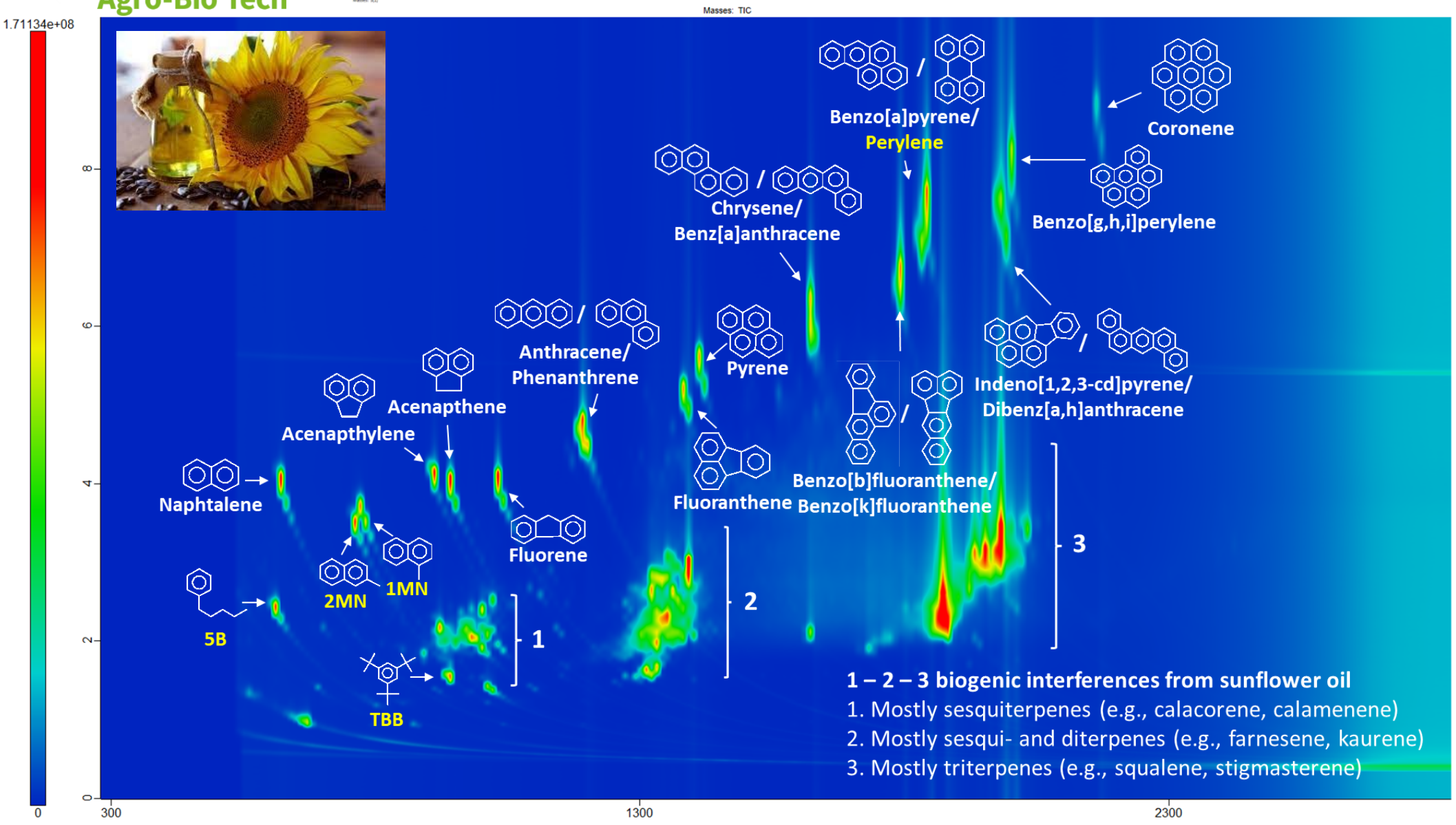
ALTERNATIVE TO EPOXIDATION: LC-GC×GC

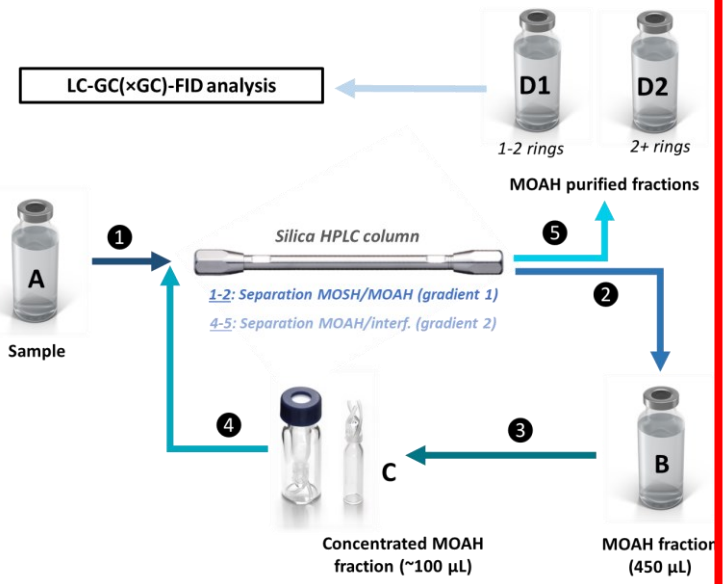
1 BEFORE LC PURIFICATION



ALTERNATIVE TO EPOXIDATION: LC-GC×GC

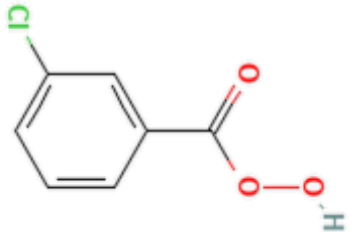
Epoxidation for the removal of interferences

LC Purification



Nestola 2017

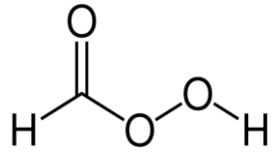
- Epoxidizing agent: **mCPBA**



- **BUT** commercial mCPBA contains impurities which may interfere with MOAH analysis ☹
- → Need to **purify** in the lab

Nestola 2022

- Epoxidizing agent: **performic acid** (formed *in situ* using formic acid and hydrogen peroxide)

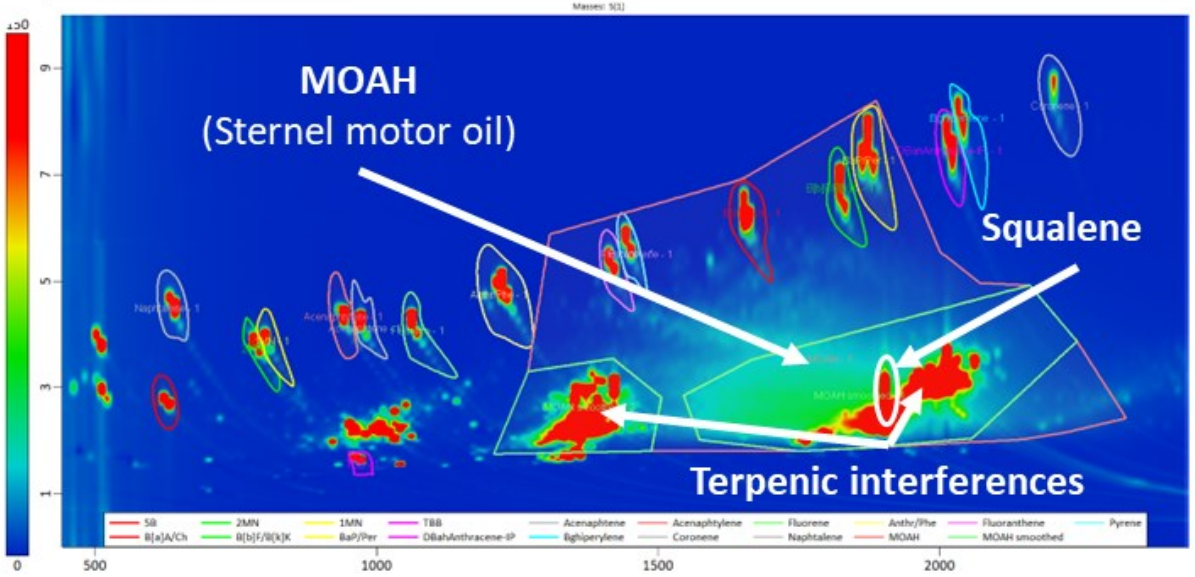


Note: a SPE clean-up on a silica column needs to be performed prior to the epoxidation step

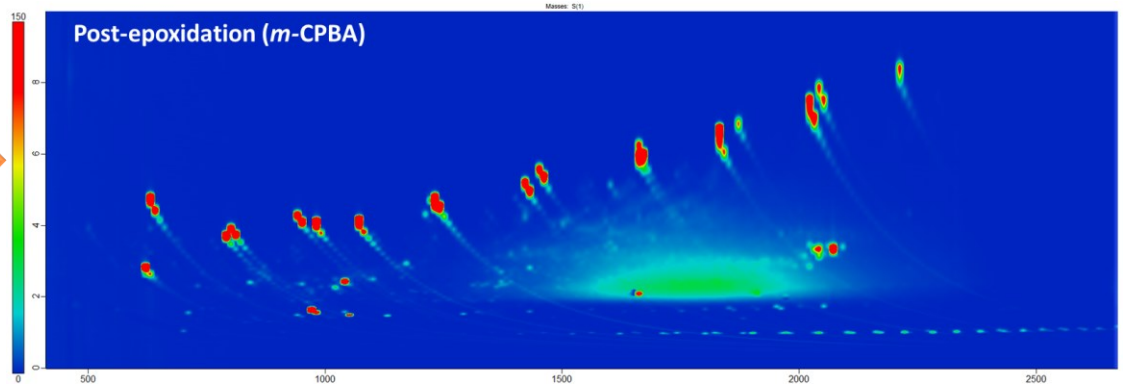
ALTERNATIVE TO EPOXIDATION: LC-GC×GC

1 BEFORE LC PURIFICATION

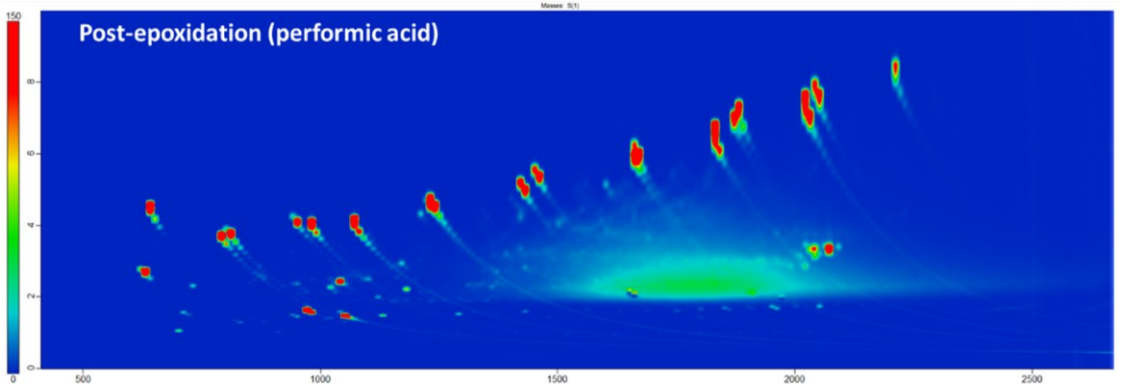
Standard PAHs + IS



Epoxidation mCPBA



Epoxidation performic acid



ALTERNATIVE TO EPOXIDATION: LC-GC×GC

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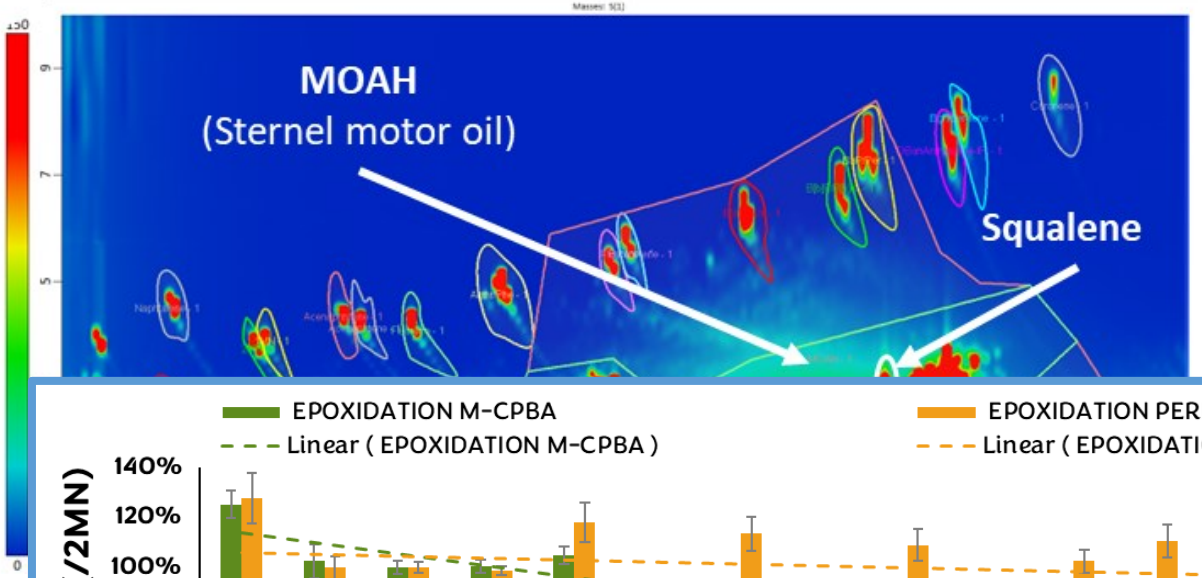
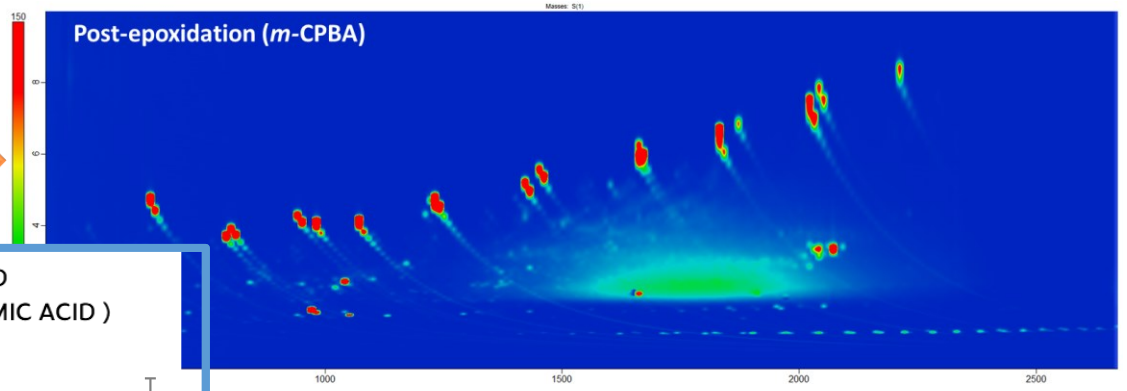
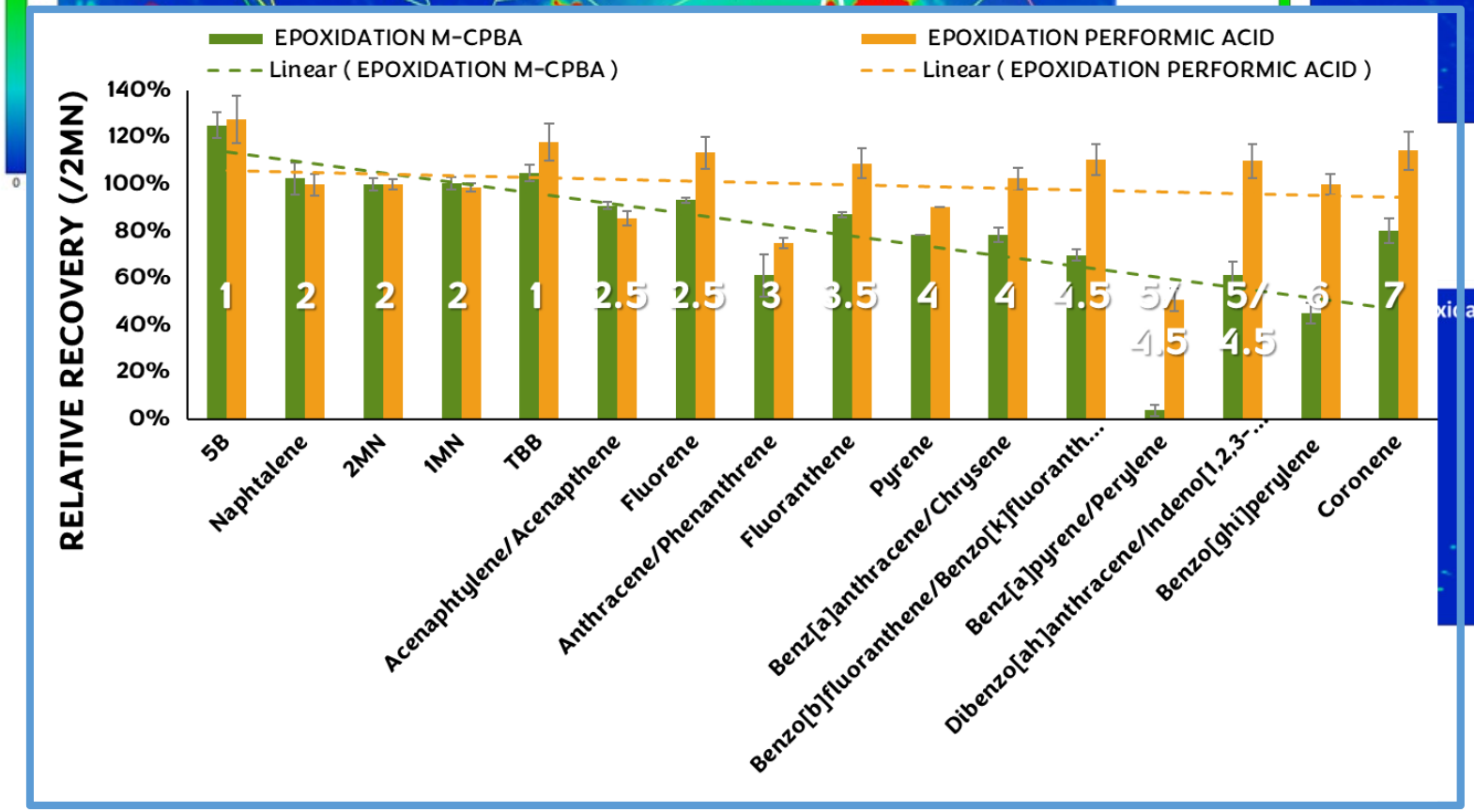
1 BEFORE LC PURIFICATION

Standard PAHs + IS

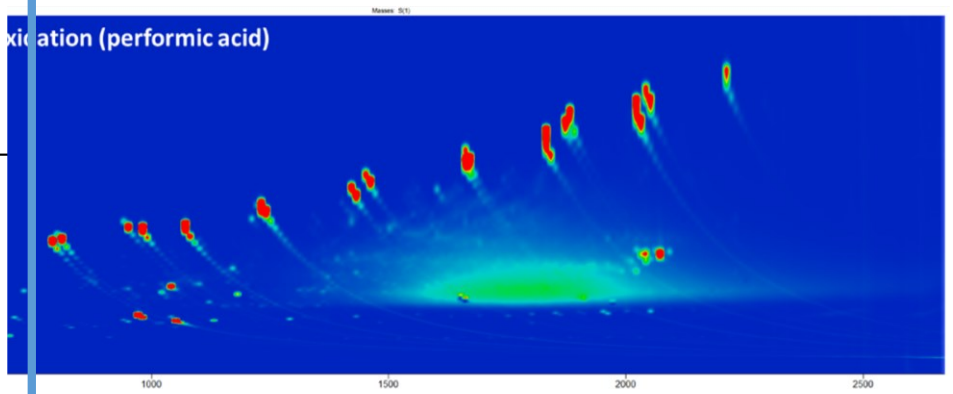


Epoxidation mCPBA





Epoxidation performic acid



ALTERNATIVE TO EPOXIDATION: LC-GC×GC

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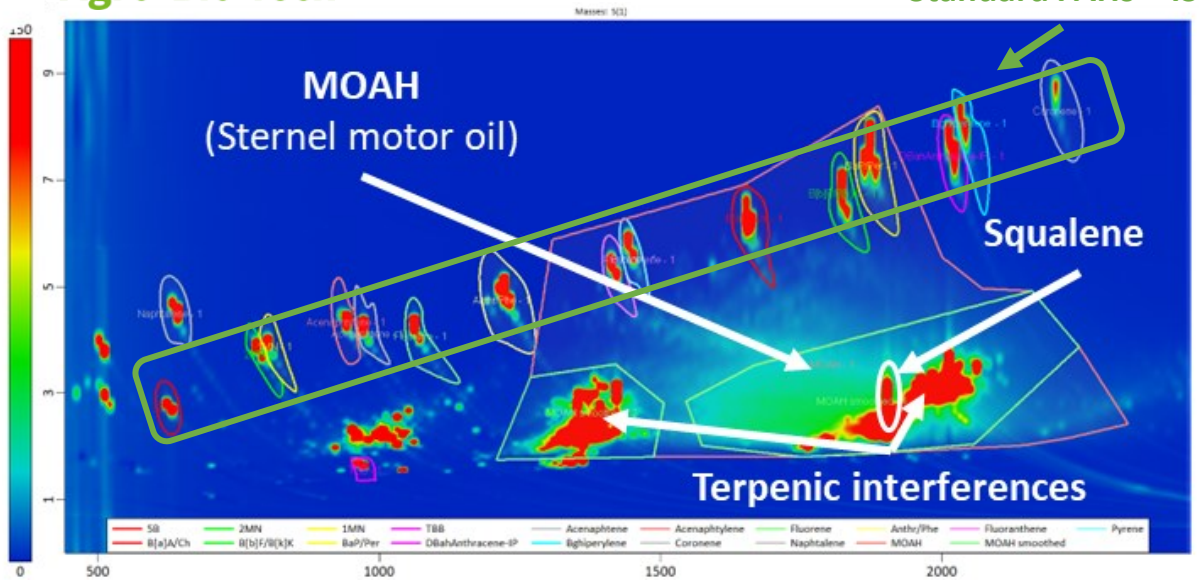
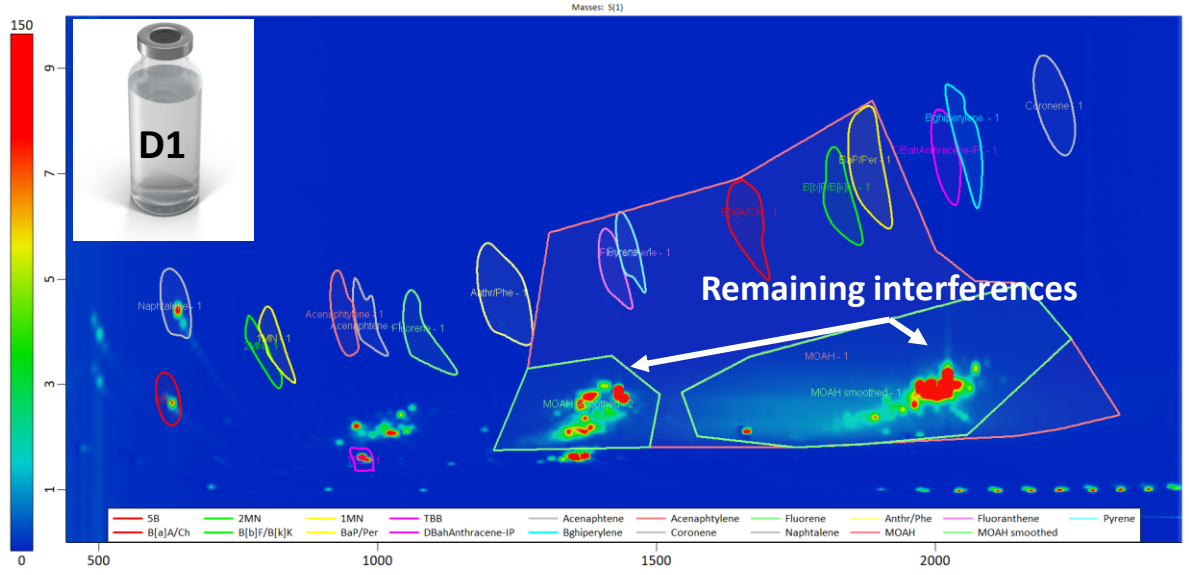
LC purification

1 BEFORE LC PURIFICATION

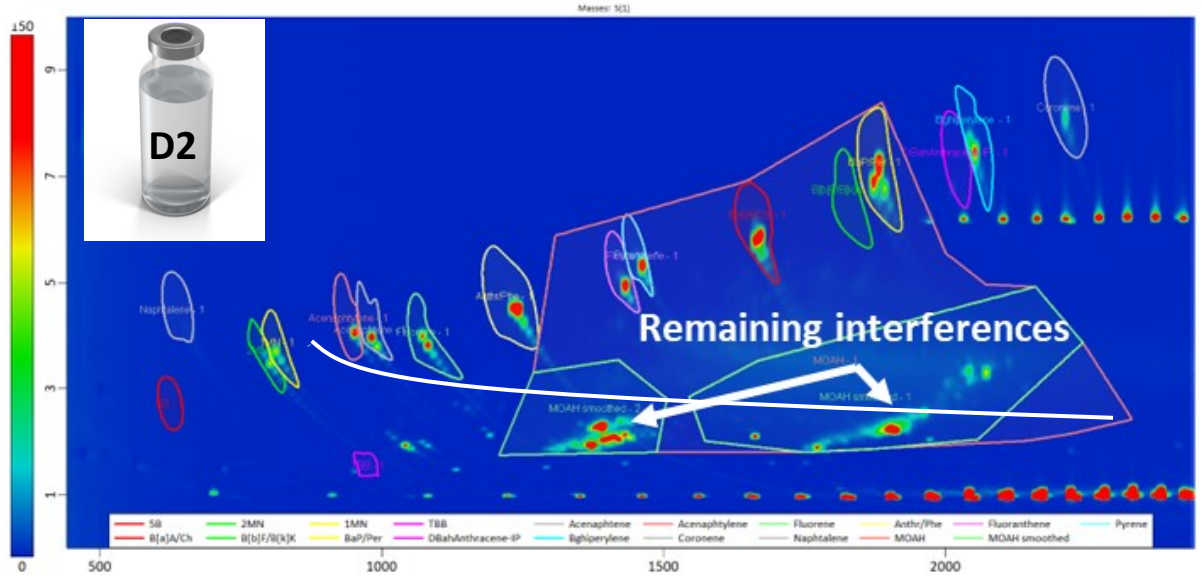
Standard PAHs + IS



2 AFTER LC PURIFICATION – 1-2 rings fraction



3 AFTER LC PURIFICATION – >2 rings fraction



ALTERNATIVE TO EPOXIDATION: LC-GC×GC

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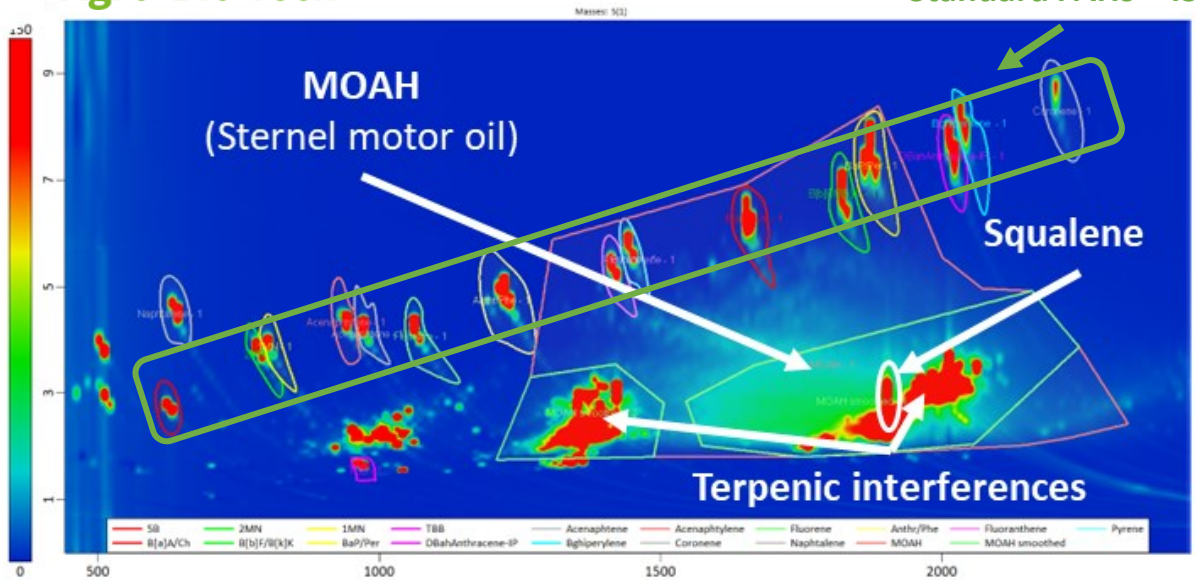
LC purification

1 BEFORE LC PURIFICATION

Standard PAHs + IS

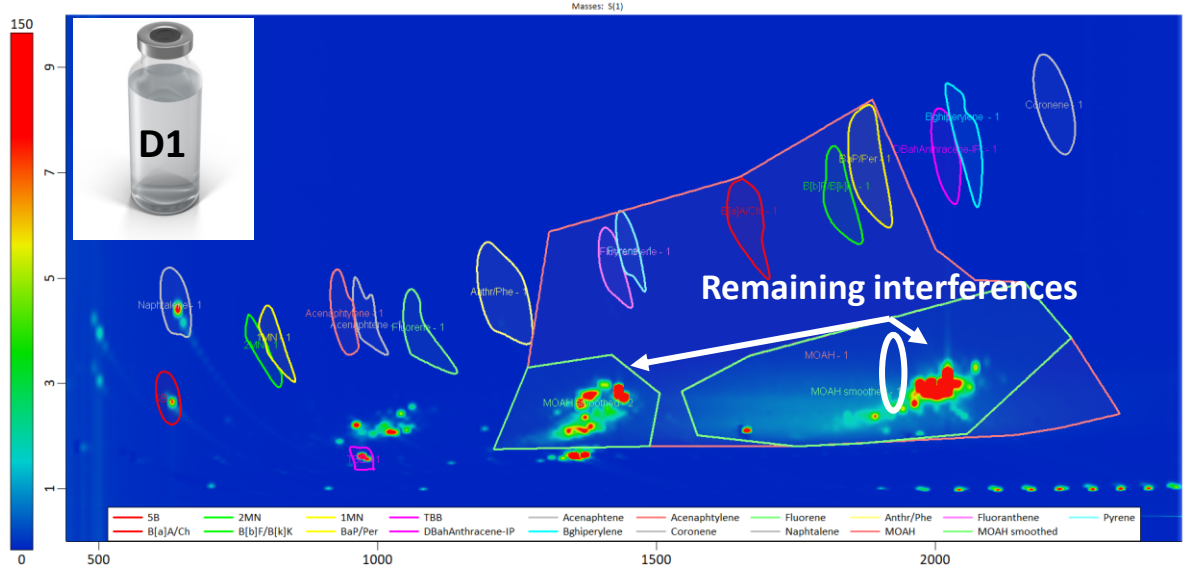


- Squalene completely retained
- Some interferences remain (of terpenic origin)
- Compounds coeluting with the toxicologically relevant MOAH fraction (>2.5 AR) are importantly reduced

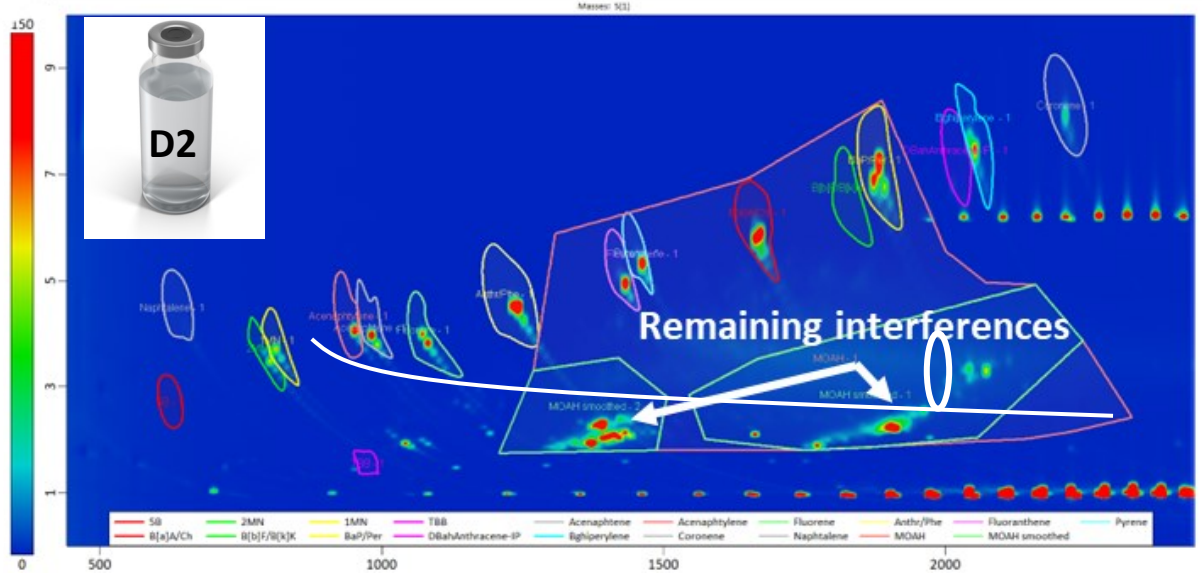
→ possible to quantify the MOAH with a **simple trimming of their peaks during integration**

→ smaller impact on the quantification than if epoxidation was applied

2 AFTER LC PURIFICATION – 1-2 rings fraction



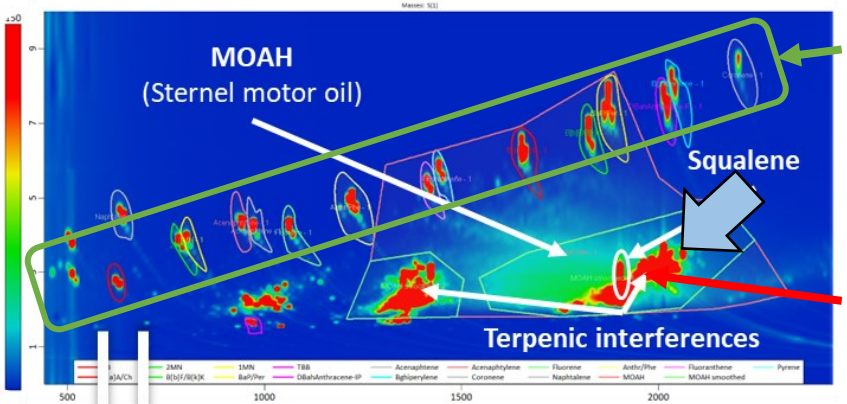
3 AFTER LC PURIFICATION – >2 rings fraction



ALTERNATIVE TO EPOX: LC-GC×GC

LC purification

1 BEFORE LC PURIFICATION

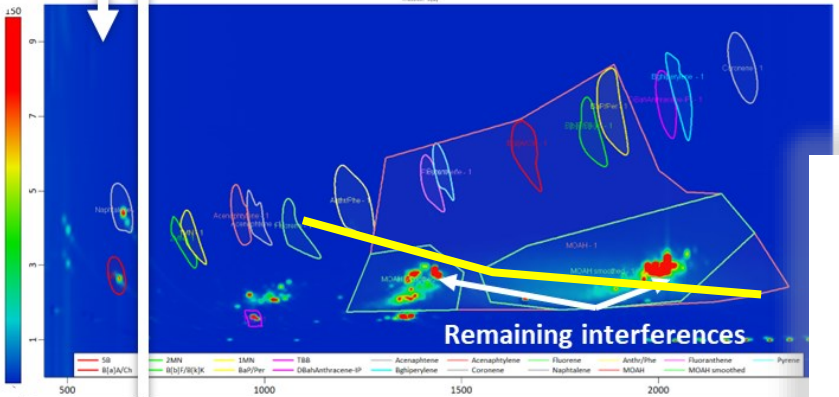


Standard PAHs + IS

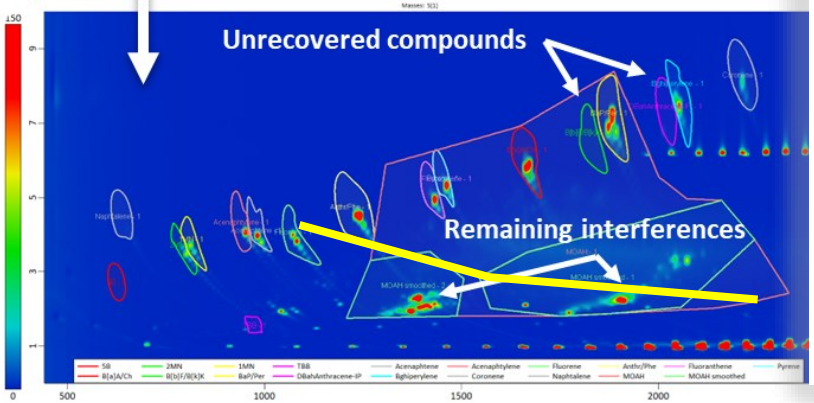
Before purification

Interferences

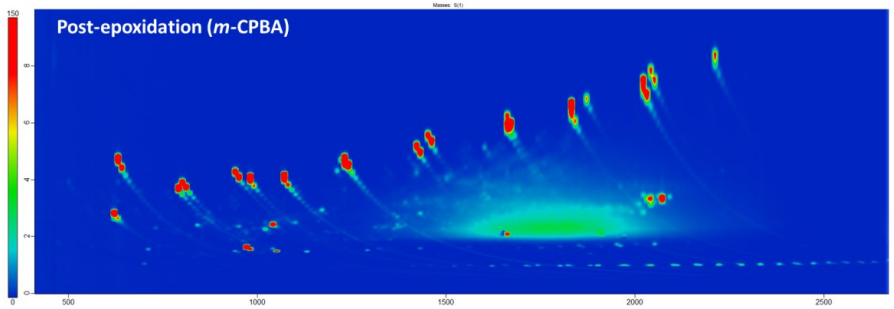
2 AFTER LC PURIFICATION – 1-2 rings fraction



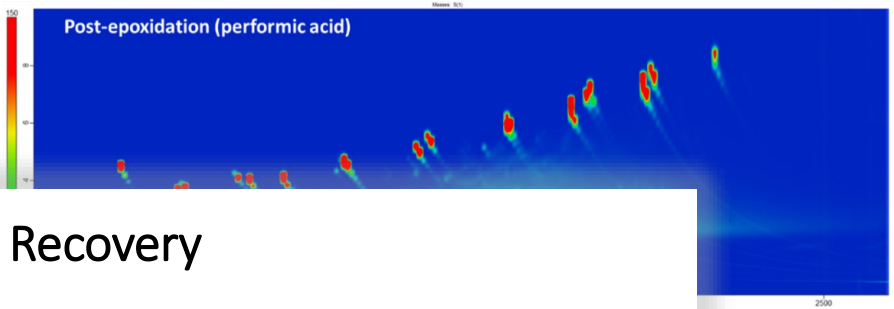
3 AFTER LC PURIFICATION – >2 rings fraction

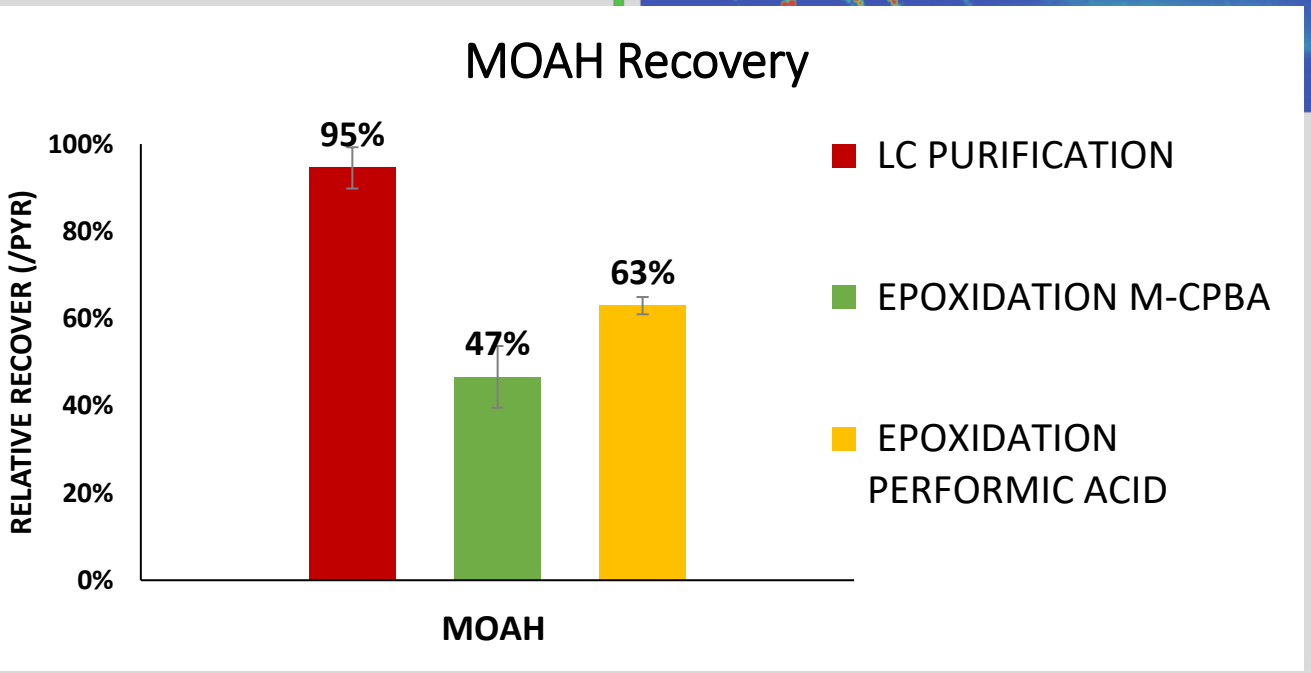


Epoxidation mCPBA



Epoxidation performic acid





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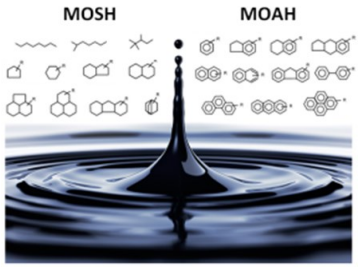
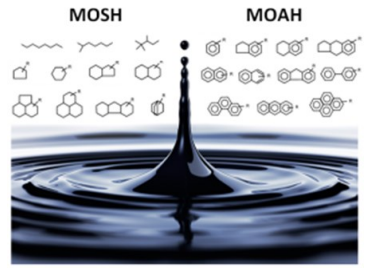
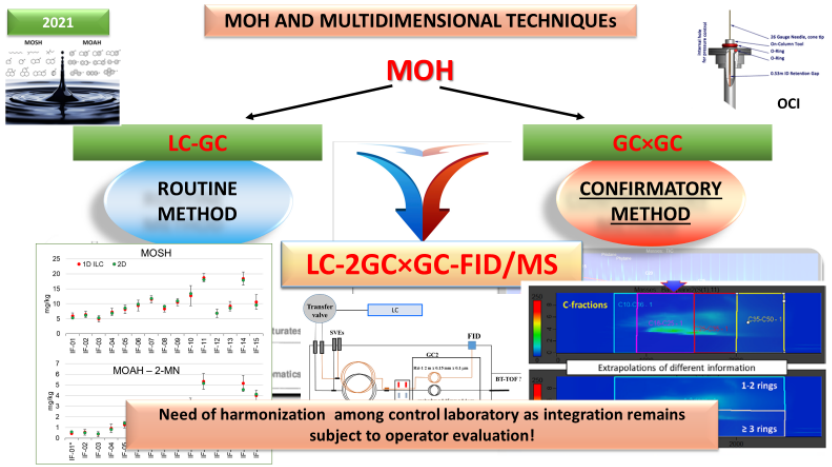
CONCLUSION AND FUTURE DIRECTIONS



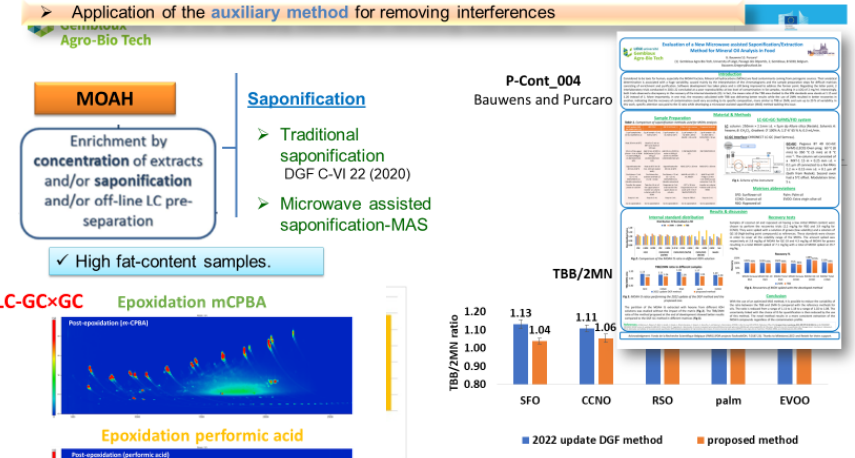
CONCLUSION AND FUTURE DIRECTIONS

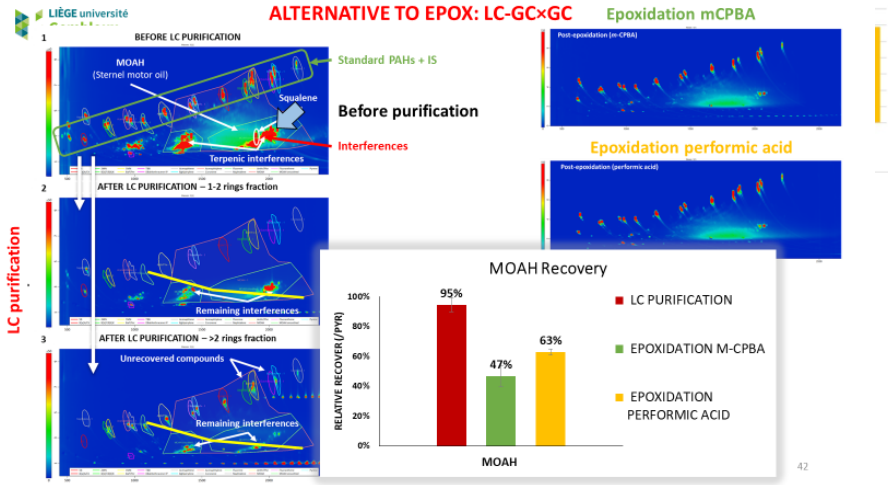




✓ **GC×GC-FID** is a highly promising tool to increase the information on MOSH & MOAH and respond to toxicological related questions

➢ GC×GC-FID harmonization needed





✓ Improvement of **sample preparation** is highly needed to:

- ➢ Reduce operator interpretation
- ➢ Handle complex matrices (e.g., essential oils)
- ➢ Avoid artifacts and biased results

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19th Euro Fed Lipid Congress and Expo
17 – 20 September 2023
Poznań · Poland
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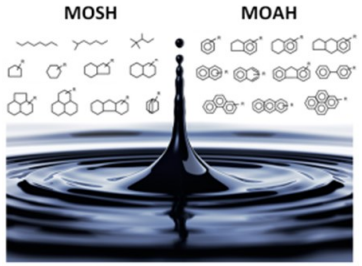

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Steven Mascrez

Grégory Bauwens

Damien Eggermont

Donatella Ferrara

Aleksandra Gorska

Visiting students:

Andrea Schincaglia

Nicolo Salgarella

Chen Feng

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RESTEK Pure Chromatography

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SepSolve Analytical

SHIMADZU Excellence in Science

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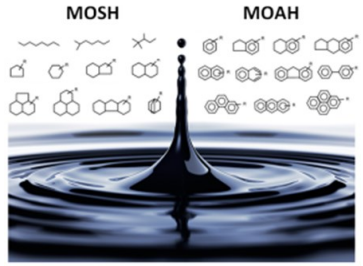

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