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THE ROLE OF GC×GC IN MINERAL OIL ANALYSIS. APPLICATION OF A COMPLETE LC-GC×GC-FID/TOFMS PLATFORM

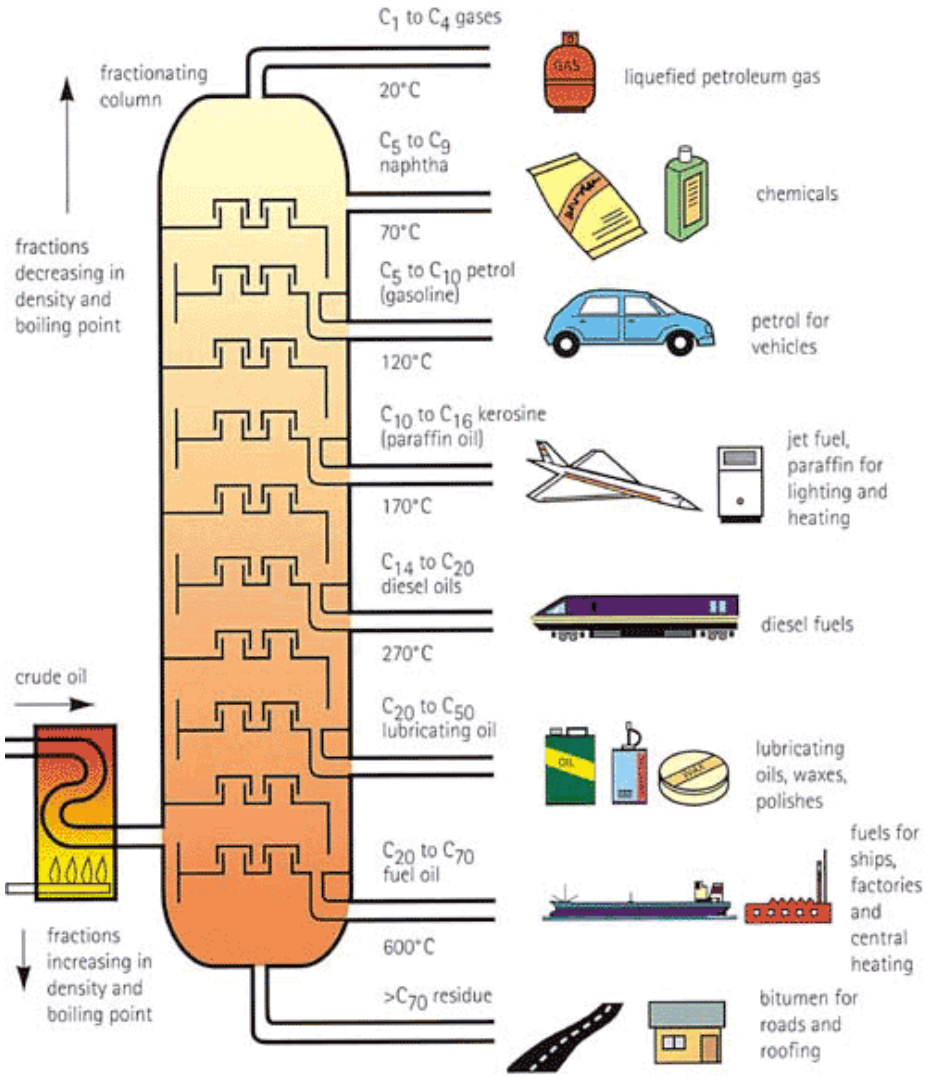
Giorgia Purcaro, Grégory Bauwens, Aleksandra Gorska

Gembloux Agro Bio-Tech, University of Liège, Belgium

gpurcaro@uliege.be

MINERAL OIL HYDROCARBONS (MOH): DEFINITION*

a wide range of products deriving from petroleum distillation fractions

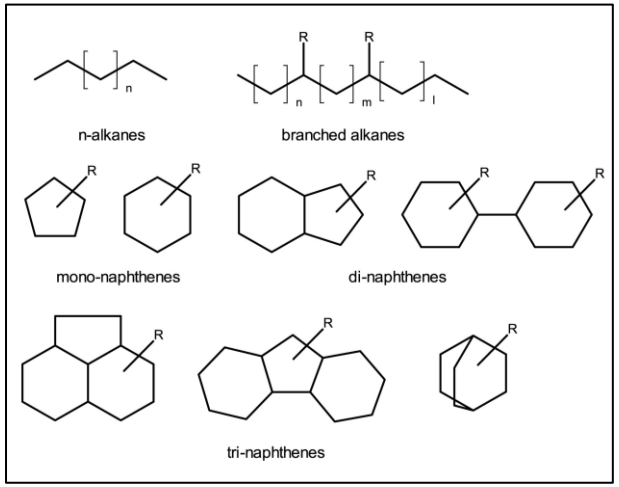


Containing mainly:

MOSH

Mineral oil saturated hydrocarbons

-n-alkane
- isoalkane
- cycloalkane

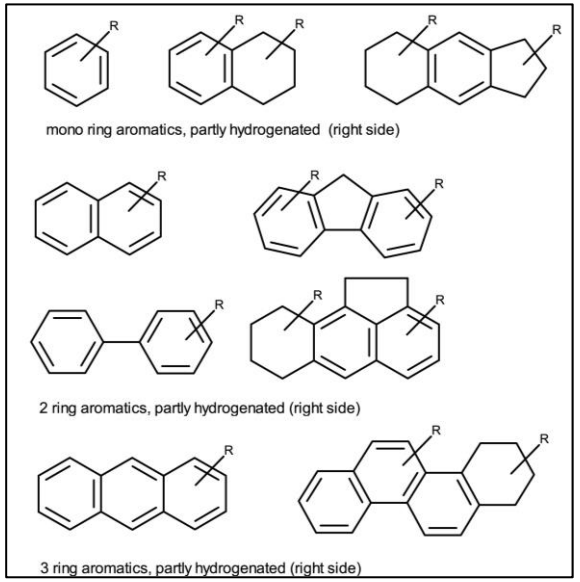


MOAH

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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Brief overview on the mineral oil analysis

1989

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

J High Resol Chromatogr. 1989 12:591–598.

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

MOH characteristic hump

As well as detection of pristane and phytane

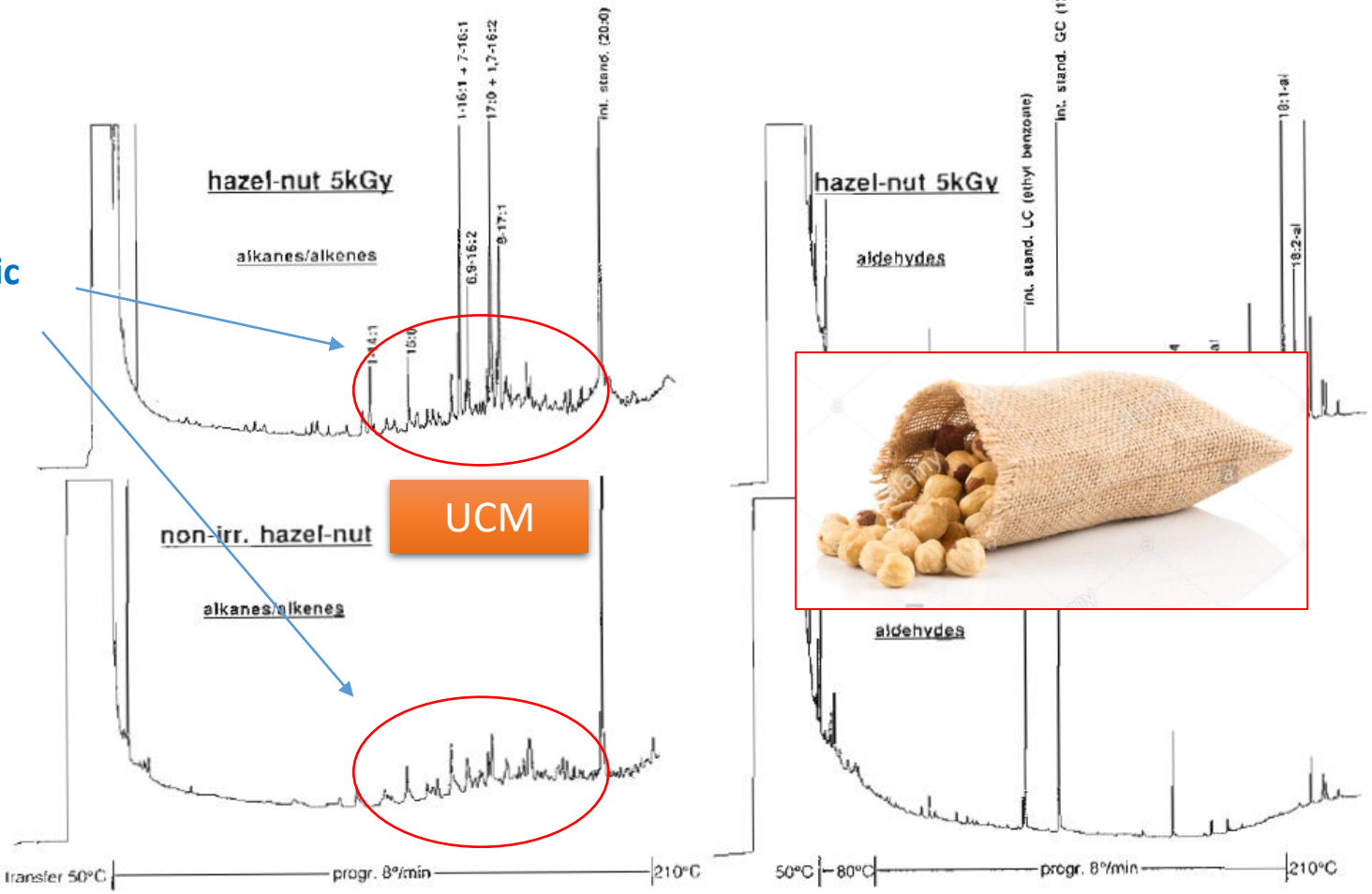

Figure 6

Alkane/alkene and aldehyde fractions of irradiated and non-irradiated hazelnuts. The poorly resolved alkanes in the matrix of the alkane/alkene fraction probably indicate a contamination of the nuts with mineral oil.

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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

1989 **2008/2009**

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

LC-GC&MOH

PUBLIC OPINION

2009

foodwatch

Test 2015

Food Packaging Forum

NEWS

Mineral oils in food

Foodwatch study shows contamination of ... introduction of functional ...

Home News Resources Food Packaging & Health Events About Us

... in paper or board with mineral oils; online petition launched to call for EU-wide

... an email petition to take action ... testing of 120 dry food products ... and with a long shelf-life (e.g. ... carbons (MOSH) and mineral oil ... mutagens and ...

RELATED ARTICLES

EFSA publishes opinion on mineral ...

NEWS | 24.10.2019

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

2019

MINERAL OIL IN FOODS

Food Packaging Forum

NEWS

Aromatic M... from adve...

A German Consumer Protecti... contaminated with aromatic ...

November 26, 2012 Charlotte ...

The German Consumer Protection Agency Stiftung Warer... chocolate of 9 advent calendars, which are suspected t... calendars. Some calendars contained chocolate with ... chocolate. The agency suspects the recycled carton ... culprit. Recycled paper often contains residues of ... printed Christmas motives may be partially respo... carcinogenic. The other types of mineral oils pr... inflammation in the liver in animal studies. Ov... single one of the 24 advent calendars tested ...

Read more ...

High number of calendars made out of virgin fibre board

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Sunflower oil: contamination with mine

Published: 28 April 2008

Migration of mineral o

BfR Opinion No. 008/201

efsa
European Food Safety Authority

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*.

1989 **2008/2009** **2012**

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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
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LC-GC&MOH

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Migration of mineral o

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Home Newsroom Sunflower oil: contamination with mineral oil from Ukraine

Sunflower oil: contamination with mine

Published: 28 April 2008

efsa
European Food Safety Authority

EFSA Journal 2012;10(6):2704

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LC-GC&MOH

TECHNICAL REPORT

APPROVED: 15 November 2019

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

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

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Supporting Publications

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)

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Sunflower oil: contamination with mine
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Scientific O
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Eur
This scientific output, publi
2012*.

Public Consultation
PC-0400
Title
Draft scientific opinion on the update of the risk assessment of mineral oil hydrocarbons in food
Full Name
Public Consultation on the draft scientific opinion on the update of the risk assessment of mineral oil hydrocarbons in food
Public Consultation Number
PC-0400
Public Consultation Details
Food Domain
Contaminants
Status
Open
Link To Document
Start Date
15/03/2023
End Date
30/04/2023

1989 2008/2009 2012 2019 2023

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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)

OUTCOMES OF THE EFSA OPINION 2023

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

MOSH

MOSH can accumulate in human body, but seems to be not of concern

- **More data on human MOSH tissue concentrations** or **development and use of biomarkers of exposure** are needed, particularly from individuals born after 1995.

MOAH

MOAH potentially genotoxic and cancerogenic, particularly the 3 and + aromatic rings

- 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**

➢ **Improvement of analytical methodology**

for better characterisation of MOSH&MOAH and consistency in reporting

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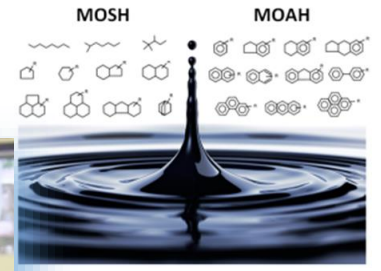
MOH AND MULTIDIMENSIONAL TECHNIQUEs



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

REVIEW ARTICLE

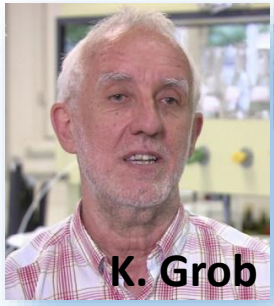
JOURNAL OF SEPARATION SCIENCE

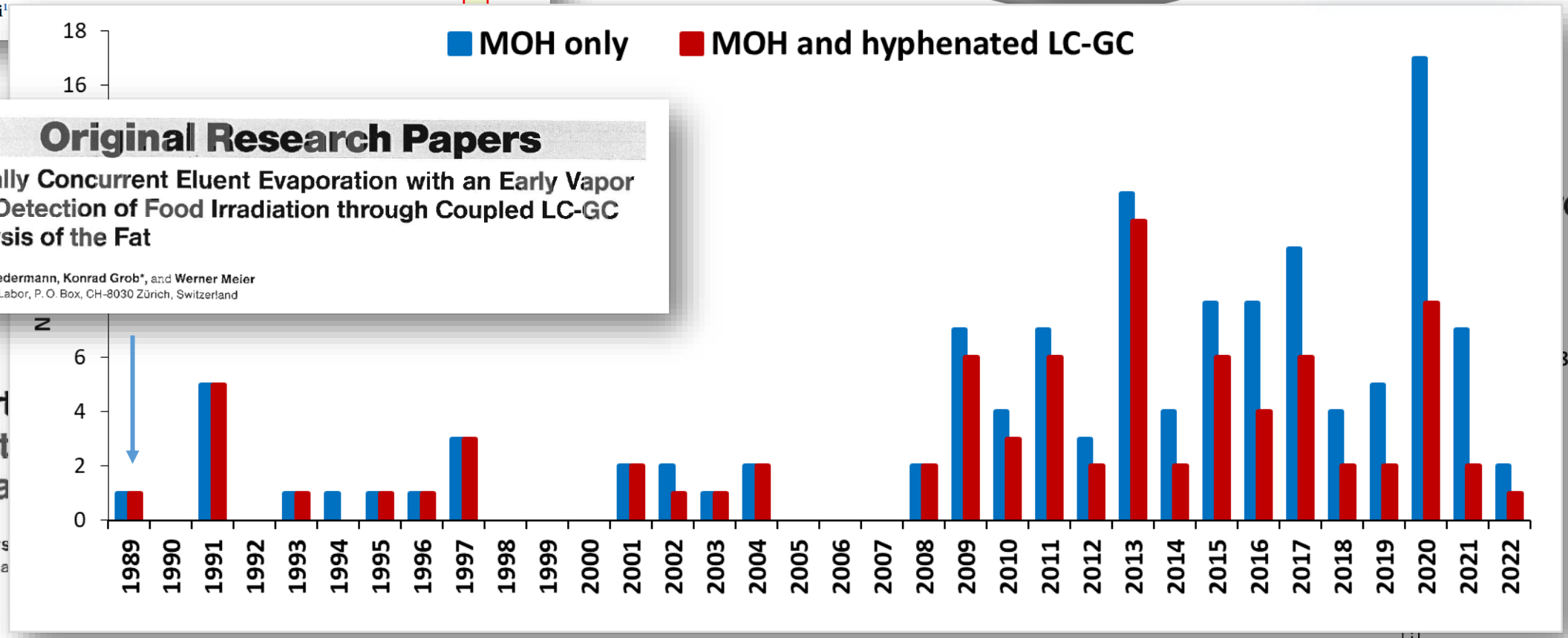
Evolution of hyphenated techniques for mineral oil analysis in food

Nicola Sdrigotti[1]

Mineral oil

LC-GC





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OGRAPHY

34 295:55-61

RETENTION GAP

Gas-liquid Partition Chromatography: the Separation and Micro-estimation of Volatile Fatty Acids from Formic Acid to Dodecanoic Acid

By A. T. JAMES and A. J. P. MARTIN
National Institute for Medical Research, Mill Hill, London, N.W. 7

(*Received 5 June* 1951)

Journal of Chromatographic Science, Vol. 18, October 1980

Multidimensional H
Liquid Chromatogra

Ronald E. Majors
Varian Instrument Group, 2700 Mitchell

Figure 6. Principle of Varian Model 8070 LC/GC Interface.

1906 GC 1951 GC-GC 1958 LC-GC 1978 1980 1984 1989 1991 2009 1941 LC

LC-GC&MOH

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY
ARTICLE

J. Agric. Food Chem. **2009**, *57*, 8711–8721 **8711**
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

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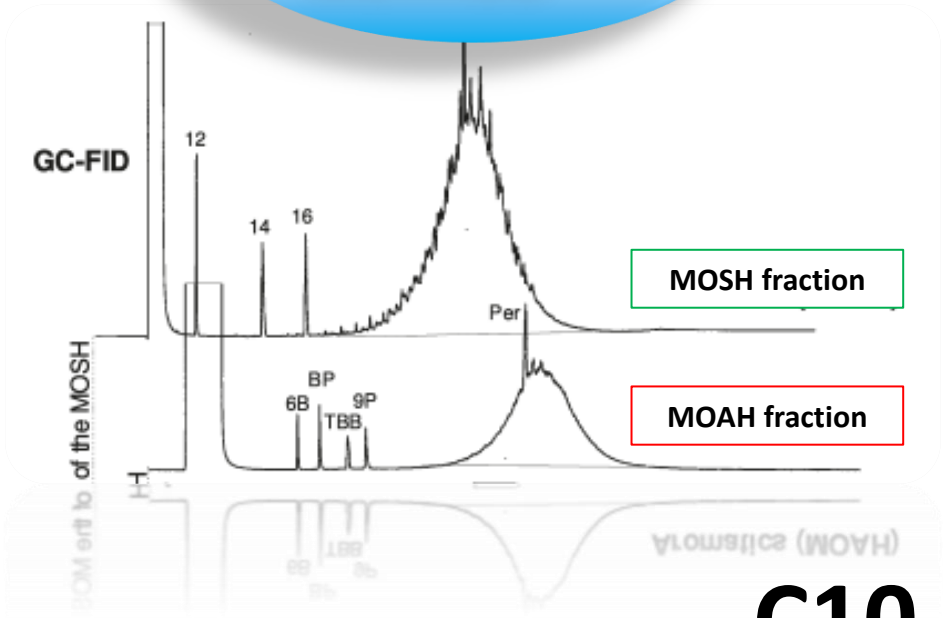
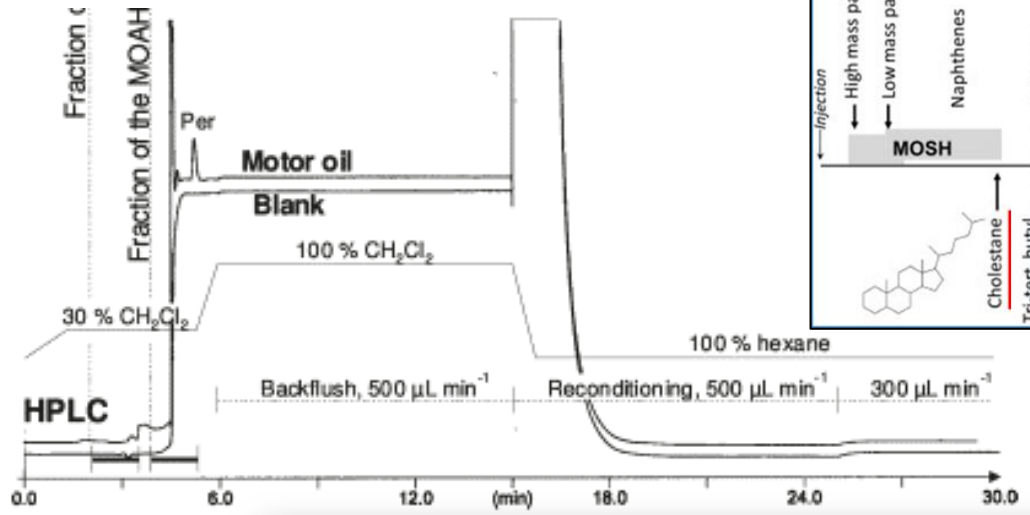
MOH AND MULTIDIMENSIONAL TECHNIQUEs

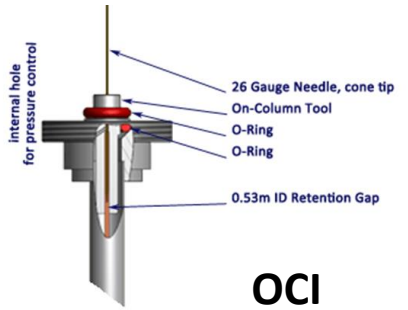
MOH

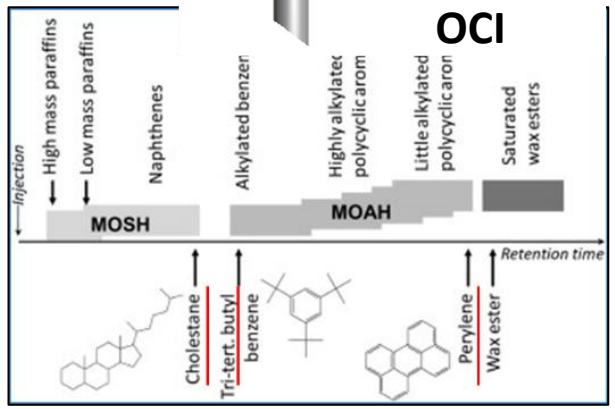
LC-GC

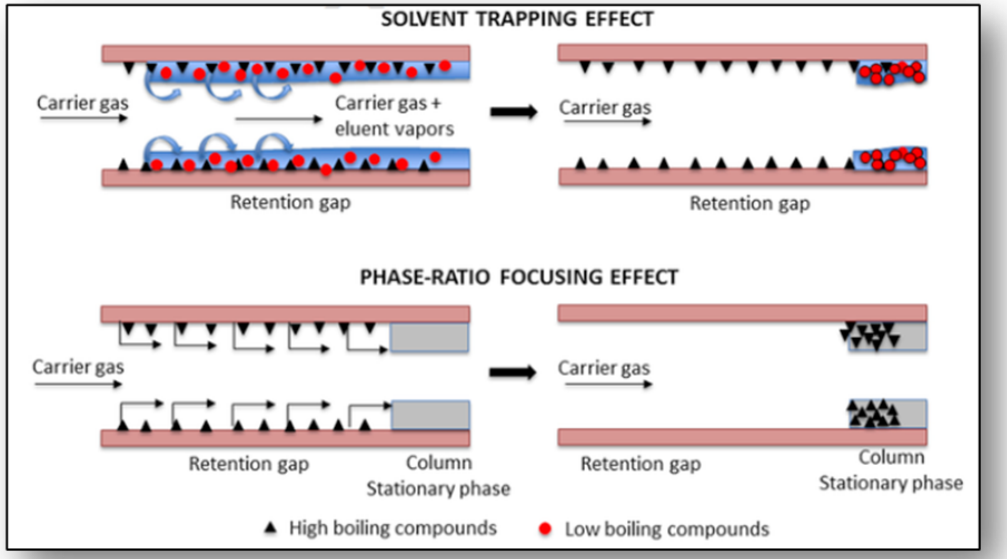
ROUTINE METHOD











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3726 *J. Sep. Sci.* 2009, *32*, 3726–3737

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Official Food Control Authority of the Canton of Zurich, Zurich, Switzerland

Received May 23, 2009
Revised August 3, 2009

Research Article

Comprehensive two-dimensional GC after HPLC preseparation for the characterization of aromatic hydrocarbons of mineral oil origin in contaminated sunflower oil

1906 1941 1978 1980 1984 1989 1991 2009

GC×GC&MOH

LC-GC&MOH

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY ARTICLE

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DOI:10.1021/jf901375e

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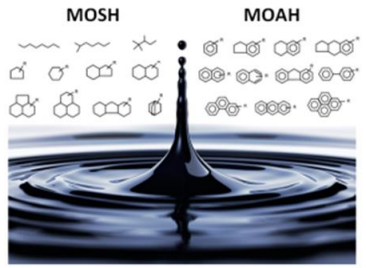
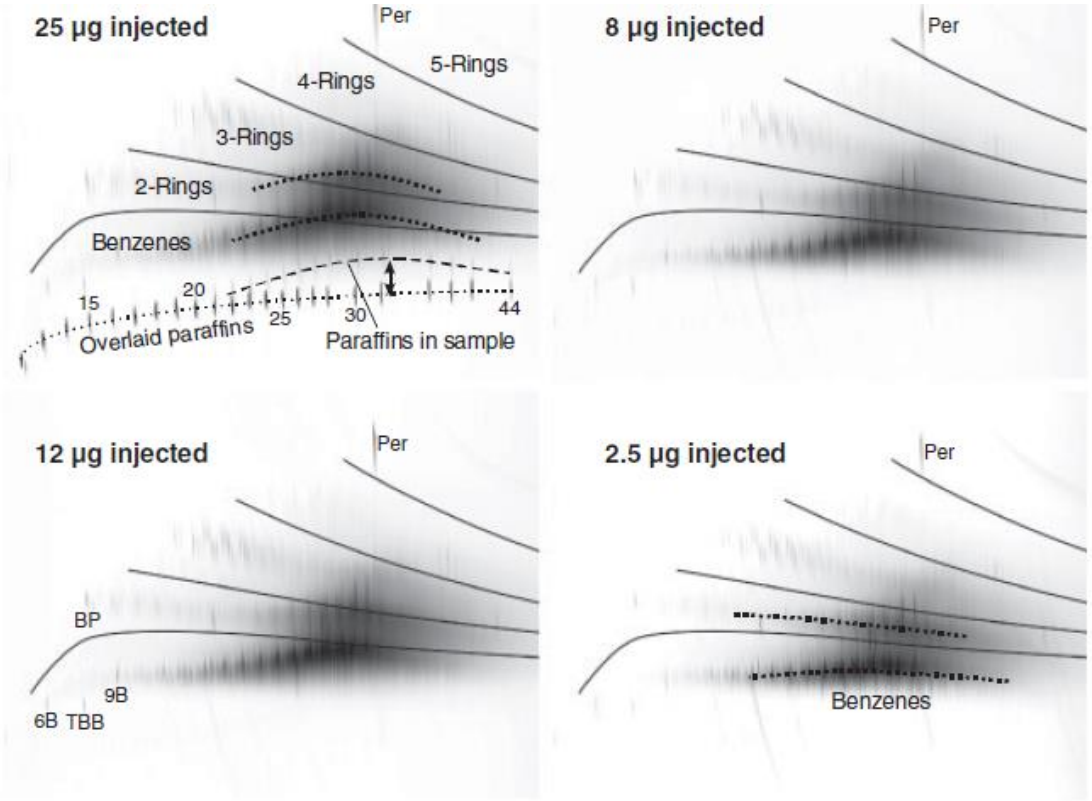
MOH AND MULTIDIMENSIONAL TECHNIQUEs

GC×GC



- **MOSH and MOAH characterization**



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Research Article

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Figure 6. Overloading of the 2nd dimension GC: GC × GC-FID plots of a sunflower oil contaminated with 20 000 mg/kg MOAH. Upper left: overlay of 25 µg MOAH with a mixture of *n*-alkanes (interconnected by dotted line at bottom). In the heavily loaded zone, the *n*-alkanes added to the MOAH show increased retention (arc with dashed line), as do the benzenes and 2-ring components (bold dotted lines). For adequate grouping of the components by ring number, overloading must be avoided (bottom right; 2.5 µg MOAH). Per, 6B, TBB, 9B and BP, internal standards for quantitation and verification of the cuts of the HPLC preseparation [4], as well as for the control of the retention times in the GC × GC plots.

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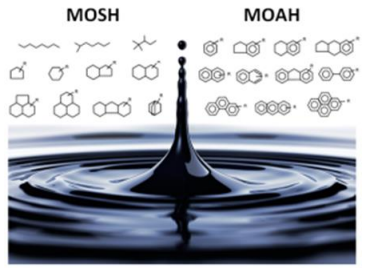
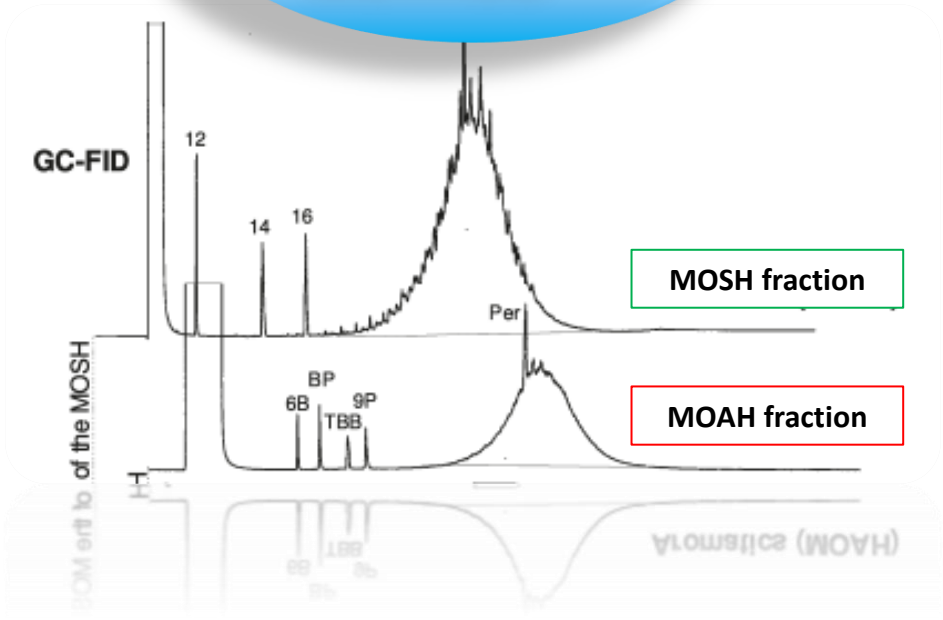
MOH AND MULTIDIMENSIONAL TECHNIQUEs



MOH

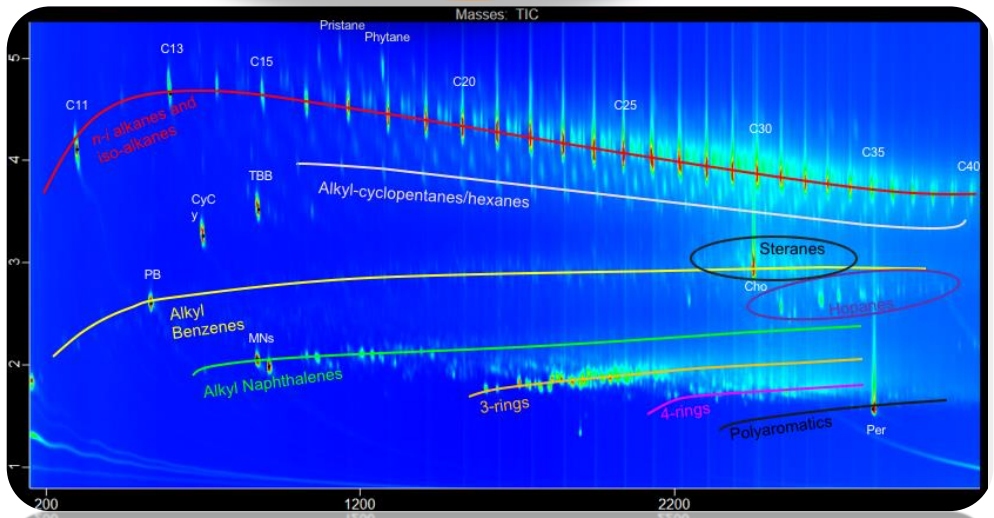
LC-GC

ROUTINE METHOD



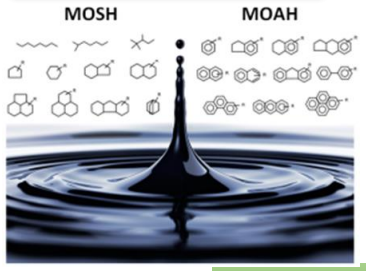
GC×GC-MS

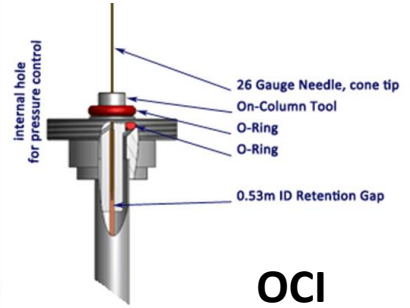
CONFIRMATORY METHOD



MOH AND MULTIDIMENSIONAL TECHNIQUEs

2021





OCI

MOH

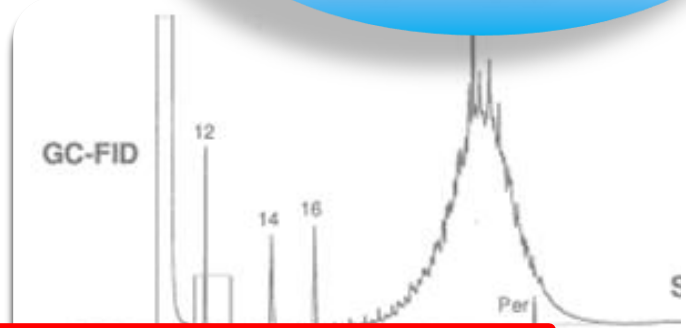
LC-GC

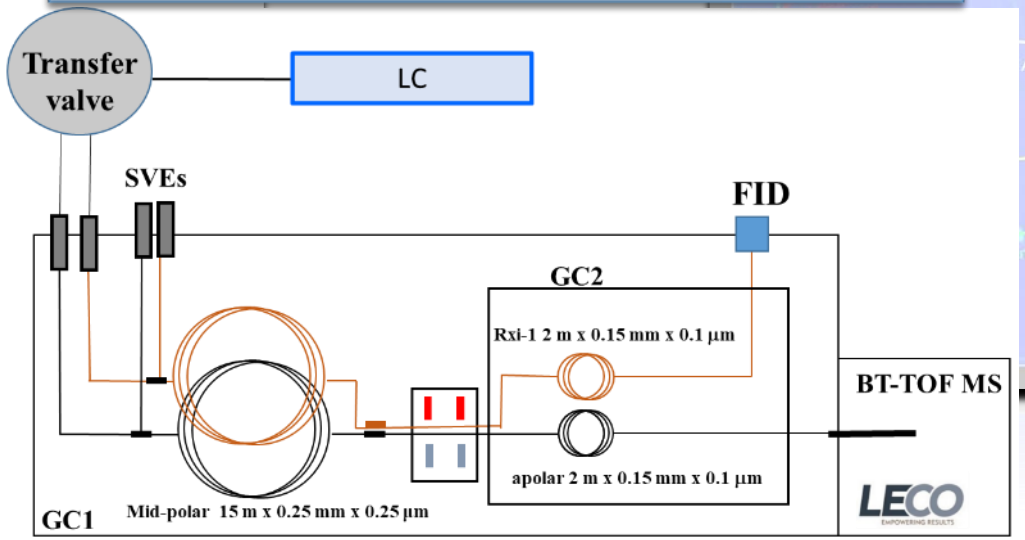
ROUTINE METHOD

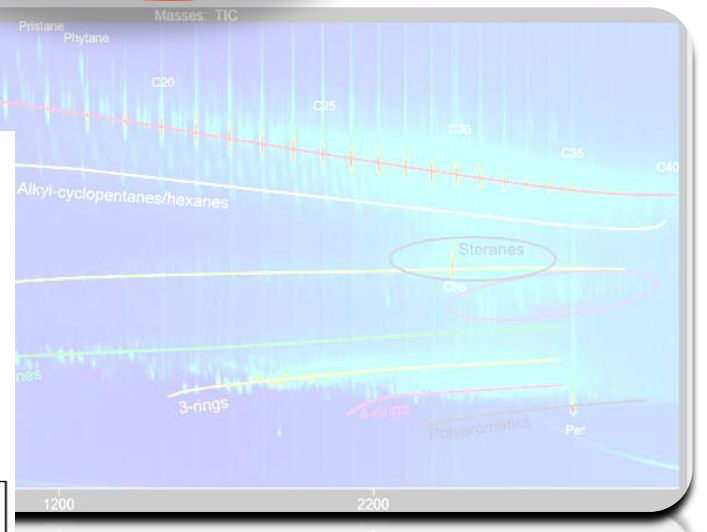
GC×GC

CONFIRMATORY METHOD

LC-2GC×GC-FID/MS







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MOH AND MULTIDIMENSIONAL TECHNIQUEs

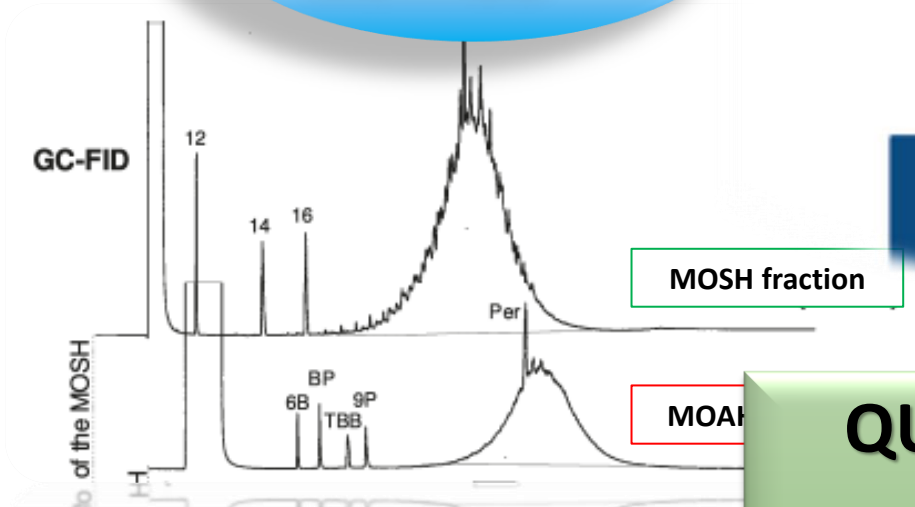
MOH

LC-GC

ROUTINE METHOD

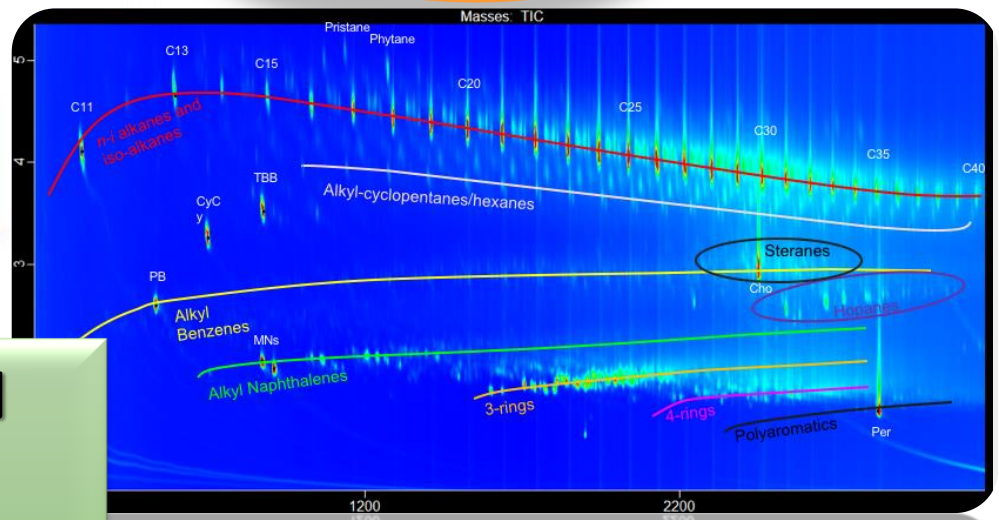
GC×GC

CONFIRMATORY METHOD



MOSH fraction

MOAH

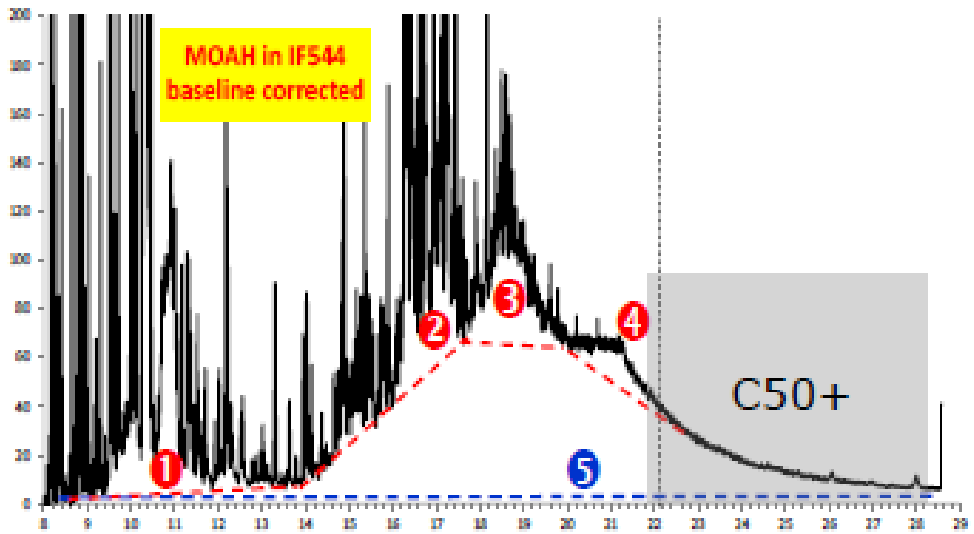


QUANTIFICATION in GC×GC

LC-GC for MOSH and MOAH integration

Integration:

- ***5*** pay attention to baseline correction → **Baseline subtraction**
- ***1, 2, 3 , 4*** should not be considered → **Trimming/smoothing of the riding peaks**



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

Data Interpretation

+

Data Integration

20 % of the total variability

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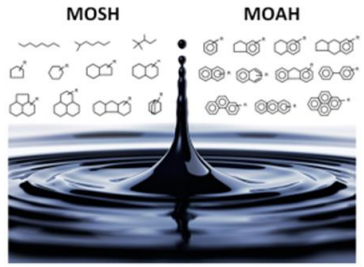
MOH & DATA INTEGRATION

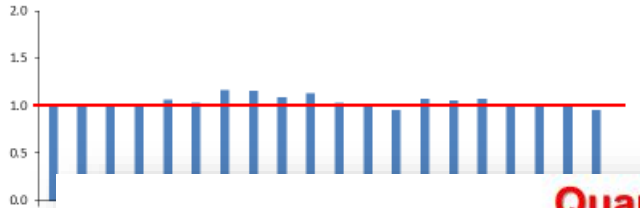


Quantification

Paraffin Mixture + n-Alkanes Mix

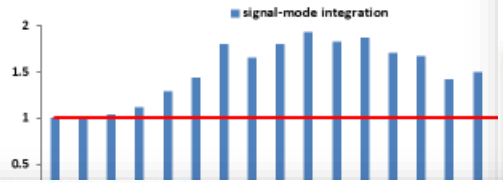
LC-GC-FID

1D



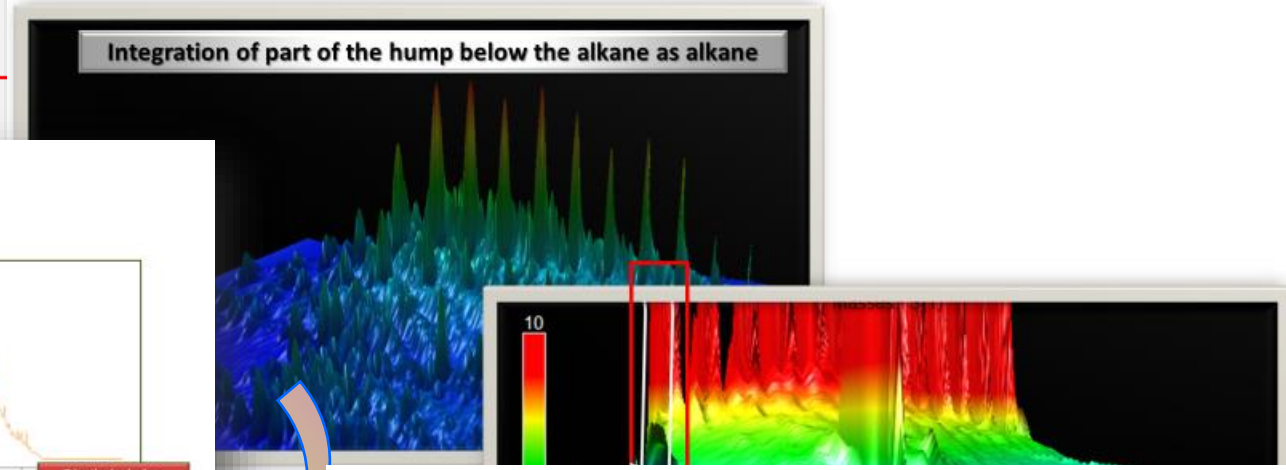
LC-GC×GC-FID

2D

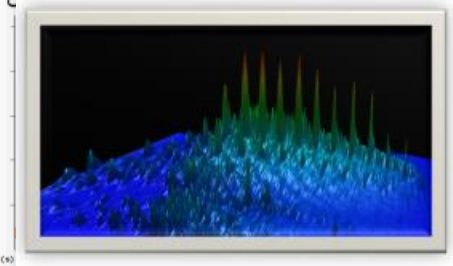


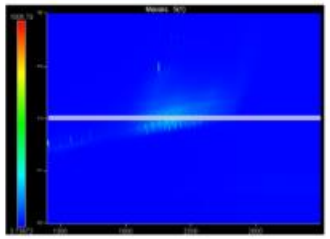
Quantification
LC-GC×GC-FID

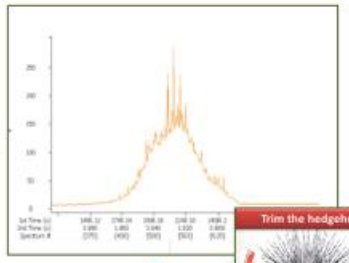
Integration of part of the hump below the alkane as alkane

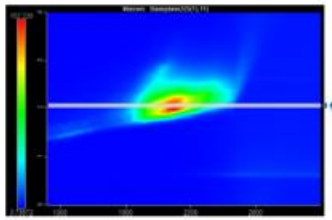


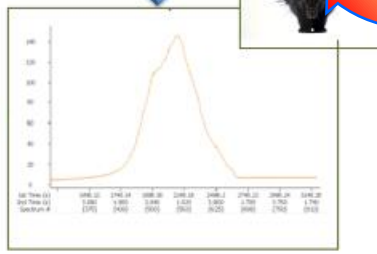
Quantification
LC-GC×GC-FID*











*patent pending

Journal of Chromatography A 1643 (2021) 462044

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

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LC-GC×GC-FID VALIDATION

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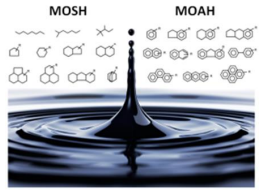
LC-GC×GC-FID VALIDATION




European Commission

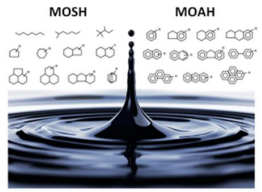
JRC TECHNICAL REPORT

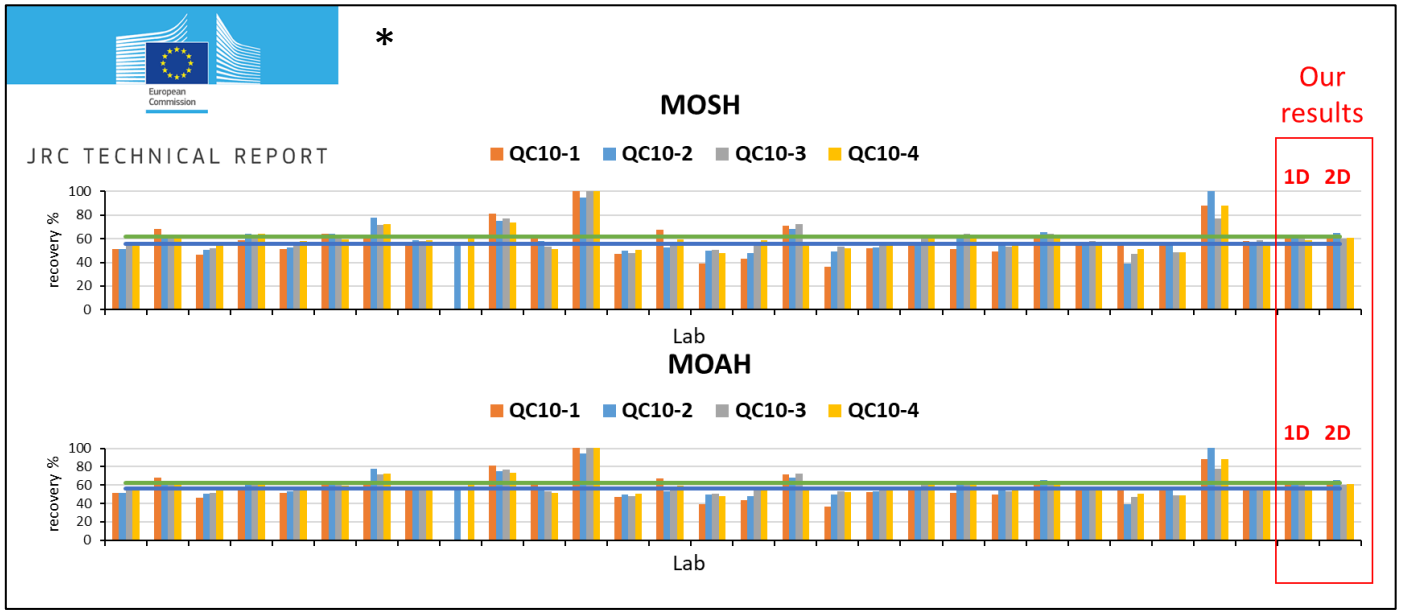
Determination of MOSH/MOAH in Shell SN500* mineral oil

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





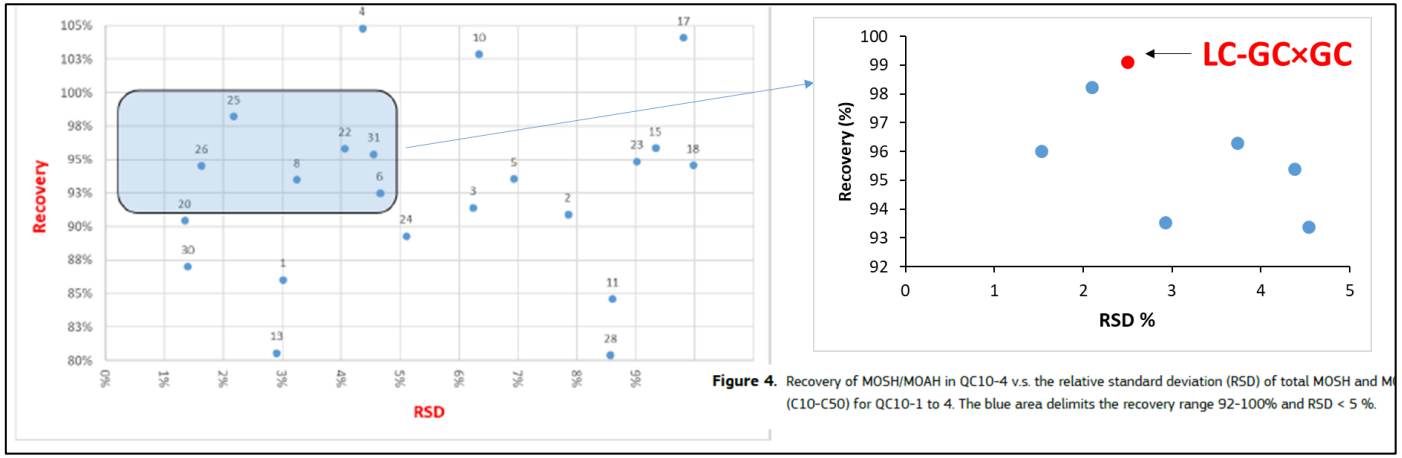


Figure 4. Recovery of MOSH/MOAH in QC10-4 v.s. the relative standard deviation (RSD) of total MOSH and M (C10-C50) for QC10-1 to 4. The blue area delimits the recovery range 92-100% and RSD < 5 %.

✓ **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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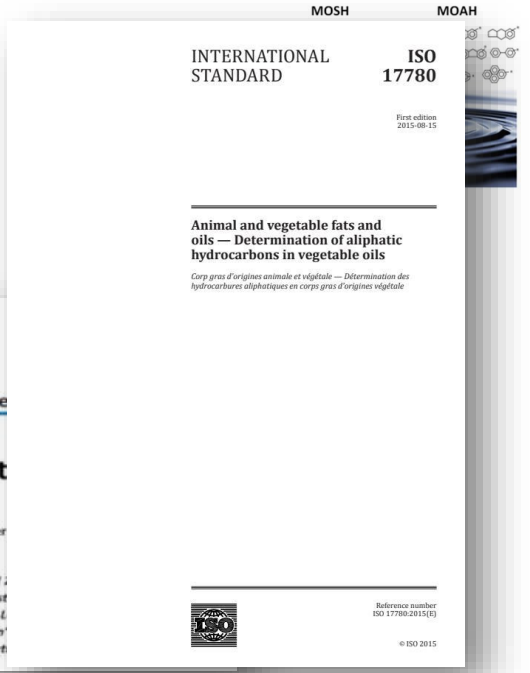
EXISTING STANDARD METHODS

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



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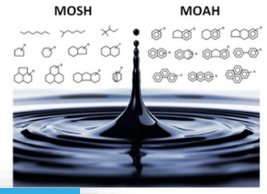
LC-GC×GC-FID VALIDATION



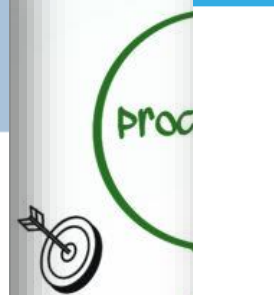
European Commission

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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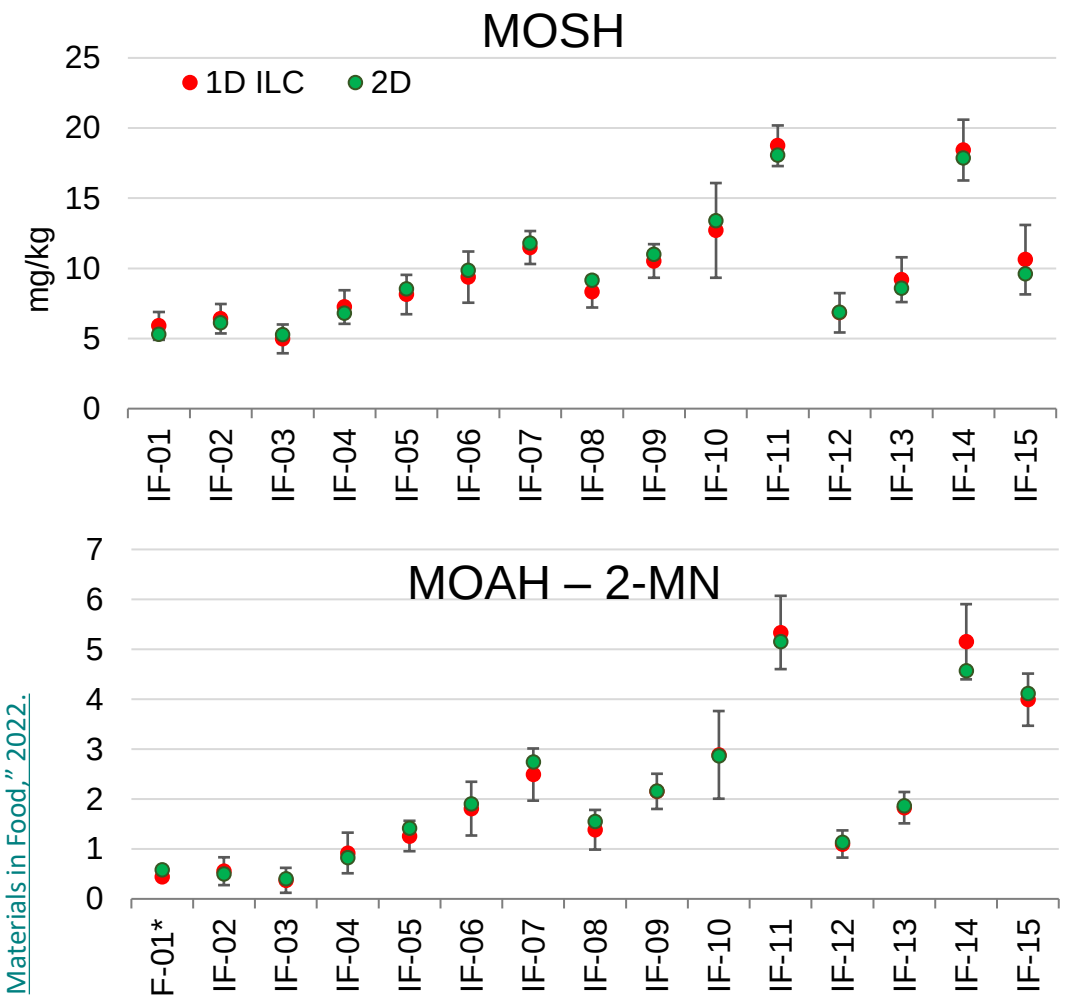
LC-GC×GC-FID/MS

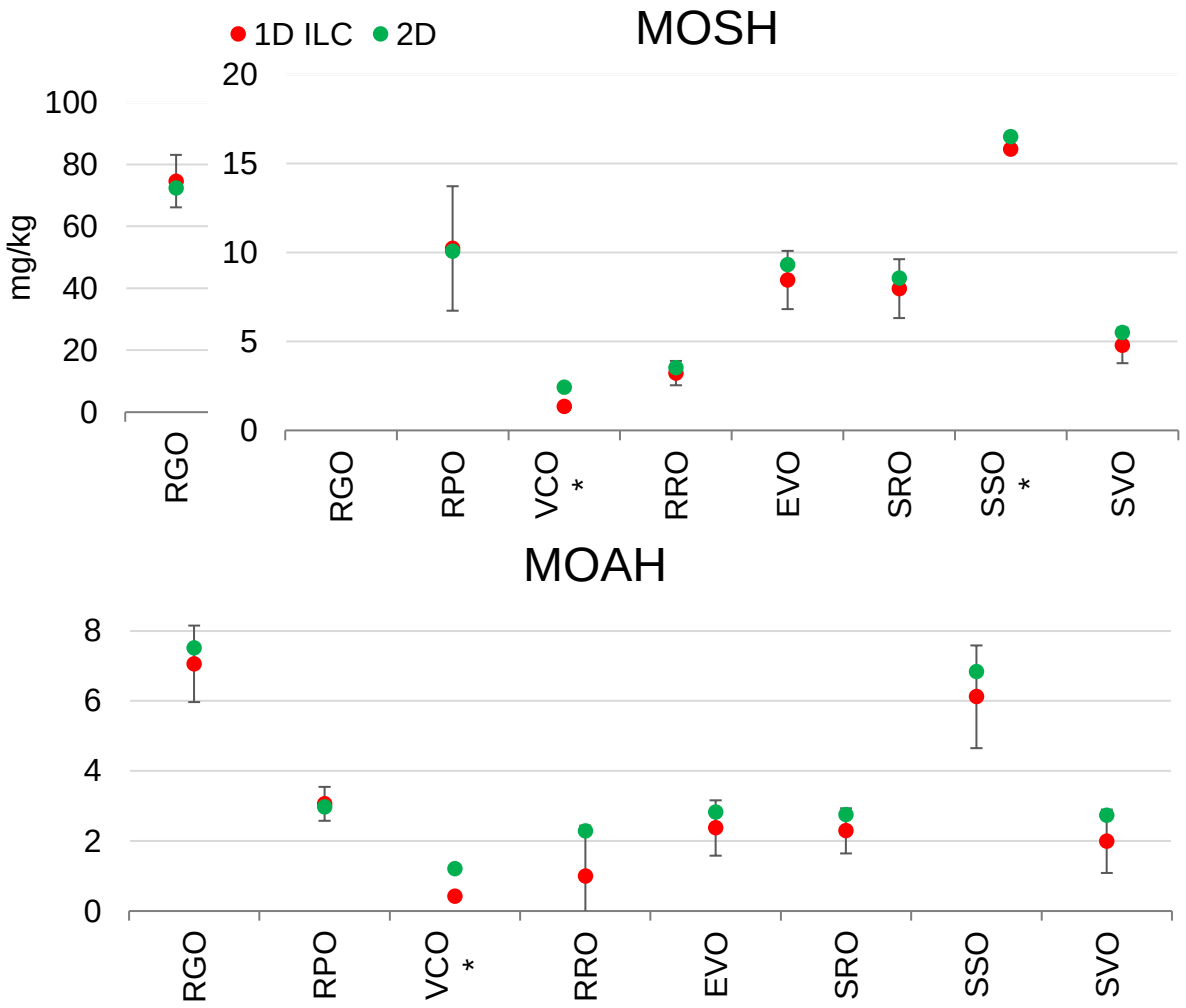
Data Integration

Data Interpretation

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



revision of DGF-EN 16995:2017



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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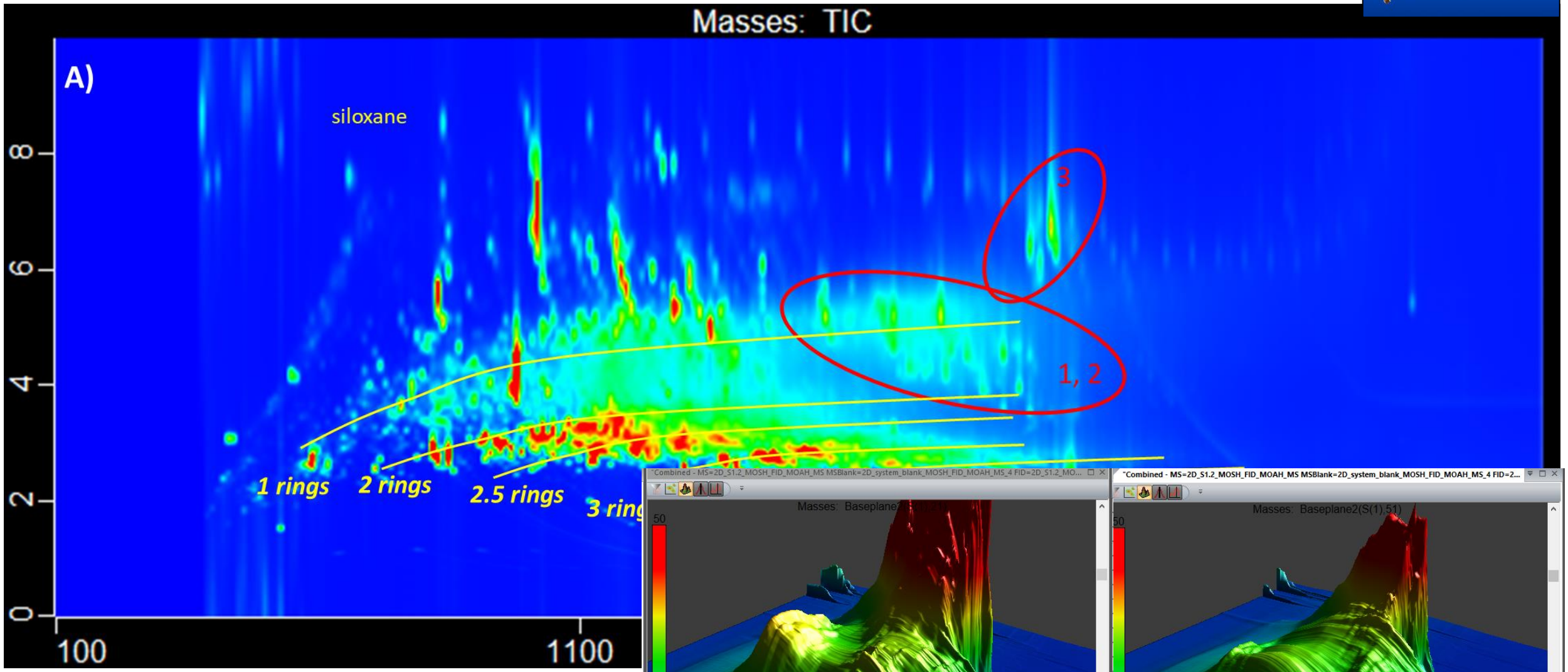
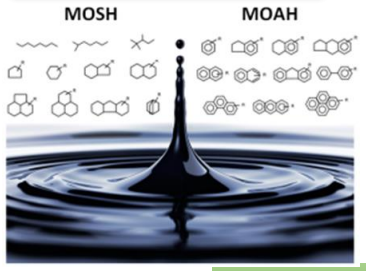
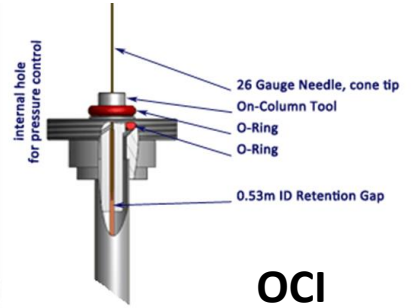
Advantage of GC×GC-ToFMS



MOH AND MULTIDIMENSIONAL TECHNIQUEs

2021





OCI

MOH

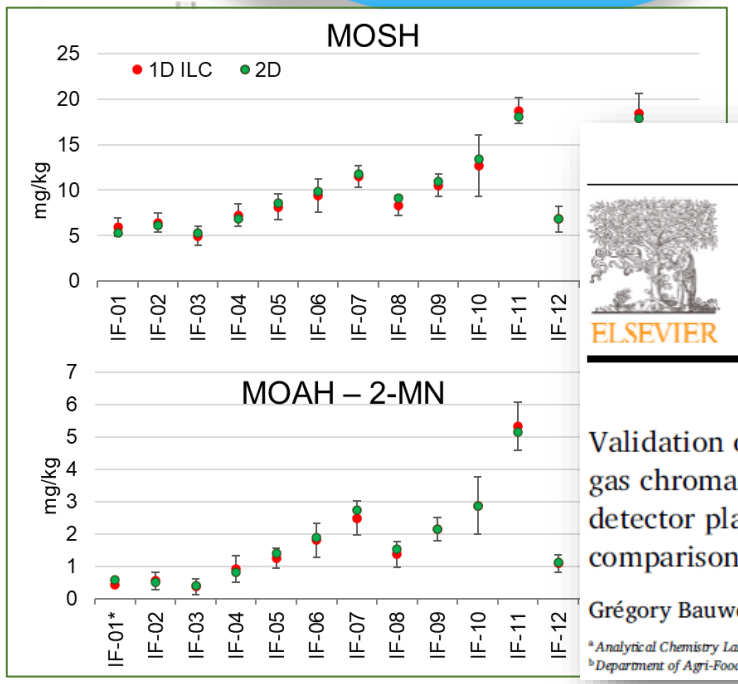
LC-GC

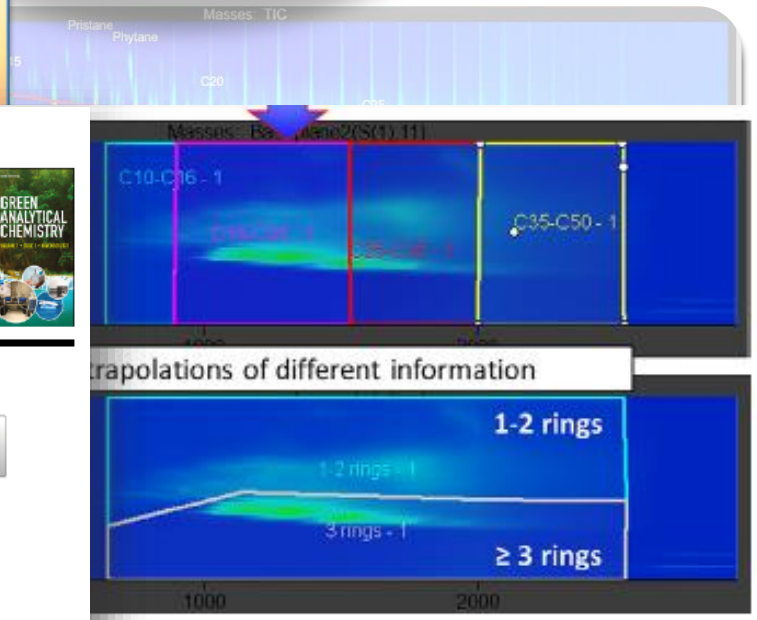
ROUTINE METHOD

GC×GC

CONFIRMATORY METHOD

LC-2GC×GC-FID/MS





Green Analytical Chemistry 4 (2023) 100047

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Green Analytical Chemistry

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

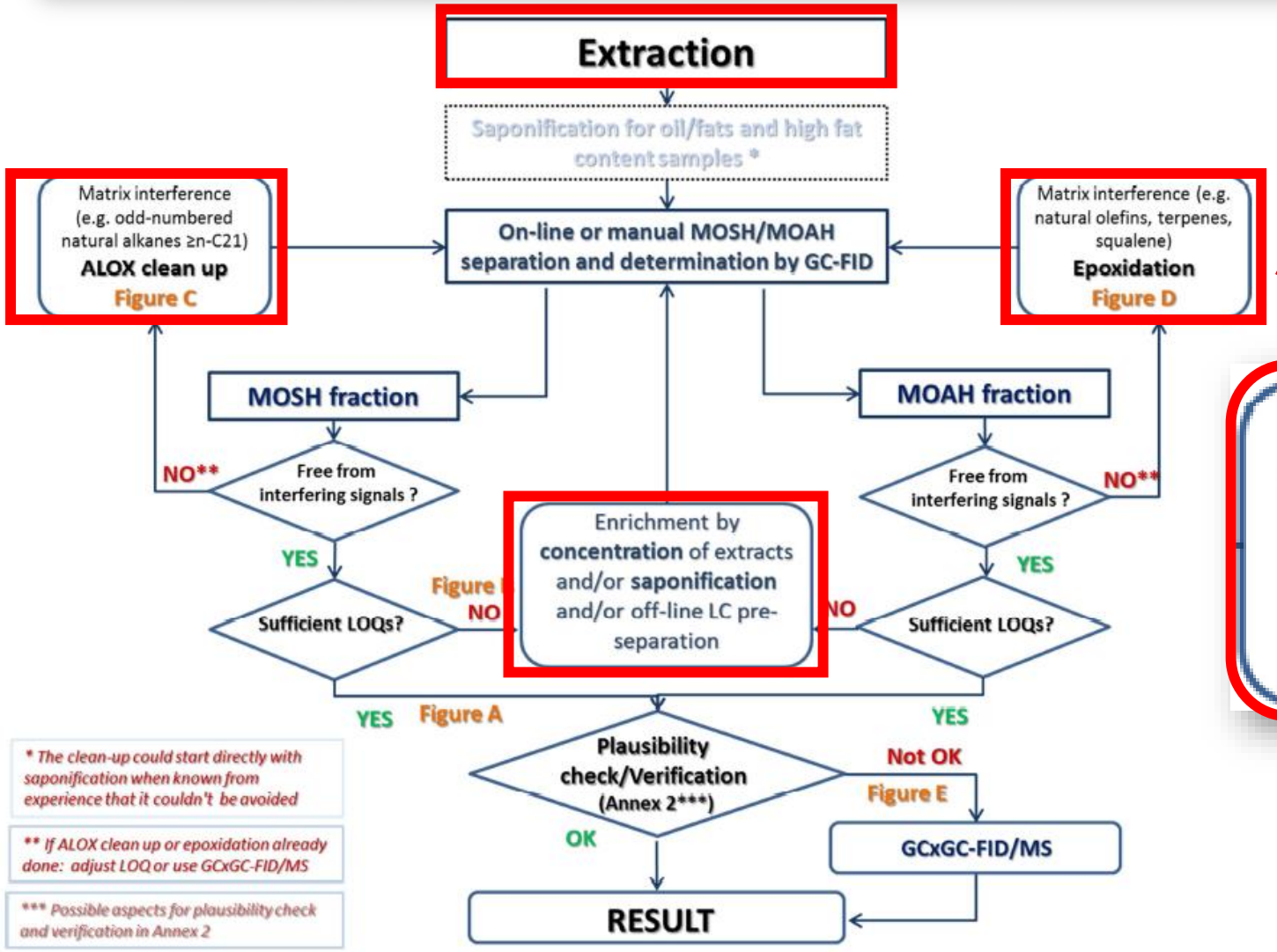
Validation of the liquid chromatography-comprehensive multidimensional gas chromatography-time-of-flight mass spectrometer/flame ionization detector platform for mineral oil analysis exploiting interlaboratory comparison data

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

➢ Outline of the Analytical approach

Sample Preparation



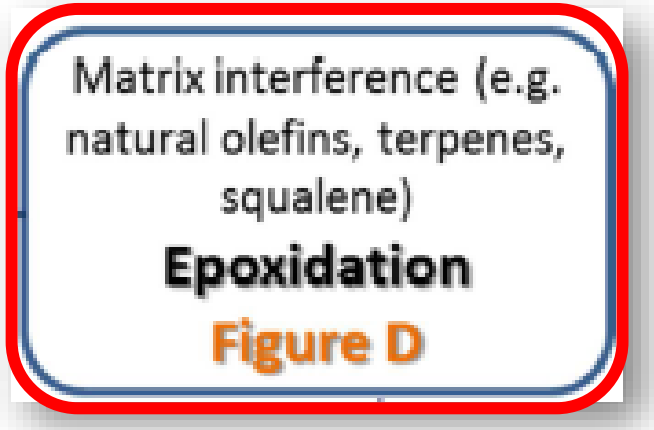
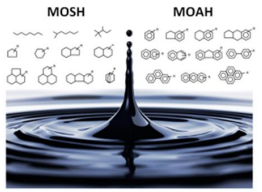


Figure 1 Decision tree on the use of auxiliary methods. Figures A-E can be found in Annex I. ALOX is aluminium oxide.

✓*Need for matrix-tailored sample prep protocols*

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

About 20% of MOAH losses

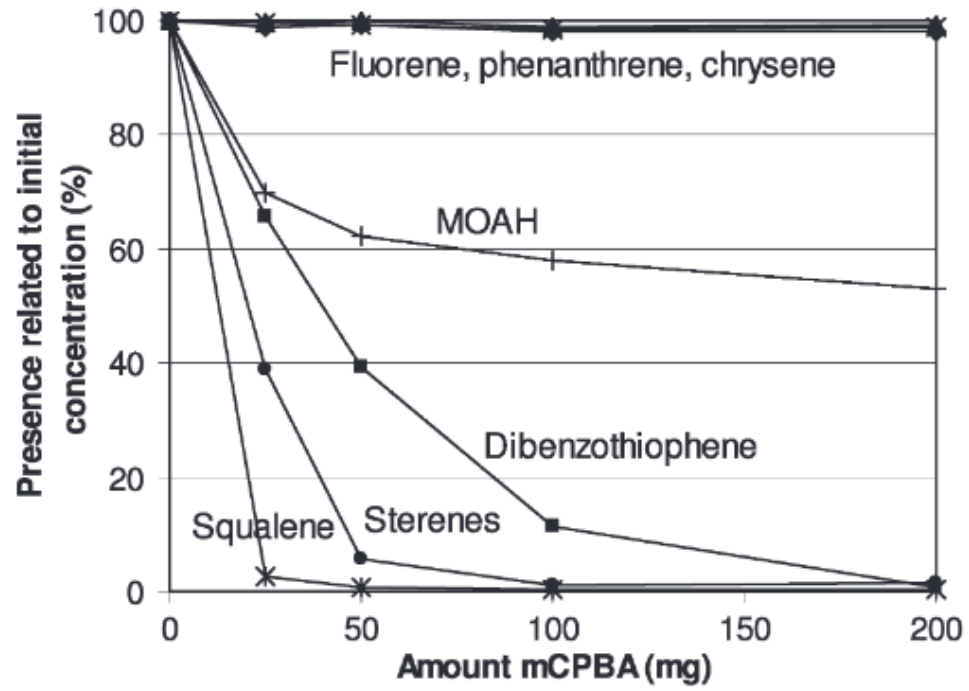


Figure 3. Epoxidation with different amounts of peracid. More peracid better removes squalene and the sterenes, but also the loss of certain aromatics increases.

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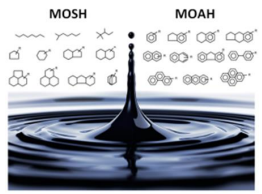
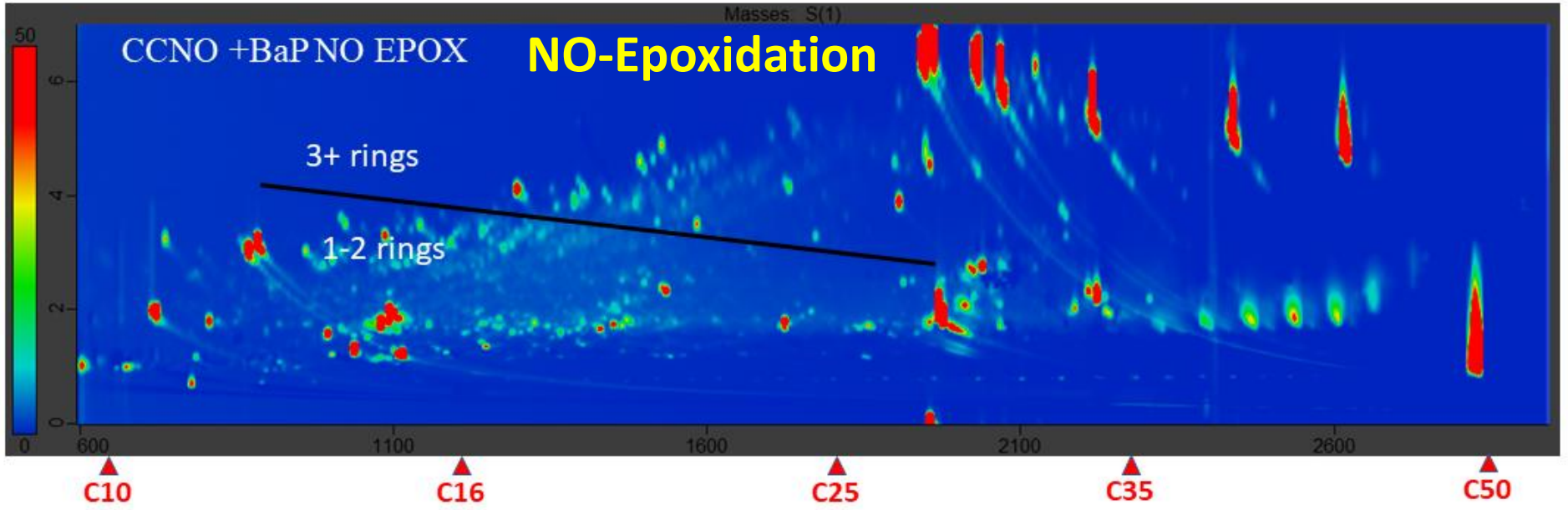
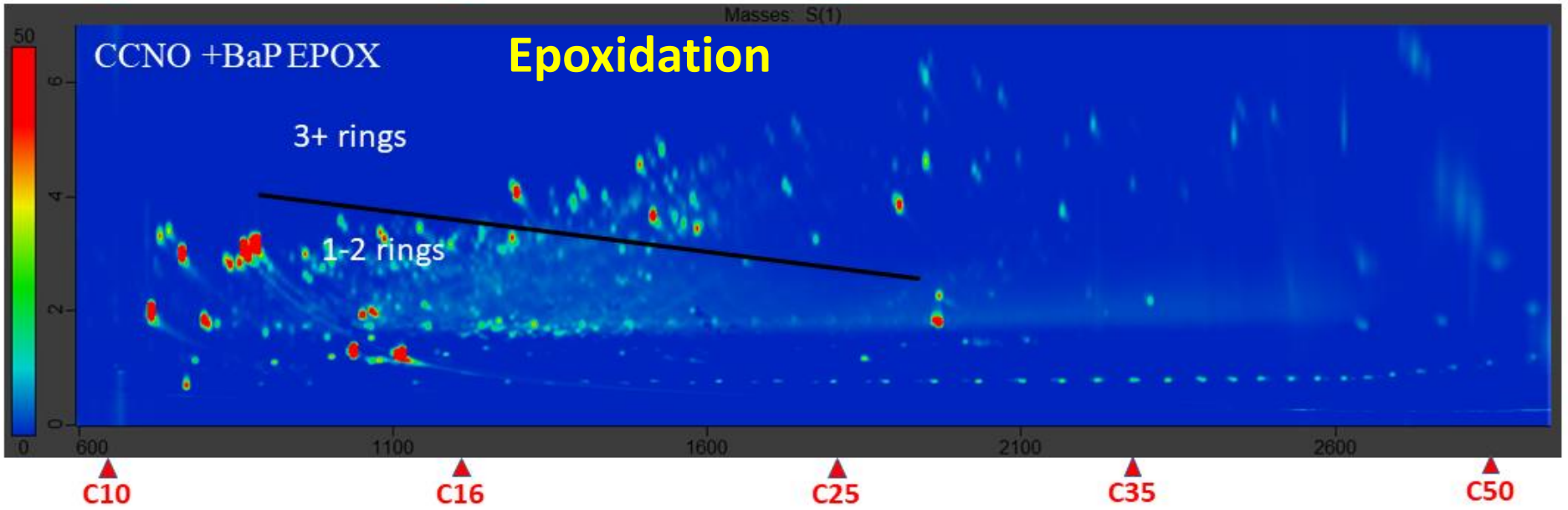
LC-GC×GC-FID/MS & EPOXIDATION

Sample Preparation

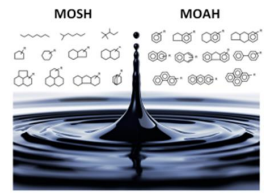


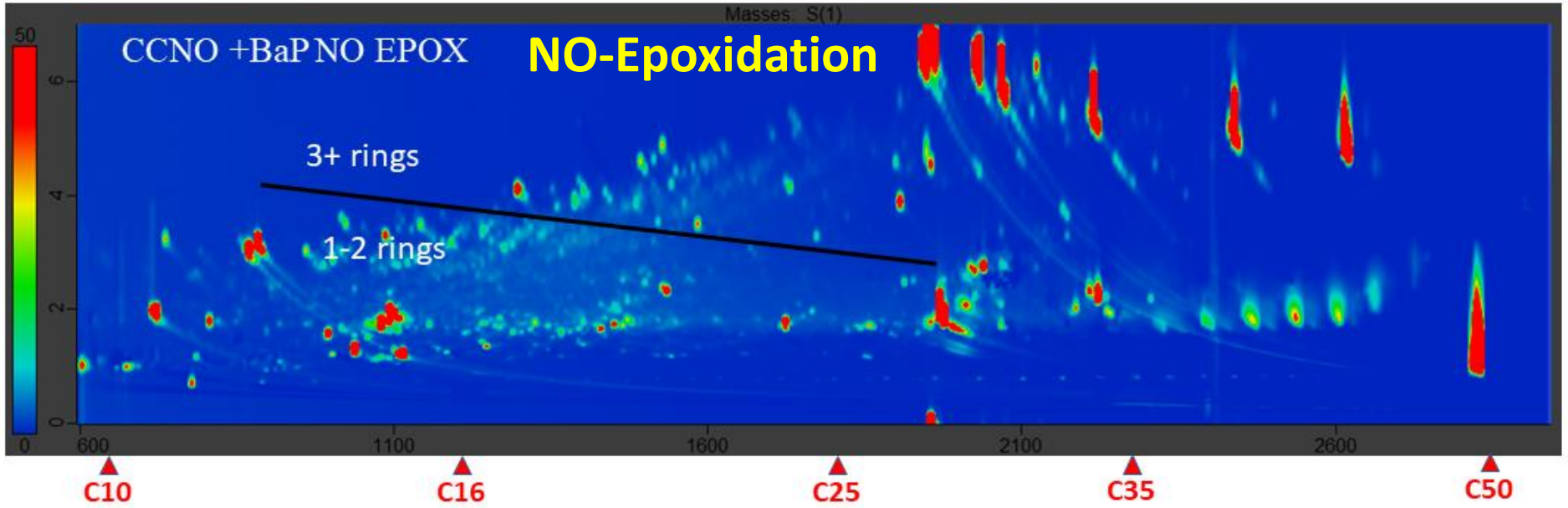


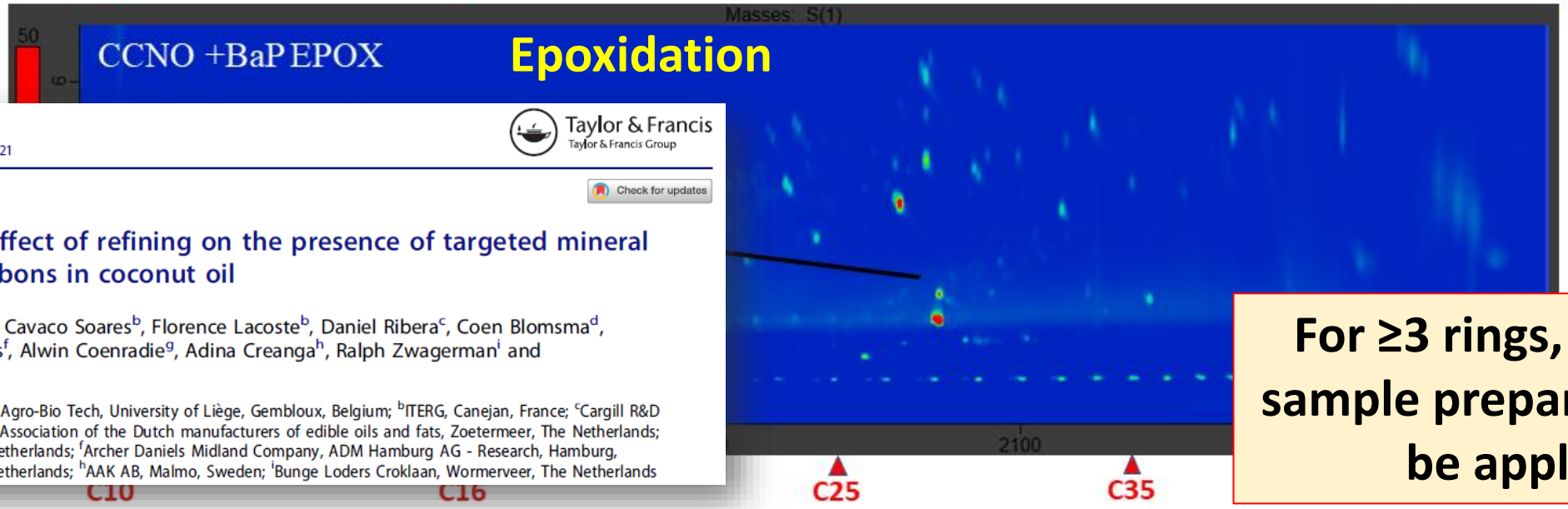


LC-GC×GC-FID/MS & EPOXIDATION

Sample Preparation







FOOD ADDITIVES & CONTAMINANTS: PART A
https://doi.org/10.1080/19440049.2022.2164621

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Check for updates

Investigation of the effect of refining on the presence of targeted mineral oil aromatic hydrocarbons in coconut oil

Grégory Bauwens[a], Alexandre Cavaco Soares[b], Florence Lacoste[b], Daniel Ribera[c], Coen Blomsma[d], Iekje Berg[e], Fernando Campos[f], Alwin Coenradie[g], Adina Creanga[h], Ralph Zwagerman[i] and Giorgia Purcaro[a]

[a]Analytical Chemistry Lab, Gembloux Agro-Bio Tech, University of Liège, Gembloux, Belgium; [b]ITERG, Canejan, France; [c]Cargill R&D Center, Vivoorde, Belgium; [d]VERNOF, Association of the Dutch manufacturers of edible oils and fats, Zoetermeer, The Netherlands; [e]Sime Darby Oils, Zwijndrecht, The Netherlands; [f]Archer Daniels Midland Company, ADM Hamburg AG - Research, Hamburg, Germany; [g]Olenex, Rotterdam, The Netherlands; [h]AAK AB, Malmo, Sweden; [i]Bunge Loders Croklaan, Wormerveer, The Netherlands

For ≥3 rings, simplify sample preparation can be applied

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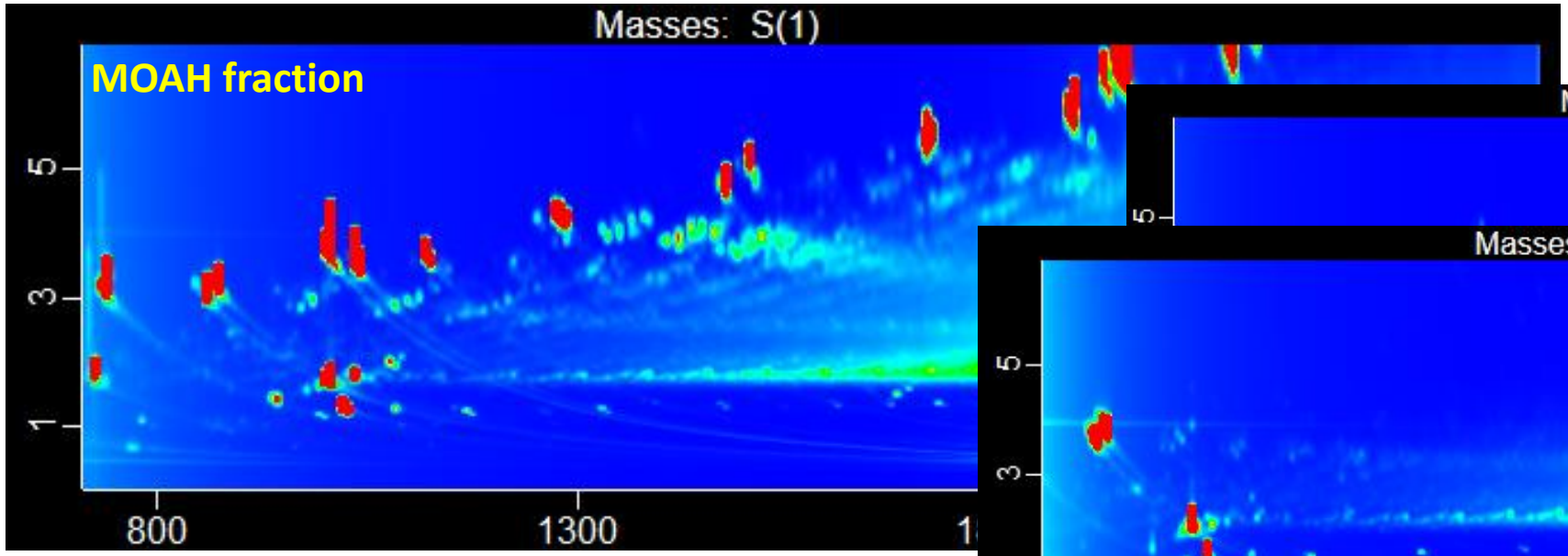
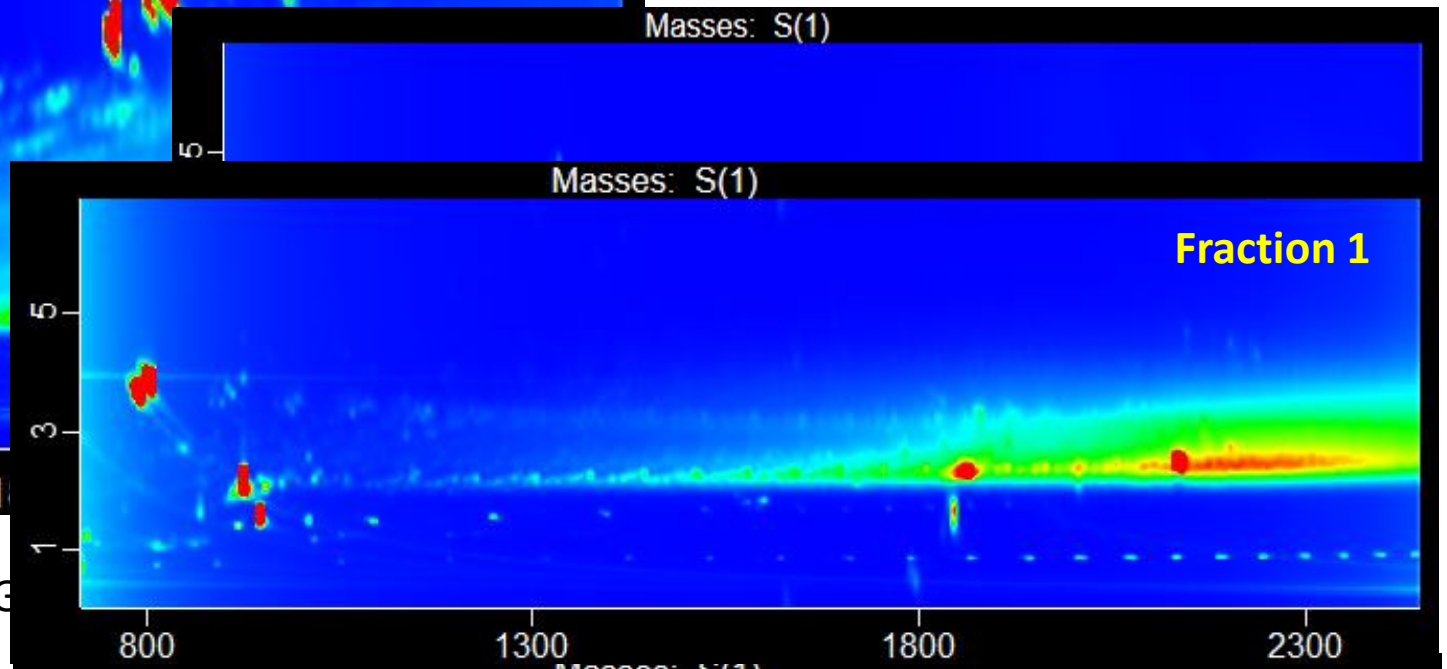
ALTERNATIVE TO EPOXIDATION: LC-GC×GC

Sample Preparation

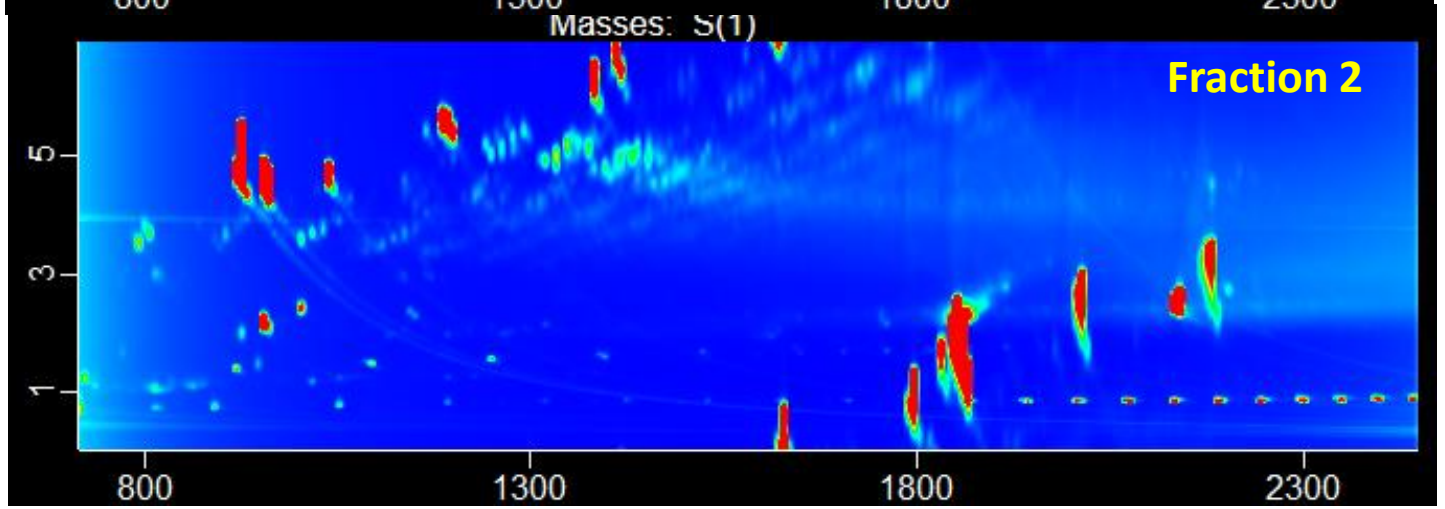




MOSH&MOAH IS+ EPA PAHs + Coronene + QC10 +HVG

Interferences

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



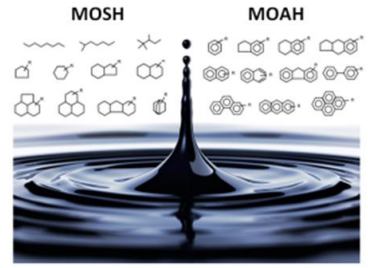
Recovery of Fr1+Fr2

104±4%

CONCLUSION
OPEN QUESTIONS AND FUTURE DIRECTIONS

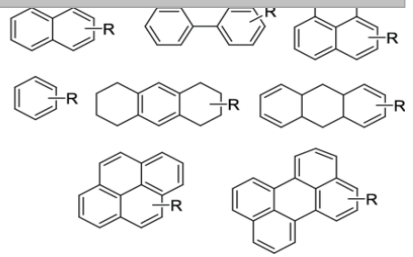
MOSH

MOAH



MOAH

- In depth characterization and further studies highly needed for a full toxicological characterization



TECHNICAL REPORT

2019

efsa European Food Safety Authority Supporting Publications

APPROVED: 15 November 2019

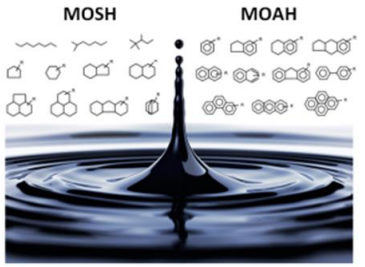
doi:10.2903/sp.efsa.2019.EN-1741

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)

Recommendations

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

CONCLUSION
LC-GCxGC-FID/TOFMS



MOSH

MOAH

MOAH

- In depth characterization and further studies highly needed for a full toxicological characterization

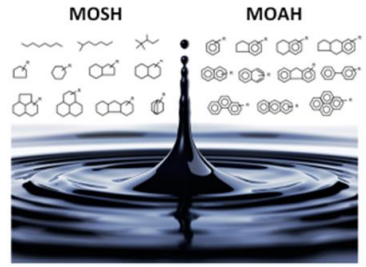
- Reduction of data uncertainty

Sample Preparation

Data Interpretation

Data Integration

CONCLUSION
LC-GCxGC-FID/TOFMS



MOSH | **MOAH**

- In depth characterization and further studies highly needed for a full toxicological characterization
- Reduction of data uncertainty

Sample Preparation | **Data Interpretation** | **Data Integration**

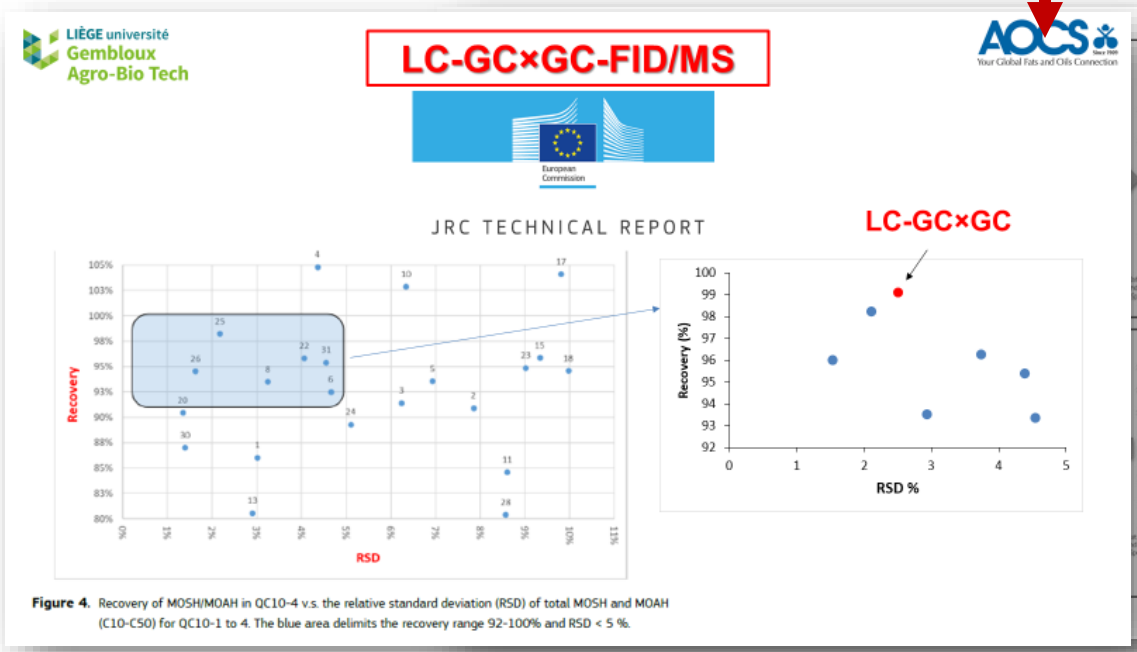
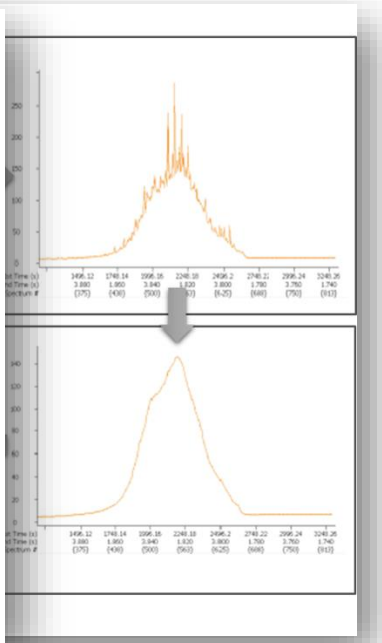
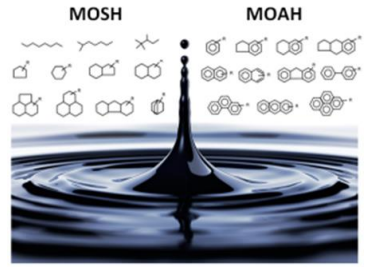


Figure 4. Recovery of MOSH/MOAH in QC10-4 v.s. the relative standard deviation (RSD) of total MOSH and MOAH (C10-C50) for QC10-1 to 4. The blue area delimits the recovery range 92-100% and RSD < 5 %.



CONCLUSION
LC-GCxGC-FID/TOFMS



MOSH

MOAH

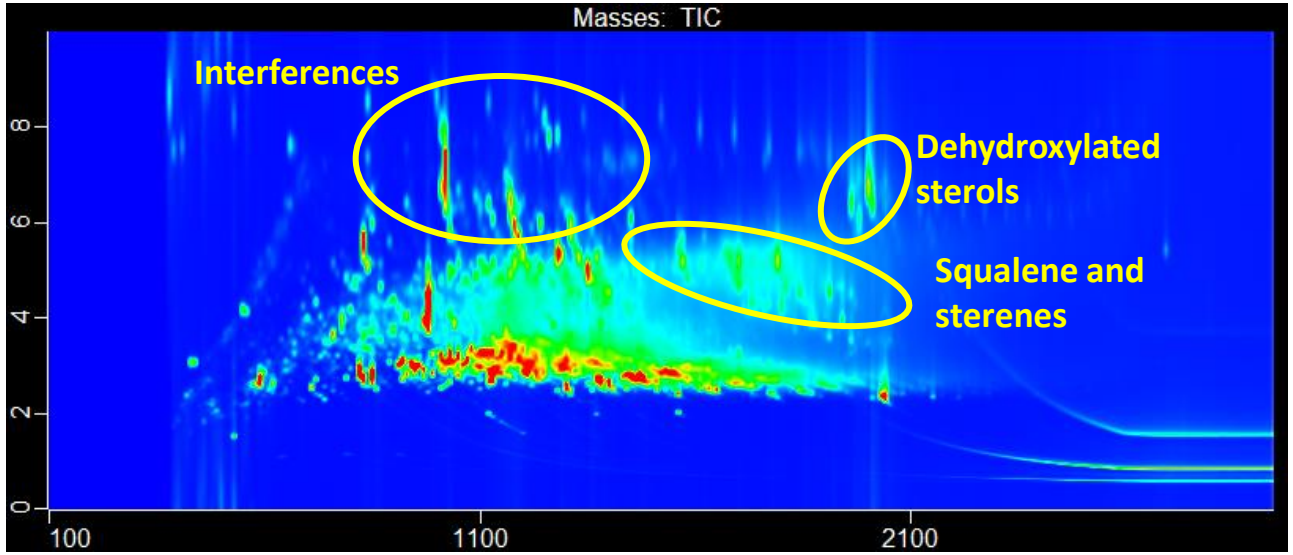
- In depth characterization and further studies highly needed for a full toxicological characterization

- Reduction of data uncertainty

Sample Preparation

Data Interpretation

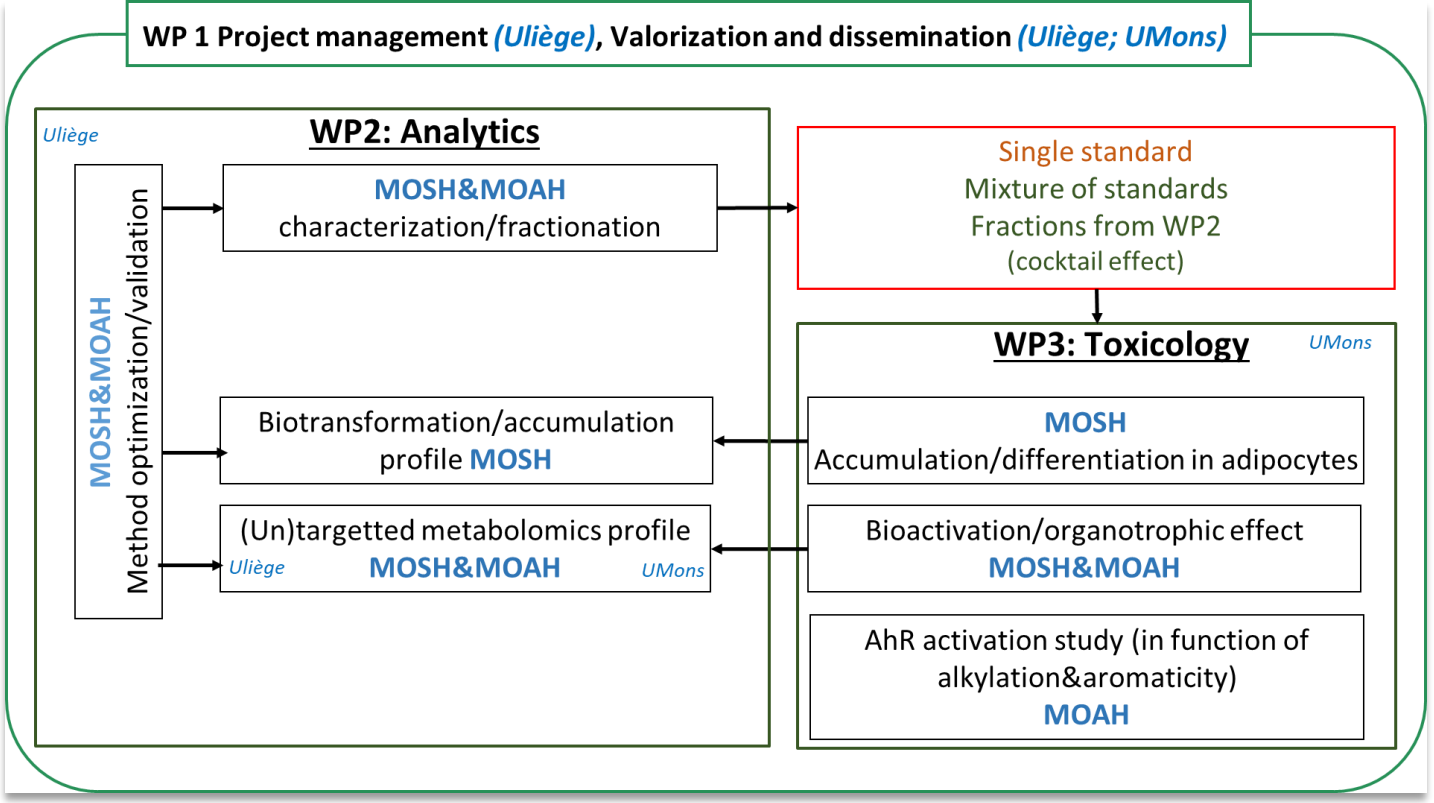
Data Integration



WHERE ARE WE GOING?

TOXICOLOGY

ANALYTICS



FRS-FNRS-Research Project 2022 (T.0187.23)
Title: “Multidisciplinary approach to enhance MOSH and MOAH understanding (**ToxAnaMOH**)”

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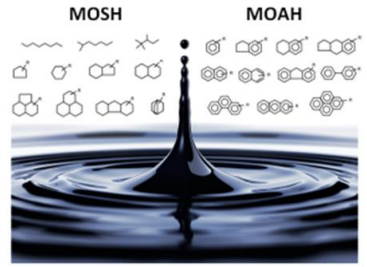
Agro-Bio Tech



My research group:

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Visiting students:

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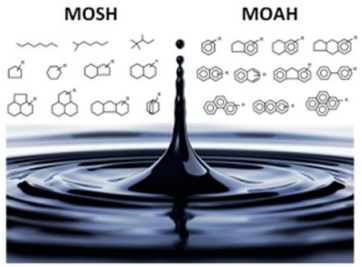

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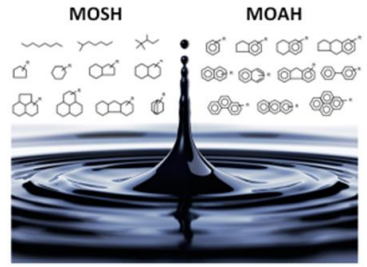

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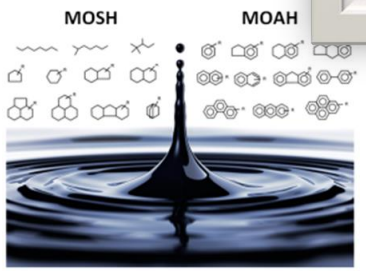

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