LIÈGE université
Gembloux
Agro-Bio Tech

ACESSS
Academic Center of Excellence in Separation Science and Sensing

Flash presentation

MOSH&MOAH

in **food ingredients** and **additives**

and the advantages of using

LC / GC×GC (-FID/TOFMS)

for their analysis

Aleksandra Gorska, Carlo Bellinghieri, Paula Albendea, Giorgia Purcaro

Analytical Chemistry – Gembloux Agro-Bio Tech

February 3rd 2025

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MOSH & MOAH

Mineral oil saturated hydrocarbons

Mineral oil aromatic hydrocarbons

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Ubiquitous, petroleum-derived food contaminants

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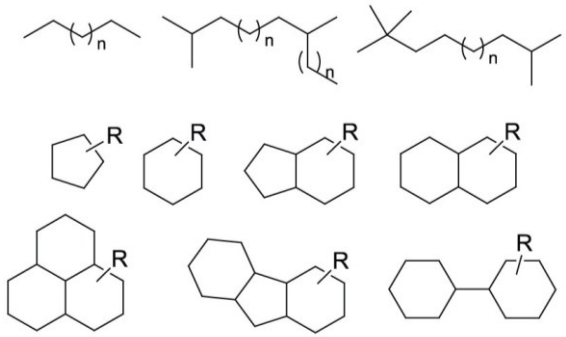
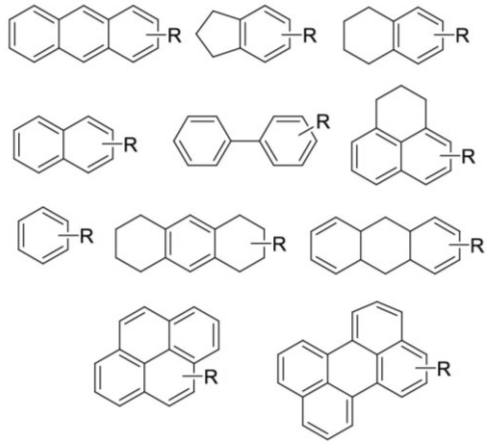


Linear and branched alkanes
Branched cycloalkanes



Branched mono- or polyaromatic compounds

MOSH & MOAH

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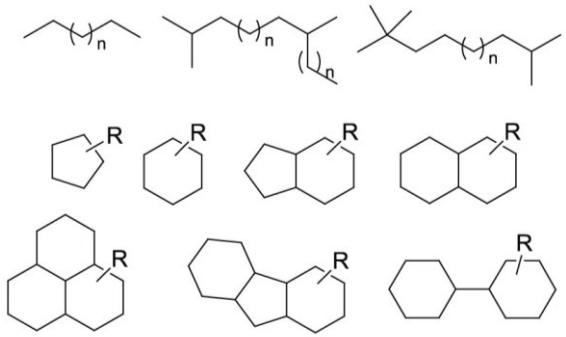

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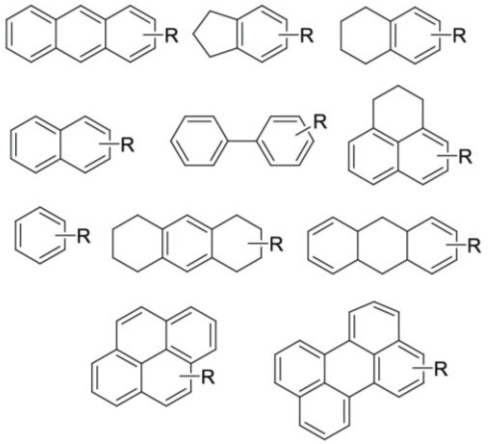

Branched mono- or polyaromatic compounds

- **Toxicological properties**
  - **MOSH** accumulate in human tissues
  - **MOAH** are associated with **genotoxic** properties (compounds with **≥ 3 AR**)

MOSH & MOAH

Mineral oil saturated hydrocarbons **Mineral oil aromatic hydrocarbons**

Ubiquitous, petroleum-derived food contaminants

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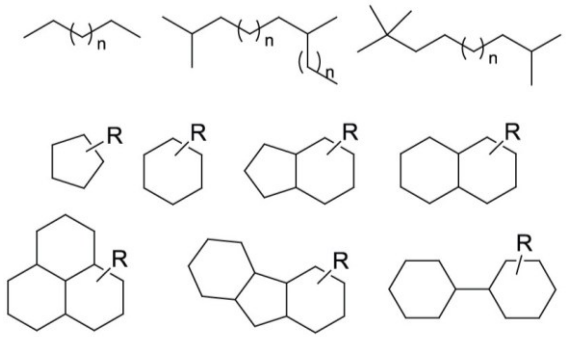

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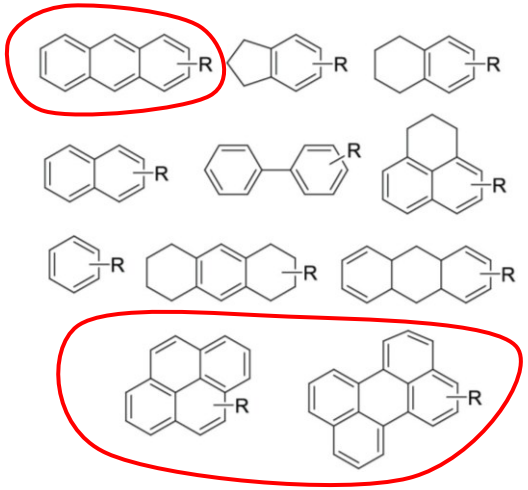

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We wanted to tackle this gap by gathering data on MOSH/MOAH …

Method for MOSH/MOAH analysis in food

Method for MOSH/MOAH analysis in food

Food sample

Method for MOSH/MOAH analysis in food

Food sample

↓

Extraction

Method for MOSH/MOAH analysis in food

Food sample
↓
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Purification

Method for MOSH/MOAH analysis in food

Food sample

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Extraction

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HPLC-GC-FID

Method for MOSH/MOAH analysis in food

Food sample

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↓

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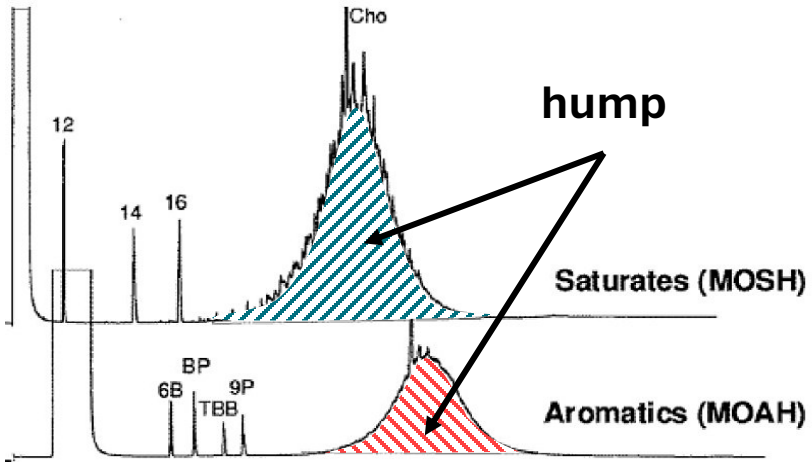
HPLC-GC-FID



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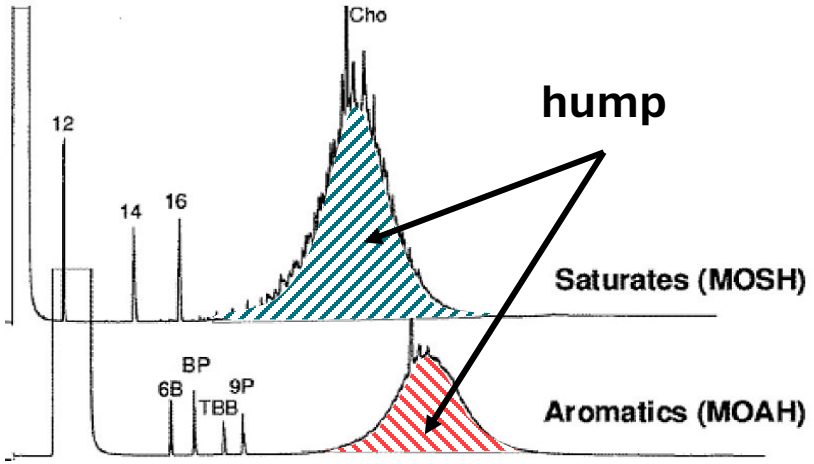

Food sample
↓
Extraction
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Purification
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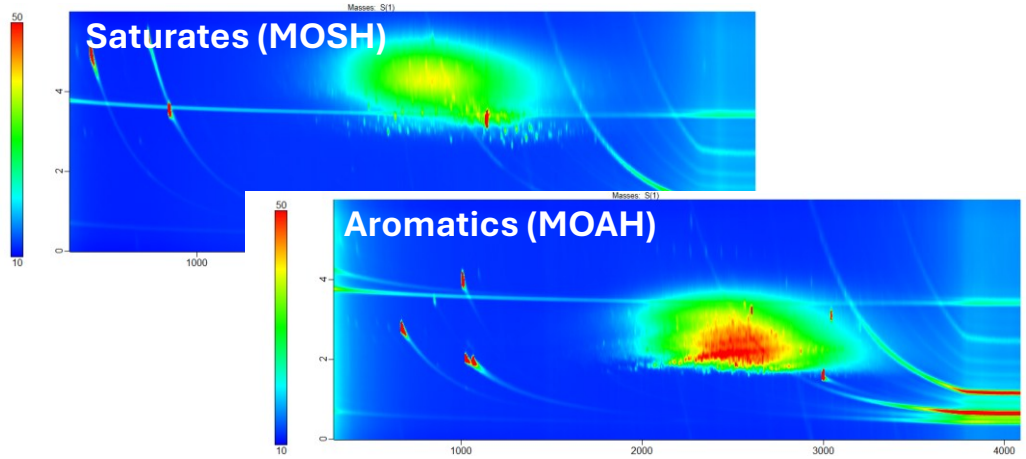
HPLC/GC×GC-FID/TOFMS

Preferred for the project



Goal of the presentation

Illustrate the **advantages** of working with **HPLC/GC×GC-FID/TOFMS** instead of HPLC-GC-FID to tackle the request of EFSA

Description of the system

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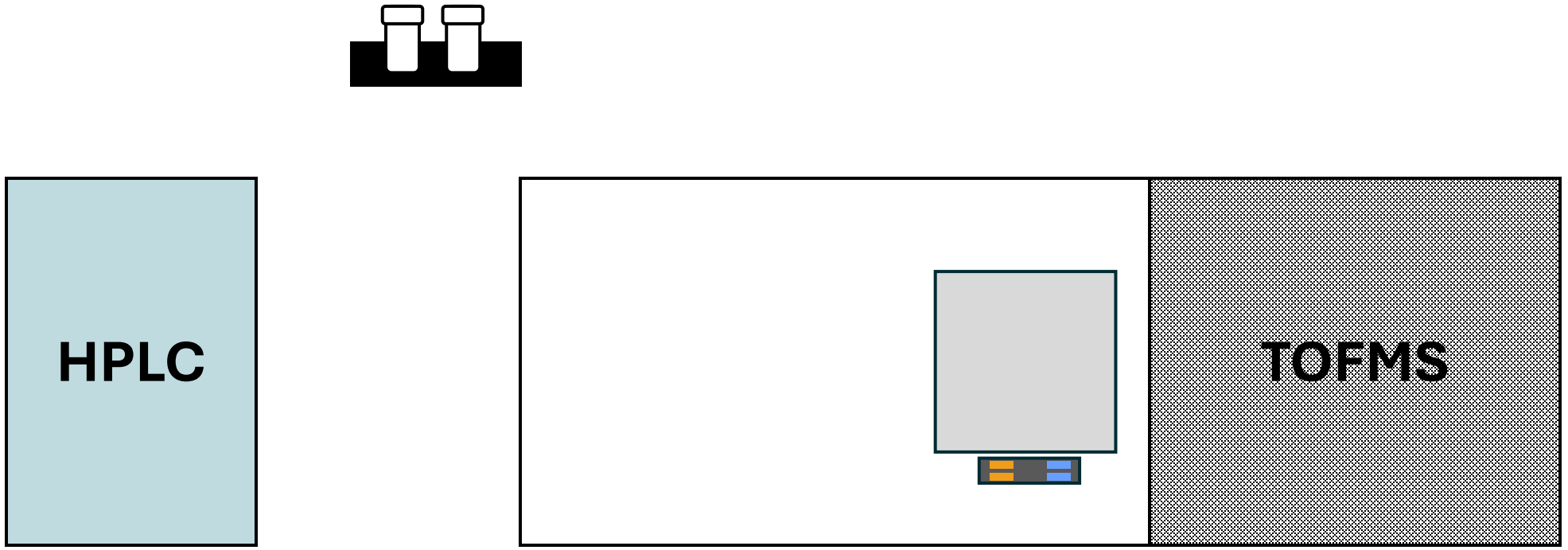

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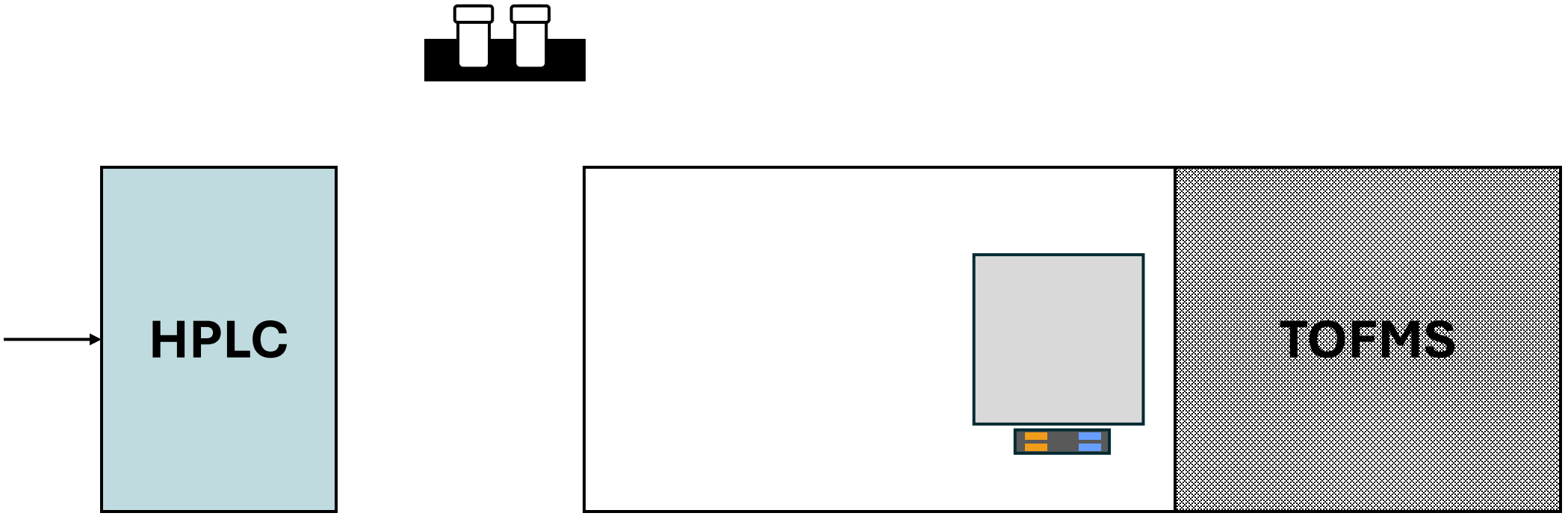

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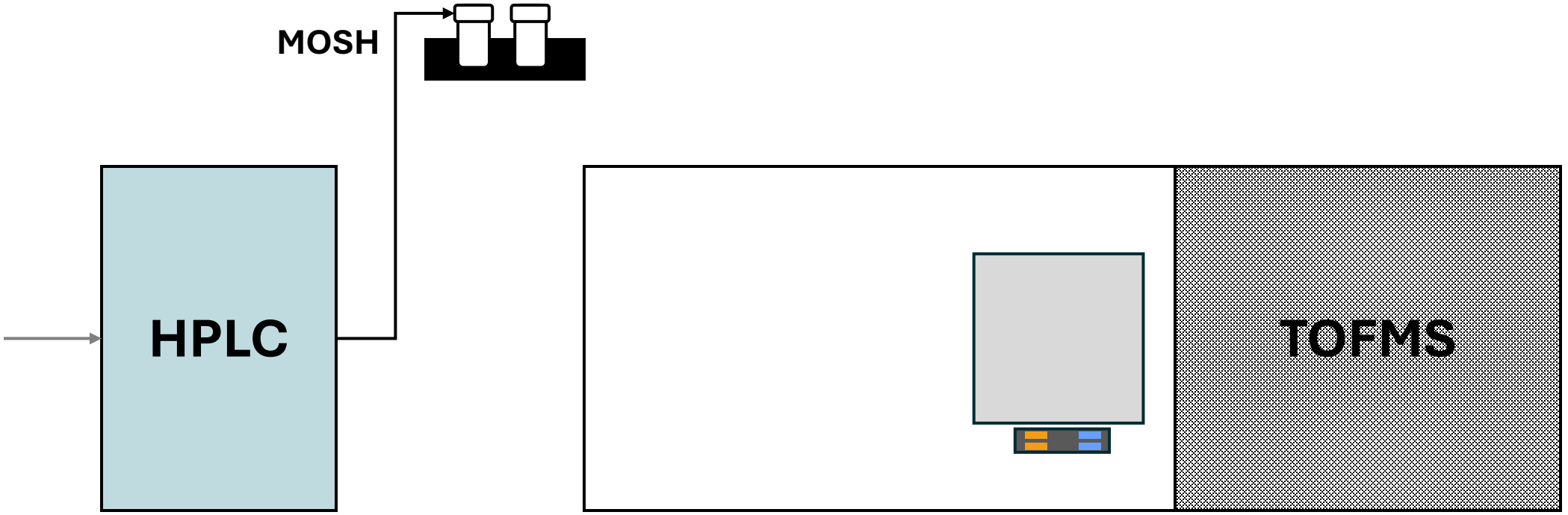

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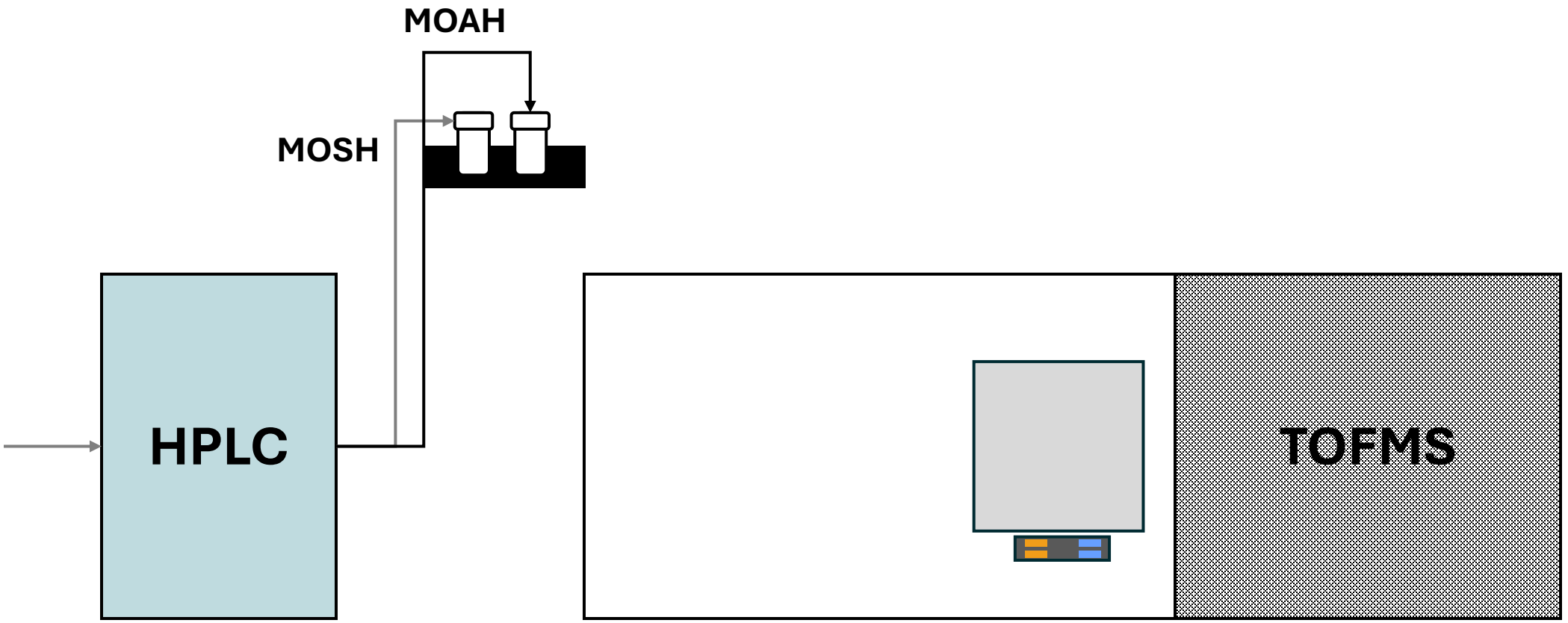

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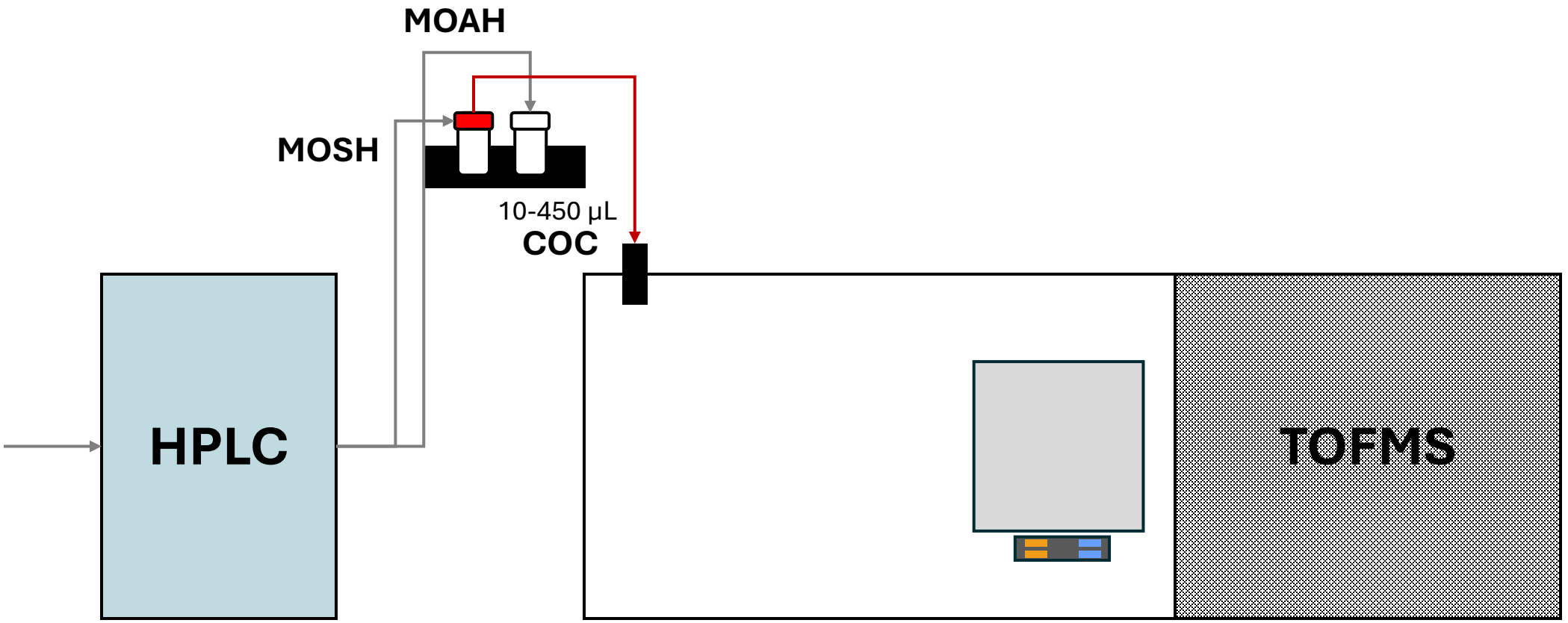

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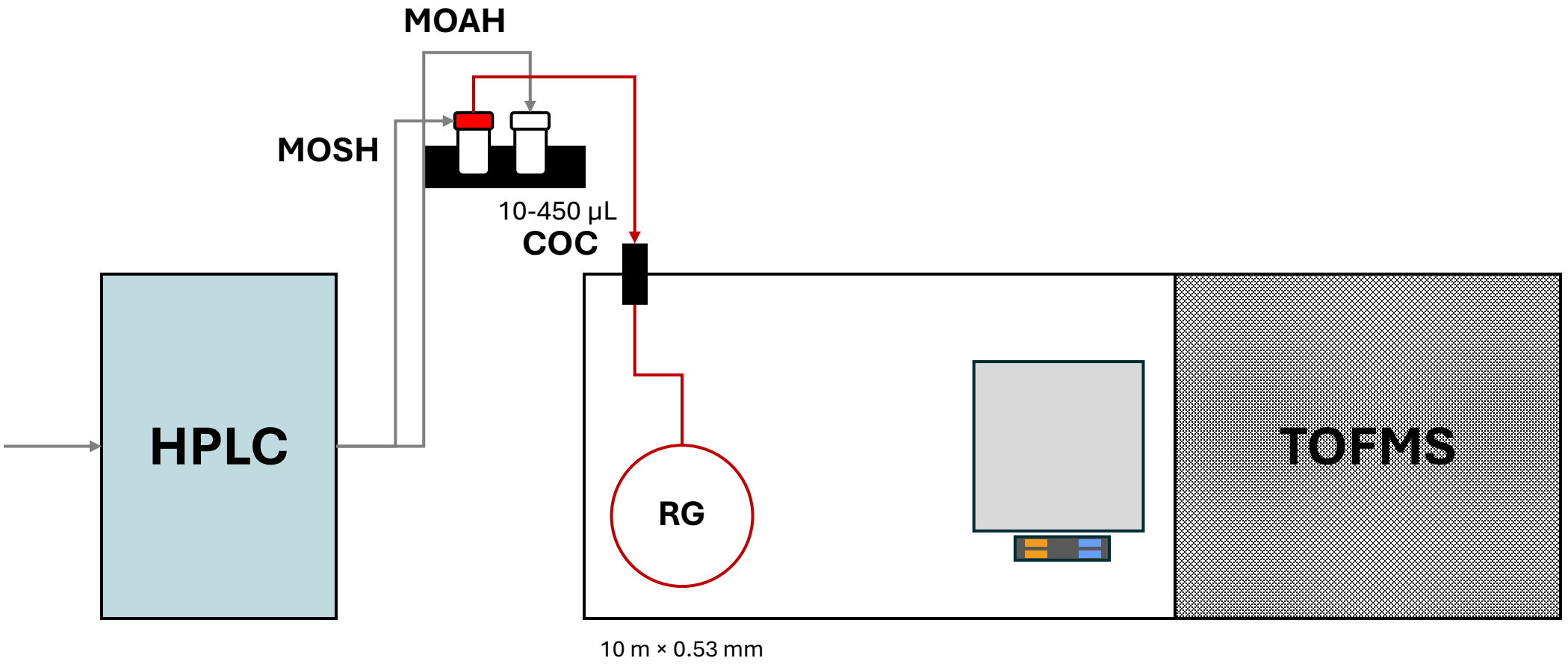

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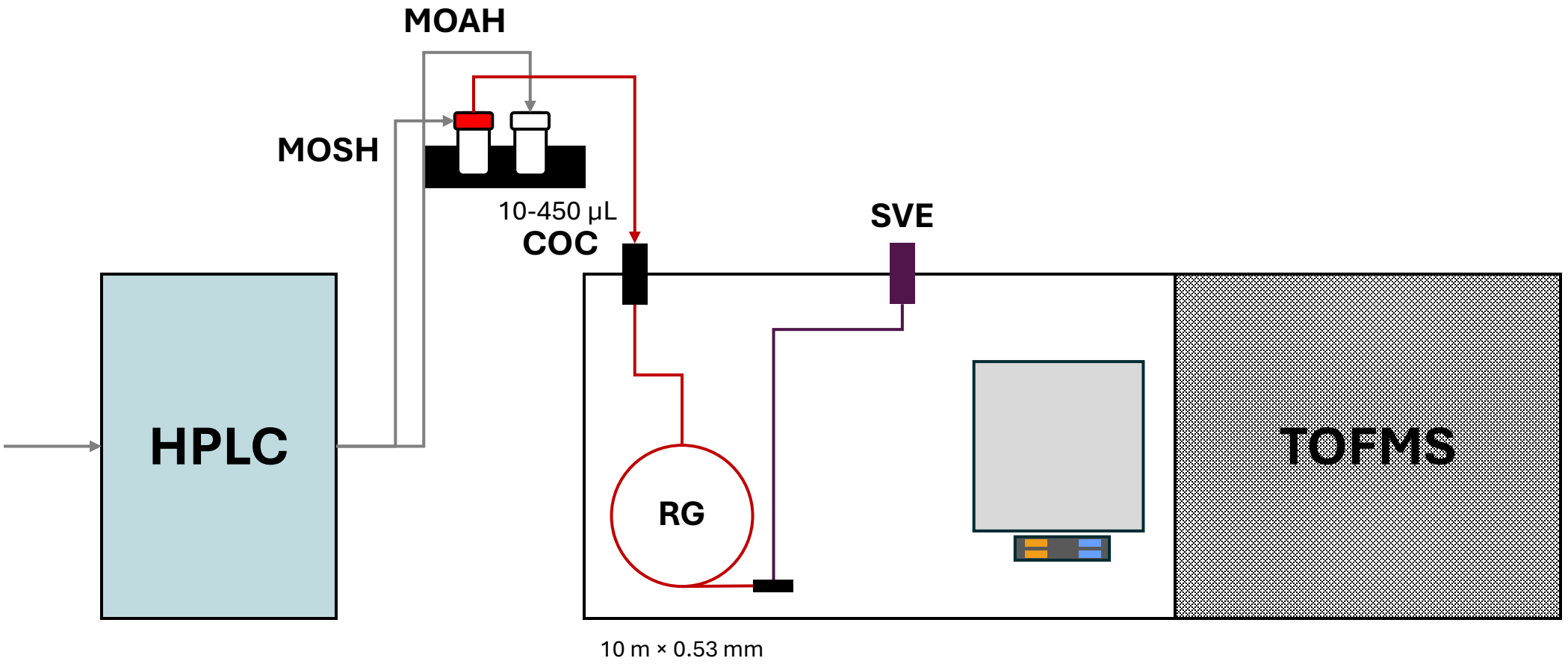

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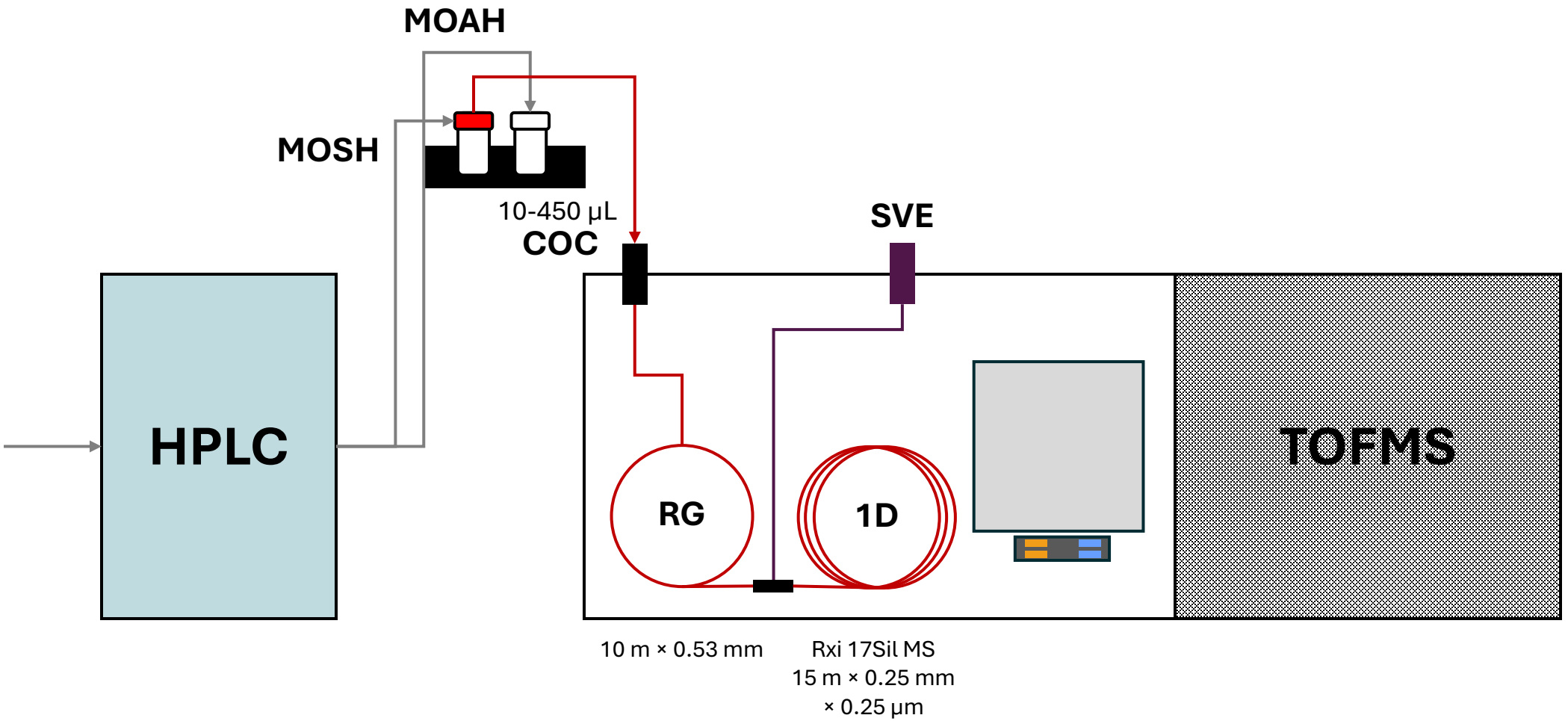

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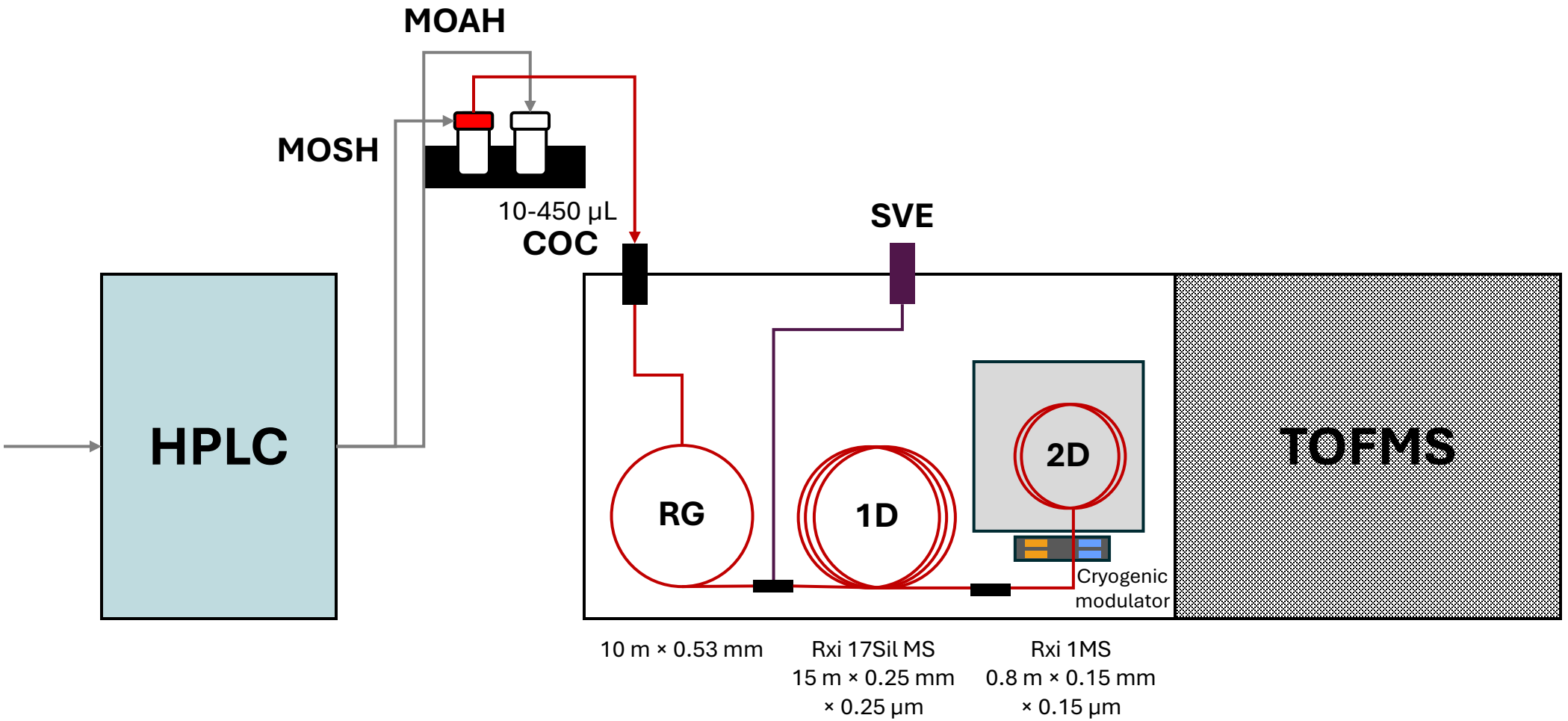

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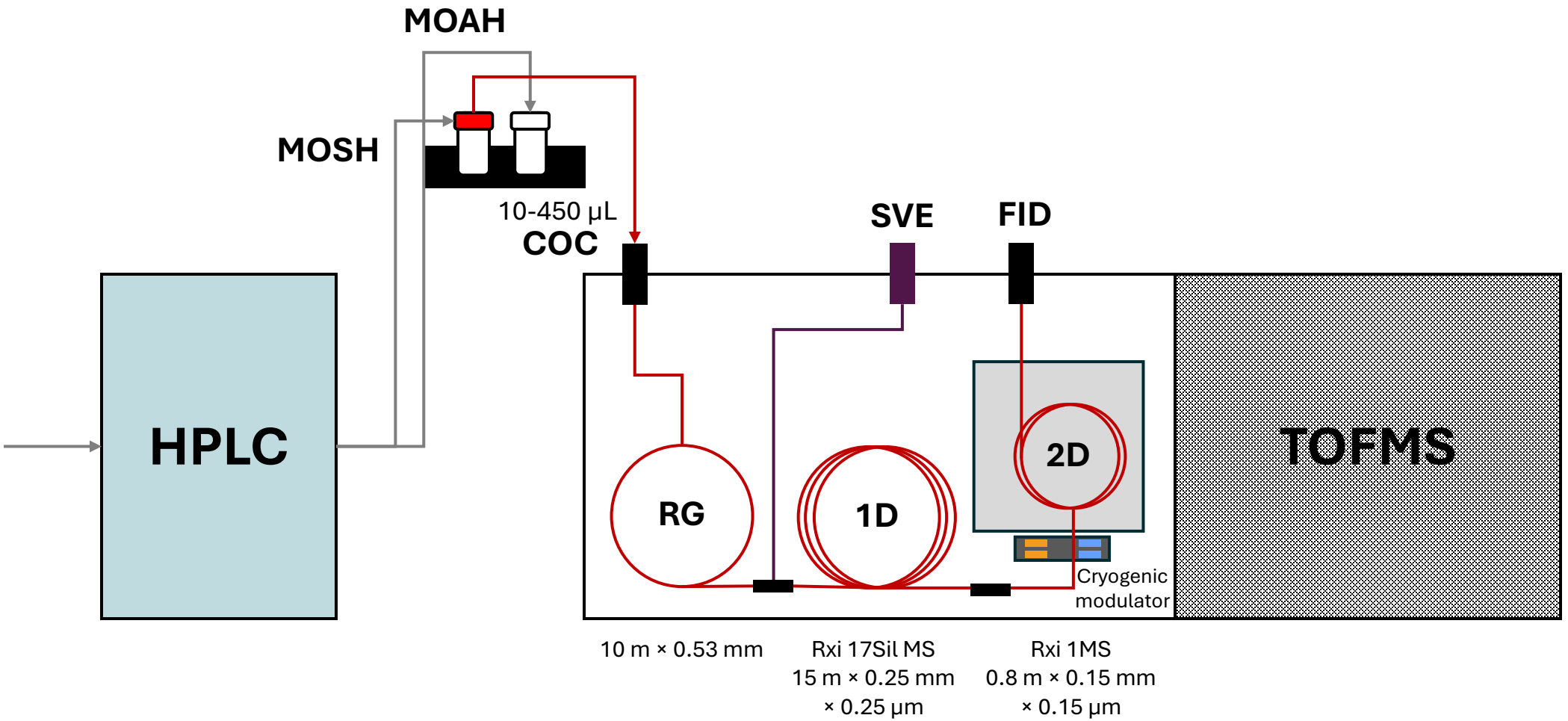

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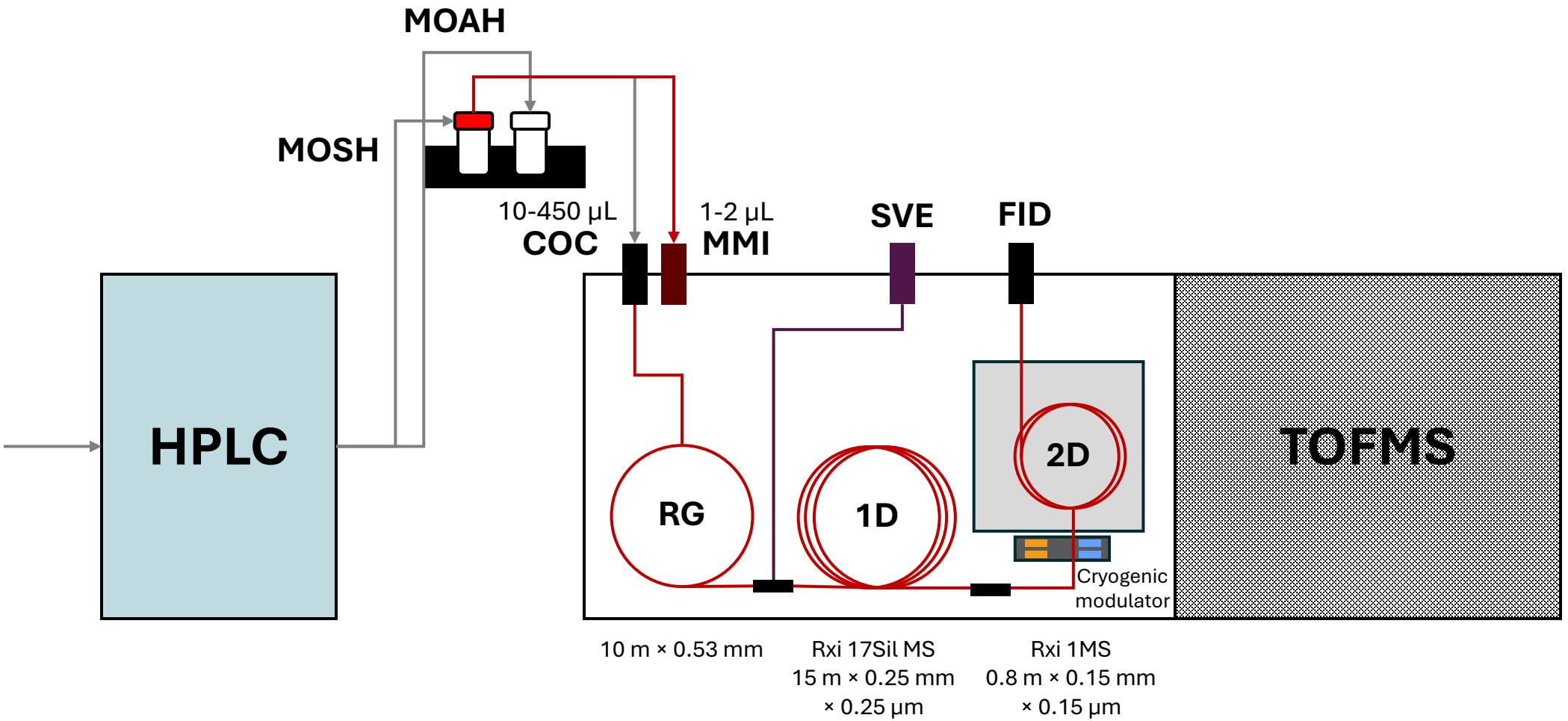

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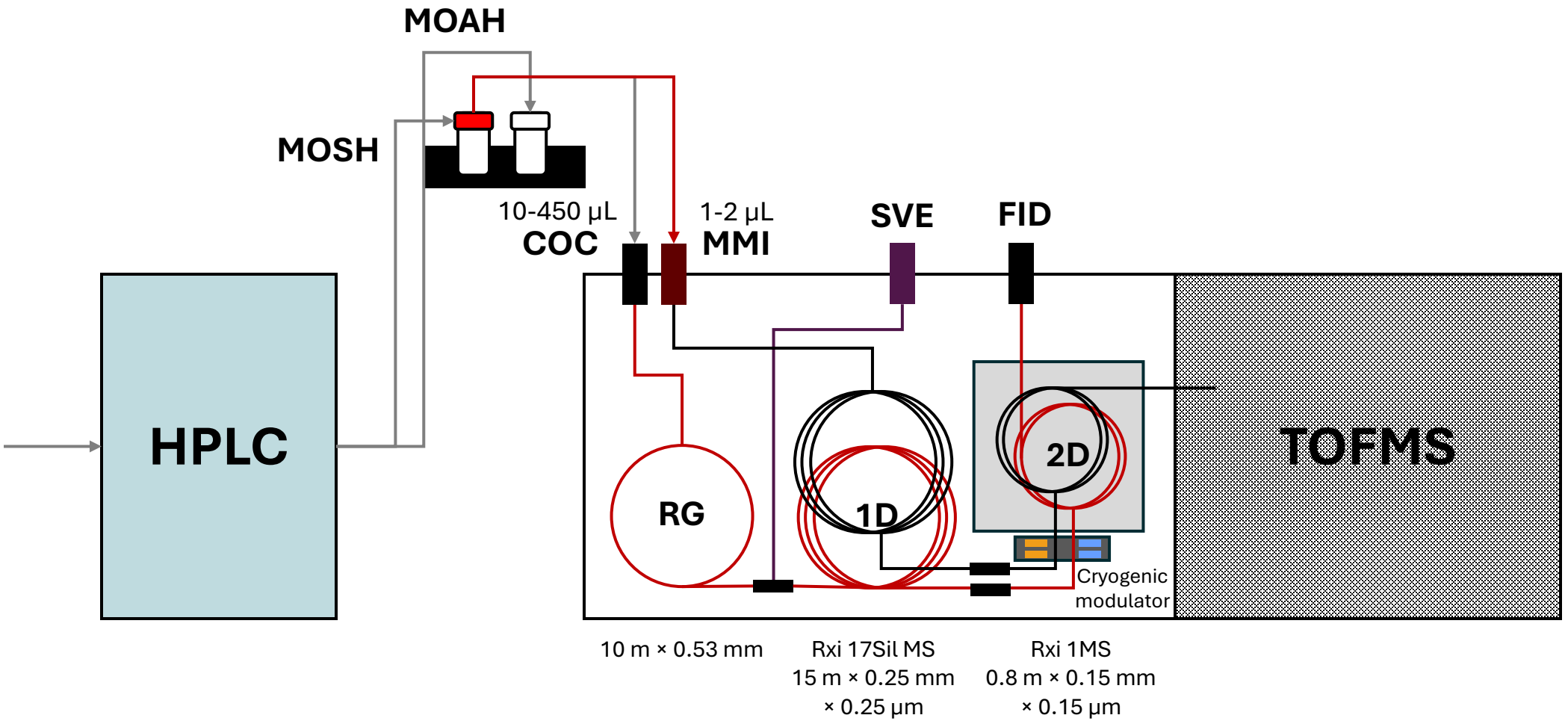

Avantages of HPLC-GC×GC-FID/TOFMS

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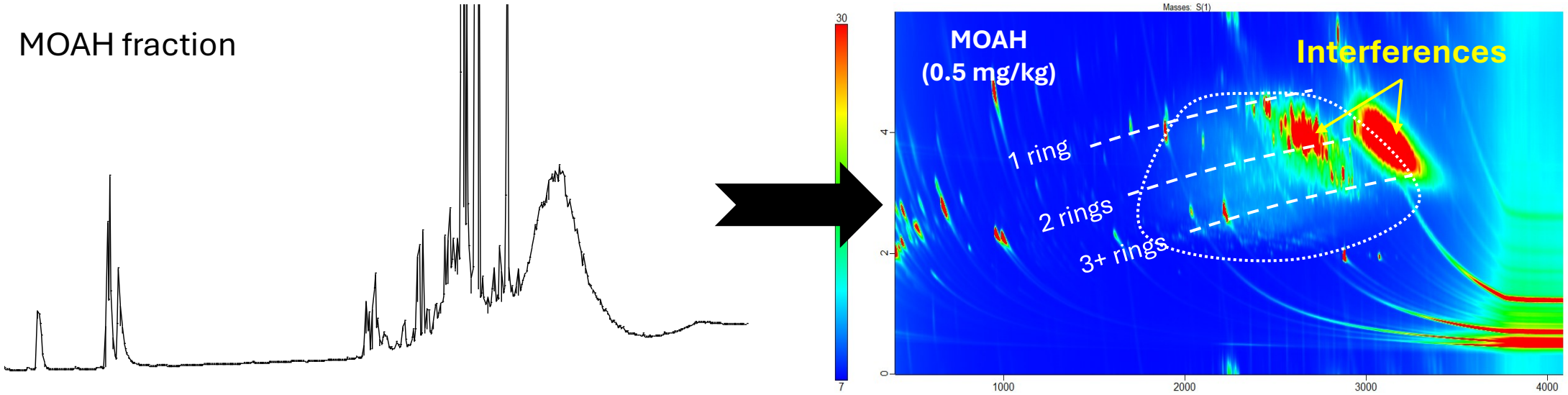
1 **Better structural characterization and easier interpretation of chromatograms**

Avantages of HPLC-GC×GC-FID/TOFMS

1 Better structural characterization and easier interpretation of chromatograms

a. Structured GC×GC chromatograms

Facilitate the determination of present MOSH/MOAH structures and the identification of interferences



*MOAH fraction of **E471** (emulsifier: mono- and diglycerides of fatty acids)*

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Avantages of HPLC-GC×GC-FID/TOFMS

1 Better structural characterization and easier interpretation of chromatograms

a. Structured GC×GC chromatograms

Facilitate the determination of present MOSH/MOAH structures and the identification of interferences

Better characterize the contamination (e.g. reporting the number of rings of MOAH) is **one of the recommendations** given by the EFSA in its Scientific Opinion on mineral oil in food

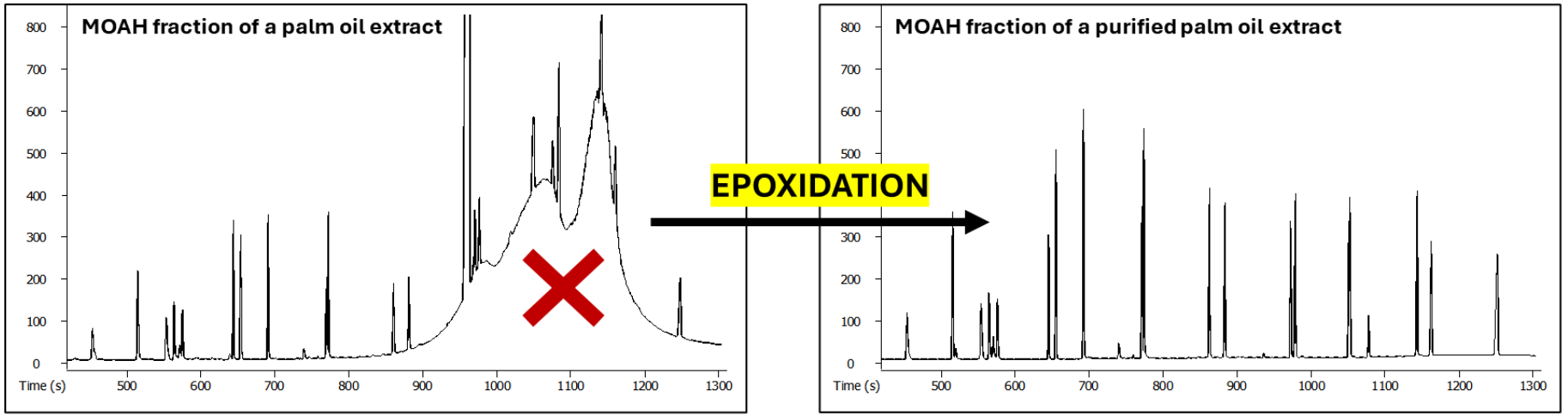
efsa
EUROPEAN FOOD SAFETY AUTHORITY

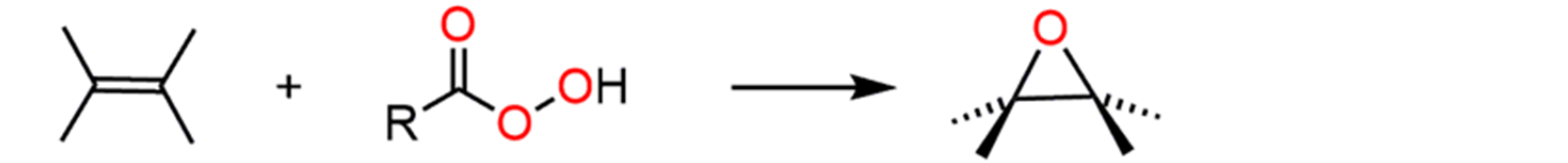
Avantages of HPLC-GC×GC-FID/TOFMS

1 Better structural characterization and easier interpretation of chromatograms

b. Removal of interferences

Usual purification of MOAH interferences





Double bond of the olefin + Peroxide → Formation of an epoxy bond ----> **Olefin becomes more polar**

Avantages of HPLC-GC×GC-FID/TOFMS

1 Better structural characterization and easier interpretation of chromatograms

b. Removal of interferences

As presented in Gorska *et al.* (2025), it is possible to remove interfering compounds coeluting with MOAH using the HPLC part, keeping the same column and eluents as for MOSH/MOAH fractionation

ELSEVIER

Journal of Chromatography A

Volume 1743, 22 February 2025, 465684

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

Aleksandra Gorska [a], Grégory Bauwens [a], Marco Beccaria [b], Giorgia Purcaro [a]

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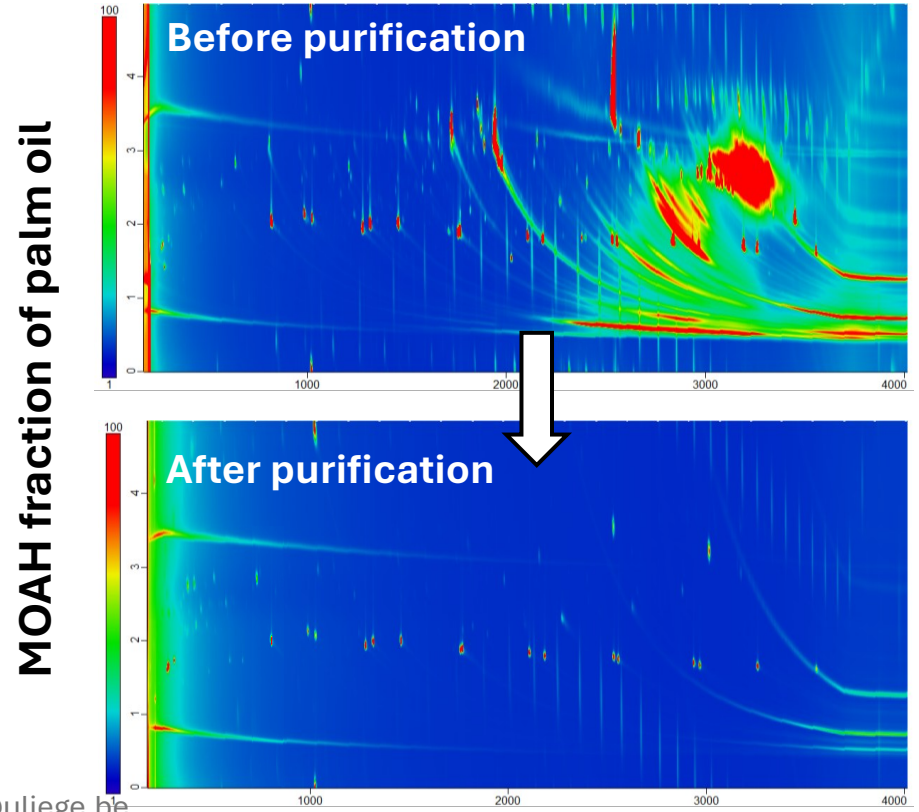
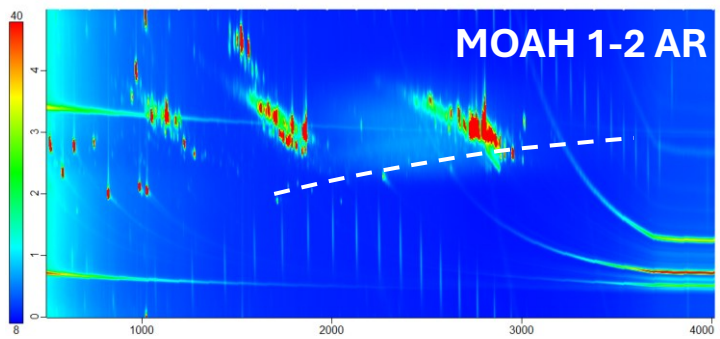


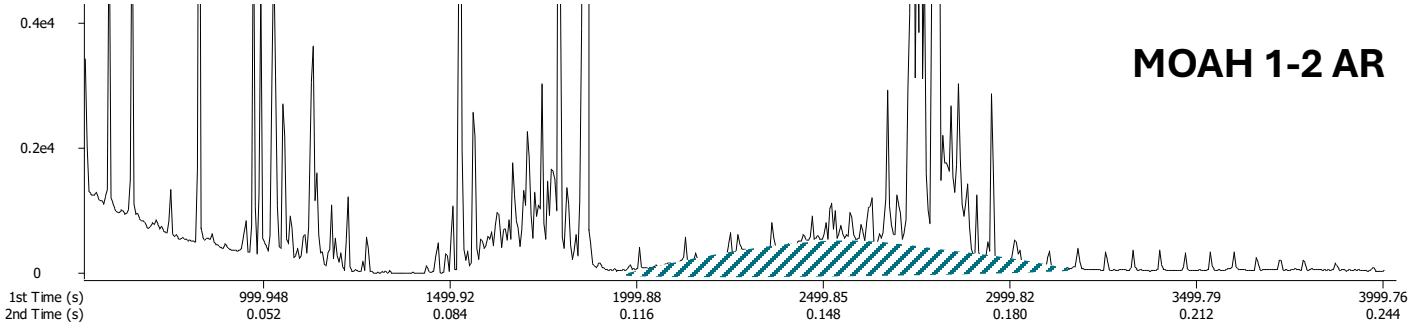
Avantages of HPLC-GC×GC-FID/TOFMS

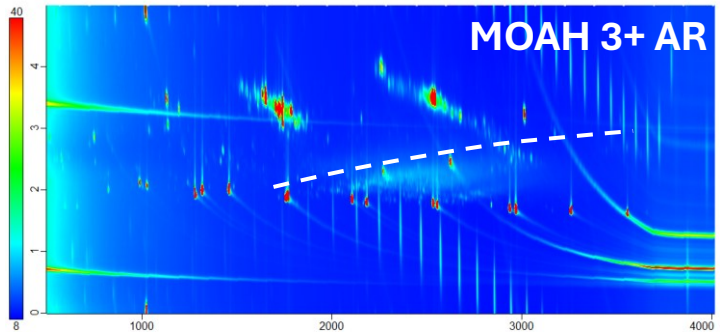
1 Better structural characterization and easier interpretation of chromatograms

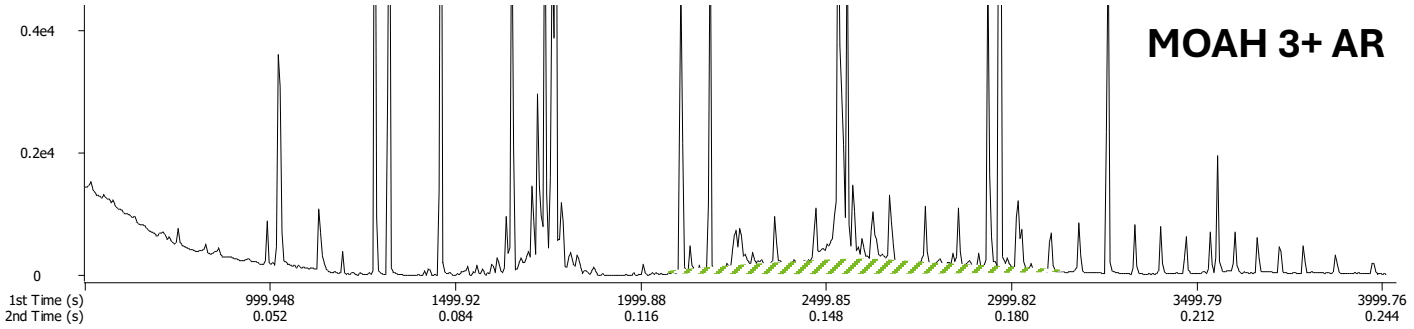
c. Separation of MOAH by number of aromatic rings by HPLC

Using the same HPLC gradient as used for purification, it is also possible to fractionate MOAH into a 1-2 and 3+ aromatic rings fractions before GC analysis









Possible determination of 3+ ring MOAH without GC×GC!

Avantages of HPLC-GC×GC-FID/TOFMS

2 Identification of markers helping to identify the source of contamination

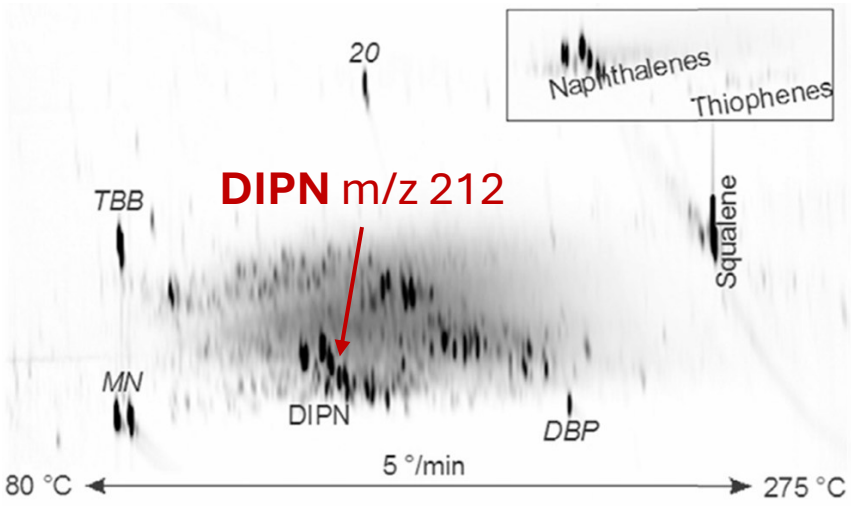
Visual (GC×GC pattern) and MS markers

The combination of the structured elution pattern with mass spectrometric detection allows for the identification of markers, supporting the identification of the origin of MOSH/MOAH contamination.

Table 1
Summary of the characteristics and their potential interpretation.

Characteristic	Indication
Repeat units of 1 C-atoms	MOSH
Pristane, phytane	MOSH
n-Alkyl cyclopentanes/hexanes	Mineral oil
Repeat units of 2 C-atoms	POSH from PE
Peak clusters clearly above n-alkanes	POSH from PP
MOSH and MOAH of same volatility range	Single contaminant
Diisopropyl naphthalenes (DIPN)	Recycled paperboard
Dibenzothiophenes	Little refined oil
Percentage of MOAH	Degree of raffination
Clearly separated band in MOAH	No hydrogenation
Perhydro pyrenes	Hydrogenated oil
"Gray cloud", slanted bands of naphthenes	Hydrogenated oil
Upper limit of mass range	Migration conditions

Example: DIPN indicates for migration of MOH from recycled paperboard (food packaging)



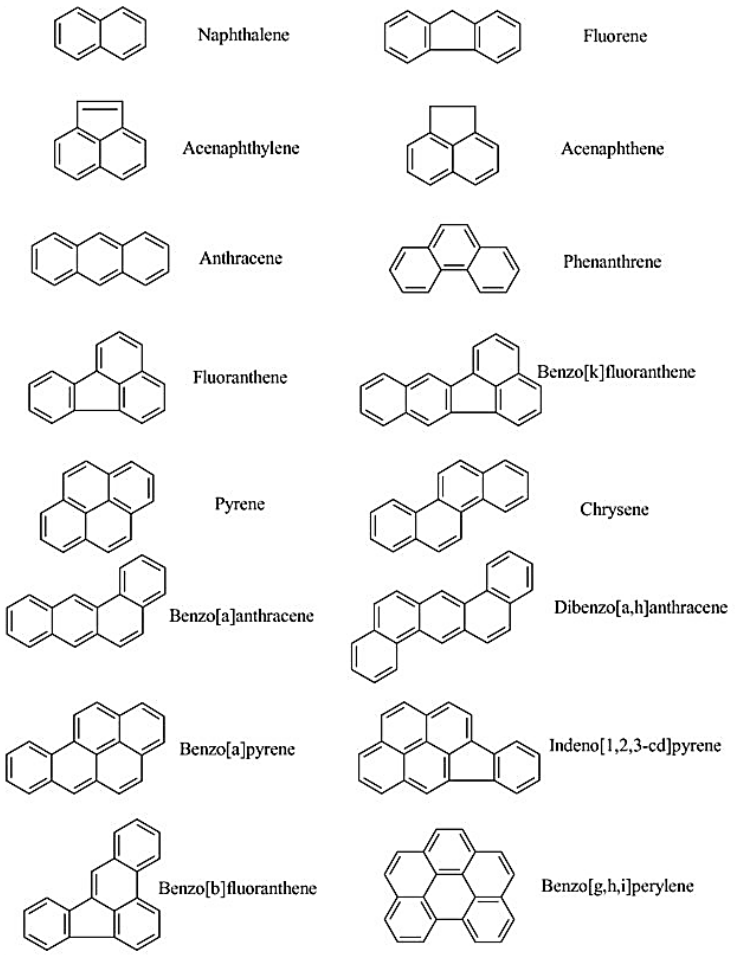
M. Biedermann, K. Grob / J. Chromatogr. A 1375 (2015) 146–153

NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Context



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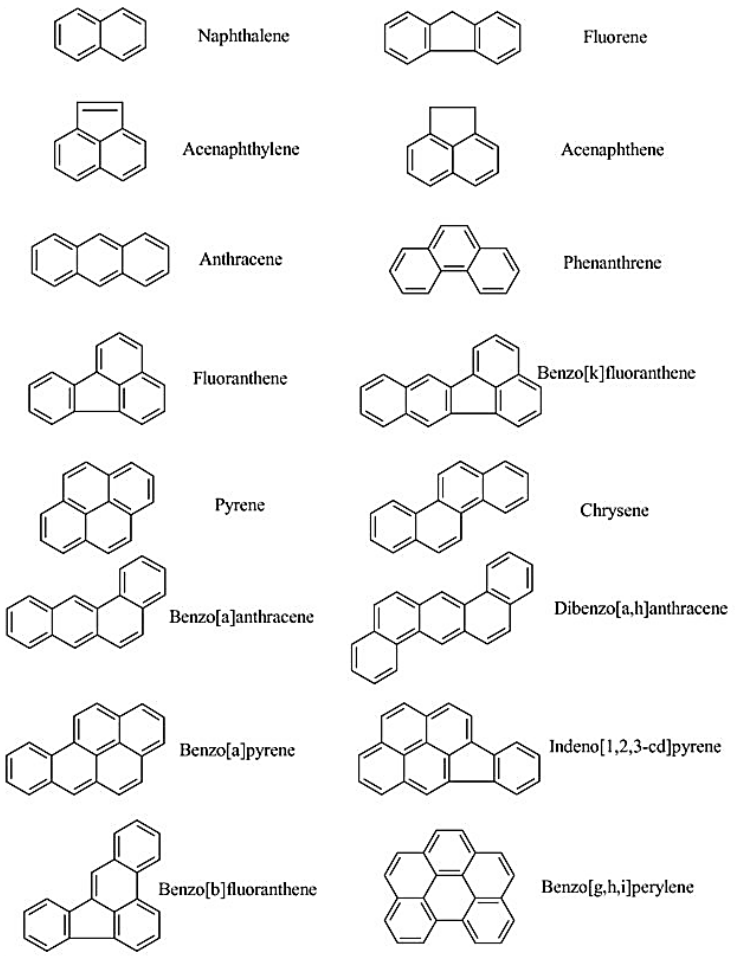

Polycyclic aromatic hydrocarbons (PAHs) are carcinogenic compounds that are formed during incomplete combustion of organic material.

They are **structurally similar to MOAH** (MOAH are the alkylated versions of PAHs), yet they have very **different analytical requirements**.

NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Different analytical requirements

Analytical requirements for MOAH

Table 4. Performance requirements for total MOSH and total MOAH analysis: maximum analytical LOQ (max LOQ) of the method, acceptable ranges for recovery (R_{rec}) of mineral oil from samples, and relative standard intermediate precision (RSD_{ip})

Categories	Associated foods (#)	Max LOQ [mg/kg]	R_{rec} range [%]	RSD_{ip} [%]
Dry, low-fat content (< 4% fat/oil)	bread and rolls; breakfast cereals; grains for human consumption; pasta, products derived from cereals	0.50	80 - 110	15
Higher fat/oil content (4% - 50% fat/oil)	fine bakery ware; confectionery (incl. chocolate) and cocoa; fish meat, fish products (canned fish); oilseeds; pulses; sausages; tree nuts	1.0	80* - 110	20

LOQ
0.5-1.0 mg/kg

Analytical requirements for PAHs

Table 7

Parameter	Criterion
Applicability	Foods specified in ►M7 ↓ Regulation (EU) 2023/915 ◄
Specificity	Free from matrix or spectral interferences, verification of positive detection
Repeatability (RSD_r)	$HORRAT_r$ less than 2
Reproducibility (RSD_R)	$HORRAT_R$ less than 2
Recovery	50-120 %
LOD	≤ 0,30 µg/kg for each of the four substances
LOQ	≤ 0,90 µg/kg for each of the four substances

LOQ
0.9 µg/kg

NEW

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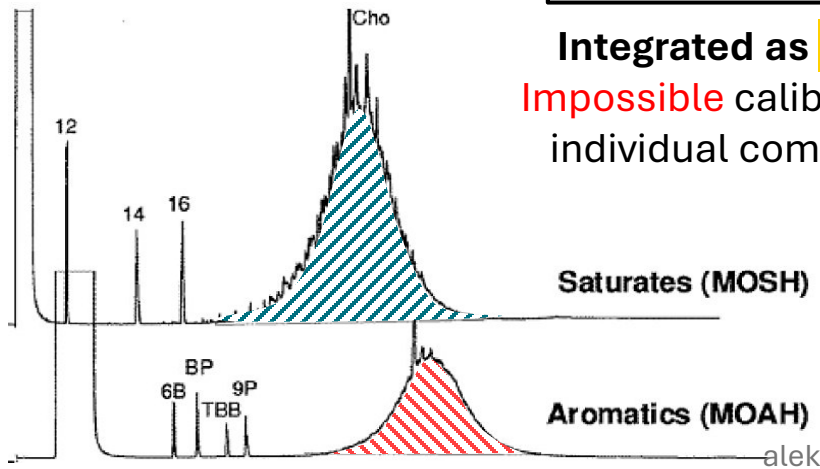
LOQ
0.5-1.0 mg/kg



Integrated as humps
Impossible calibration for individual compounds

Analytical requirements for PAHs

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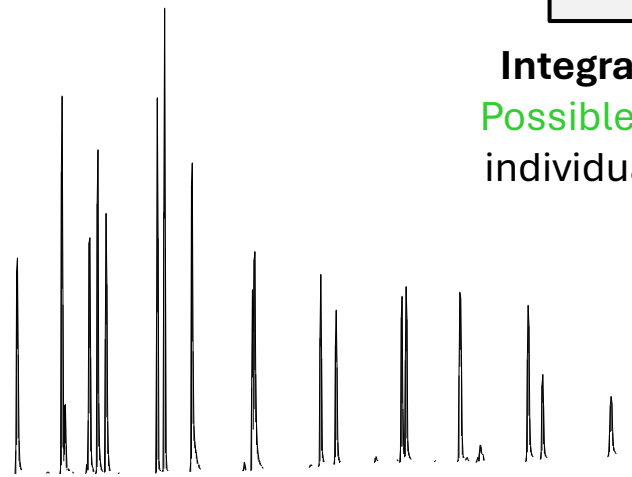
Reproducibility (RSD_R)	$HORRAT_R$ less than 2
Recovery	50-120 %
LOD	≤ 0,30 µg/kg for each of the four substances
LOQ	≤ 0,90 µg/kg for each of the four substances

LOQ
0.9 µg/kg



Integrated as peaks
Possible calibration for individual compounds

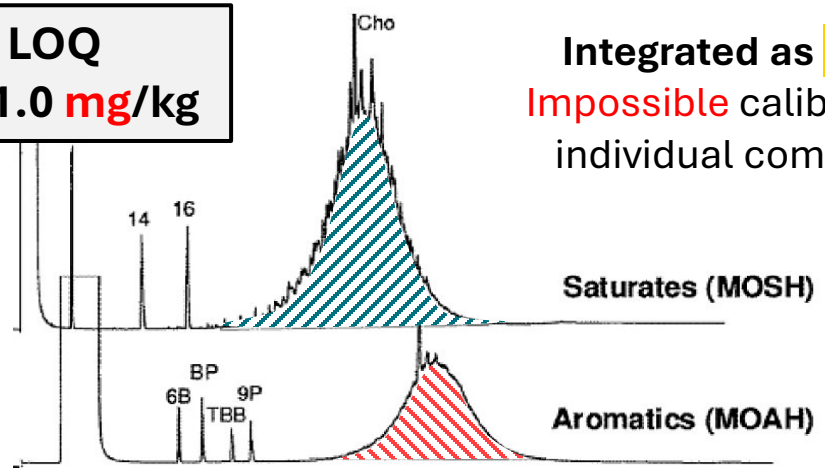
NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Different analytical requirements

LOQ
0.5-1.0 mg/kg



Integrated as humps
Impossible calibration for individual compounds

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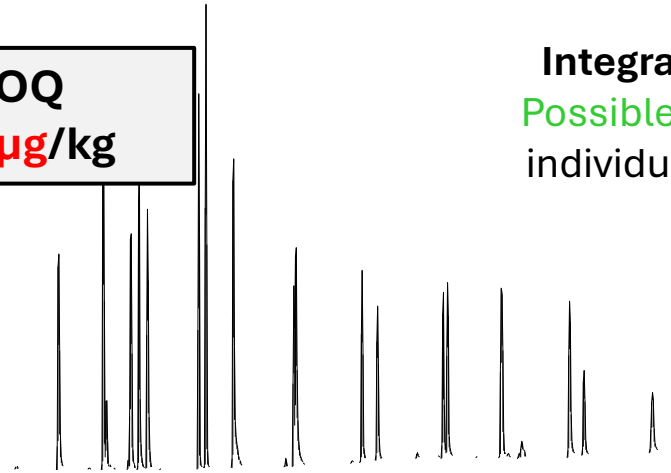
0.9 µg/kg



Integrated as peaks
Possible calibration for individual compounds

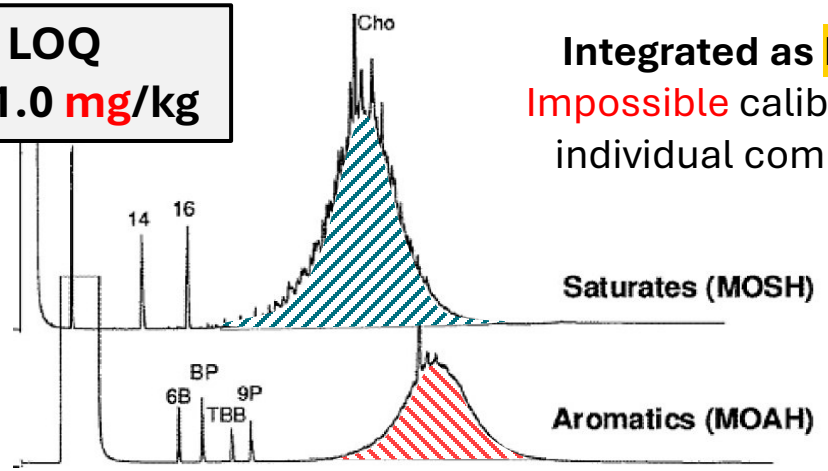
NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Different analytical requirements

LOQ
0.5-1.0 mg/kg



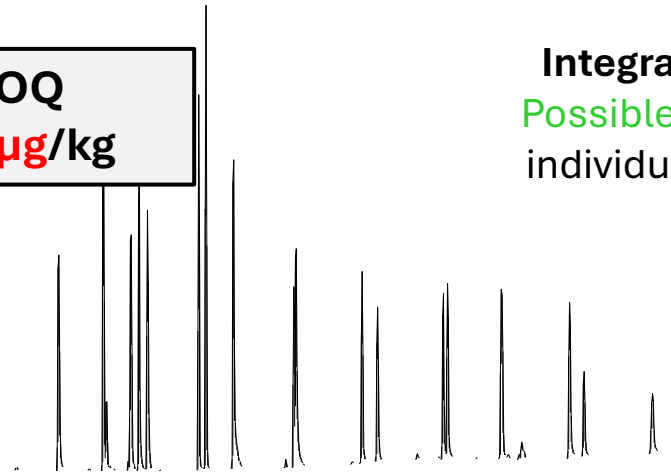
Integrated as humps
Impossible calibration for individual compounds

Need for a detector that gives a **similar response** for all hydrocarbons

→ **FID** does the job

Not the case of MS, where the response factor varies depending on the compound!

LOQ
0.9 µg/kg



Integrated as peaks
Possible calibration for individual compounds

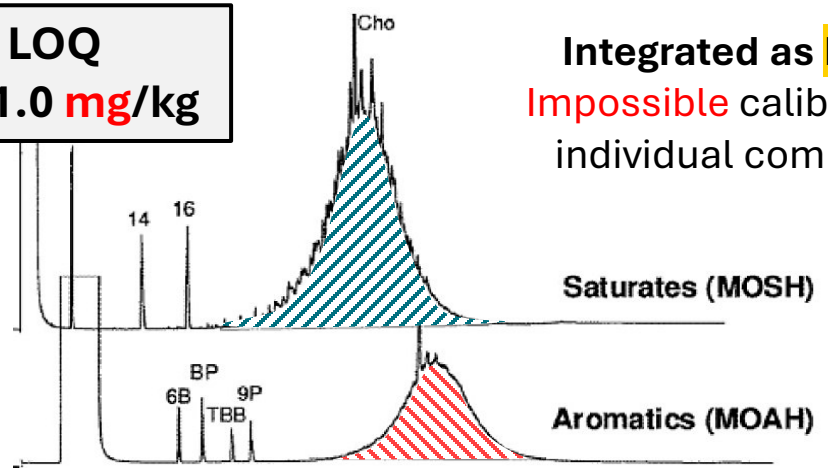
NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Different analytical requirements

LOQ
0.5-1.0 mg/kg



Integrated as humps
Impossible calibration for individual compounds

Need for a detector that gives a **similar response** for all hydrocarbons

→ **FID** does the job

Not the case of MS, where the response factor varies depending on the compound!

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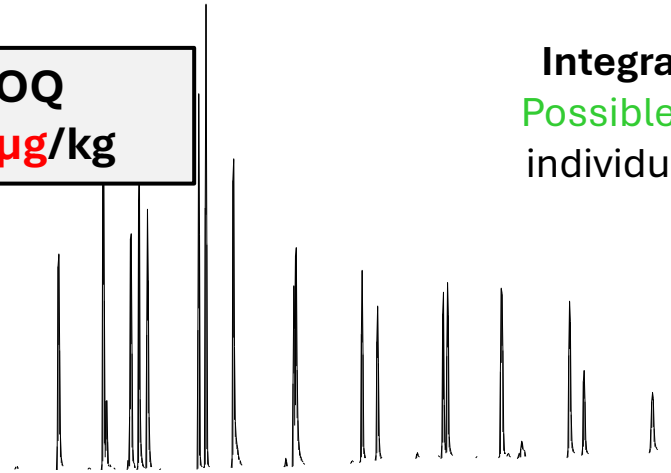
0.9 µg/kg



Integrated as peaks
Possible calibration for individual compounds

Need for a detector that is **selective** enough and that **reaches the LOQ**

→ **MS** is adapted

FID is not sensitive nor specific enough

NEW

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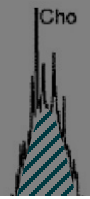

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The HPLC/GC×GC-FID/TOFMS system can handle both analyses at once

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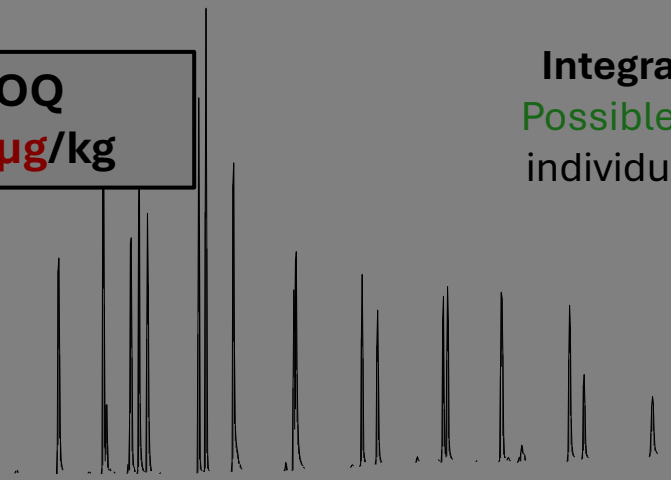

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NEW

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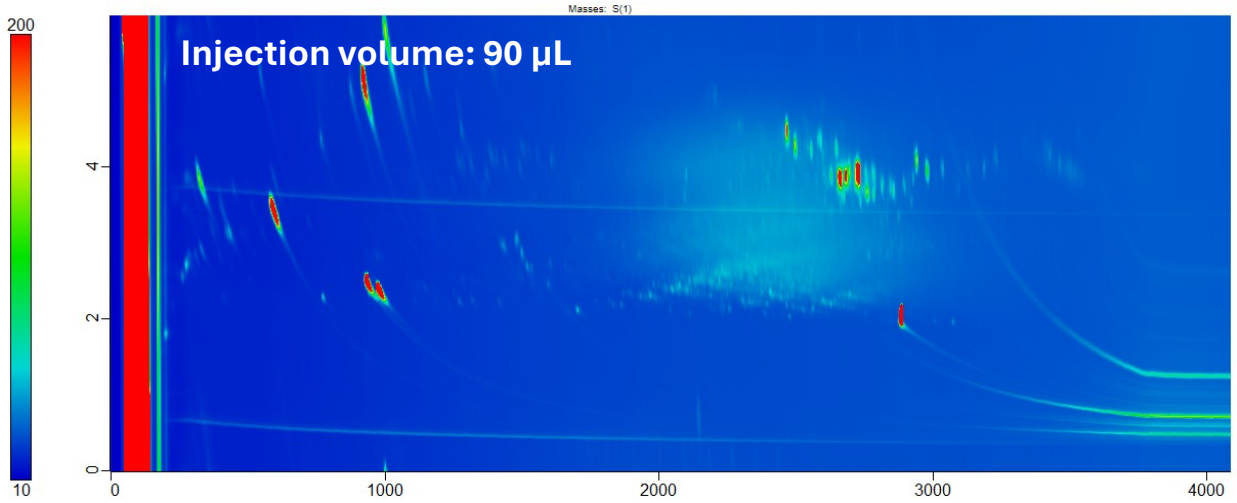
Example of MOAH and PAHs analysis in soya lecithin

HPLC/GC×GC-FID



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

NEW

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Example of MOAH and PAHs analysis in soya lecithin

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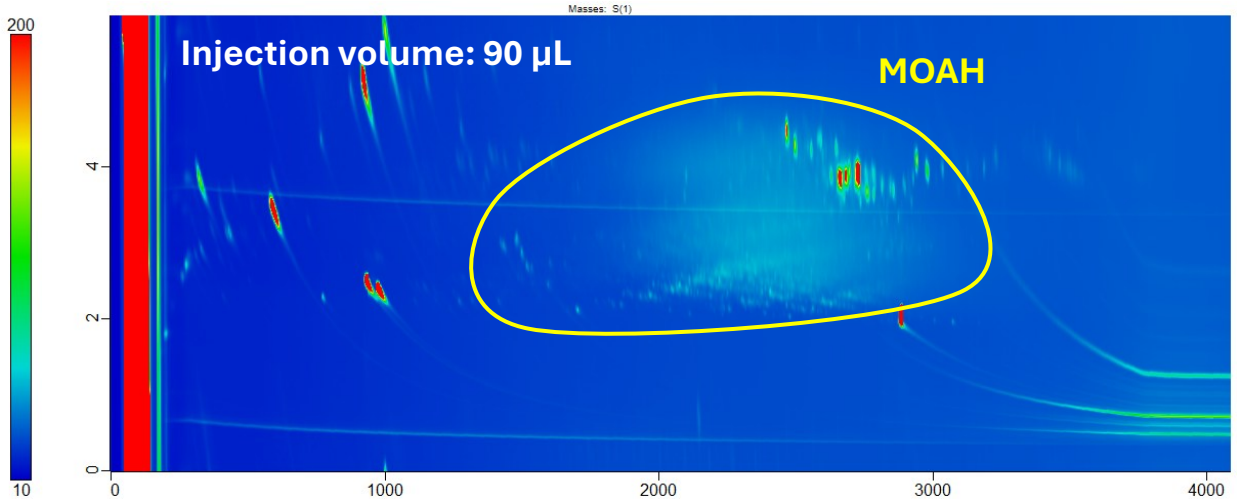

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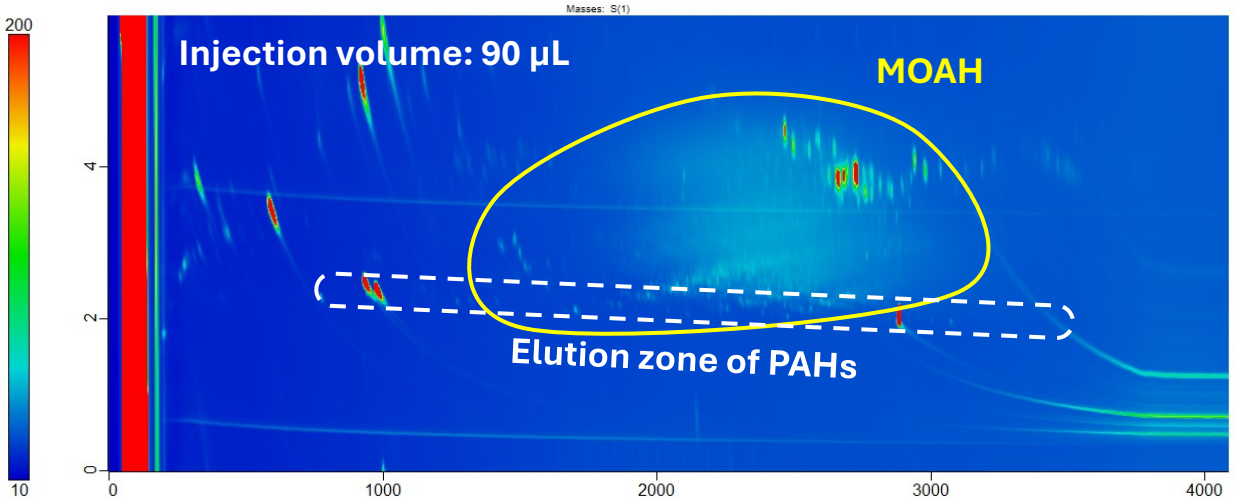

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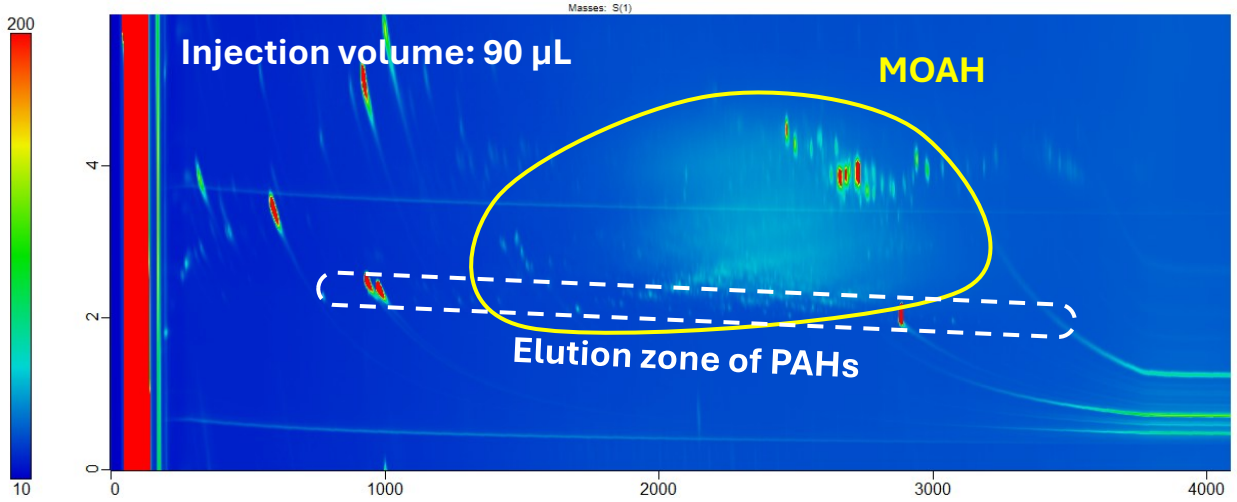
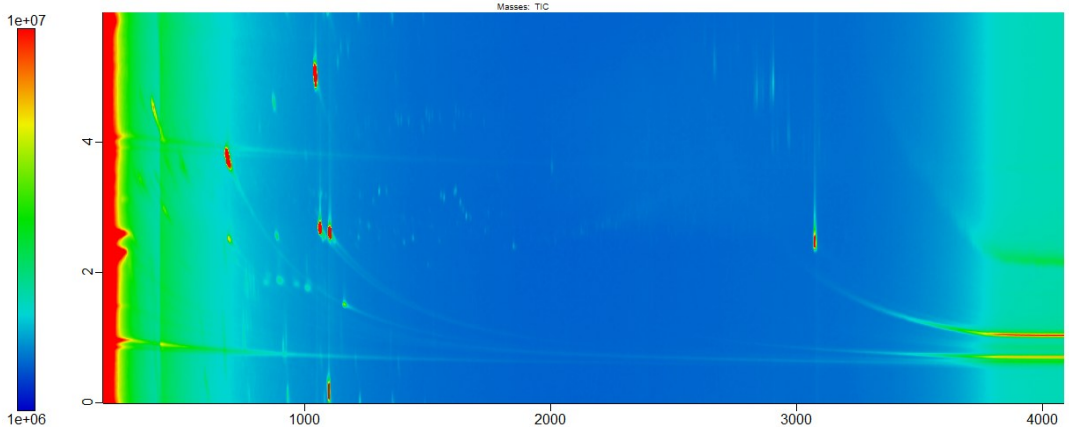


HPLC/GC×GC-TOFMS – TIC



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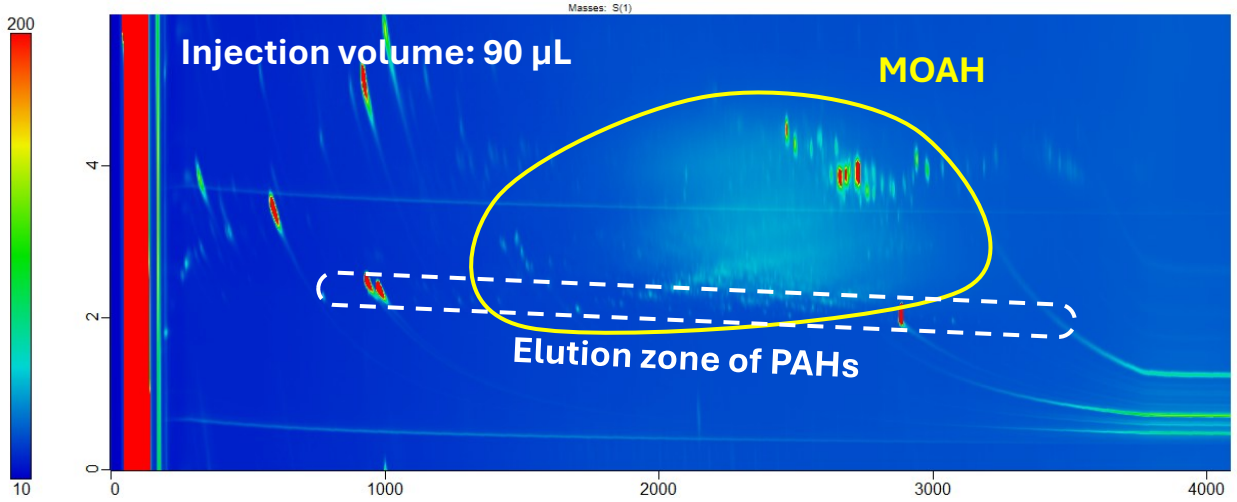

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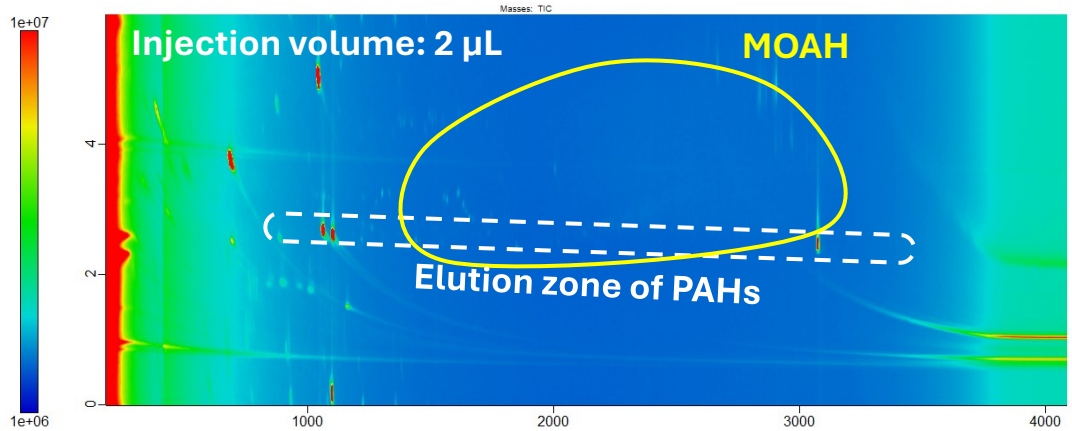

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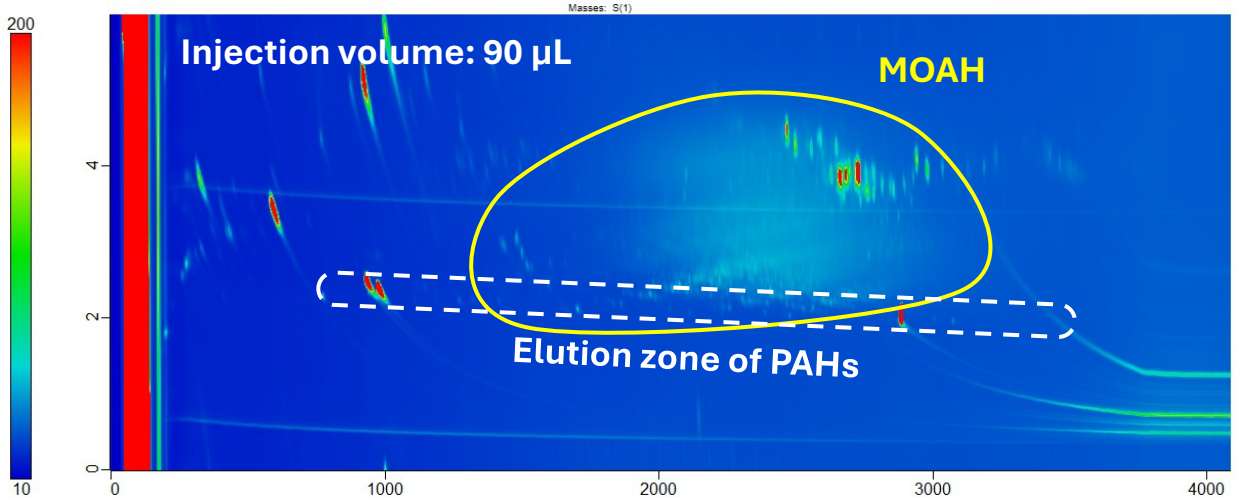

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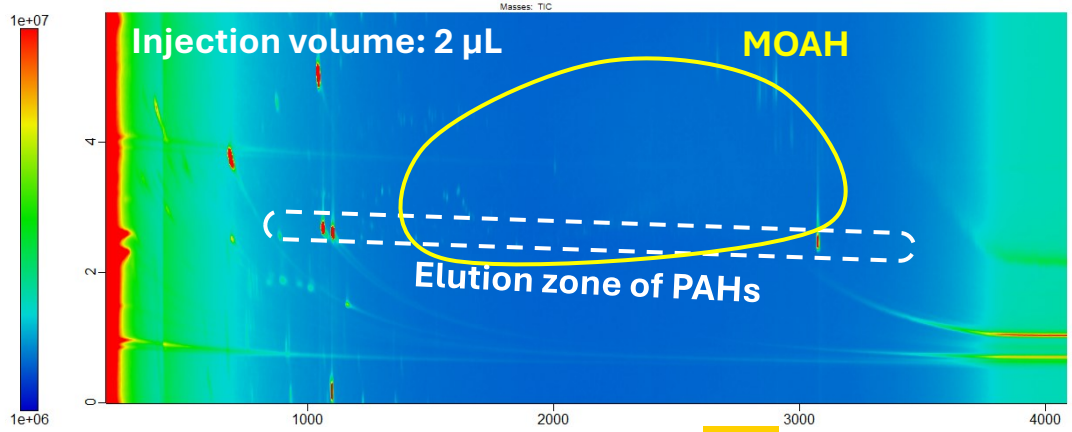
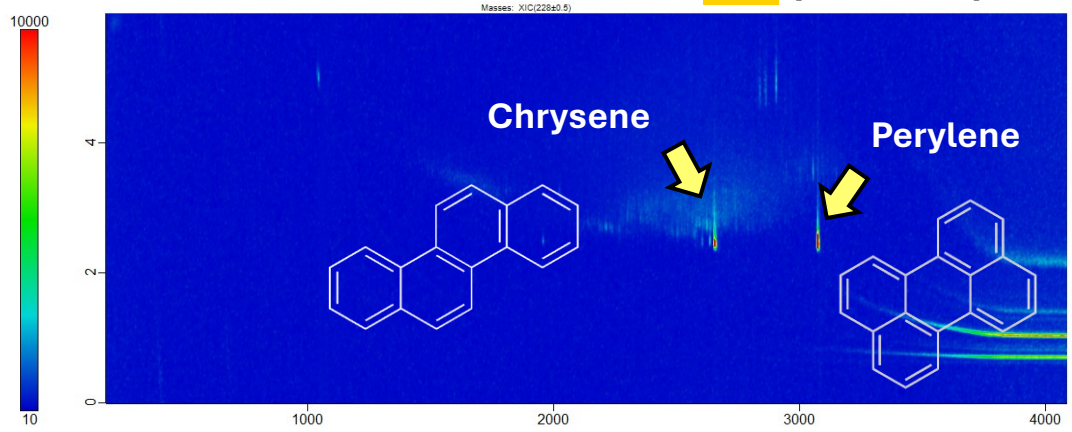


HPLC/GC×GC-TOFMS – EIC (m/z 228)



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In MS, we see the PAHs using EIC

aleksandra.gorska@uliege.be

NEW

Avantages of HPLC-GC×GC-FID/TOFMS

3 Simultaneous analysis of MOAH and PAHs

Example of MOAH and PAHs analysis in soya lecithin

PAHs determination possible because of the substitution of the usual purification method (epoxidation) by the LC purification

Food sample

↓

Extraction

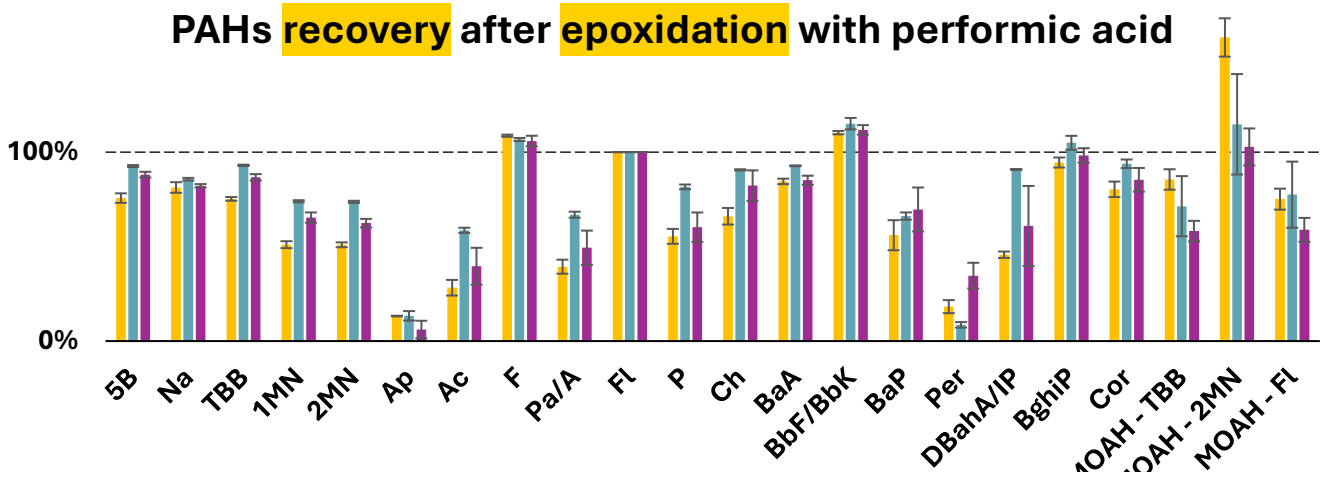
↓

Purification

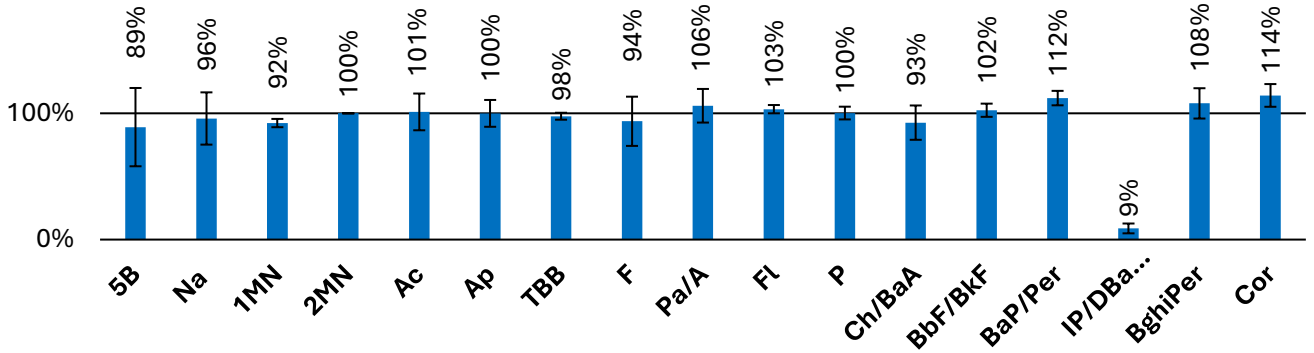
↓

HPLC/GC×GC-FID/TOFMS

PAHs recovery after epoxidation with performic acid



PAHs recovery after LC purification



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These points would not have been reached with the routine HPLC-GC-FID method.

More complex is not always better, but in this case, it is!

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Thank you for your attention

And many thanks to the Analytical Chemistry team of Gembloux Agro-Bio Tech

Prof. Giorgia Purcaro
Sophie Vancraenenbroeck
Damien Eggermont
Donatella Ferrara
Paula Albendea
Damien Pierret
Carlo Bellinghieri
Nicola Ruin

RT 24/05 IMPOFAD – Impurities in oil- or fat-derived food additives and compound foods