



Mineral Oil Hydrocarbon Contamination in Oils - Experiences of Eurofins Hamburg -

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Analytical method Customer project with Greek extra virgin olive oils Data (extract) of 2018 and 2019



Common methods in the market for quantification:

- Online "swiss" Method (developed since 1989)
 → Extraction, clean-up, LC-GC-FID
- Offline "BfR" Method (2012)
 → Extraction, clean-up, column fractionation, GC-FID
- Online "ISO" Method (ISO 17780:2015)
 → Oils and fats, MOSH, 50 mg/kg to 1000 mg/kg
 → Extraction, LC-GC-FID
- Online "CEN" Method (DIN EN 16995:2017-08)
 - \rightarrow mineral oil in veg. oils and foodstuffs on basis of veg. oils
 - → Extraction, clean-up, LC-GC-FID
 - → Eurofins method (modified)



Qualitative methods: Confirmation by Mass Spectrometer



 online LC-GC-MS for identification of specific masses (source, kind of mineral oil)

LC-GCxGC-MS 2-

dimensional separation of MOSH, POSH and MOAH (e.g. > 3 rings), plus identification of specific masses



General scheme of the method





Sample preparation

Extraction

n-Hexan

 \rightarrow for fats & oils

Clean up

Silica gel (for MOSH/MOAH) Aluminumoxid (for MOSH)

Epoxidation (for MOAH)

- \rightarrow removing of simple fats
- → removing the natural long chain (> C20) n-Alkanes (lower values!)
- → partially removing of the natural Olefins like Squalene (Triterpens) / Vitamin E / Carotinoides
- Currently working on introduction of saponification: samples with Lecithine, theoretically lower LOQ











Epoxidation e.g. Squalene (triterpene)







Internal standards for verification of the correct LC separation





1. complete hump area of the unresolved components



3. calculation of the concentration using the internal standards



Eurofins WEJ Contaminants (Hamburg) validated LOQs (LC-GC-FID)

Matrix	MOSH/POSH MOAH	
Oils & fats	1 mg/kg	1 mg/kg

Matrixes interferences (e.g. hydrocarbons from the plant matrix – triterpenes, etc.) may increase the LOQ

Project Olive Oil from Crete vs. Greece



Customer project with Greek extra virgin olive oils

Project Olive Oil from Crete vs. Greece (Results)



	AREAS OF CRETE		AREAS OUTSIDE CRETE		
	CHANIA	HERAKLION	AREA 1	AREA 2	AREA 3
MOSH/POSH (C10 - C16)	ND	ND	ND	ND	ND
MOSH/POSH (C17 - C24)	1,3 mg/kg	1,1 mg/kg	2,2 mg/kg	3,6 mg/kg	5,2 mg/kg
MOSH/POSH (C25 – C35)	3,5 mg/kg	3,4 mg/kg	11,2 mg/kg	13,6 mg/kg	23,0 mg/kg
MOAH	ND	ND	1,5 mg/kg	2,3 mg/kg	4,4 mg/kg



- There is a very serious olive oil cross-contamination "problem" with MOAH and MOSH/POSH.
- Serman retailers are even forced to recall products if the cross-contamination levels are above acceptable limits.
 (MOSH/POSH < 4 -10 mg/kg and MOAH non-detectable)
- Significant higher values in olive oils coming from areas outside of Crete
- Lubricants based on mineral oils used on chainsaws (used for tree cutting by farmers outside Crete) and olive fruit harvesting machinery come in direct contact with the olive fruit.





Data of 2018 and 2019 of selected oils



• Oils & fats in 2018 and 2019: ca. 8500 samples

 Extra virgin olive oils: ca. 1400 samples MOAH (C10 – C62): < 1 mg/kg (1000 samples), 1 mg/kg (200 samples) – 62 mg/kg
 MOSH/POSH (C10 – C62): < 1 mg/kg (62 samples), 1 – 4 mg/kg (850 samples), > 4 – 10 mg/kg (250 samples) – 550 mg/kg



Oils & fats in 2018 and 2019: ca. 8500 samples

 Coconut oil: ca. 545 samples
 MOAH (C10 – C62): < 1 mg/kg (327 samples), 1 – 1,5 mg/kg (31 samples) – 90 mg/kg
 MOSH/POSH (C10 – C62): < 1 mg/kg (50 samples), 1 – 4 mg/kg (122 samples), > 4 – 10 mg/kg (146 samples) – 180 mg/kg



Oils & fats in 2018 and 2019: ca. 8500 samples

• Avocado oil: ca. 55 samples

MOAH (C10 – C62): < 1 mg/kg (41 samples) -61 mg/kg MOSH/POSH (C10 – C62): < 1 mg/kg (7 samples), 1 – 4 mg/kg (9 samples), > 4 – 10 mg/kg (26 samples) – 150 mg/kg



Thank you for your kind attention!

