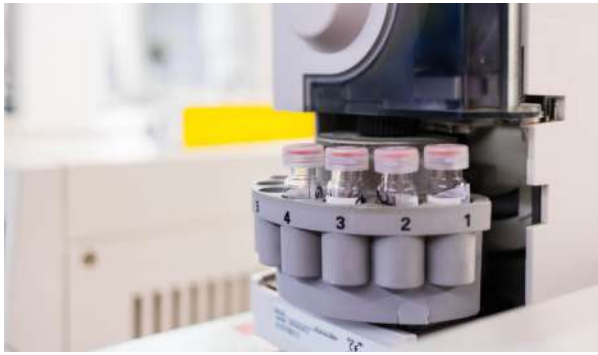
chemiservice
LABORATORIO DI ANALISI CHIMICHE E MICROBIOLOGICHE

MOSH AND MOAH: VALIDATION OF THE ANALYTICAL METHOD AND OCCURRENCE ON OILS

ARIANNA LUISI

Bologna, 12th December 2019

About us



- **Chemiservice srl** is a private and independent laboratory, specialized in analytical services of chemistry, microbiology and biochemistry for food, environmental, industrial and cosmetic sectors.
- Thanks to the experience acquired in 30 years and the consolidated business administration, Chemiservice can offer many services in support of both agricultural and industrial small and large companies.

The laboratory is expert on chemical and product analyses of oils and fats for human consumption, raw materials and by-products.
Chemical analysis of fatty matrices represents the core business of Chemiservice laboratory. In this the sector we reached the most significant expertise and experience for both years of activity and particular interest devoted to this subject.
More specifically, virgin and refined olive oils, vegetable seed oils (both for human consumption and different uses) represent the main activity object.
The services offered by the laboratory for this kind of matrices are inspired by many targets. If possible, the laboratory applies recognized official methods or validated and accredited internal methods. Almost all analyses are accredited.
Chemiservice srl is accredited by ACCREDIA (since 1999) as analytical laboratory operating in compliance with UNI CEI EN ISO/IEC 17025.

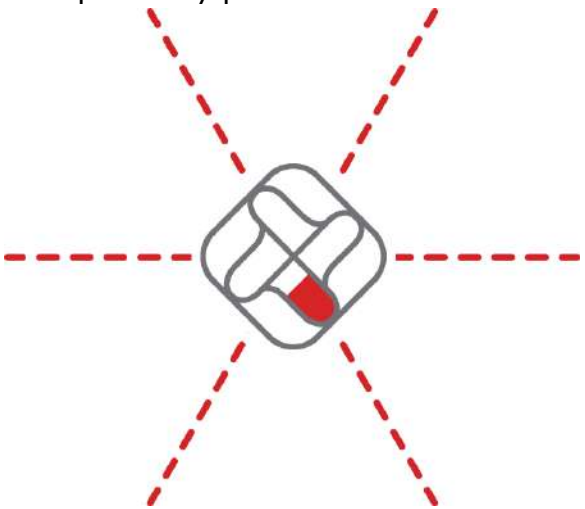
Services

ANALYSIS ON OLIVE OILS, VEGETABLE OILS AND FATS

Chemical and classification analyses on oils and fats for human consuption, raw materials and process by-products

ANALYSIS OF FOOD CONTAMINANTS

Pesticides (multiresidual analysis), PAH, Heavy metals, Phthalates, Mineral oils, MOSH, MOAH, Acrylamide



FOOD ANALYSIS

Chemical, biochemical and physical analysis on matrices for human and animal consumption, raw materials, semifinished and finished products.

Analysis for the detection of GMO and allergens

MICROBIOLOGICAL AND BIOCHEMICAL ANALYSIS

Detection of bacterial/fungal contaminants and technical support on products for human and animal consuption, water (drinkable, waste, process, swimming and thermal), surfaces of production environment, cosmetic products

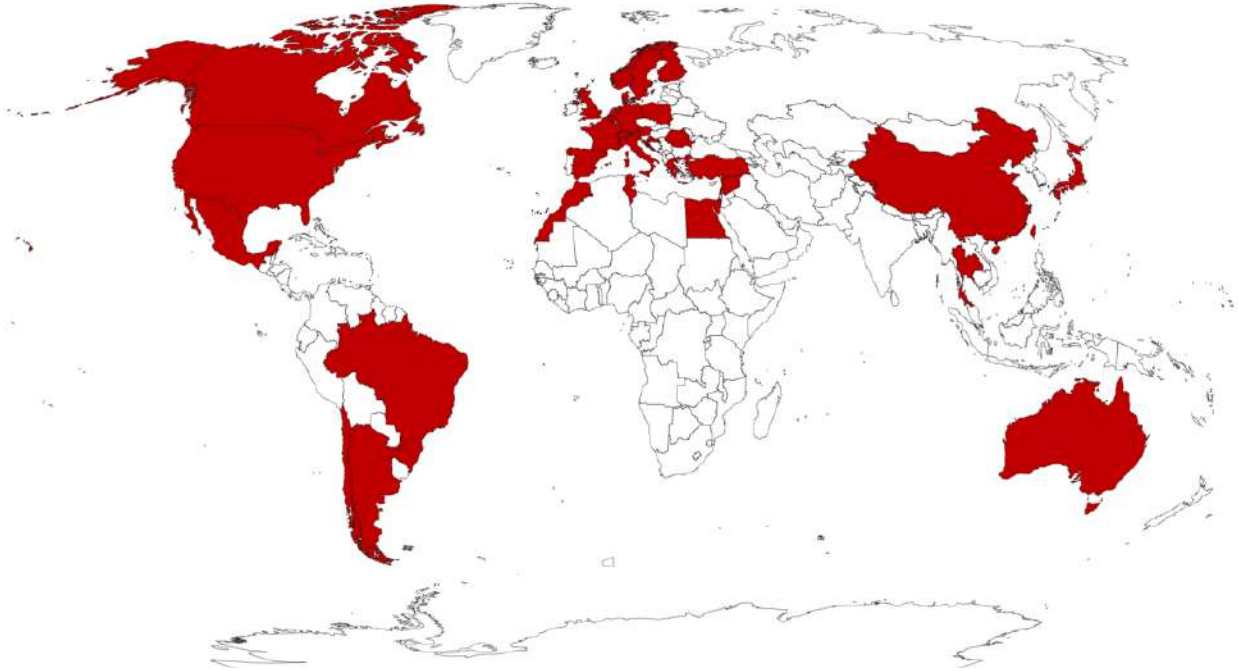
TECHNICAL-LEGAL ADVICE AND CONSULTANCY SERVICES FOR QUALITY

CHEMICAL ANALYSIS ON NO-FOOD PRODUCTS

Chemical and physical analysis on environmental matrices (air, soil, water), wastes and muds, construction materials and cosmetic products. The team, made of specialized technicians and professionals, is involved in both laboratory analysis and sample collection.

Chemiservice in the word

- Numbers of Chemiservice (year 2018)
- ***Surface of laboratory**: about 2.000 m²*
- ***Customers**: 6000+*
- ***Analyzed samples**: 33.000+*
- ***Analysis carried out**: 100.000+*
- ***Analysis for contaminants:** 15.000+*
- ***Analysis for commercial classification of olive oils:** 30.000+*



Bologna, 12th December 2019

INTRODUCTION

- In April 2008, the alert on the contamination by mineral oils of sunflower oil for human consumption of Ukrainian origin was notified. At that time, the maximum limit of 50 mg/kg was set for mineral oils in sunflower oil of Ukraine origin.
- Following EC and FEDIOL guidelines and with regard to the characteristics of the analytical method, Chemiservice tested and validated an internal method using GC-FID even in that stage.
- Already in June 2012 EFSA published a scientific opinion about mineral oils food contamination, updated in August 2013.
- In 2014-2015 Chemiservice took part also in the interlaboratory validation of ISO 17780:2015 "Animal and vegetable fats and oils — Determination of aliphatic hydrocarbons in vegetable oils" official method.
- July 2017 method 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
- **In 2019 JRC Technical Reports "Guidance on sampling, analysis and data reporting for monitoring of mineral oil hydrocarbons in food and food contact materials".**
- 15th November 2019 EFSA - 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)

No legal limit, No EU regulations for MOSH and MOAH in OILS and FOOD

MOSH (C10-C16)	< 1 mg/kg
MOSH (C17-C24)	< 4 mg/kg
MOSH (C25-C35)	< 10 mg/kg
MOSH (C10-C35)	< 14 mg/kg
MOAH (C10-C35)	< 1 mg/kg

Since 2015, our customers signed specifications on MOSH and MOAH monitoring for the export to some North European market.

BfR (2011):
-12 mg/kg food for MOSH C10-C16
-4 mg/kg food for MOSH C16-C20

BMEL (2011-14): during 4 years the Federal Ministry for Nutrition and Agriculture drafted 3 ordinances for packaging made of recycled fibers
The last one in July2014 provided maximum limits for mineral oil residues in packaging and food in contact with packaging not complying with the proposed limits. Limits for foodstuffs were established at:
MOSH C20-C35 max 2 mg/kg foodstuff
MOAH C16-C35 max 0.5 mg/kg

The attention has been focused particularly on MOAH and, besides monitoring, the request consists in assessing the causes of a possible contamination and applying corrective measures to reduce detected levels.

No legal limit, No EU regulations for MOSH and MOAH in OILS and FOOD

- However, in this framework still being defined, large-scale retail trade (GDO) has focused on imposing to producers and resellers specifications to whom reference can be made for the evaluation of the aforementioned contamination. The majority of Chemiservice's customers must respond to specific contractual agreements for vegetable oils and, more specifically, for extra virgin olive oils.

3.A - imballo	Gli imballi in carta/cartone in fibra riciclata, in ogni caso, possono contenere MOSH max 24 mg / kg MOAH max 6 mg / kg
3.B - alimento	Nell'alimento i valori limiti ammissibili per MOSH e MOAH sono: MOSH max 2 mg / kg MOAH max 0,5 mg / kg

- **The 4th BMEL (2017) draft ordinance suggested:**

0.5 mg/kg limit for MOAH

no limit for MOSH

ANALYTICAL METHOD APPLIED

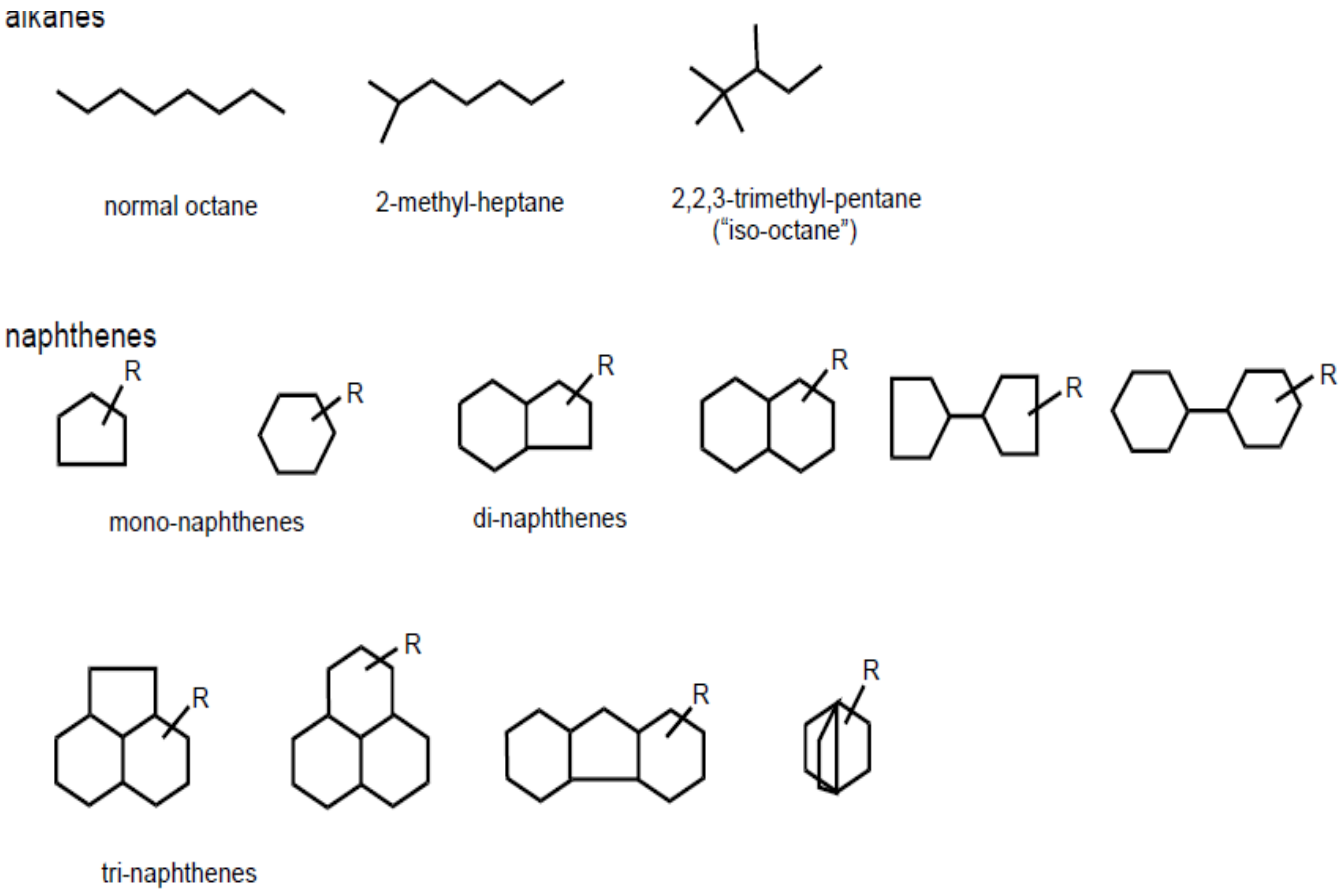
PROCEDURA DI PROVA METODO INTERNO Laboratorio di Prova: Chemiservice srl	MI/C12 Revisione 02
Determinazione di MOSH e MOAH in olio extravergine di oliva e oli vegetali	18/04/2019

OFF-LINE method

Chemiservice developed and validated an internal method using solid liquid separation on chromatographic column and injection into gas chromatograph with on-column injector and FID detector.

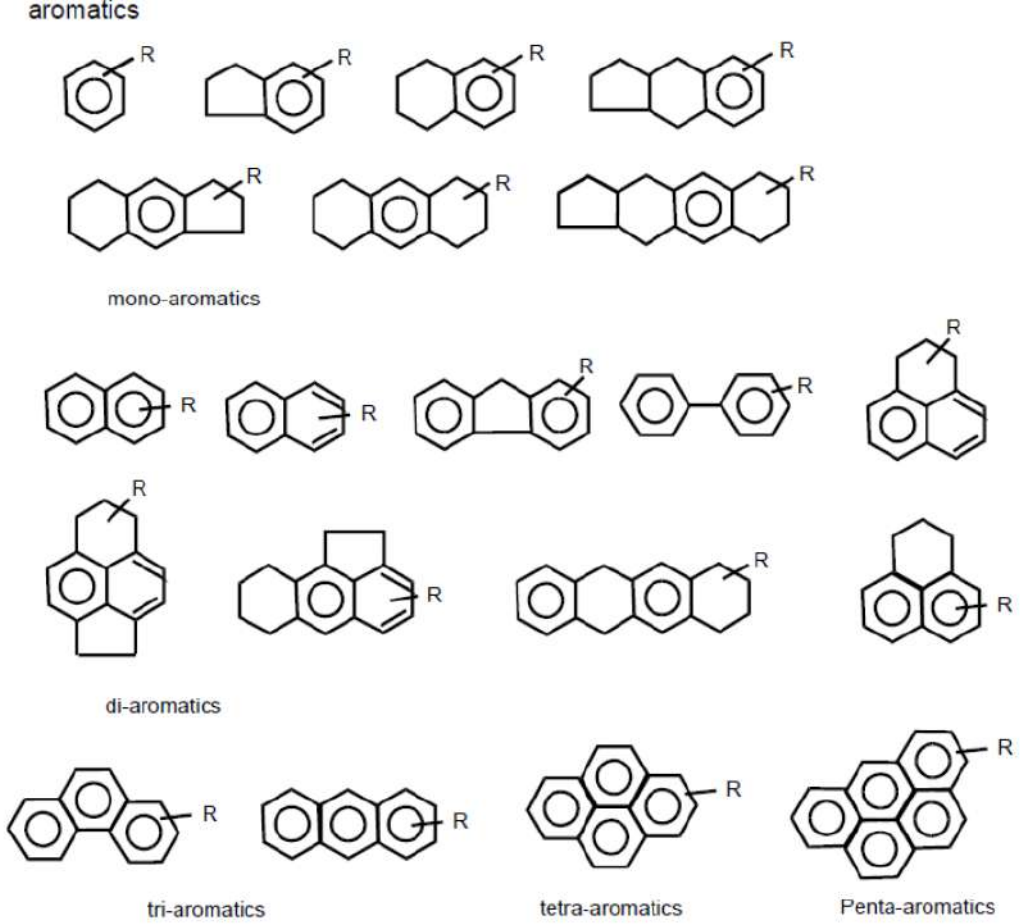
In the preliminary stages, the method was applied and validated to analyze virgin olive oils. Recently, application has been extended also to other vegetable oils, adding further purification stages.

MOSH comprise paraffins (open chain hydrocarbons) and naphthenes (cyclic hydrocarbons), which are mostly highly alkylated and originate either directly from mineral oil or are formed during refining by hydrogenation of aromatic compounds or other conversion processes.



9 Mineral oil saturated hydrocarbons (MOSH)

MOAH contain at least one aromatic ring. They include polyaromatic compounds, but should be distinguished from the compounds commonly termed polyaromatic hydrocarbons (PAH), such as benzopyrenes, which are formed at high temperatures.



10 Mineral oil aromatic hydrocarbons (MOAH)

Oils and fats

Analytical procedure FLOW CHART

Purification step: Epoxidation

Passage through activated alluminium oxide phase

Weighted Rapresentative 0,5 grams of oil + IS and QC standard + n-Hexane

Separation on glass chromatographic column (1 cm inner diameter) filled with 1 gram of silica gel with silver nitrate at 10% + 6,5 grams of silica gel 60-200 µm (70-230 mesh).

Conditioning of chromatographic column and elution of dead volume

Elution with hexane and collection of MOSH fraction

Elution with n-hexane /dichlorometane 75%/25% (v/v) and collection of MOAH fraction

Add 200 µl of isooctane (to avoid the evaporation of volatile compounds) to two eluted fractions and concentrate in Rotavapor at pressure ≥ 260 mbar and temperature ≤40°C until 200 µl. Insert with a siringe the concentrated fractions into two vials with conical insert.

Concentration of the two fractions in rotavapor at controlled pressure and temperature (until 200 µl)

Injection in GC-FID on-column (2 µl)

Quality control materials and Internal standard used

MOSH

MOAH

MIX 1
n-Decane
Bicyclohexyl
n-Tridecane
(100 mg/L in n-hexane)

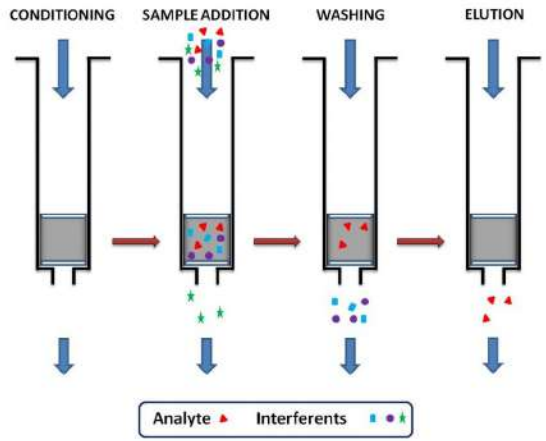
MIX 2
5-alpha-Cholestane
n-Tetracontane (IS)
(40 mg/L in n-hexane)

MIX 3
Pentylbenzene
1-Methyl-Naphthalene
2-Methyl-Naphthalene
1,3,5-Tri-tert-butylbenzene
Perylene (IS)
(100 mg/L in Toluene/n-hexane 50-50 v/v %)

At 0,5 grams of oil add 250 µl of MIX 1 + 100 µl of MIX 2 + 50 µl of MIX 3.

20 mg/kg for MOSH
10 mg/kg for MOAH

Solid phase extraction procedure



conditioning

- n-hexane/dichlorometane
- n-hexane

Sample loading

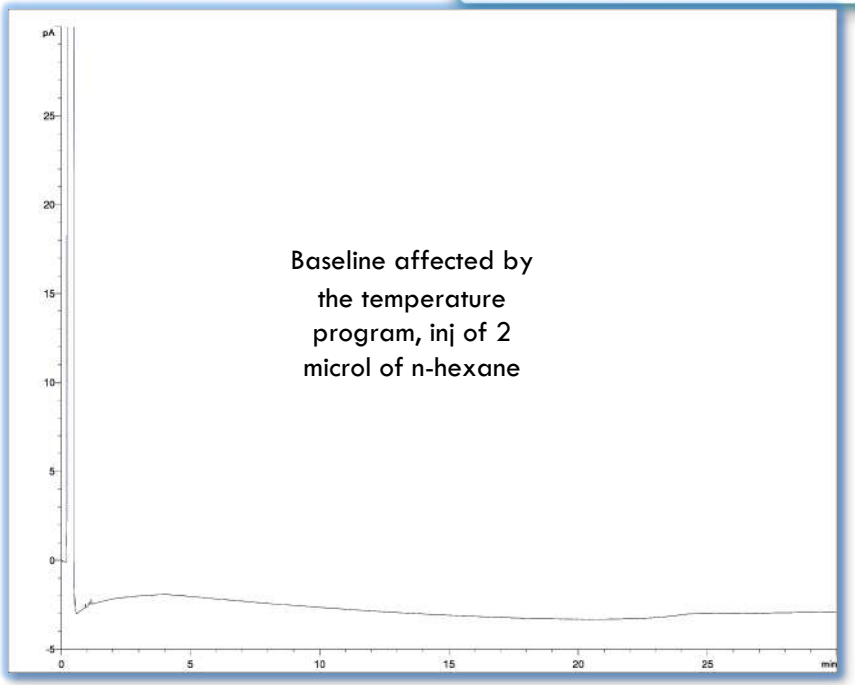
- Insert the oil with standards into column, discarting eluate.
- Wash the vial with 1 ml of hexane, insert into column, elute discarting eluate.
- Add 1 ml into column letting the oil flow on sides (to bring all oil into silica gel), elute discarting eluate.

Elution

- Add n-hexane and recover the fraction of MOSH in a 50 ml flask with emery neck.
- Add a mix hexane-dichloromethane (75%-25% v/v) and recover the fraction of MOAH in a 50 ml flask with emery neck. All MOAH are recovered if the yellow stripe due to the presence of the internal standard (Perylene) is completely eluted.

Gas chromatography Analysis

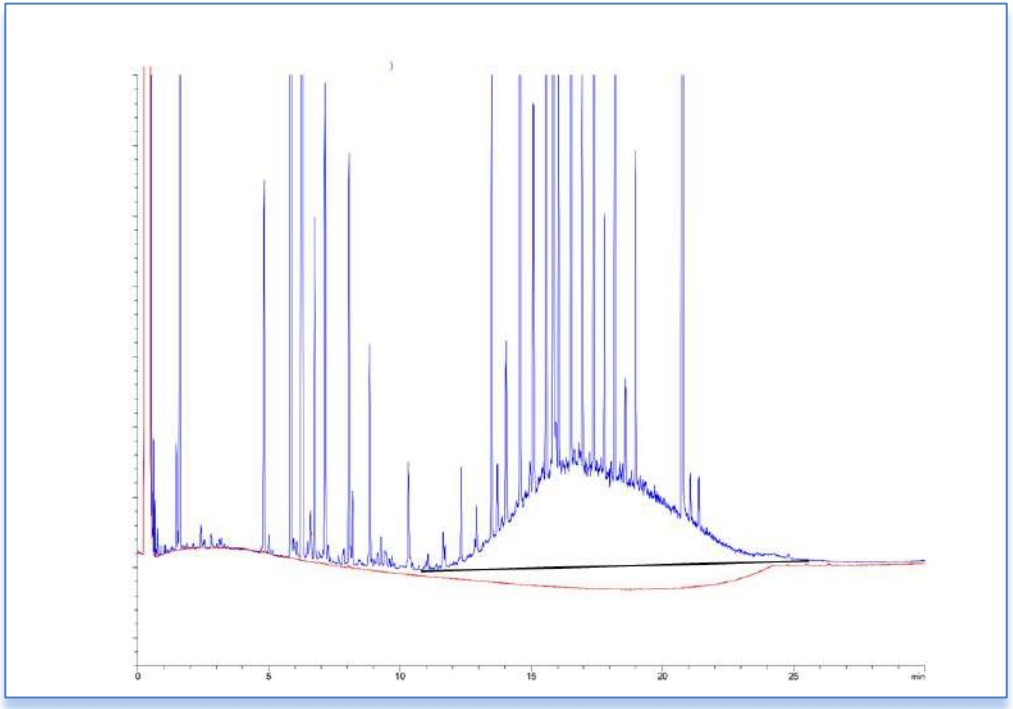
GC Agilent 7890A with double column, 2 on-column injectors, 2 FID and 2 automatic injectors.	
Carrier gas	Idrogen (65 kPa)
Detector temperature	350 ° C
GC Column	J&W DB-1HT, lungh. 15 m, diam. Int. 0.32 mm, spessore film 0.1 µm
GC Conditions	T inziale: 40°C. Isoterma di 2 minuti Incremento di 5°C/min fino a 50°C (0 minuti) Incremento 15°C/min fino 350°C (8 minuti)
Injection volume	2 µl
Software	Chemstation Agilent



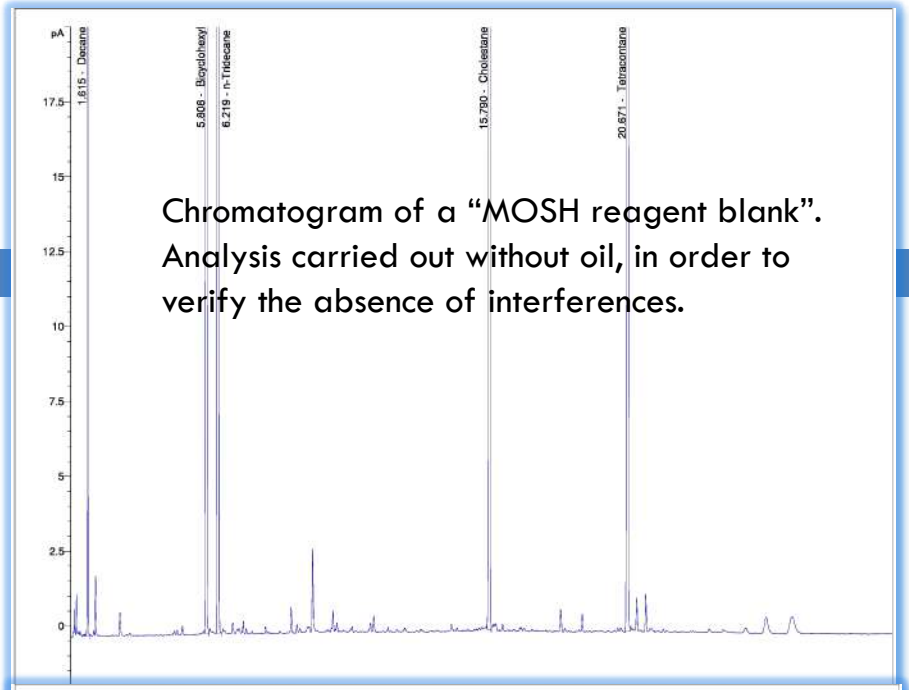
Baseline affected by the temperature program, inj of 2 microl of n-hexane

Critical points

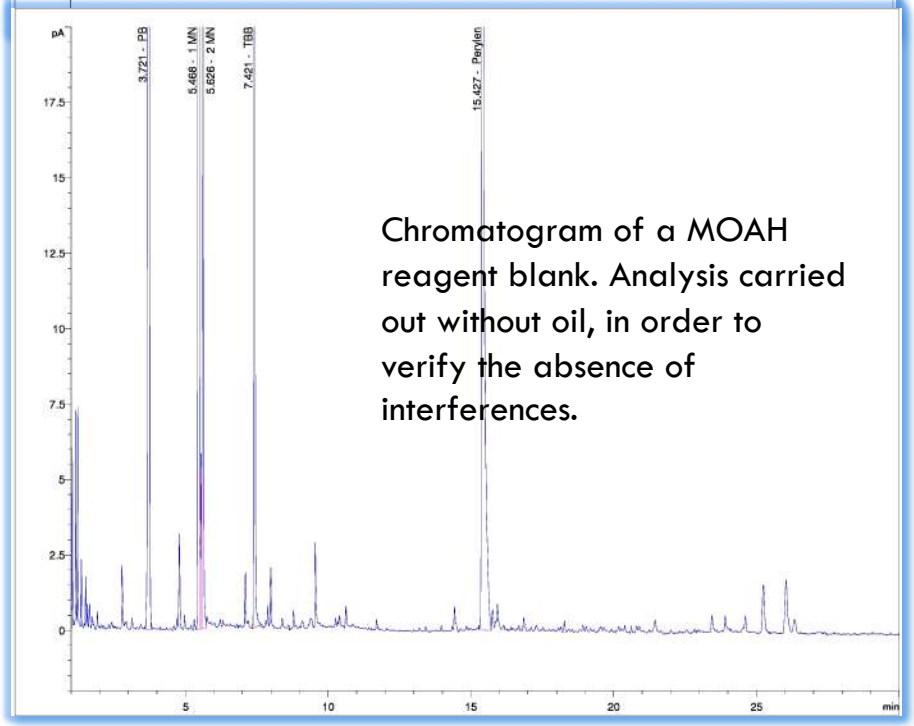
- Trend of the baseline due to the increase in temperature: deducting the baseline obtained by injection of 2 µl of solvent (n-hexane) from the chromatogram obtained injecting the fractions of MOSH and MOAH;
- Possible contamination during entire flow-work: accurate cleaning of lab glassware and control of contamination during sample processing by blanks;
- Interferences by endogenous hydrocarbons, natural paraffins (odd-numbered n-alkanes), olefins, squalene partially isomerized (refined olive oils)
- Sensitivity of method, LOQ in comparison to the maximum limits set for MOAH



Overlap of Solvent chromatogram (red line) and MOSH chromatogram of an extra virgin olive oil sample

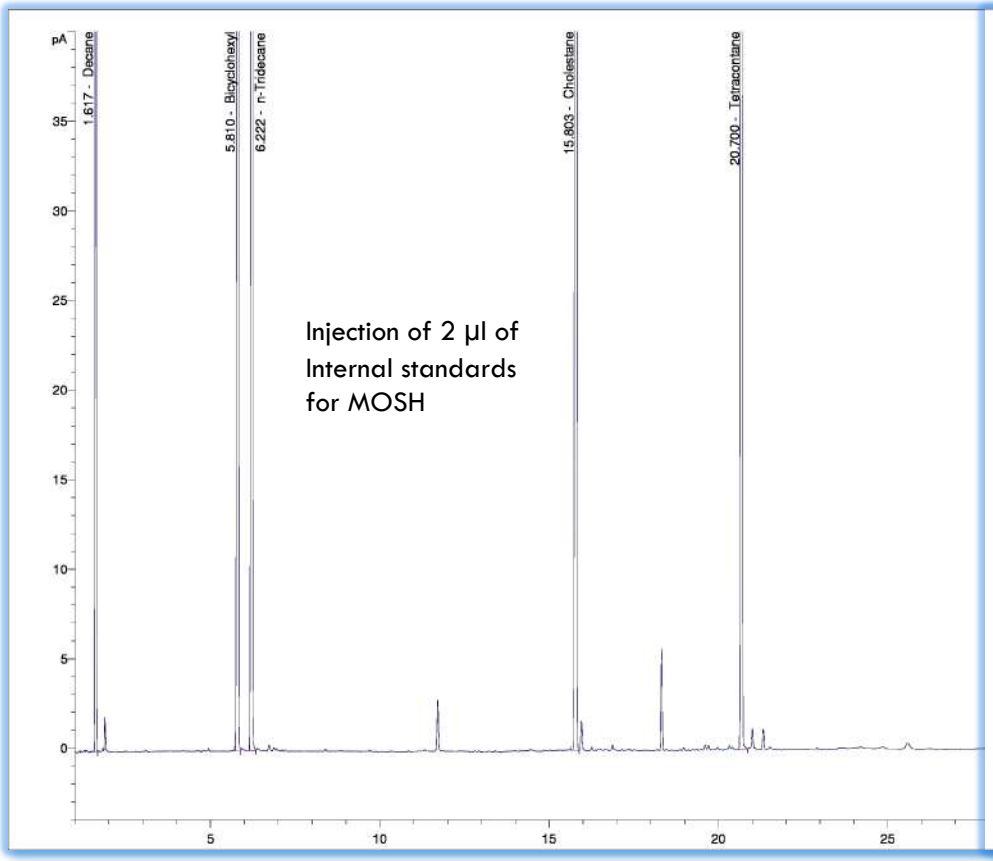


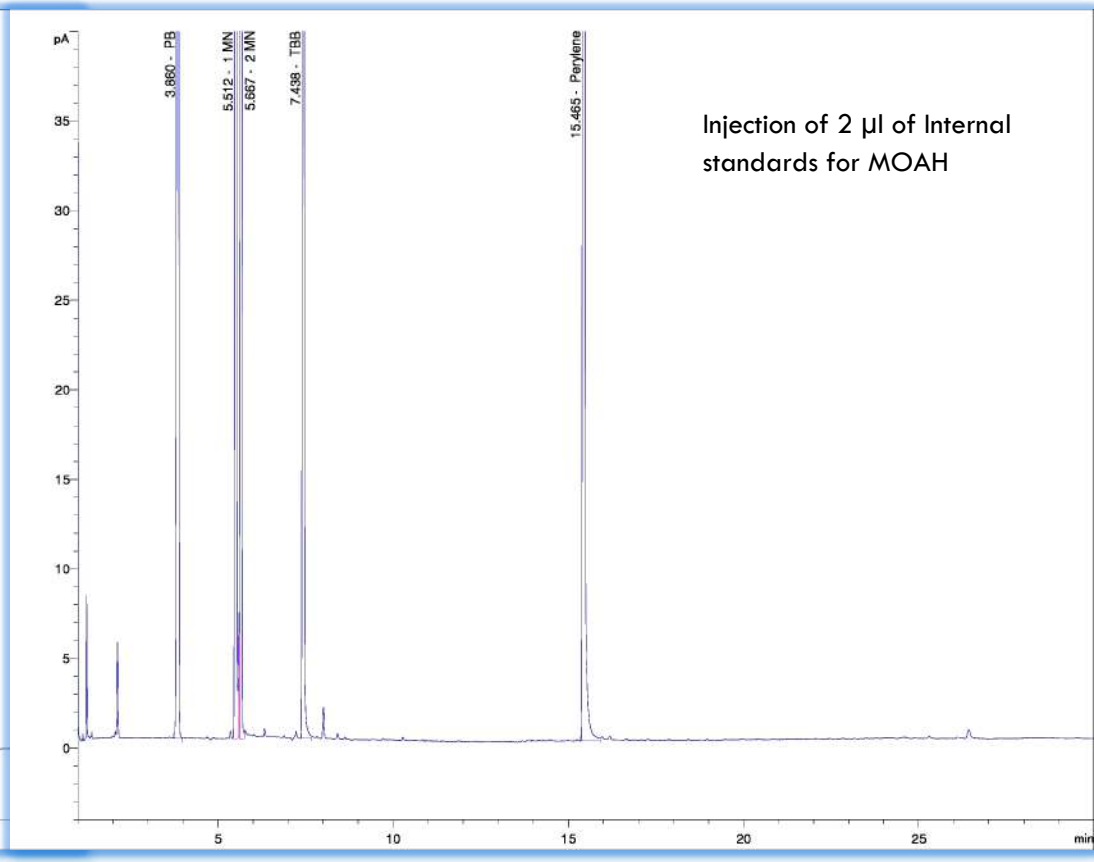
Chromatogram of a "MOSH reagent blank". Analysis carried out without oil, in order to verify the absence of interferences.



Chromatogram of a MOAH reagent blank. Analysis carried out without oil, in order to verify the absence of interferences.

Reference materials and IS





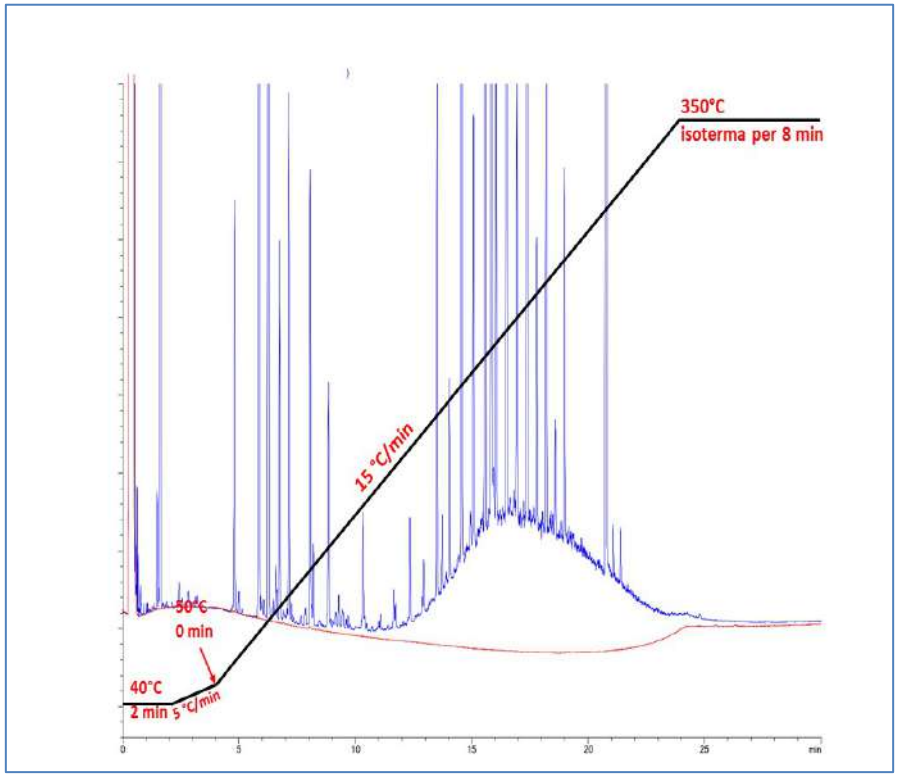


Fig. A- EVO Chromatogram: without baseline correction

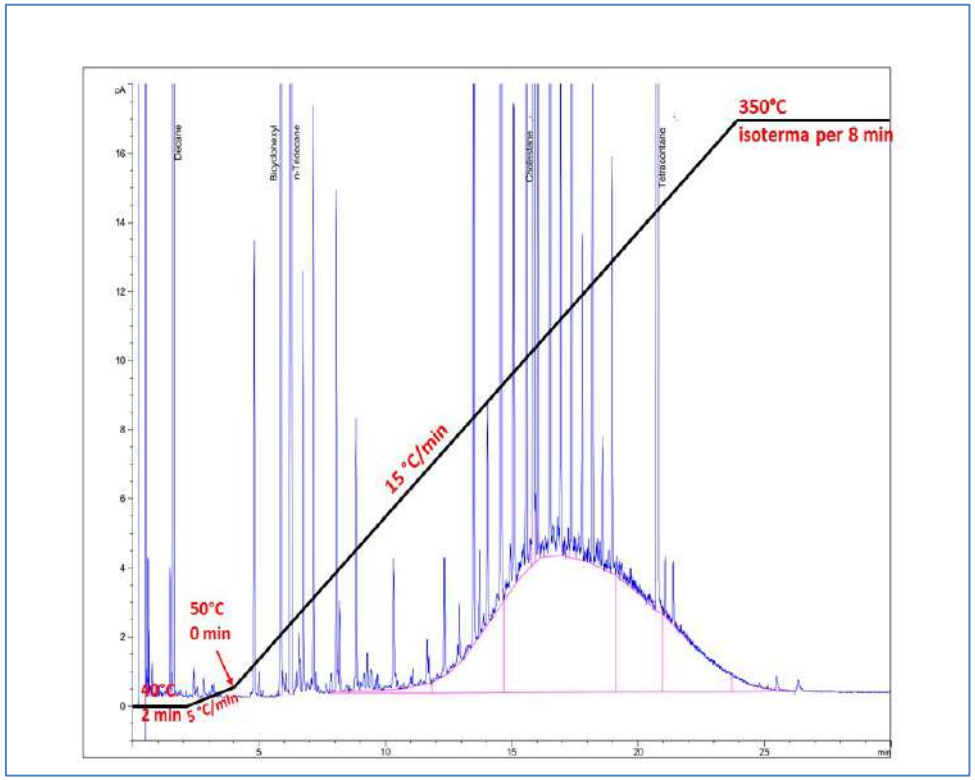
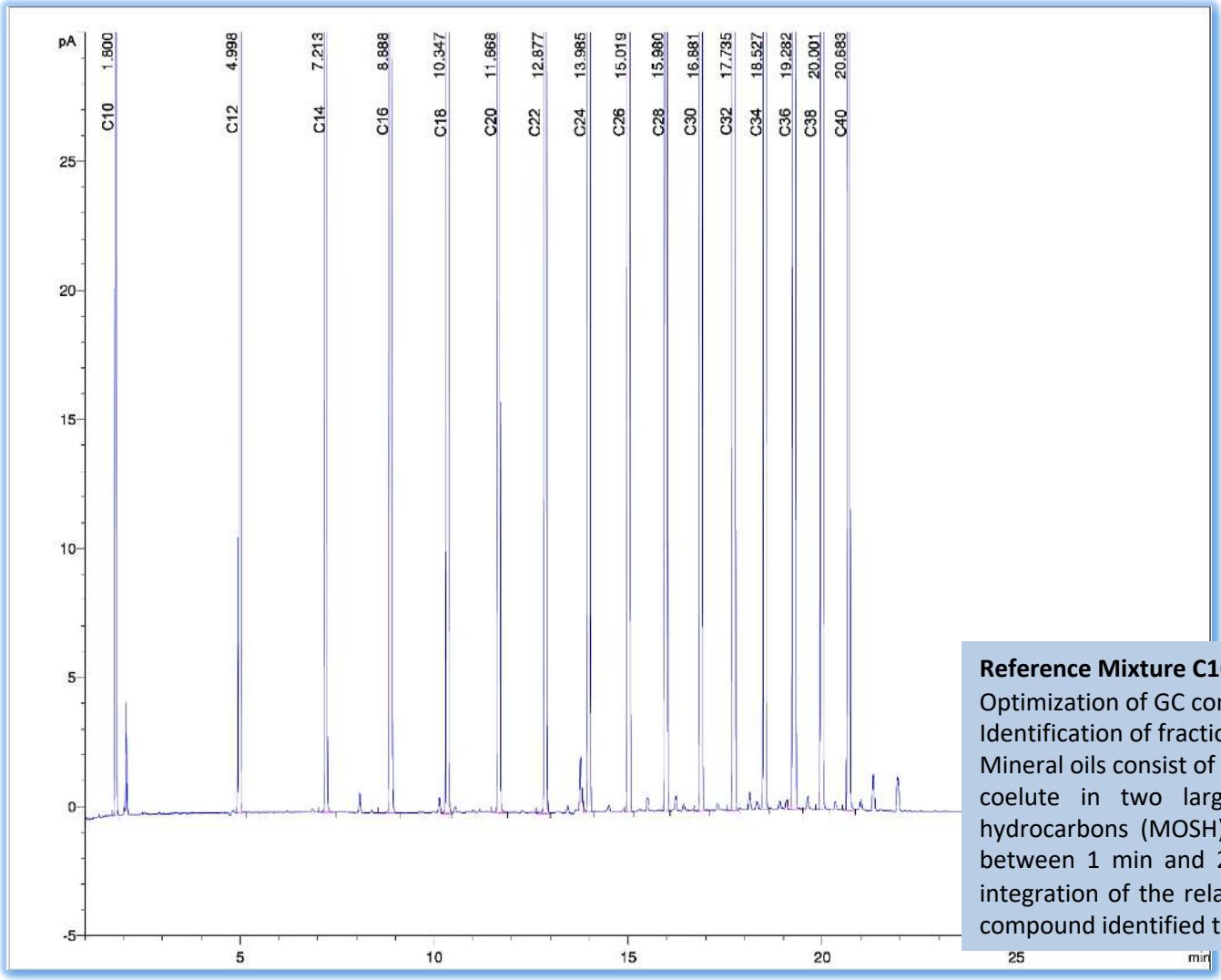


Fig B. EVO Chromatogram: with baseline correction, solvent signal subtraction



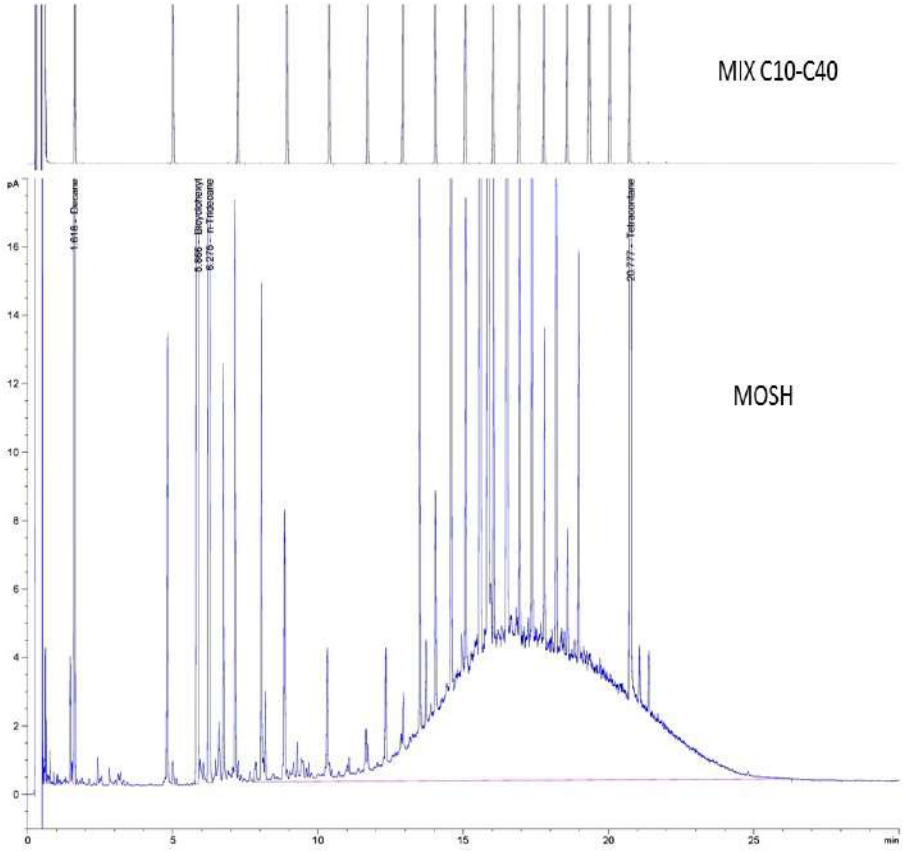
Reference Mixture C10-C40

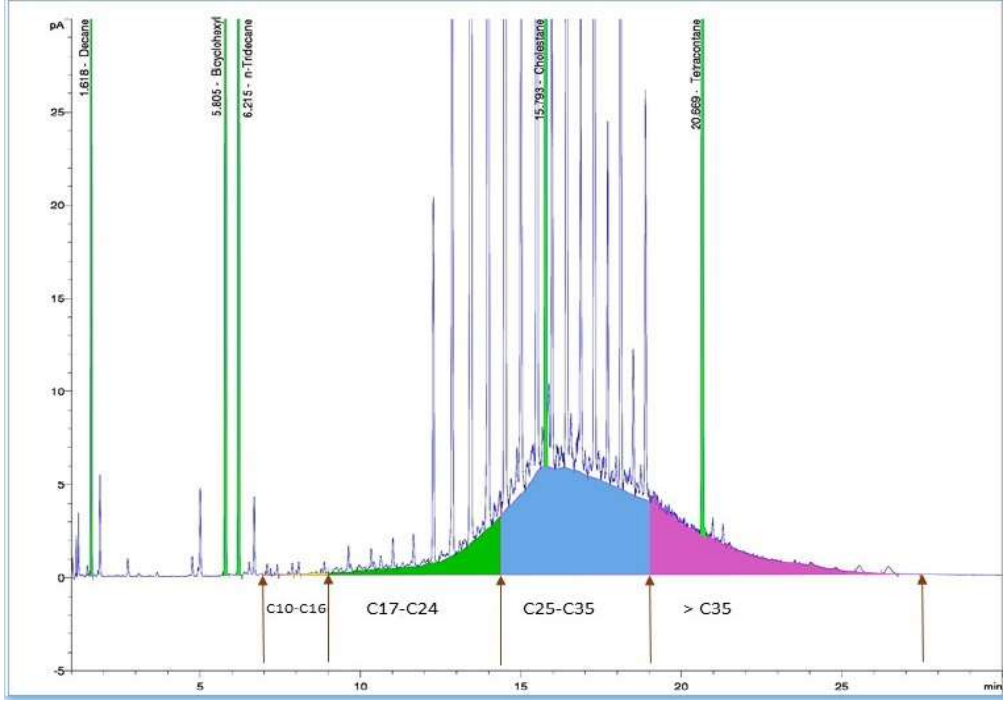
Optimization of GC conditions: Tetracontane (C40) – RT (min): 20-21

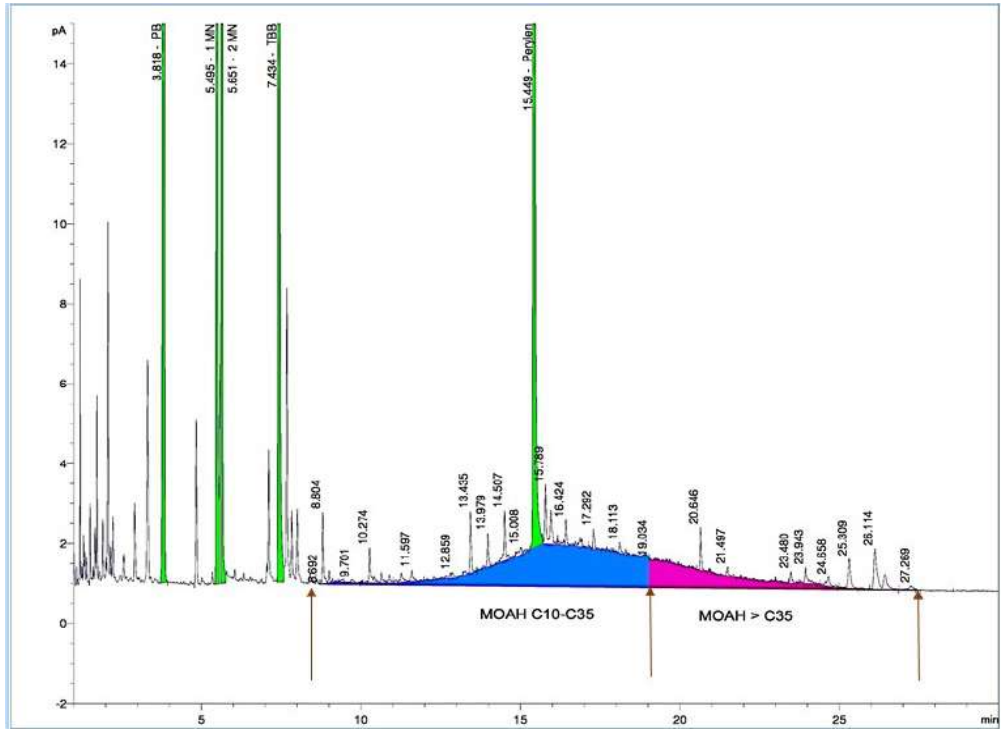
Identification of fractions:

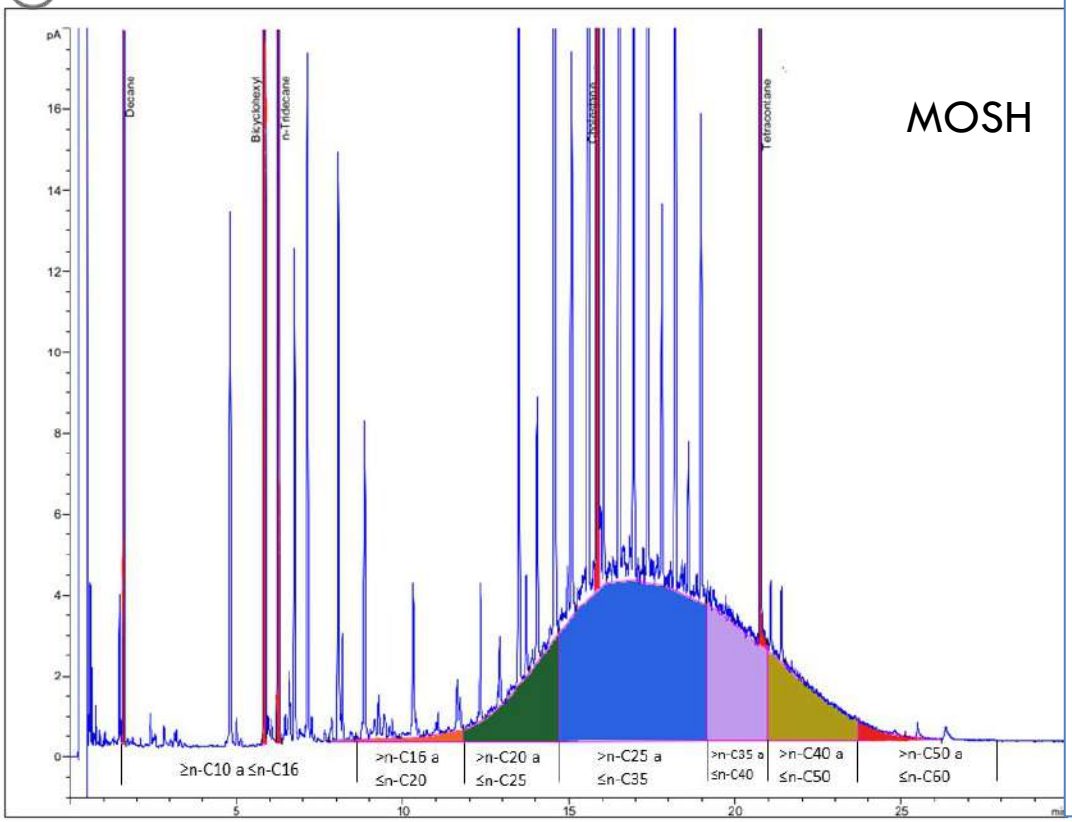
Mineral oils consist of a large variety of hydrocarbons of petroleum origin which coelute in two large unresolved bands (UCM) identified as saturated hydrocarbons (MOSH) and MOAH aromatic hydrocarbons in a time interval between 1 min and 28 min. The identification of the fractions requires the integration of the relative bands by referring to the elution time of the linear compound identified through the use of *a reference material*.

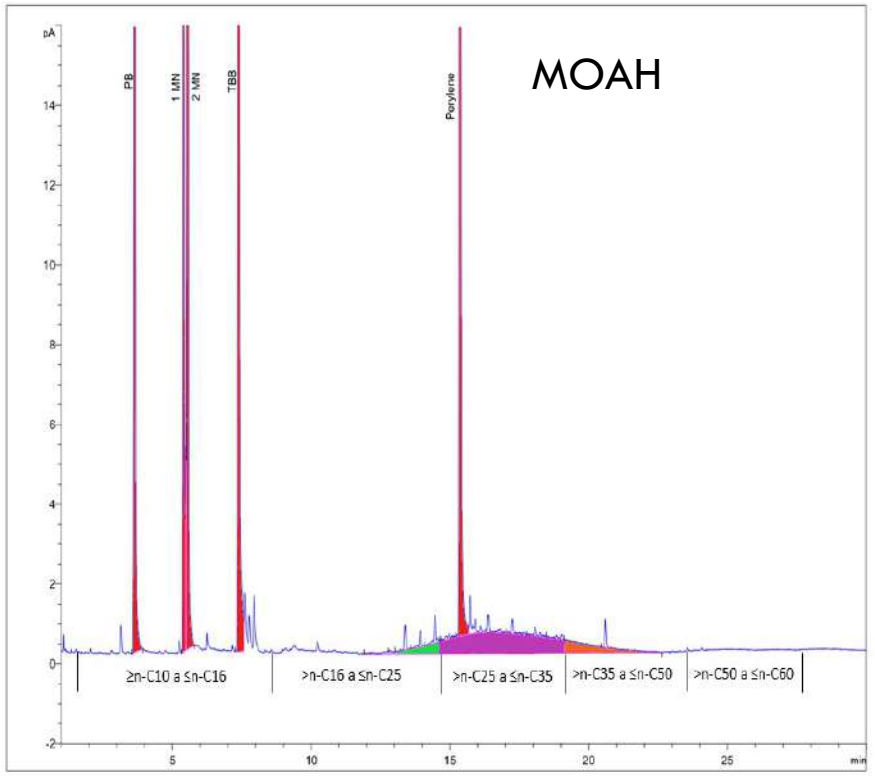
Overlap of the reference mixture chromatogram and MOSH fraction of Extra Virgin Olive Oil







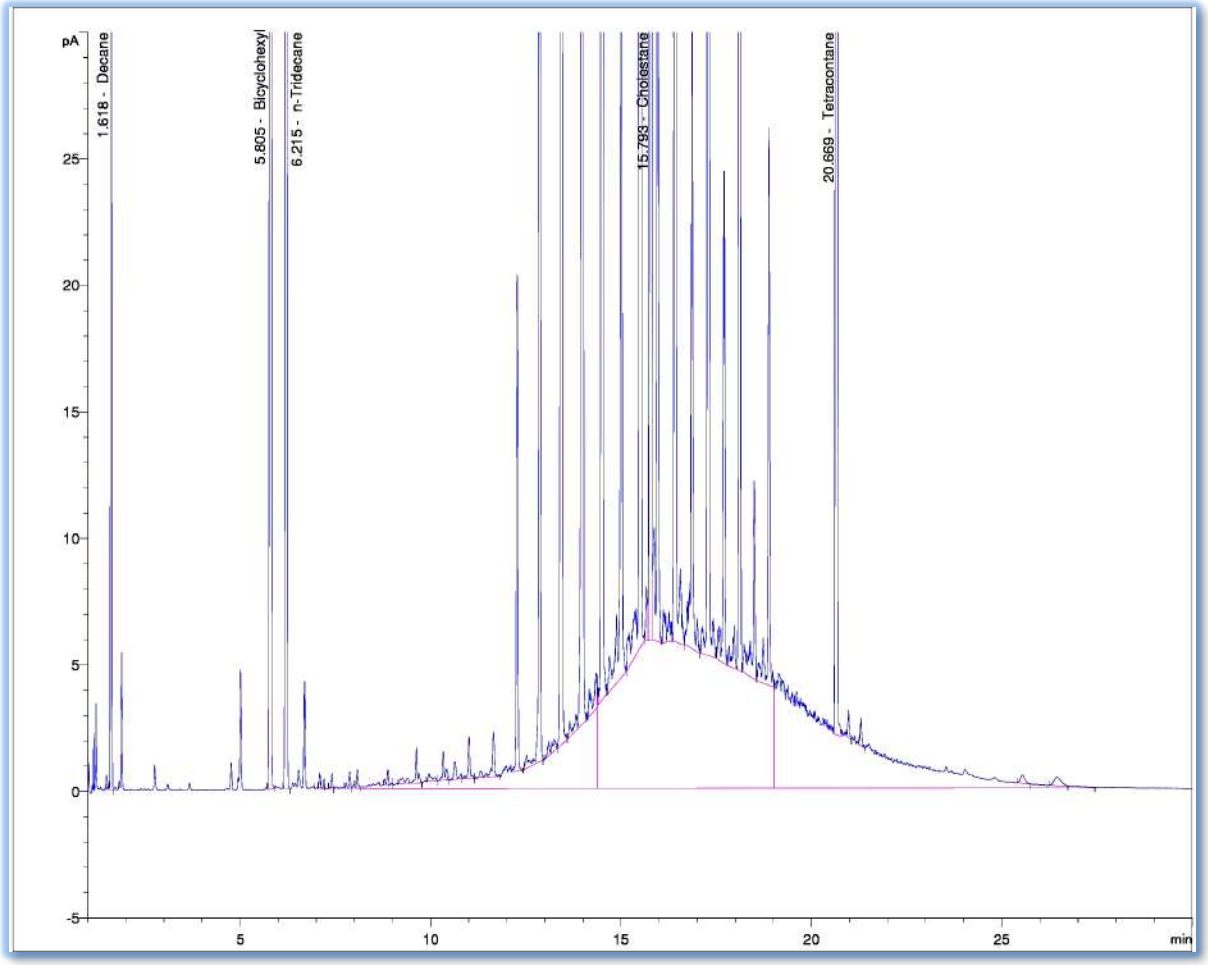




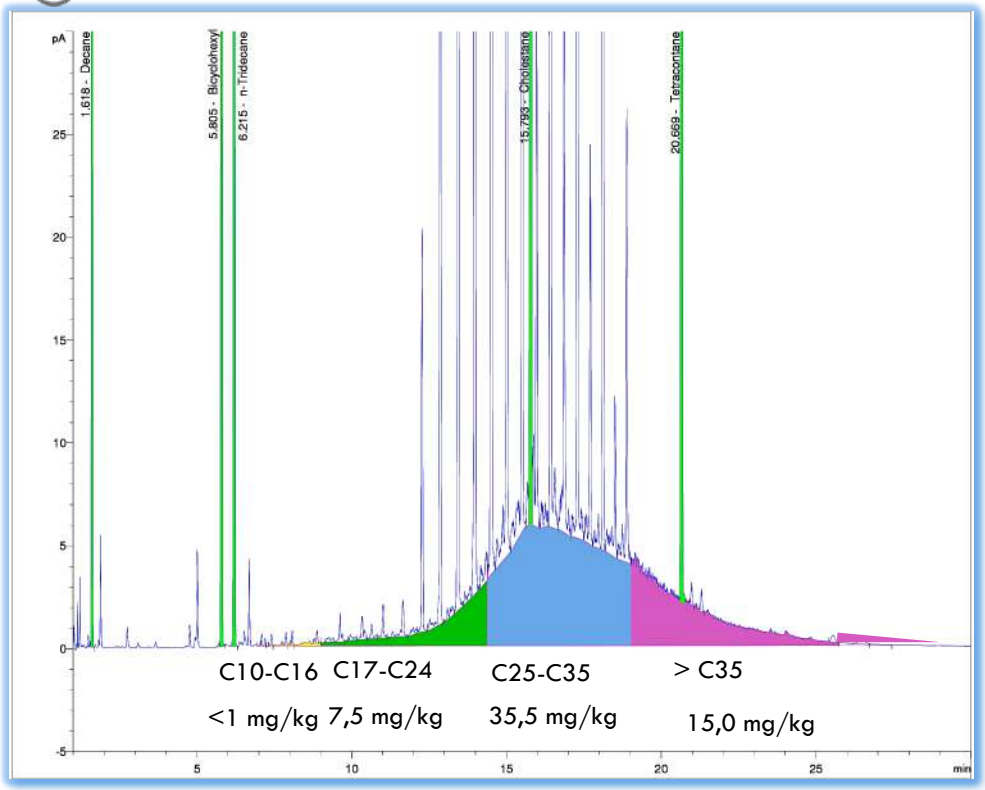
21

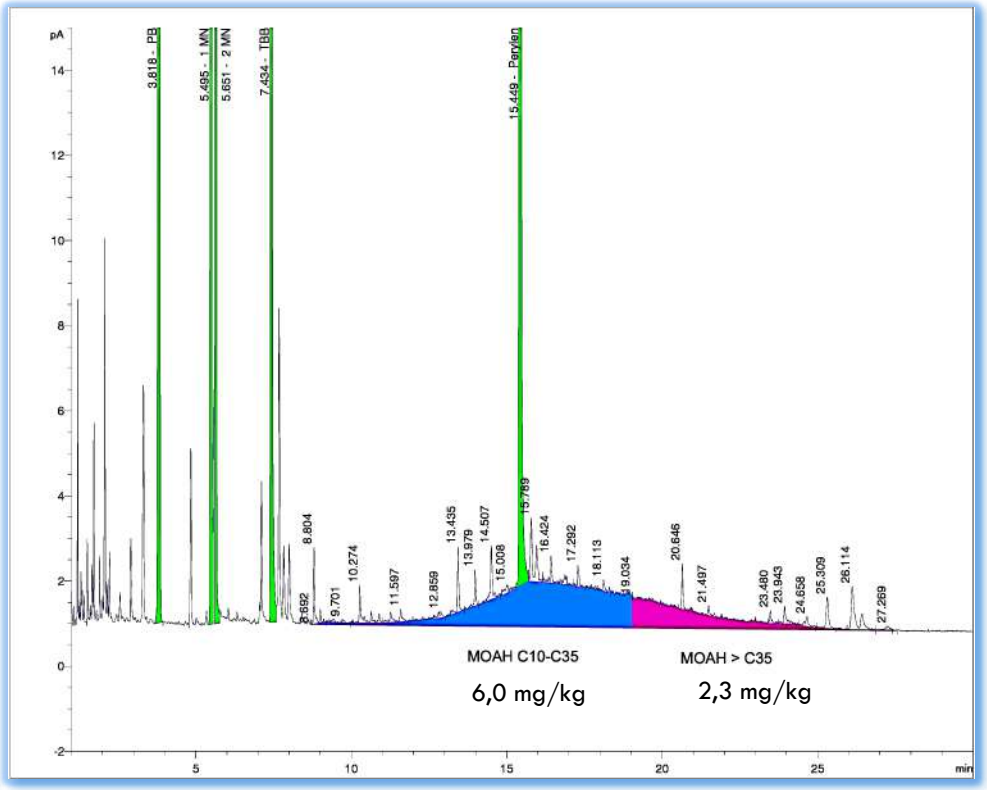
Chromatogram of extravirgin olive oil: JRC fractions

Integration of chromatograms and quantification



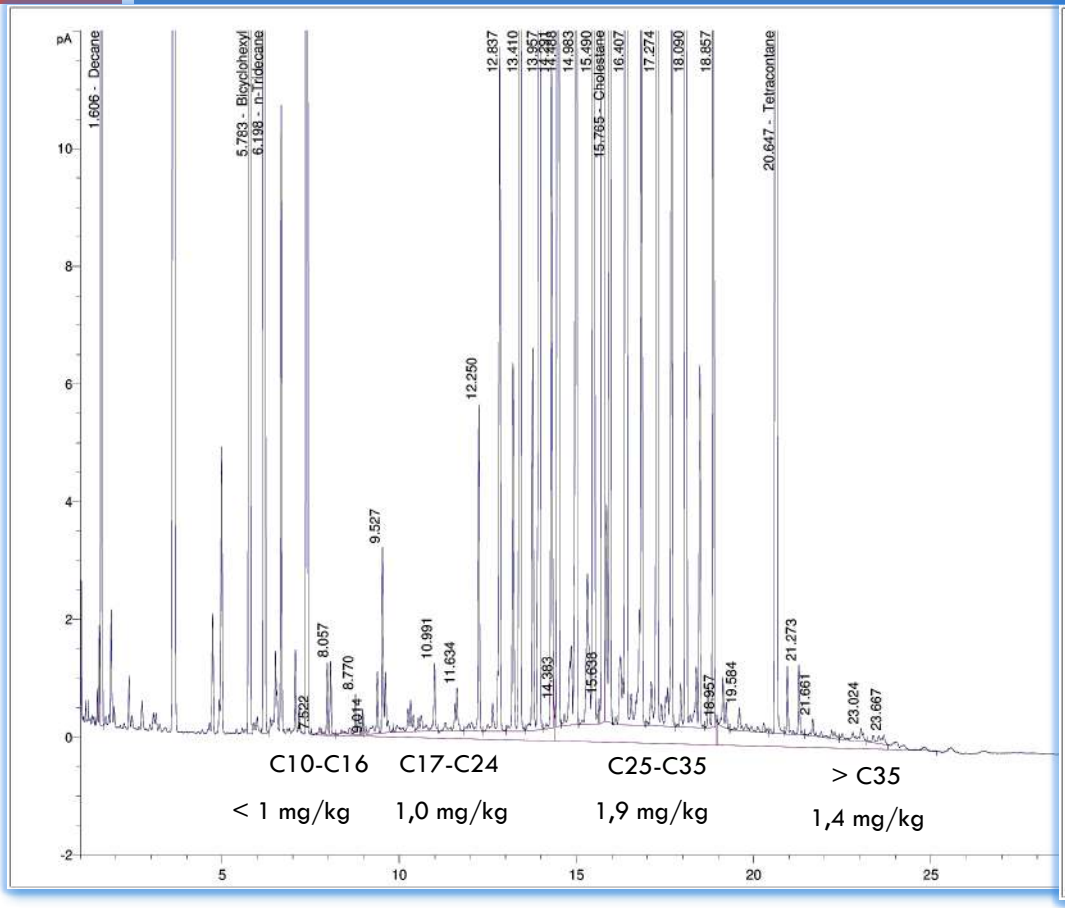
- Drawing a base line for joining the point where the area subtended to the unresolved peak rises with the point where it descends;
- Identification of fractions
- using skim function, subtraction of all peaks standing out against the mix of the unresolved complex mixture (UCM) (standard and Alkanes of endogenous origin)
- verifing method performance;
- Calculation of concentration by IS method.



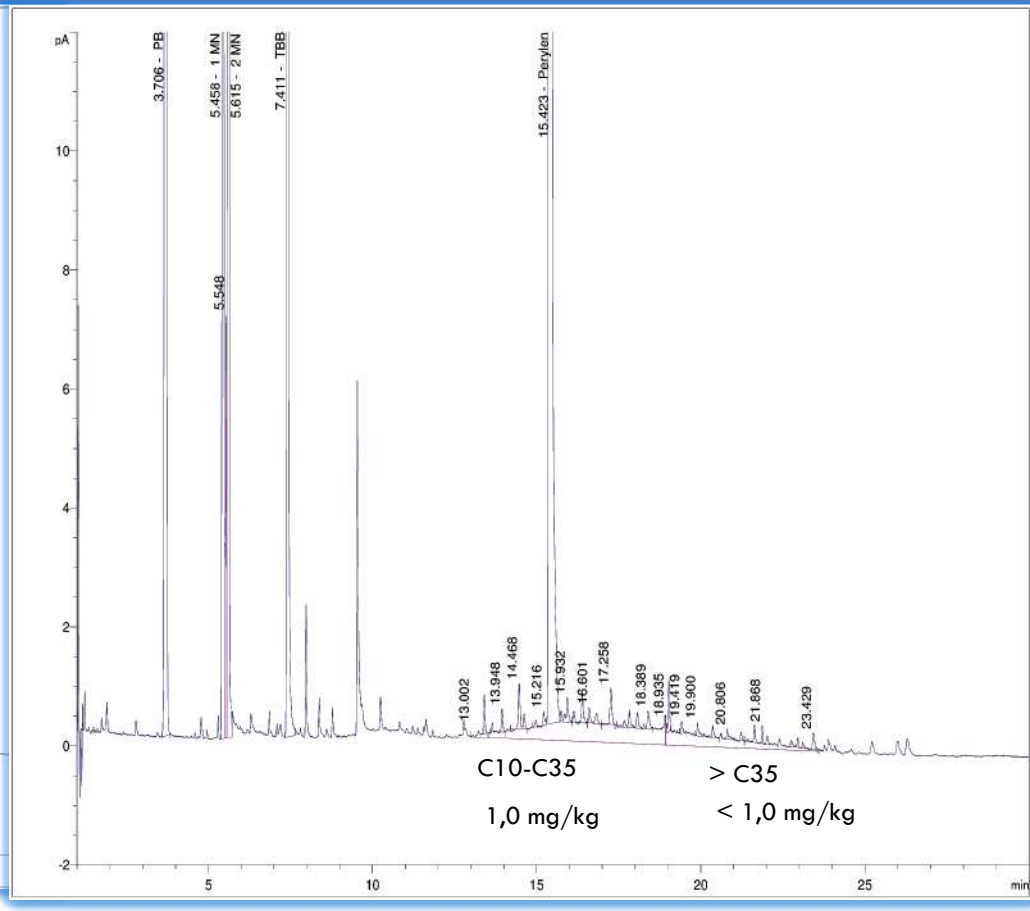


Bologna, 12th December 2019

Examples of Sample of extravirgin olive oil



MOSH of extra virgin olive oil



MOAH of oil at LOQ level.
Extra virgin olive oil

Epoxidation

Evaluation with known MOSH additions

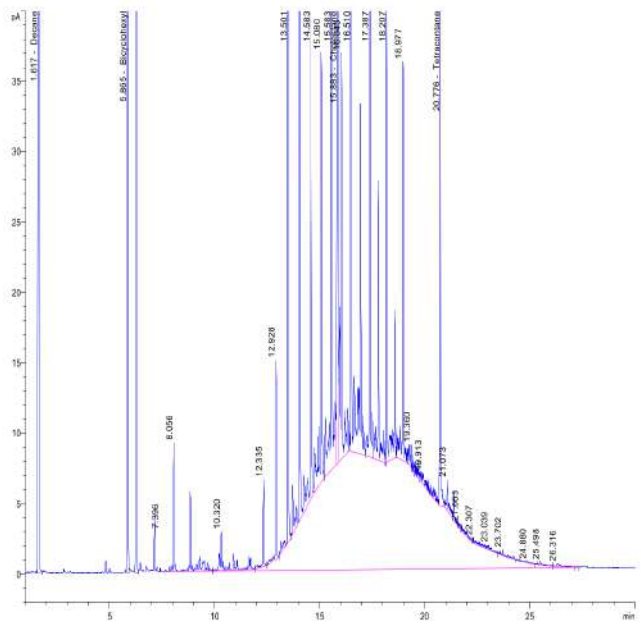
How did we prepare the known additions?

- MOSH-Using the MRC Mineral oil Heavy (Dr. Ehrenstorfer, 03009010 – CAS No 8042-47-5), known additions have been made to both extra virgin olive oil and refined olive oil.
- MOAH-purification and recovery of the MOAH fraction from waste oil, evaluation of the concentration by gravimetry

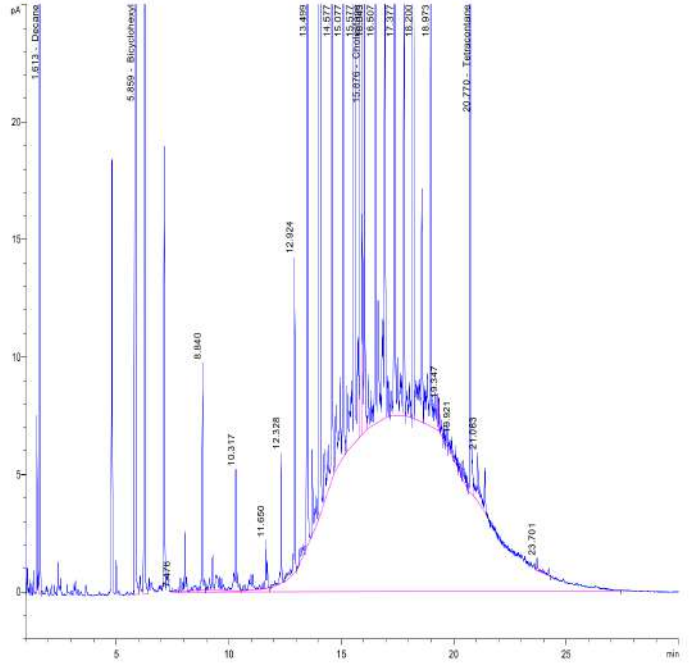
	EXTRAVIRGIN OLIVE OIL			
	spikes	Results without epoxidation	Results after epoxidation	Recovery
	mg/kg	mg/kg	mg/kg	%
MOSH C10-C60	31,7	32,2	31,2	98
MOAH C10-C60	5,0	4,9	4,5	90
	REFINED OLIVE OILS			
		mg/kg	mg/kg	
MOSH C10-C60		57,1	53,8	
MOAH C10-C60		-	3,0	

Epoxidation

MOSH Fraction of olive oil – no epoxidation

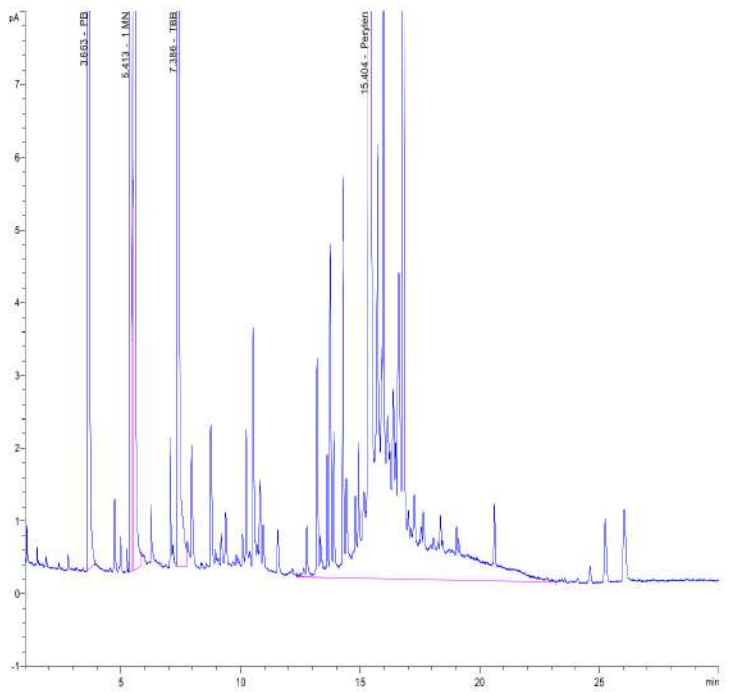


MOSH fraction of olive oil – with epoxidation

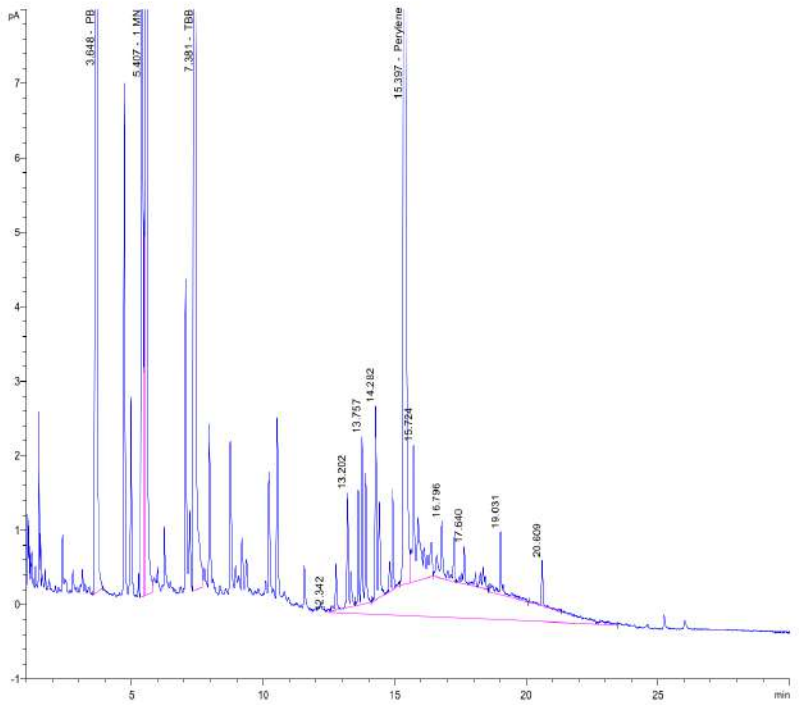


Epoxidation

MOAH Fraction of olive oil – no epoxidation

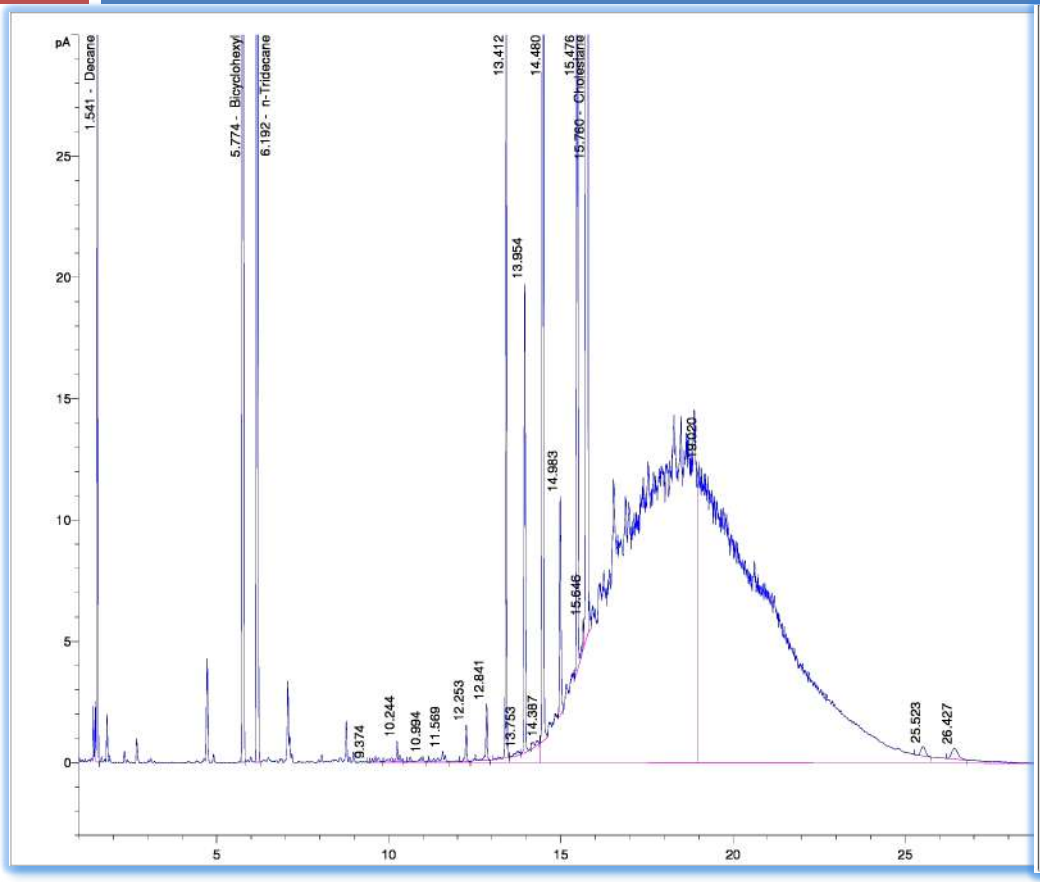


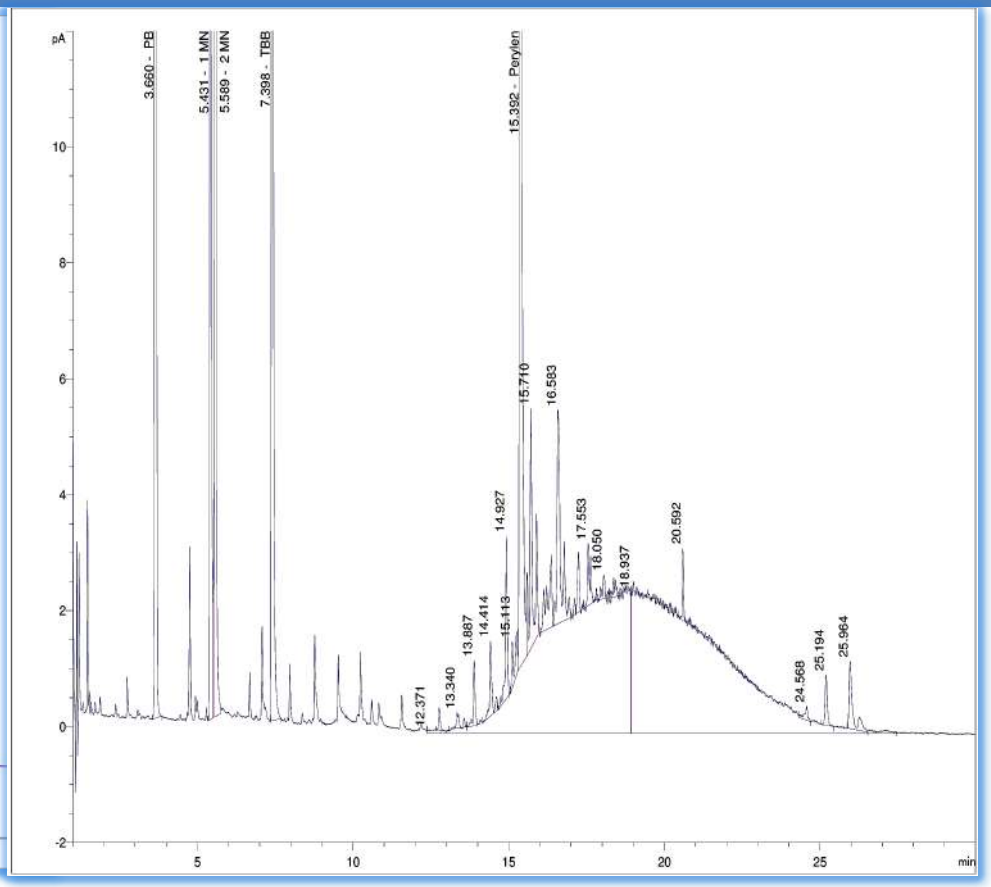
MOAH fraction of olive oil – with epoxidation



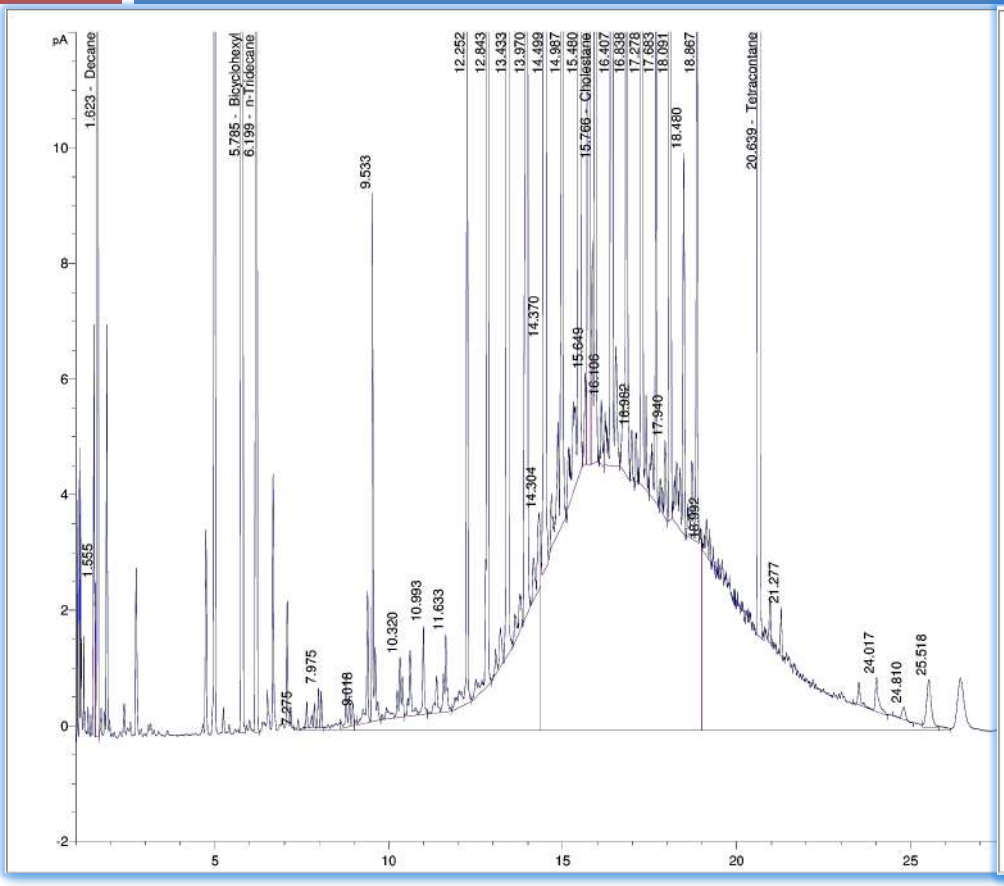
MOSH of a refined pomace oil after epoxidation and passage through Alox-activated silica gel column (calculation with bicyclohexyl)

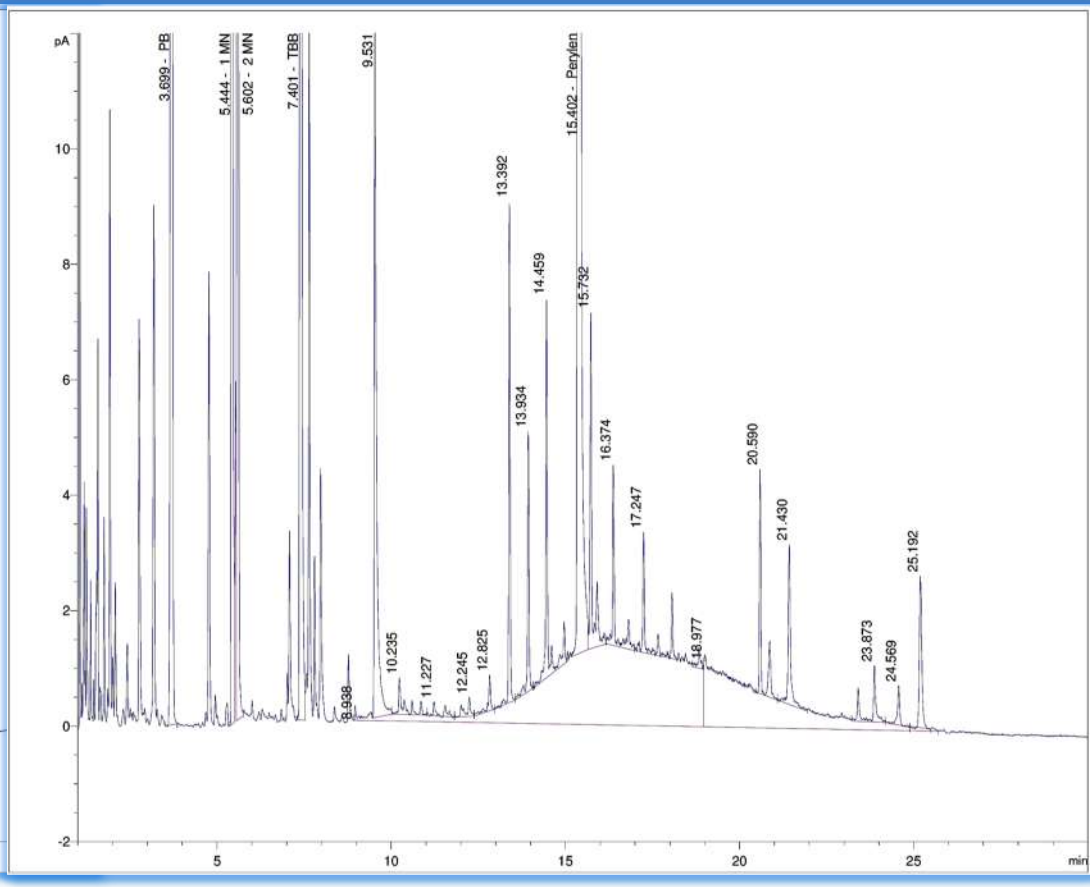
MOAH of refined pomace oil after epoxidation





Extravirgin olive oil: example 1

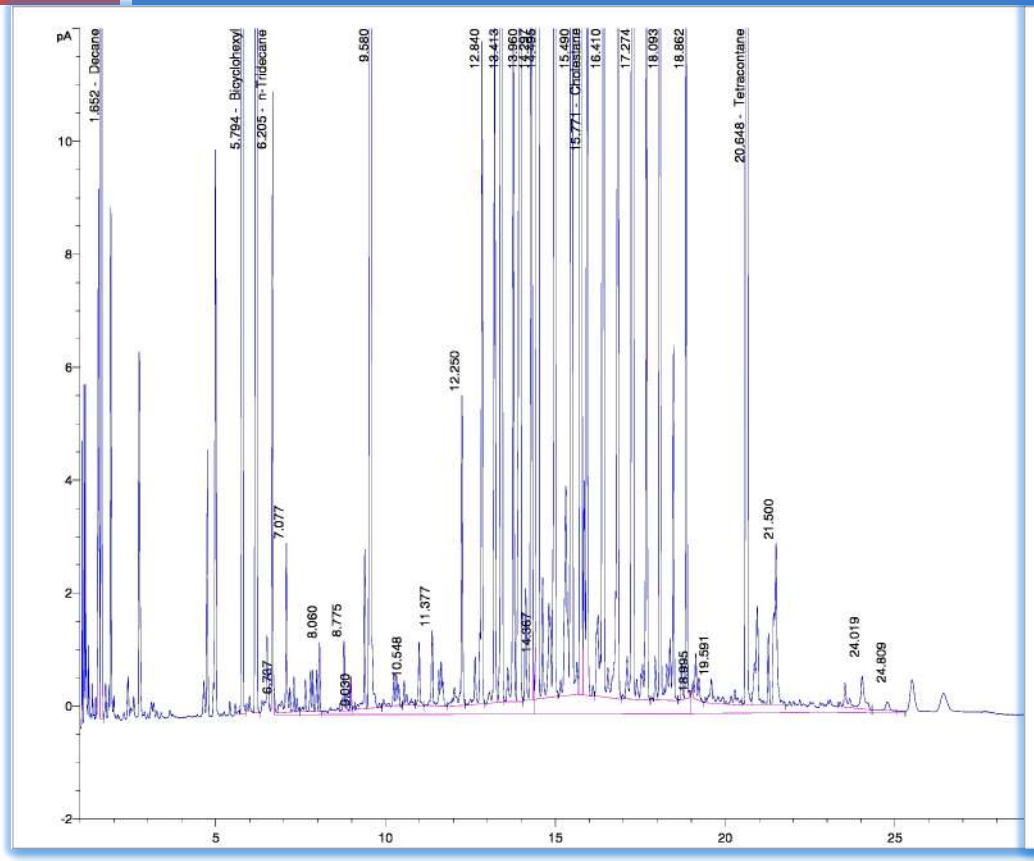


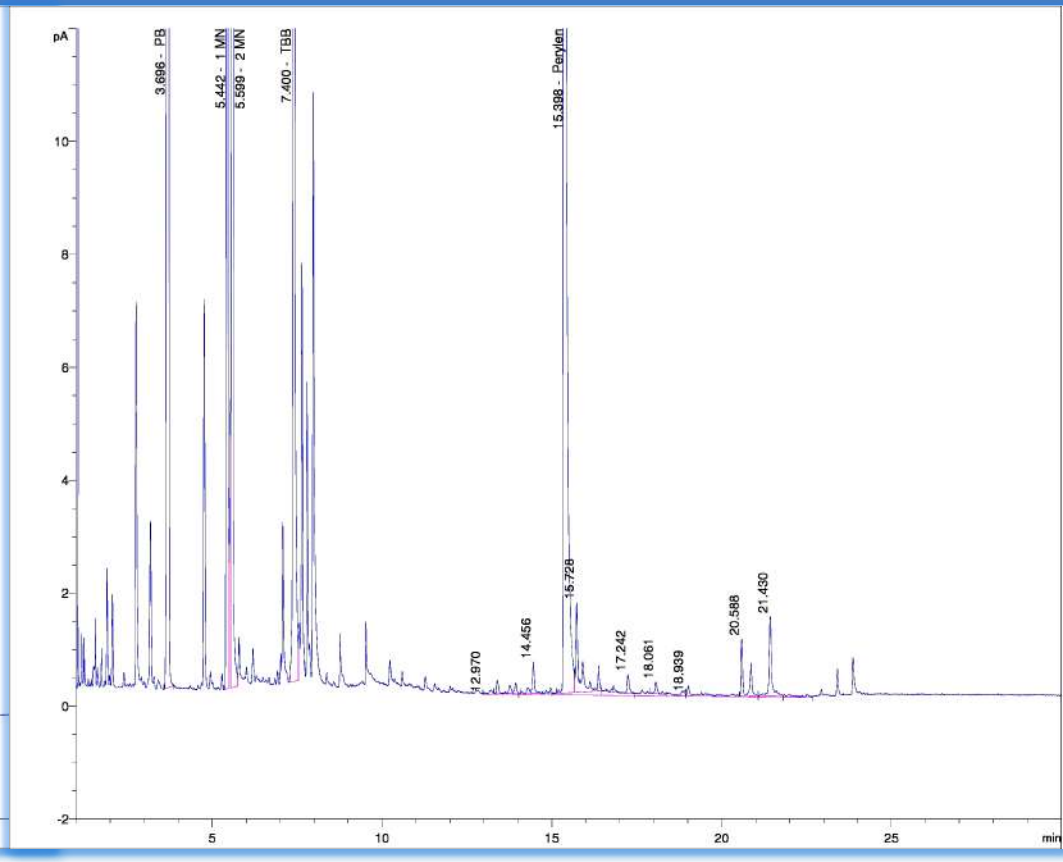


MOSH C10-C16 = < 1 mg/kg
MOSH C17-C24 = 6,9 mg/kg
MOSH C24-C35 = 35,3 mg/kg
MOSH C10-C35 = 42,2 mg/kg
TOTAL MOSH = 56,1 mg/kg

MOAH C10-C35 = 7,0 mg/kg
TOTAL MOAH = 9,7 mg/kg

Extravirgin olive oil: example 2



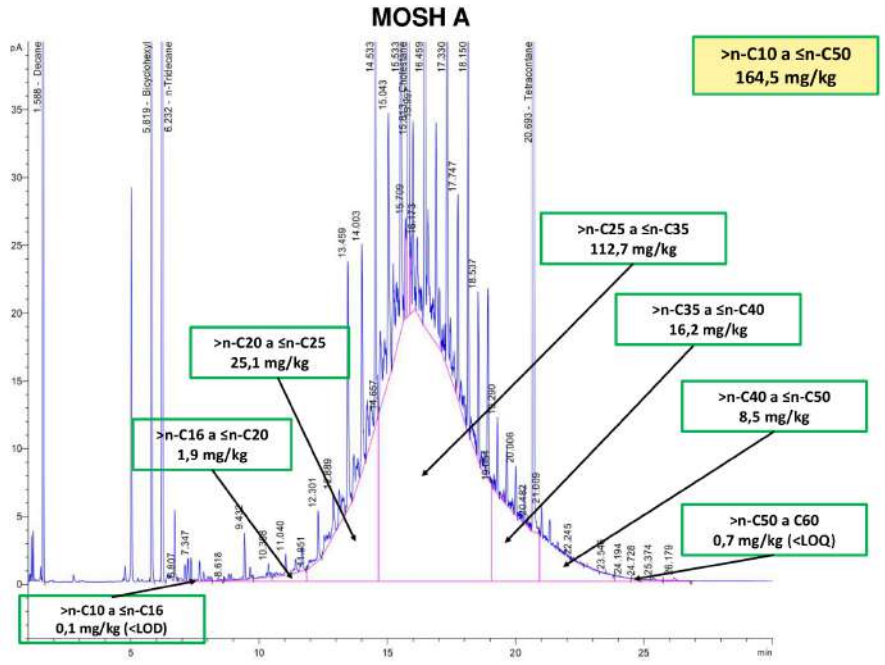


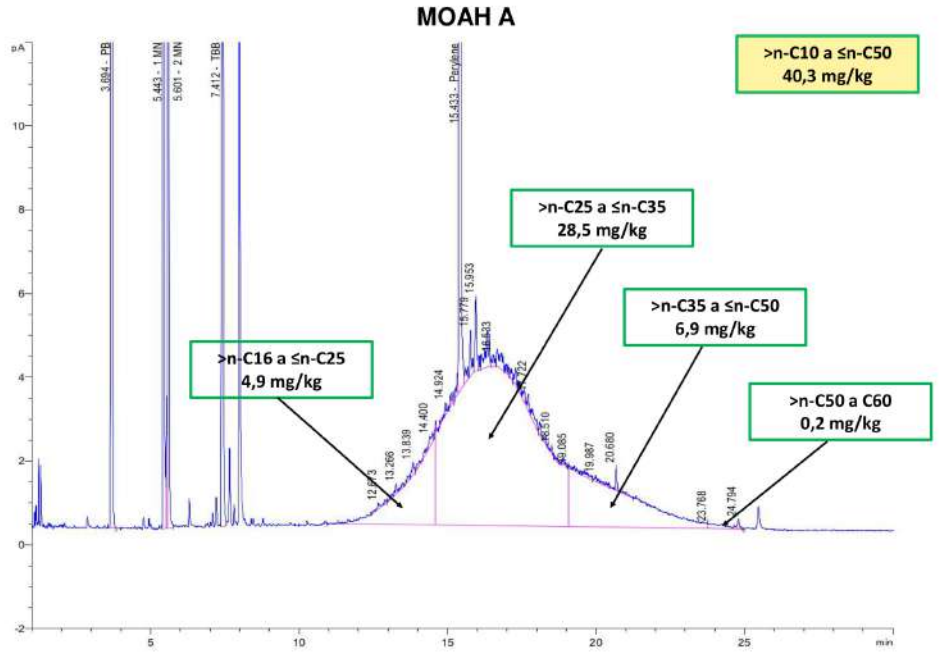
MOSH C10-C16 = < 1 mg/kg
MOSH C17-C24 = 1,3 mg/kg
MOSH C24-C35 = 1,8 mg/kg
MOSH C10-C35 = 3,1 mg/kg
TOTAL MOSH = 4,5 mg/kg

MOAH C10-C35 = < 1 mg/kg
TOTAL MOAH = < 1 mg/kg

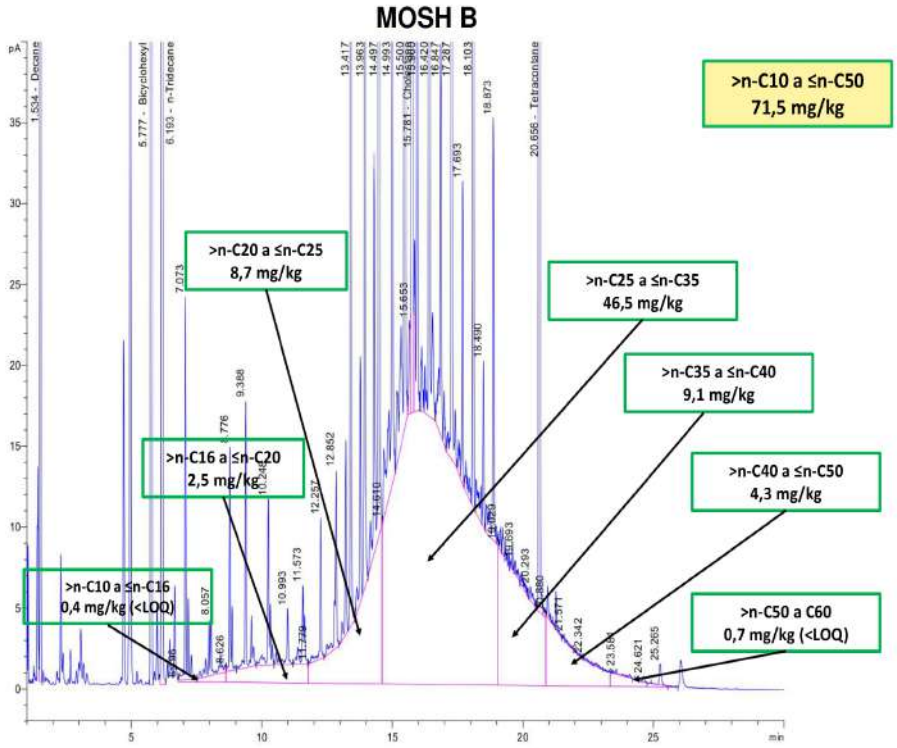
Bologna, 12th December 2019

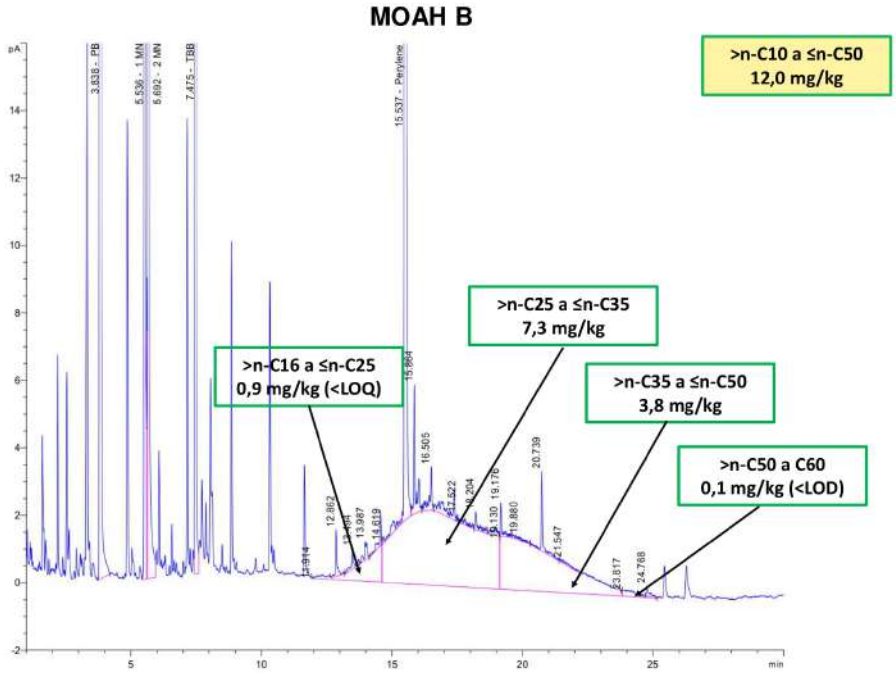
Extravirgin olive oils with high levels of MOSH and MOAH: example 3



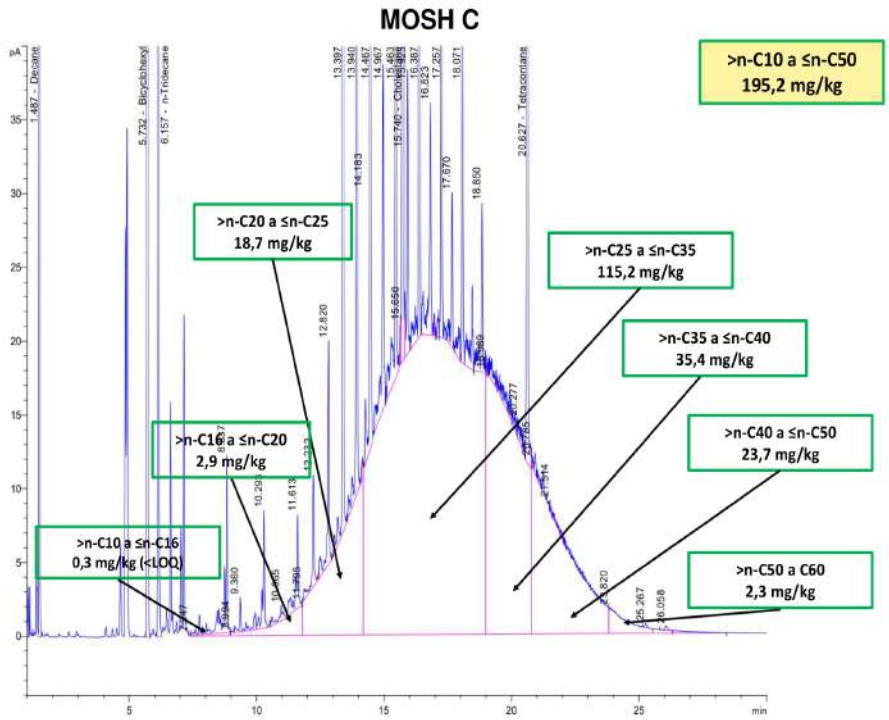


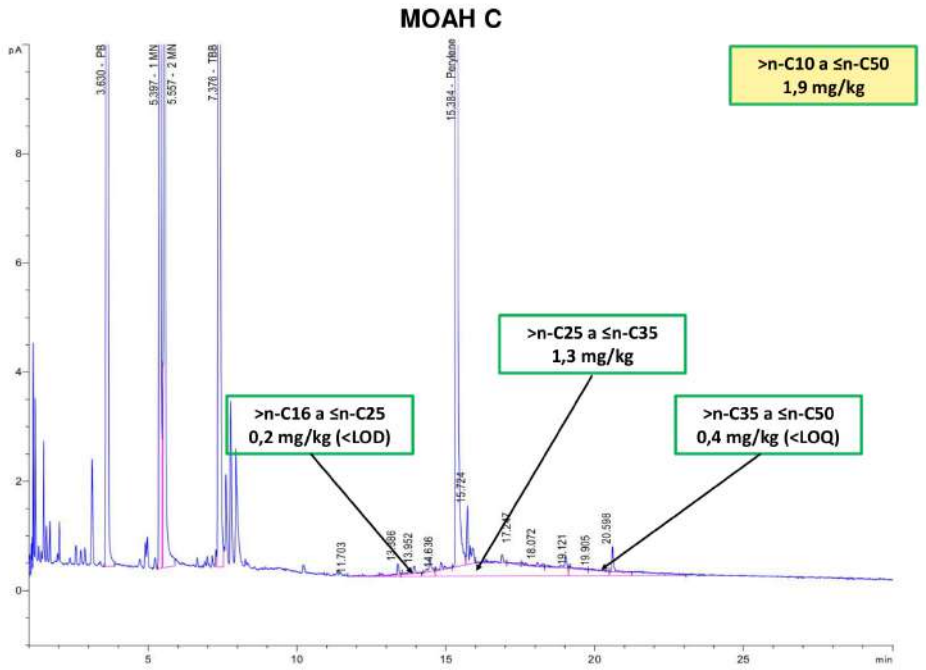
Extravirgin olive oils with high levels of MOSH and MOAH: example 4





Extravirgin olive oils with high level of MOSH but low MOAH level: example 5





METHOD PERFORMACE

- Validation for determining method characteristics and result assessment: Linearity, Limit of Quantification (LOQ), precision and recovery.
- The LOQ was assessed as lowest concentration limit at which precision parameters in terms of percentage of coefficient of variation are still verified. Precision parameters in terms of percentage of coefficient of variation: CV% lower than or equal to 20%.
- Precision was assessed in terms of strict repeatability carrying out a series of test repeated at 3 different levels of concentration for MOSH and at 3 levels for MOAH (in accordance with the availability of samples).
- Expanded measurement Uncertainty

JRC Technical Reports
- specifications

Table II Performance requirements for MOSH and MOAH analysis: maximum LOQ for each C-fraction (LOQ-max), target LOQ for each C-fraction (LOQ-t), acceptable ranges for recovery (R_{rec}) of mineral oil from samples, and intermediate precision

Categories	Associated foods #	LOQ - max [mg/kg]	LOQ -t [mg/kg]	R_{rec} [%]	interme-diate precision [%]
Dry, low-fat content (< 4% fat/oil)	bread and rolls; breakfast cereals; grains for human consumption; pasta, products derived from cereals	0.5	0.1	80 - 110	15
Higher fat/oil content (> 4% fat/oil)	fine bakery ware; confectionery (incl. chocolate) and cocoa; fish meat, fish products (canned fish); oilseeds; pulses; sausages; tree nuts	1	0.2	70 - 120	20
Fat/oils	animal fat (e.g. butter); vegetable oils	2	0.5	70 - 120	20
Paper and Board	Reporting only up to C_{35} (extraction optimised up to C_{35})	10	5	80 - 110	10

In some cases, a shift to another category may be necessary due to different fat content. This has to be stated and justified for each case.

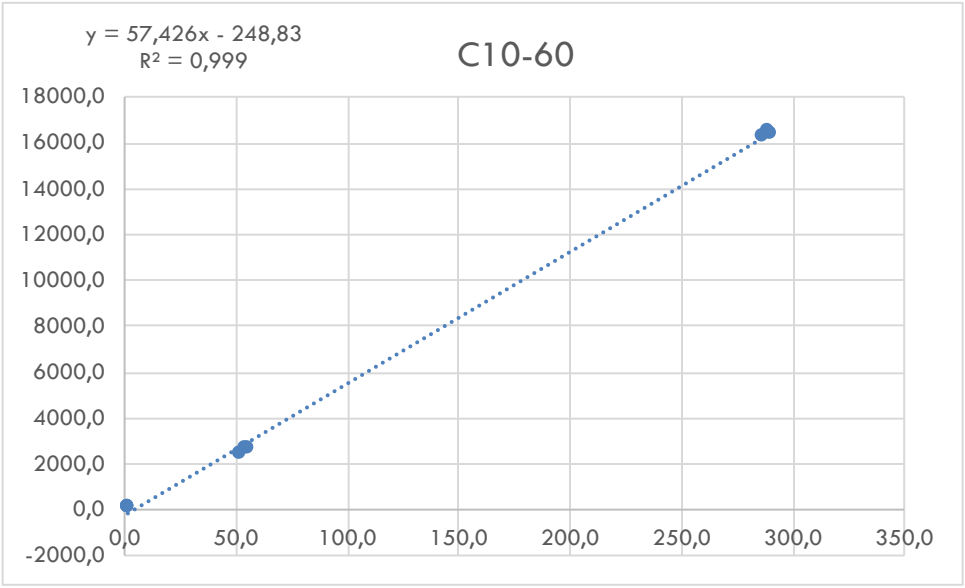
VALIDATION DATA AND RESULTS:

Linearity

Linearity

The tests were carried out on extra virgin olive oil with added MOSH (certified reference material) at 3 different concentrations of MOSH and performing the calculation both on MOSH C10-60.
The test was conducted at total MOSH concentrations of 1.4 mg / kg, 55 mg / kg, 290 mg / kg respectively; for each concentration level the determination was performed in triple.
A graph was then plotted by placing the theoretical concentration in the abscissa and the area of the bell of MOSH C10-60 in the ordinate.
The data were examined visually, the non-weighted linear regression was conducted, the regression coefficient of the best interpolating line was calculated and also the trend of residues was evaluated.

MOSH C10-60				
	C calcolata (mg/Kg)	Area	C teorica (mg/Kg)	E%
Livello1	288,7	16517,1	290,0	-0,46
	286,2	16231,0	290,0	-1,32
	290,1	16335,0	290,0	0,03
livello2	51,9	2412,5	54,8	-5,28
	55,1	2646,0	54,8	0,62
	54,3	2580,0	54,8	-0,84
livello3	1,5	64,7	1,4	4,08
	1,5	63,1	1,4	1,17
	1,5	100,1	1,4	6,86



C calcolata: concentrazione calcolata espressa in mg/Kg
C teorica: concentrazione teorica espressa in mg/Kg
E%: errore percentuale della concentrazione

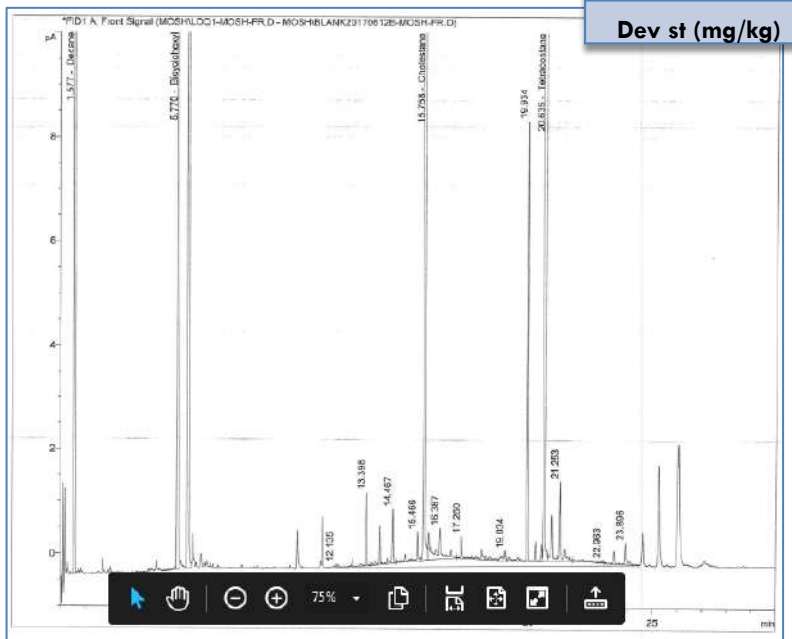
VALIDATION DATA AND RESULTS:
Limit of quantification (LOQ)

MOSH C10-35

The limit of quantification of the method was determined experimentally by performing 8 repeated tests on a "blank" extra virgin olive oil matrix added with a well-known MOSH mixture.

The tests were carried out at a theoretical concentration of 1.4 ppm on the total MOSH and at a relative concentration of MOSH C10-35 equal to 1 ppm. The calculations were conducted both on the data expressed as a total and on the fraction C10-35.

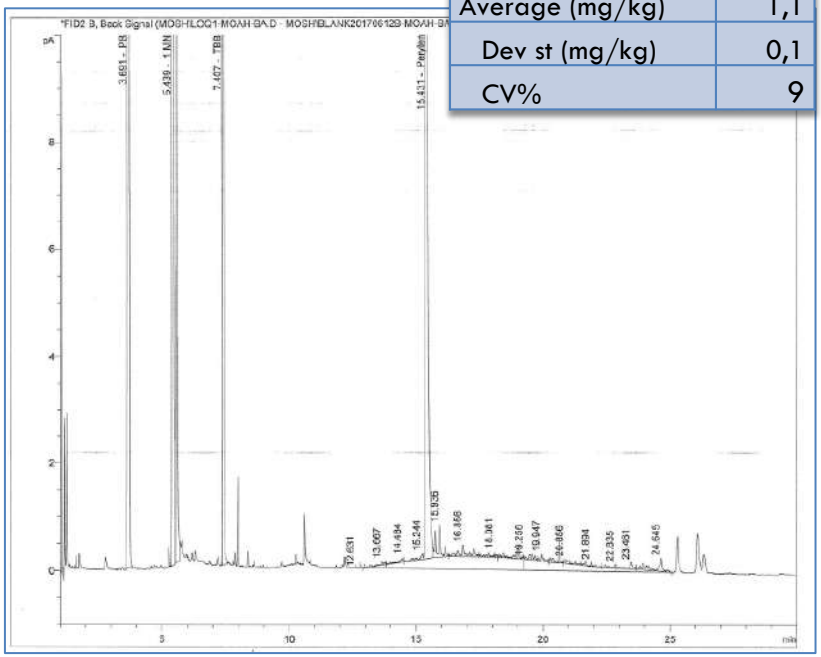
MOSH tot (average) mg/kg	1,50
MOSH C10-35 (average) mg/kg	1,0
CV% (MOSH C10-35)	9
Rec% medio (MOSH tot)	104,2
Dev st (mg/kg)	0,082



MOAH

Currently there are no reference materials for the aromatic mineral oil fraction (MOAH) commercially available, therefore the limit of quantification of the method was verified experimentally by performing 8 repeated tests on a real sample of extra virgin olive oil at a concentration approximately equal to 1 ppm .

MOAH C10-C35	
Average (mg/kg)	1,1
Dev st (mg/kg)	0,1
CV%	9



VALIDATION DATA AND RESULTS

Precision (CV%), accuracy (R%), uncertainty (U)

MOSH / MOAH - Precision was estimated by performing repeated tests on extra virgin olive oils at different levels of contamination.

At least 8 tests were carried out by two different operators for each level

MOSH	livello 1	livello 2	livello 3	livello 4
Average (mg/kg)	0,95	5,2	45,4	199
Sr	0,082	0,226	1,14	7,91
CV %	8,6	4,4	2,35	3,98
n	8	8	8	8

MOAH	livello 1	livello 2	livello 3
Average (mg/kg)	1,14	6,44	30,3
Sr	0,092	0,233	1,92
CV %	8,05	3,61	6,32
n	8	8	8

MOSH - Recovery was estimated by performing 8 repeated tests on a synthetic sample at a theoretical MOSH concentration equal to 54.8 mg / kg consisting of extra virgin olive oil added with a reference material mixture of saturated mineral oils.

Prova N	x_s (mg/Kg)	Rec%
1	51,9	95
2	55,1	101
3	54,3	99
4	55,2	101
5	54,1	99
6	54,1	99
7	55,2	101
8	52	95
Rec % Medio		**99**
Average (mg/Kg)		**54,76**

Uncertainty - Analytical results are reported as x±U (where x is analytical result and U is expanded uncertainty with a coverege factor k=2 evaluated at level of confidence 95%). A U value of 40% was obtained for an extravirgin olive oil fortified with 3 levels of MOSH.

The validation was also carried out on refined Olive Oils to verify the epoxidation phase. We obtained compliant results.

Accuracy evaluation by partecipation to a proficiency test

Method ring test P1815-MRT

MOSH/MOAH in rapeseed oil and chocolate muesli

June - August 2018

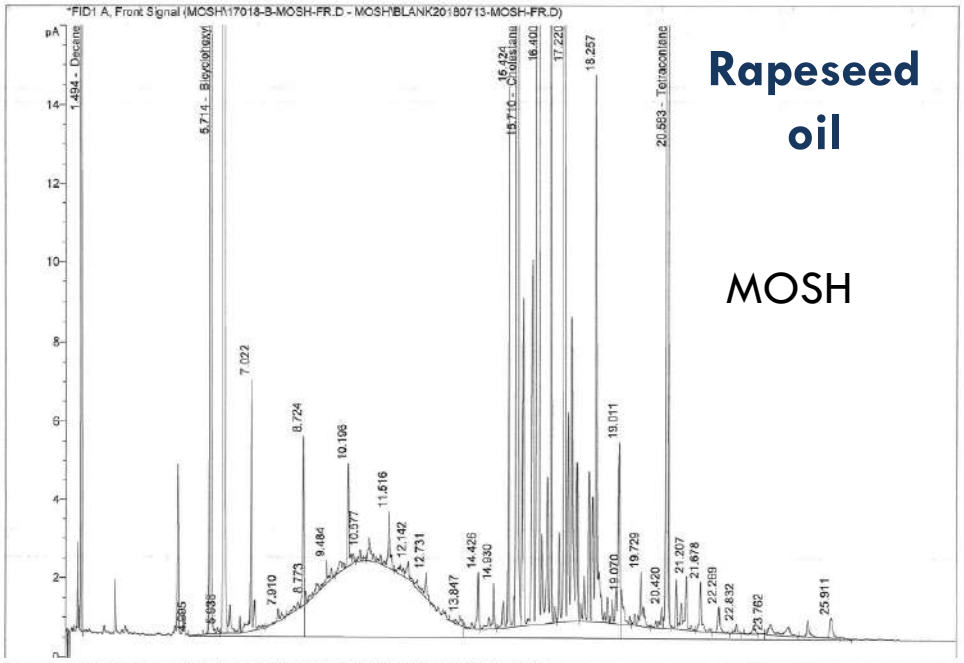
CHEMISERVICE SRL

Monopoli, Italy

Laboratory code 14

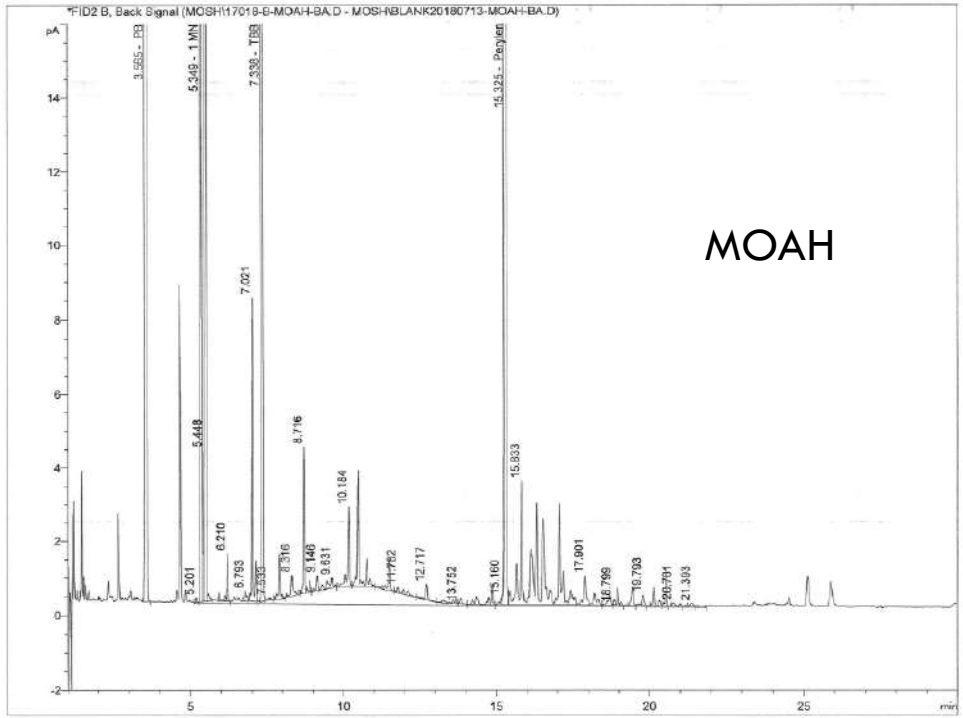
Matrix	Parameter	Result [mg/kg]	z-score	Comparability criterion passed	Result in % of the spiked level	Trueness criterion passed
Oil	Total MOSH	11.0	2.0	yes	110	yes
	Total MOAH	3.0	0.0	yes	79	yes
Muesli	Total MOSH	15.7	-1.4	yes	-	-
	Total MOAH	2.3	-1.4	yes	-	-

Hamburg, 10 October 2018



Rapeseed oil

MOSH



MOAH

Accuracy evaluation by partecipation to a proficiency test

Method ring test P1815-MRT

MOSH/MOAH in rapeseed oil and chocolate muesli

June - August 2018

CHEMISERVICE SRL

Monopoli, Italy

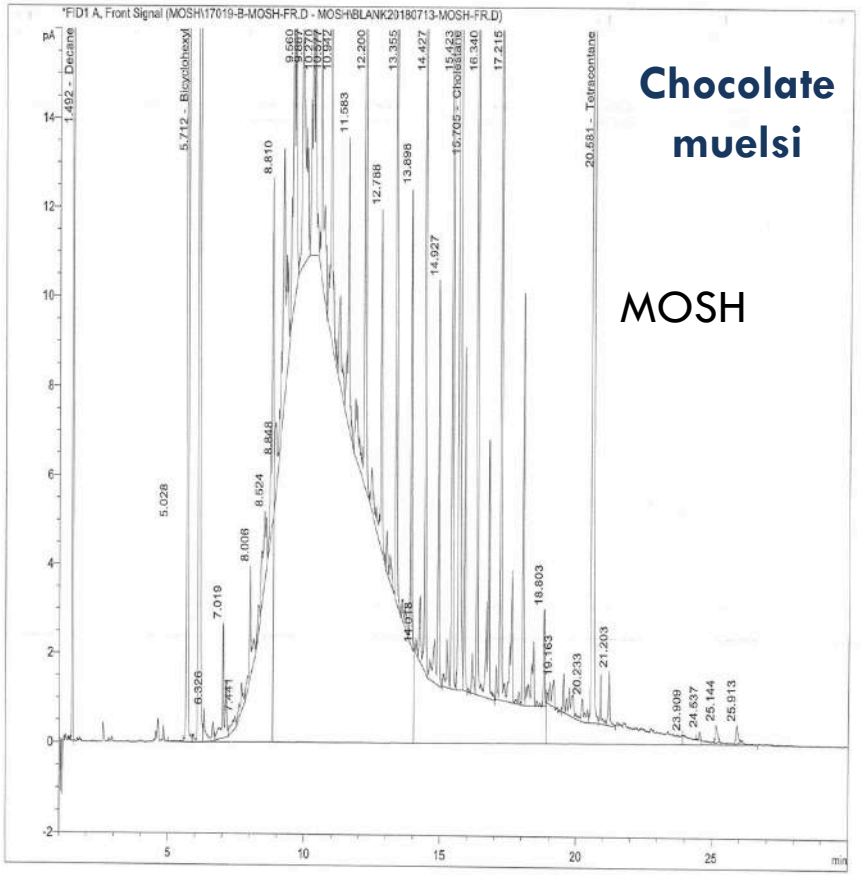
Laboratory code 14

Matrix	Parameter	Result [mg/kg]	z-score	Comparability criterion passed	Result in % of the spiked level	Trueness criterion passed
Oil	Total MOSH	11.0	2.0	yes	110	yes
	Total MOAH	3.0	0.0	yes	79	yes
Muesli	Total MOSH	15.7	-1.4	yes	-	-
	Total MOAH	2.3	-1.4	yes	-	-

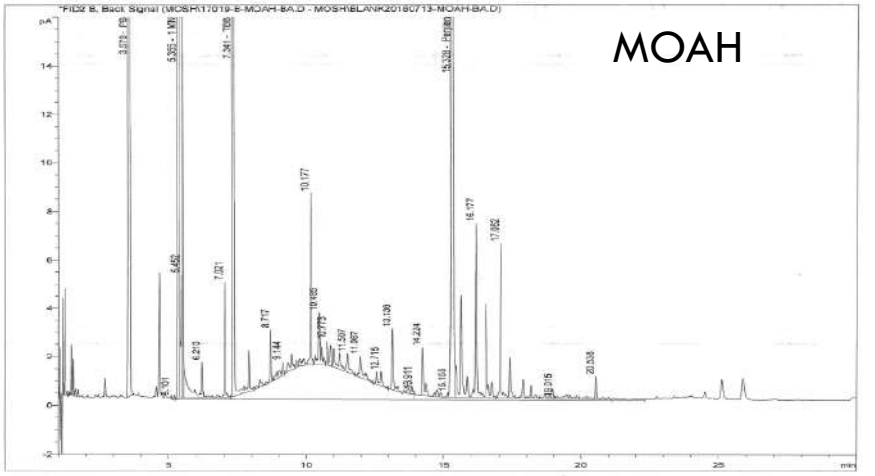
Hamburg, 10 October 2018

Chocolate muelsi

MOSH

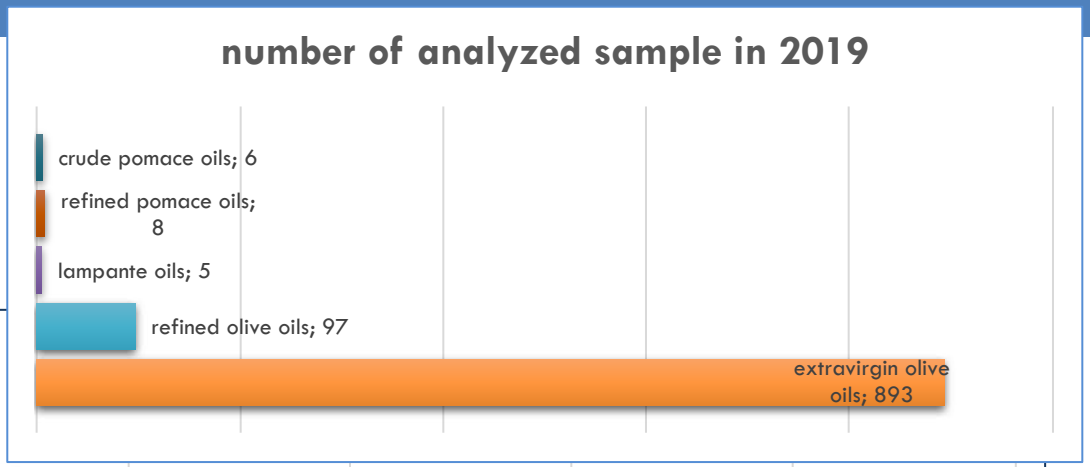


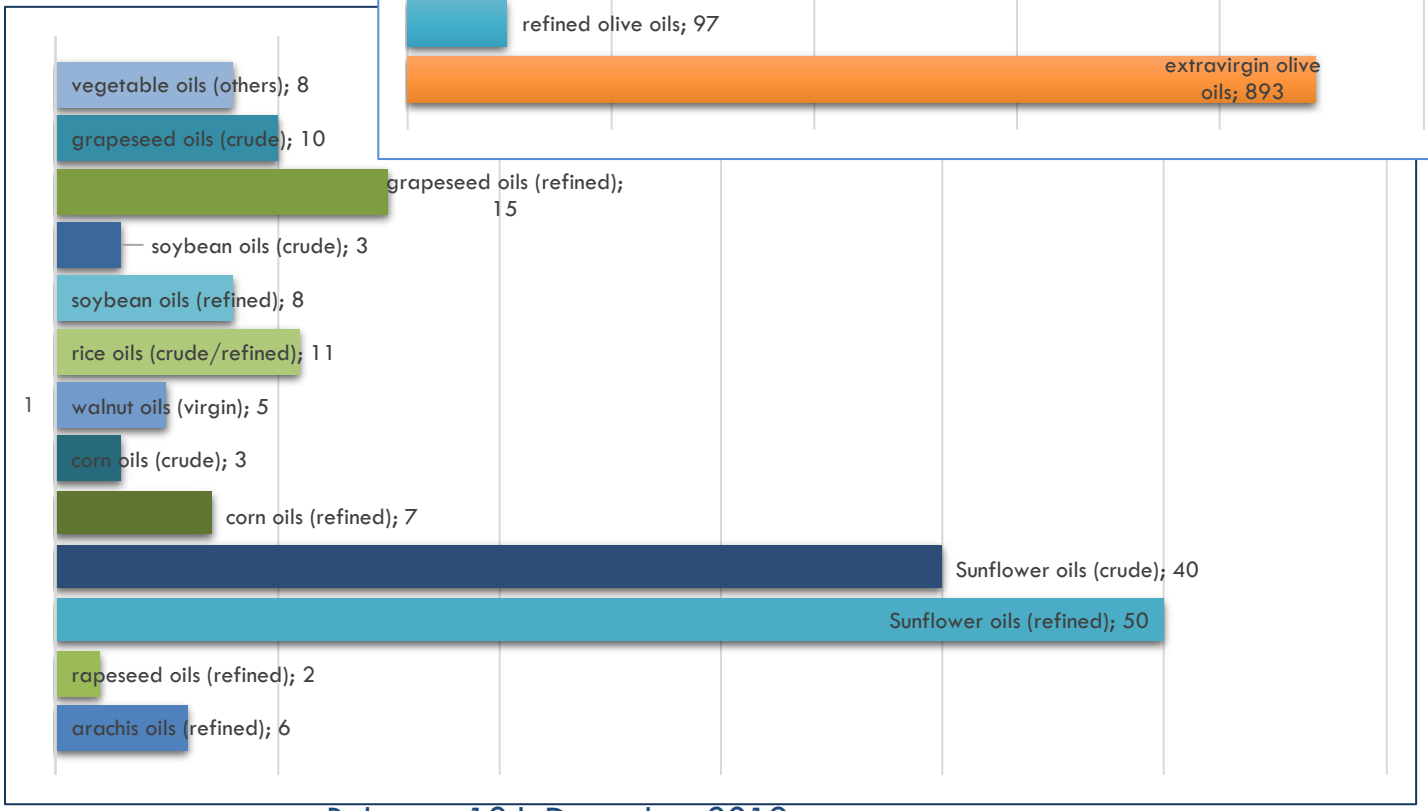
MOAH



2019: Type of oils and fats analyzed

	% on total sample (1302)
extravirgin olive oils	**89**
refined olive oils	10
lampante oils	0
refined pomace oils	1
crude pomace oils	1
olive oils (total)	**77**
arachis oils (refined)	4
rapeseed oils (refined)	1
Sunflower oils (refined)	30
Sunflower oils (crude)	24
corn oils (refined)	4
corn oils (crude)	2
walnut oils (virgin)	3
rice oils (crude/refined)	7
soybean oils (refined)	5
soybean oils (crude)	2
grapeseed oils (refined)	9
grapeseed oils (crude)	6
vegetable oils (others)	5
vegetable oils (total)	**13**



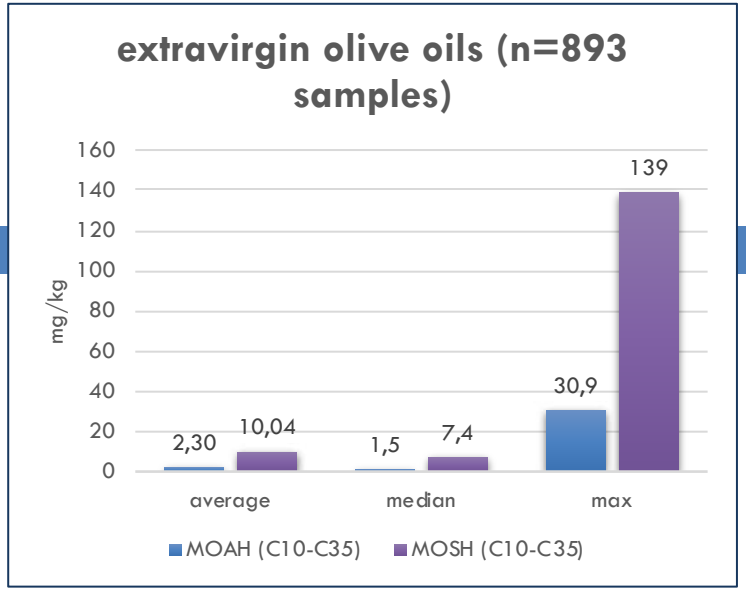


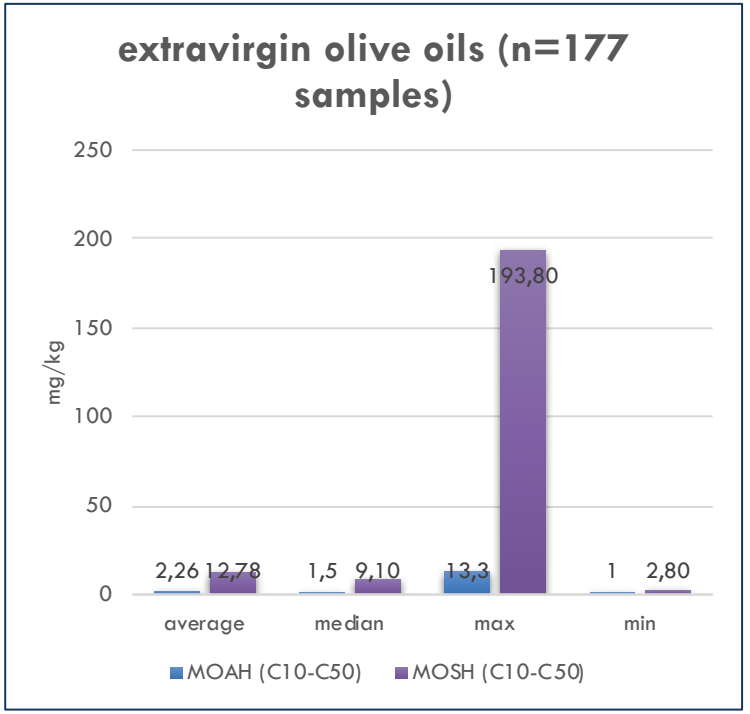
Extra virgin olive oils

MOAH (C10-C35)				MOSH (C10-C35)		
	num	%			num	%
totali	893			totali	893	
< LOQ	**563**	**63**		< LOQ	2	0
>= LOQ	330	37		>= LOQ	891	100
				LOQ- 2 mg/kg	31	3,5
				>2 - <=10 mg/kg	**614**	**68,8**
				>10 - <=50 mg/kg	231	25,9
				> 50 mg/kg	15	1,7

all samples of extra virgin olive oil show the highest concentration in MOSH fraction included between C25-C35, MOSH fraction (C10-C16) is never present: it is confirmed by the chromatographic profile of the analyzed oils

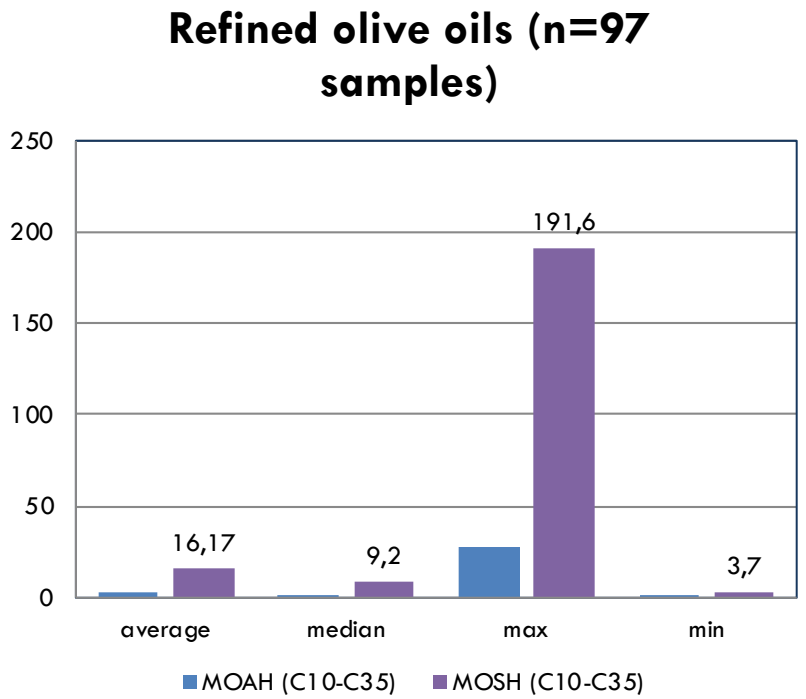
MOAH (C10-C50)				MOSH (C10-C50)		
	num	%			num	%
totali	177			totali	177	
< LOQ	78	44		< LOQ	0	0
>= LOQ	**99**	**56**		>= LOQ	177	100
				LOQ- 2 mg/kg	1	1
				>2 - <=10 mg/kg	**96**	**54**
				>10 - <=50 mg/kg	75	42
				> 50 mg/kg	5	3



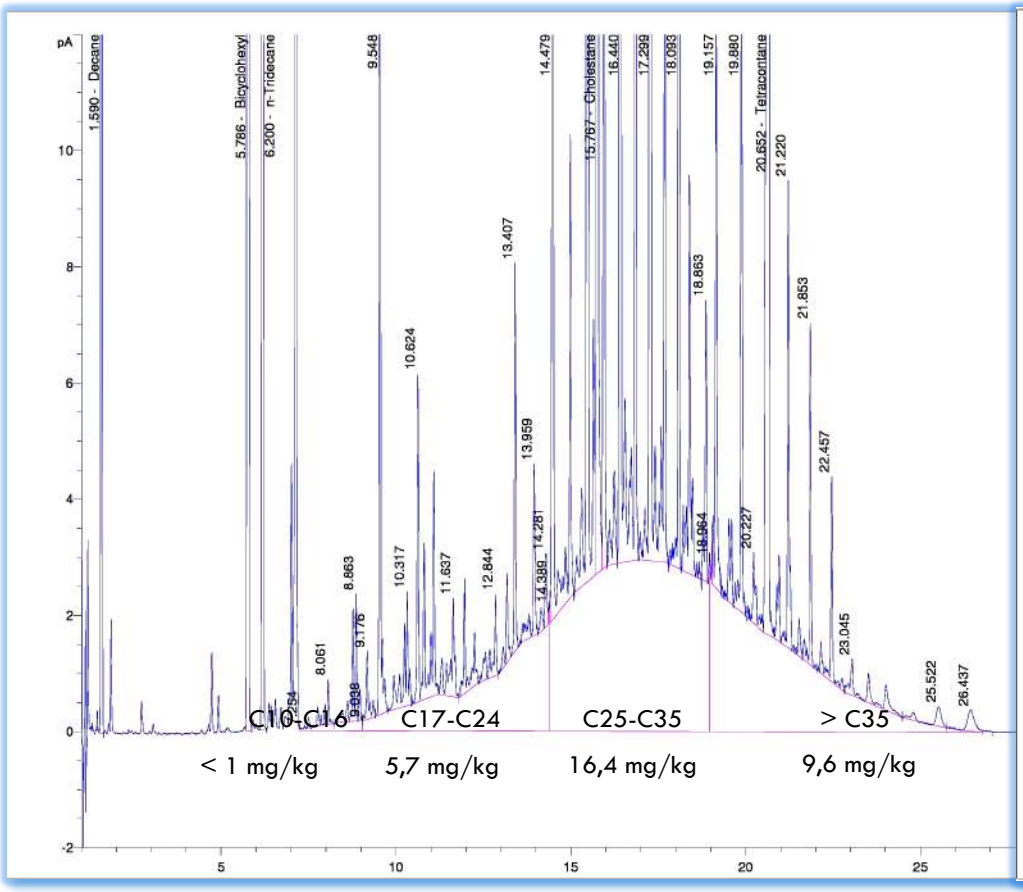


Refined olive oils

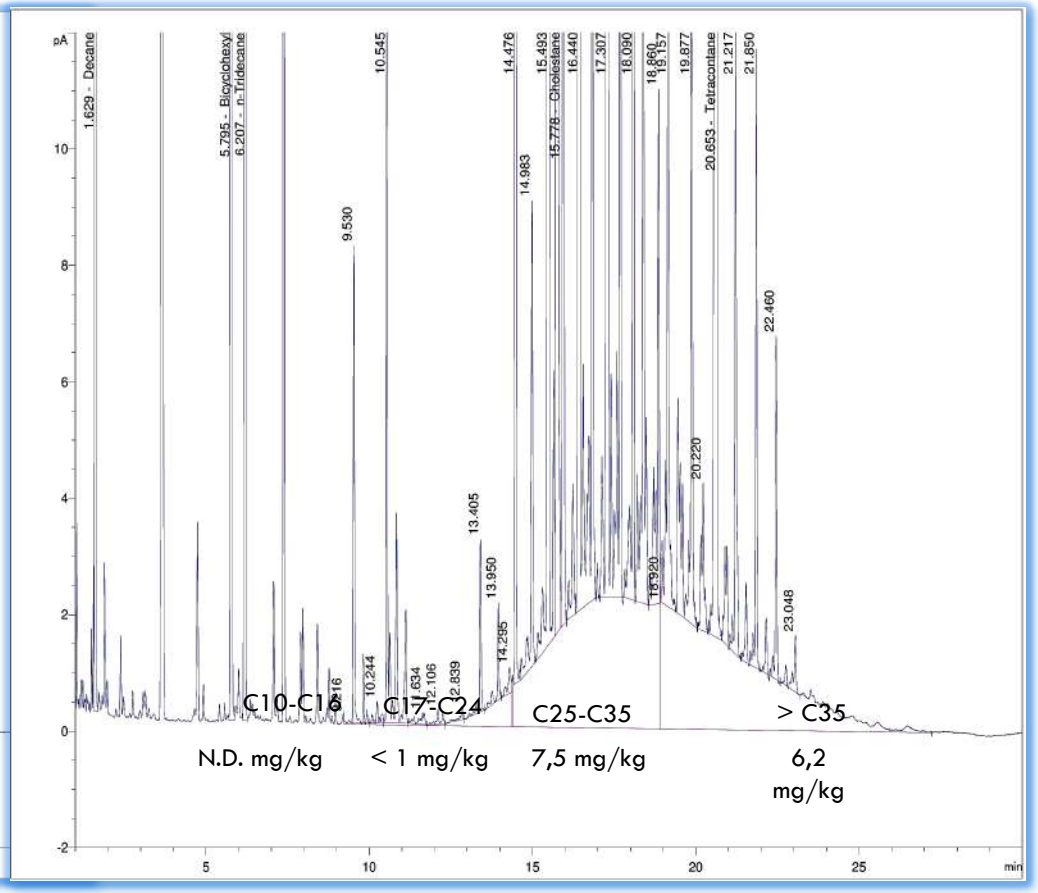
refined olive oils						
MOAH (C10-C35)				MOSH (C10-C35)		
	num	%			num	%
totali	97			totali	97	
< LOQ	44	45		< LOQ	0	0
>= LOQ	**53**	**55**		>= LOQ	97	100
				LOQ- 2 mg/kg	1	1
				>2 - <=10 mg/kg	**58**	**60**
				>10 - <=50 mg/kg	34	35
				> 50 mg/kg	4	4



2019	Number of sample	MOSH (C10-C35) media (mg/kg)
Sunflower oils (refined)	30	5,2
Sunflower oils (crude)	25	10,4
grapeseed oils (refined)	9	26,1
grapeseed oils (crude)	6	45,1



MOSH of a crude sunflower oil



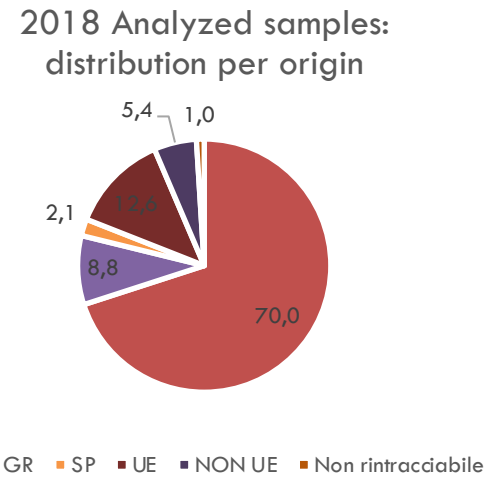
MOSH of refined sunflower oil

2018: Extravirgin olive oils analyzed, classified by origin

		num	% (su totale campioni analizzati)
	Totale dati	1214	
per origin	**IT**	**850**	**70,0**
	GR	107	8,8
	SP	26	2,1
	UE	153	12,6
	NON UE	66	5,4
	Non rintracciabile	12	1,0

	MOSH (C10-C35)					
	totale campioni	IT	GR	SP	UE	NON UE
num dati > LOQ	1209	850	107	26	152	64
media (mg/kg)	10,21	9,69	17,77	9,63	10,67	4,37
mediana (mg/kg)	7,5	7,55	13,8	6,1	7,6	2,45
min (mg/kg)	1	1	1,7	2	1	1
max (mg/kg)	86,3	64	74,8	57,9	86,7	25,5

	MOAH (C10-C35)						
	totale campioni	IT	GR	SP	UE	NON UE	
num dati > LOQ	327	213	54	5	51	3	
media (mg/kg)	3,00	2,50	3,86	4,90	3,50		
mediana (mg/kg)	2,1	1,90	2,90	4,9	2,3		
min (mg/kg)	1	1,00	1,10	1,5	1		
max (mg/kg)	20,9	13,50	12,80	11,8	11,6	20,9	ISRAELE



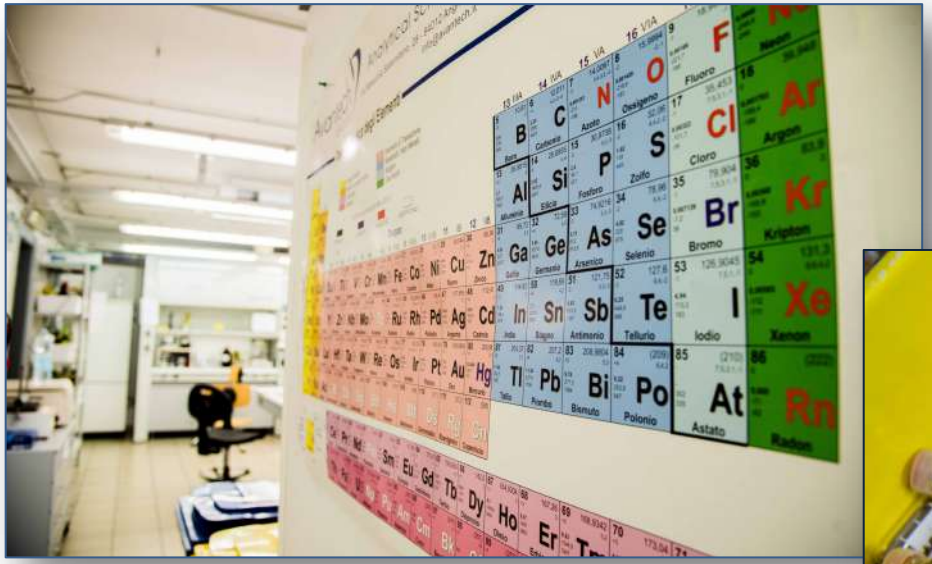
REMARKS and CONCLUSIONS

- The laboratory developed an off-line method; its validation produced good results according to JRC guidelines; experienced operators are required for a correct interpretation of the GC chromatograms.
- The off-line method returns results comparable with the on-line HPLC-GC method (verification by proficiency test); Suitable for routine, especially for virgin olive oils (number of analyzed sample per day 12-14).
- Almost half of analyzed extra virgin olive oils do not correspond to the strictest supply specifications provided by our customers, mostly for MOAH; on the basis of the experience of our laboratory, most analyzed oil samples show environmental contaminations.
- An almost always constant relationship was observed between mosh and moah (both in concentration and in distribution of fractions), when this was not verified it was necessary to make further assessments on the type of contamination.
- For the future, we have to complete the validation of the other procedures applied to different kind of foodstuffs.

References

- ISO 17780:2015 Animal and vegetable fats and oils -- Determination of aliphatic hydrocarbons in vegetable oils
- ISO Draft - Animal and vegetable fats and oils — Determination of aliphatic hydrocarbons in vegetable oils mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with online-HPLC-GC-FID - 2014-03-06
- Koni Grob. Mineral oils in vegetable oils: tools for the analysis of the saturated and the aromatic hydrocarbons.
- Moret S, Barp L, Grob K, Conte L (2011): Optimised off-line SPE GC-FID method for determination of mineral oil satured hydrocarbons (MOSH) in vegetable oils – Food Chemistry 129 (2011) 1898-1903;
- Scientific Opinion on Mineral Oil Hydrocarbons in Food, EFSA Panel on Contaminants in the Food Chain (CONTAM), EFSA Journal 2012;10(6):2704
- Optimised off-line SPE–GC–FID method for the determination of mineral oil saturated hydrocarbons (MOSH) in vegetable oils Sabrina Moret, Laura Barp a, Konrad Grob b, Lanfranco S. Conte. *Food Chemistry 129 (2011) 1898–1903*
- JRC Technical report - Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact Materials (2019)

chemiservice
LABORATORIO DI ANALISI CHIMICHE E MICROBIOLOGICHE



Thanks for your kind attention

Bologna, 12th December 2019