DETECTION OF ADULTERATION

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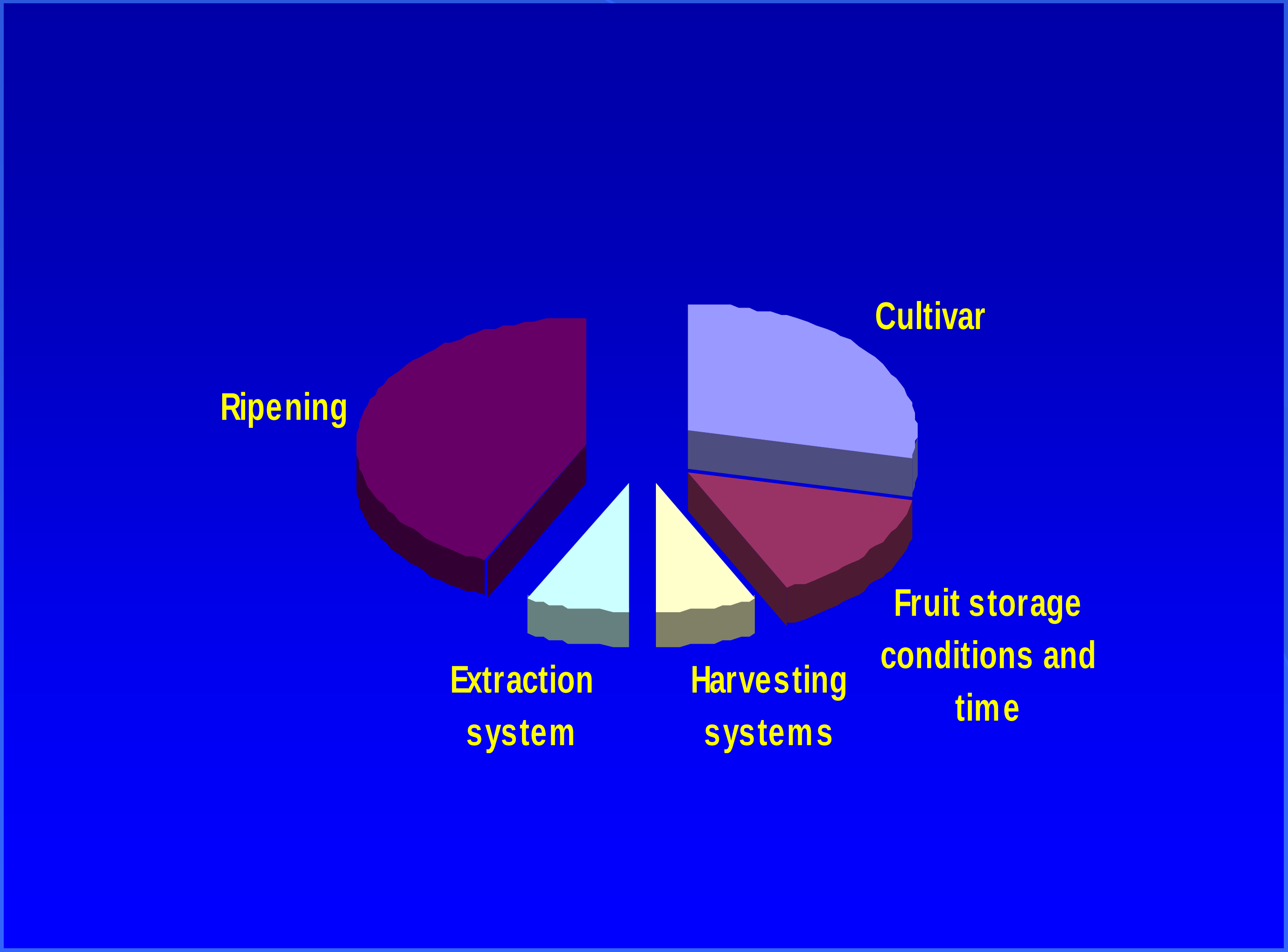
WHAT WE WILL SPEAK ABOUT

- GENERAL ASPECTS OF QUALITY
- NORMATIVE SOURCES
- SOME HISTORICAL ASPECTS OF QUALITY CONTROL
- ACTUAL SITUATION AND RECENT ADVANCES
- NEW EXPERIMENTAL APPROACHES

SOME FACTORS INFLUENCING OLIVE OILS QUALITY



GENUINENESS CHARACTERISTICS

- FATTY ACIDS COMPOSITIONS
- Δ ECN 42
- STEROLS COMPOSITION
- STERENES
- TRANS ISOMERS OF FATTY ACIDS
- WAXES
- SPECTROPHOTOMETRIC UV

- QUALITY CHARACTERISTICS
- FREE ACIDITY
- PEROXYDES INDEX
- PANEL TEST

NORMATIVE SOURCES CONCERNING OLIVE OILS

EUROPEAN UNION
REGULATION CE 136/66, 2677/85, 1915/87, 2568/91, 1683/92,1513/98, 455/01, 796/02, 1019/02 , 1989/03

INTERNATIONAL OLIVE OIL COUNCIL (IOOC)
TRADE AGREEMENT - NORM
(REVISED ONCE A YEAR)

CODEX ALIMENTARIUS (FAO OMS)
TRADE AGREEMENT - NORM
(REVISED ONCE EVERY THREE YEARS)

1. VIRGIN OLIVE OILS

Oils extracted by olive tree fruit, by means of mechanical processes or other physical processes, only, in such conditions not to modify oil modifications, None treatment other than washing, sedimentation, centrifugation and filtration is admitted. Oils extracted by means of solvents or by the use of technological coadiuvants with chemicla or biochemical action or by means of re-esterification do not belong to this class, as well as those obtained by blend of different oils. Virgin oils undergo to the following further classification:

a) Extra virgin olive oil

Virgin olive oil whose free acidity , as oleic acid, do not exceed 0,8 g per 100 g and whose other characteristics laid down to those fixed for this category

b) Virgin olive oil

Virgin olive oil whose free acidity , as oleic acid, do not exceed 2,0 g per 100 g and whose other characteristics laid down to those fixed for this category;

c) "Lampante" olive oil

Virgin olive oil whose free acidity , as oleic acid, exceed 2,0 g per 100 g and whose other characteristics laid down to those fixed for this category;

2. REFINED OLIVE OIL

Olive oil obtained by refining of virgin olive oils, whose free acidity , as oleic acid, do not exceed 0,3 g per 100 g and whose other characteristics laid down to those fixed for this category.

3. OLIVE OIL — BLEND OF REFINED OLIVE OIL AND VIRGIN OLIE OILS

Olive oil obtained by blend of refined olive oil with virgin olive oils other than lampante whose free acidity , as oleic acid, do not exceed 1,0 g per 100 g and whose other characteristics laid down to those fixed for this category.

4. RAW OLIVE POMACE OIL

Oil extracted by olive pomace by means of solvents or by physical processes, or oils whose composition correspond to the one of lampante oil, except for some parameters. Oil obtained by re esterification or blend wirh different oils o not belong to this category. Characteristics must laid down to those listed for this class of oils.

5. REFINED OLIVE POMACE OIL

Oil obtained by refining of raw olive pomace oil, whose maximum free acidity do not exceed 0,3 g per 100 g as oleic acid and whose other characteristics laid down to those fixed for this category

6. OLIVE POMACE OILS

Oil obtained by blend of refined olive pomace oils with virgin olive oil other than lampante oil, whose maximum free acidity do not exceed 1 g per 100 g and whose other characteristics laid down to those fixed for this category

THE EARLIER APPROACHES

The "Chemistry of indexes" (before 1953)

- Iodine index
- Wood's light fluoroscence
- Acidity index
- Melting point
- Refrective index
- Critical temperature of solubility in alcohol
- Saponification index
- Esters index
- Volatile acids index
- Acetyl index

Iodine index ⇨ Fatty acid composition (GLC)

Fatty acids composition depends on botanical source

Blend with mineral oils do not lead to any modification of fatty acid composition ('60)

Evaluation of amount of unsaponifiable matter

Chemical synthesis of triacylglycerols

Recovery of fatty acids from refining

Research of trans isomers by Infra Red Spectrometry (Quantitative determination in CS_2 solution)

Seed oils fractioned to get fatty acid composition similar to olive oils

Genetic modified seed crops with fatty acids similar to olive oils:

1st Safflower oil (Knowles 1967): 70 % oleic

Then Sunflower oils 70% oleic

….but genetic improvement can modify fatty acids and not sterols fraction

…from whole unsaponifiable quantitative evaluation to its fractionation in single classes

Most studied class: sterols (GC, GC-MS) by means of packed colums

ACTUAL SITUATION

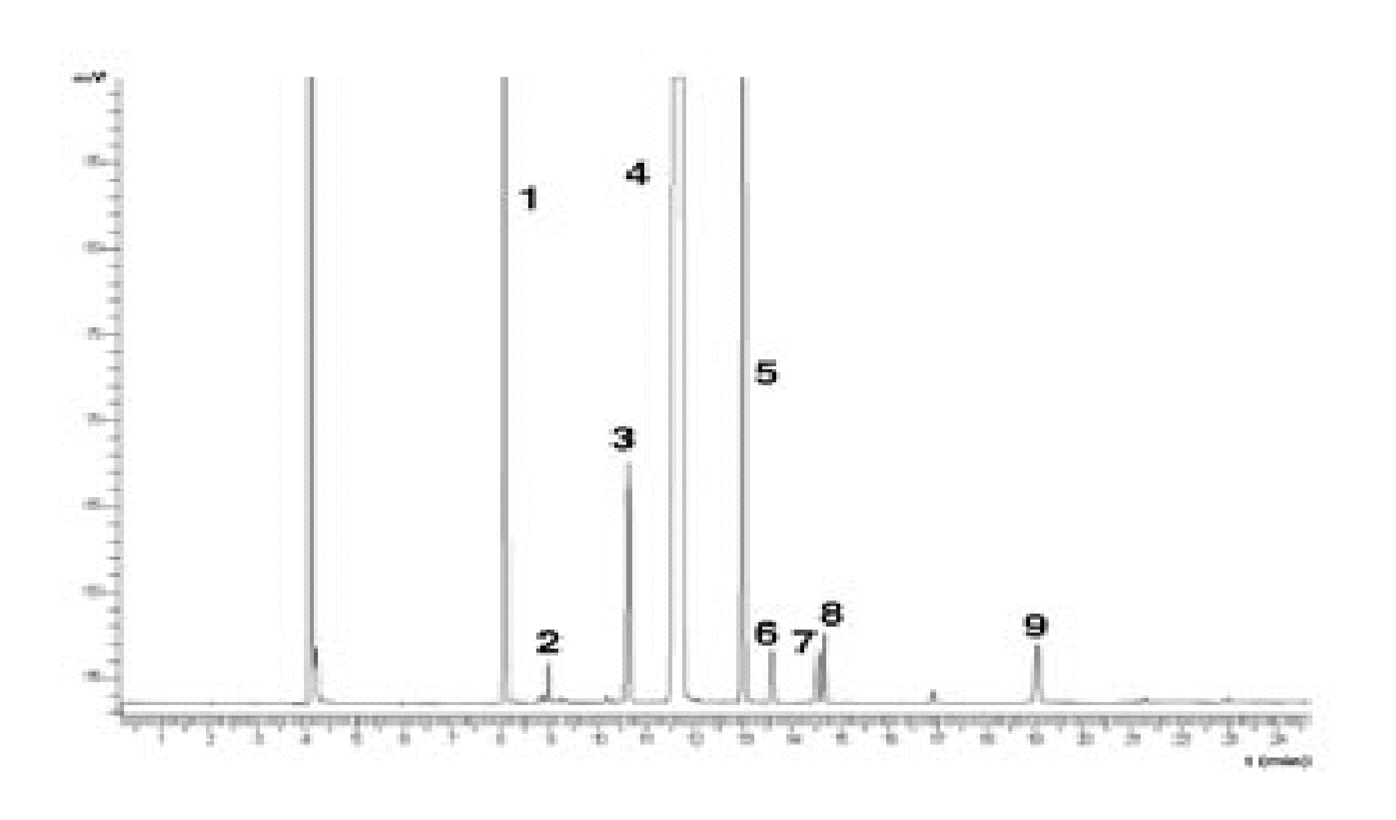
AND

RECENT ADVANCES

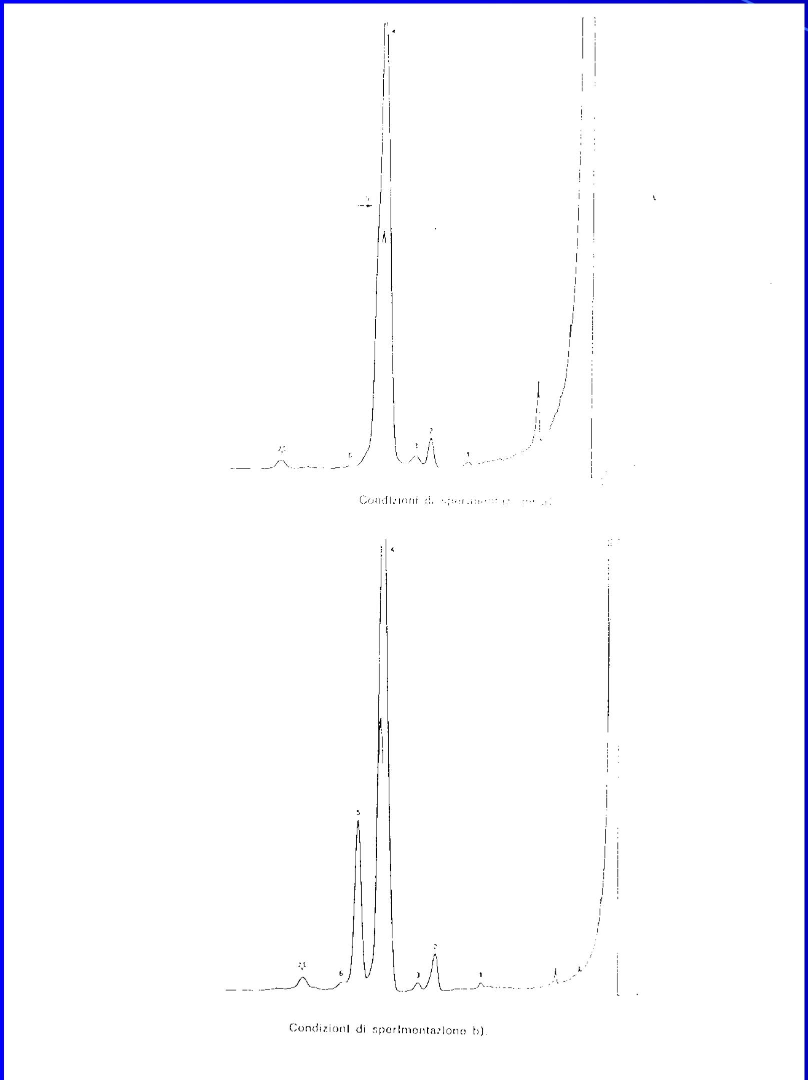
Quality and purity parameters of virgin olive oils	**CEE 2568/91**	**CEE 1989/2003**	**COI**	**CODEX 2003**
Free acidity (% , expressed as oleic acid)	≤ 1,0	≤ 0,8	≤ 1,0	≤ 0,8
Peroxides Value (meq O_2/kg olio)	≤ 20	≤ 20	≤ 20	≤ 20
K232	**≤ 2,50**	**≤ 2,40**	**-**	**Not mandatory**
K270	**≤ 0,20**	**≤ 0,22**	**≤ 0,25**	**≤ 0,22**
ΔK	≤ 0,01	≤ 0,01	≤ 0,01	≤ 0,01
C14:0 (%)	≤ 0,05	≤ 0,05	≤ 0,05	0,0 - 0,1
C16:0 (%)	-	7,5 – 20,0	7,5 – 20,0	7,5 - 20,0
C16:1 (%)	-	0,3 - 3,5	0,3 - 3,5	0,3 - 3,5
C17:0 (%)	-	≤ 0,3	≤ 0,3	≤ 0,5
C17:1 (%)	-	≤ 0,3	≤ 0,3	≤ 0,6
C18:0 (%)	-	0,5 - 5,0	0,5 - 5,0	0,5 - 5,0
C18:1 (%)	-	55,0 - 83,0	55,0 - 83,0	55,0 - 83,0
C18:2 (%)	-	3,5 – 21,0	3,5 – 21,0	3,5 - 21,0
C18:3 (%)	≤ 0,9	≤ 0,9	≤ 0,9	≤ 1,0
C20:0 (%)	≤ 0,6	≤ 0,6	≤ 0,6	≤ 0,8
C20:1 (%)	≤ 0,4	≤ 0,4	≤ 0,4	-
C22:0 (%)	≤ 0,2	≤ 0,2	≤ 0,2	≤ 0,2
C24:0 (%)	-	≤ 0,2	≤ 0,2	≤ 1,0
Cholesterol (%)	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5
Brassicasterol (%)	≤ 0,1	≤ 0,1	≤ 0,1	≤ 0,1
Campesterol (%)	≤ 4,0	≤ 4,0	≤ 4,0	≤ 4,0
Stigmasterol (%)	< CAMPE	< CAMPE	< CAMPE	< CAMPE
Δ - 7 –stigmastenol (%)	≤ 0,5	≤ 0,5	≤ 0,5	
Betasitosterol + Δ-5avenaster .+ Δ 5,23stigmastadienol + clerosterol + sitostanol + Δ5,24stigmastadienol (%)	≥93,0	≥93,0	≥ 93,0	≥ 93,0
Total Sterol content (ppm)	≥ 1000	≥ 1000	≥ 1000	≥ 1000
Waxes (ppm)	≤ 250	≤ 250	≤ 350	≤ 300
Saturated fatty acids in 2position of triglyceride(%)	**≤ 1,3**	**≤ 1,3**	**≤ 1,5**	**≤ 1,5**
Eritrodiol + uvaol (%)	≤ 4,5	≤ 4,5	≤ 4,5	≤ 4,5
ECN 42 (HPLC – calculated)	≤ 0.2-	≤ 0.2-	≤ 0,2	≤ 0,4
Stigmastadienes (ppm)	**≤ 0,15**	**≤ 0,15**	**≤ 0,10**	
R1	**-**	**-**	**> 15**	
C18:1 t(%)	≤ 0,05	≤ 0,05	≤ 0,05	
C18:2 t+ C18:3 t(%)	≤ 0,05	≤ 0,05	≤ 0,05	
Water + volatile substances (% m/m)	-	-	≤ 0,2	≤ 0,2
Flame Point	-	-	-	≥ 120°C
Iron (ppm)	-	-	≤ 3,0	≤ 5,0
Cupper (ppm)	-	-	≤ 0,1	≤ 0,4
Lead (ppm)	-	-	-	≤ 0,1
Arsenic (ppm)	-	-	-	≤ 0,1
Halogenated Solvents (ppm) (each)	≤ 0,10	≤ 0,10	≤ 0,1	≤ 0,1
Halogenated Solvents (ppm) (sum)	≤ 0,20	≤ 0,20	≤ 0,2	≤ 0,2
Saponification Number	-	-	-	184 - 196
Iodine number	-	-	-	75 - 94
Unsaponifiable matter (g/kg)	-	-	-	15
Bellier index	-	-	-	≤ 17

Analytical Parameter	**EEC Limit (2568/91)**	**Meaning**
Free Acidity (% as oleic acid)	1,0 (0,8)	Depends on triacylglycerols hydrolisys: it mainly depends on olive fruits quality
Peroxides Vale (meq O_2/kg oil)	20	PV is related to oil oxidative status, it depends on storage conditions
K232		Dealing with absorbance of conjugated dienes, these could be formed both by refining processes and by oxidative phenomena
K270	0,20	Dealing with absorbance of conjugated trienes, these could be formed both by refining processes and by oxidative phenomena
K	0,01	Differential absorbance at 270 nm rispetto alla curva di assorbanza nell' UV, an exceeding value depends on the presence of refined oils
C14:0 (%)	0,05	Higher values when seed oils are present
C18:3 (%)	0,9	Higher values when seed oils are present, mainly soja and rapeseed
C20:0 (%)	0,6	Higher values when seed oils are present, mainly soja, peanut and rapeseed
C20:1 (%)	0,4	Higher values when seed oils are present, mainly soja, peanut and rapeseed
C22:0 (%)	0,2	Higher values when seed oils are present, mainly soja, peanut and rapeseed
C24:0 (%)	0,2-	Higher values when peanut oil is present

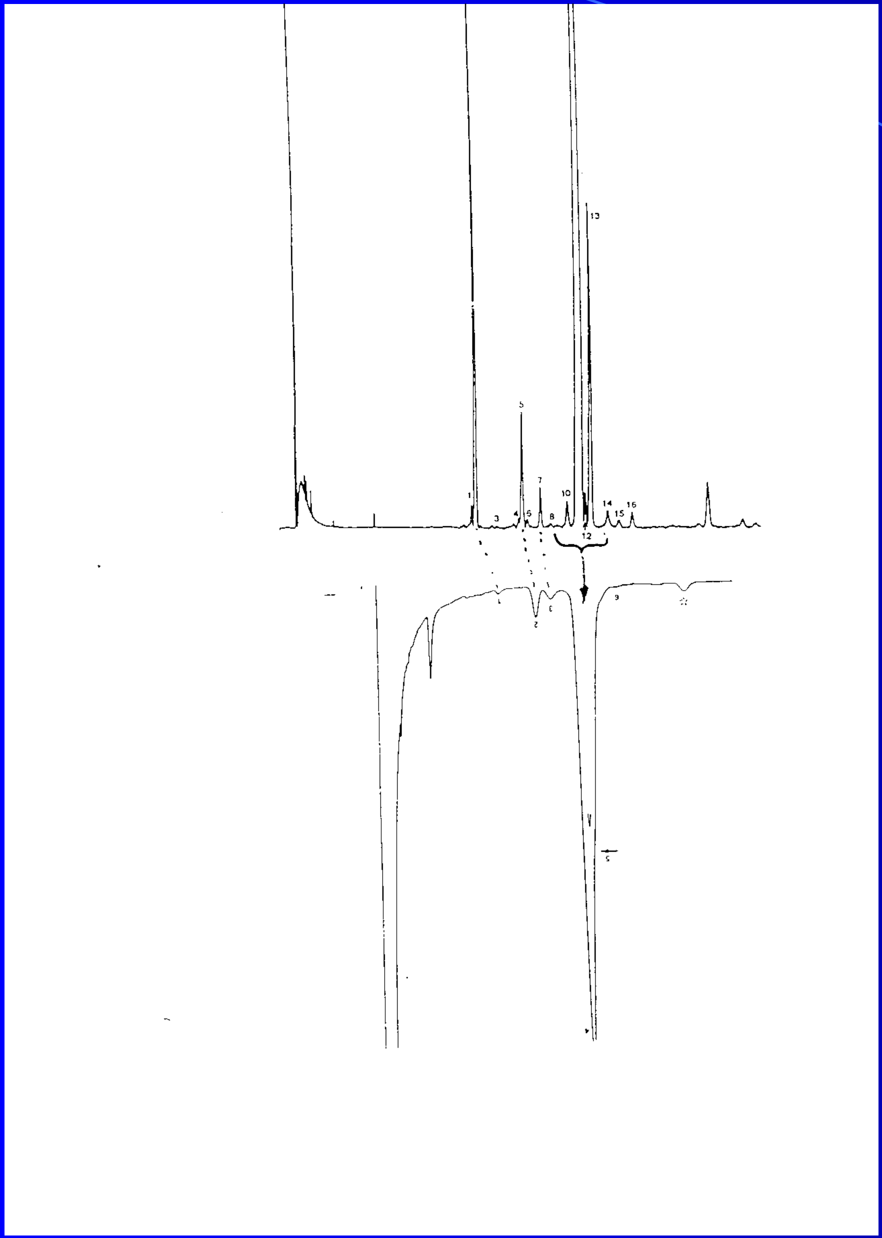
Cholesterol (%)	0,5	Higher values when extraneous fats and oils are blended (vegetable fats as fractionated palm)
Brassicasterol (%)	0,1	Higher values when *Brassicaceae* oils are blended soia o colza
Campesterol(%)	4,0	> 4,0 % when seeds oils are blended
Stigmasterol (%)	< CAMPE	In seeds oils, campesterol and stigmasterol generally are present in compaable amounts
Δ-7–stigmastenol (%)	0,5	Higher values when *Compositae* oils (sunflower and safflower) even if high oleic type are blended
Betasitosterol + Δ -5avenasterol .+ Δ 5,23stigmastadienol + clerosterol + sitostanol + Δ 5,24stigmastadienol (%)	93,0	Seeds oils usually has a lower content, when blended with olive, this content is lower
Total Sterols content (ppm)	1000	A low value can depend on blend with “desterolised” oils
Waxes (ppm)	250	Olive pomace oils contains very high amounts of waxes
Saturated Fatty Acids in 2 position of triglyceride (%)	1,3	Higher values can depend on chemical sinthesys
Erithrodiol + uvaol (%)	4,5	Olive pomace oils contains very high amounts of Erithrodiol + uvaol
ECN 42 (HPLC - calculated)	0.2-	Higher values depends on the presence of oils not deriving by fruit biosinthesys
Stigmastadienes (ppm)	0,15	Stigmastadienes (Sterenes) derive by sterols dehydration, their presence is not in agreement with not refined oils
C18:1 t(%)	0,05	Trans isomers are formed in bleaching step of refining: higher values depends on the presence of refined oils, maybe desterolised oils
C18:2 t + C18:3 t (%)	0,05	Trans isomers are formed in bleaching step of refining: higher values depends on the presence of refined oils, maybe desterolised oils



1 C16:0 2 C16:1 3 C18:0 4C18:1 5 C18:2 6 C20:0 7 C18:3 8 C20:1 9 C24:0

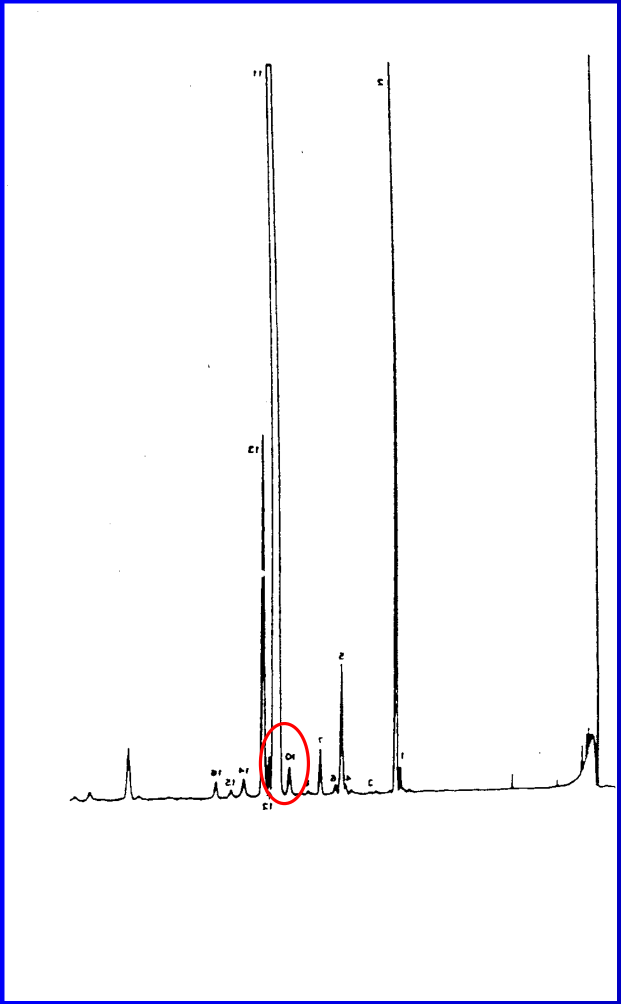


Gas chromatographic analysis by packed colums: top SE30, bottom OV17

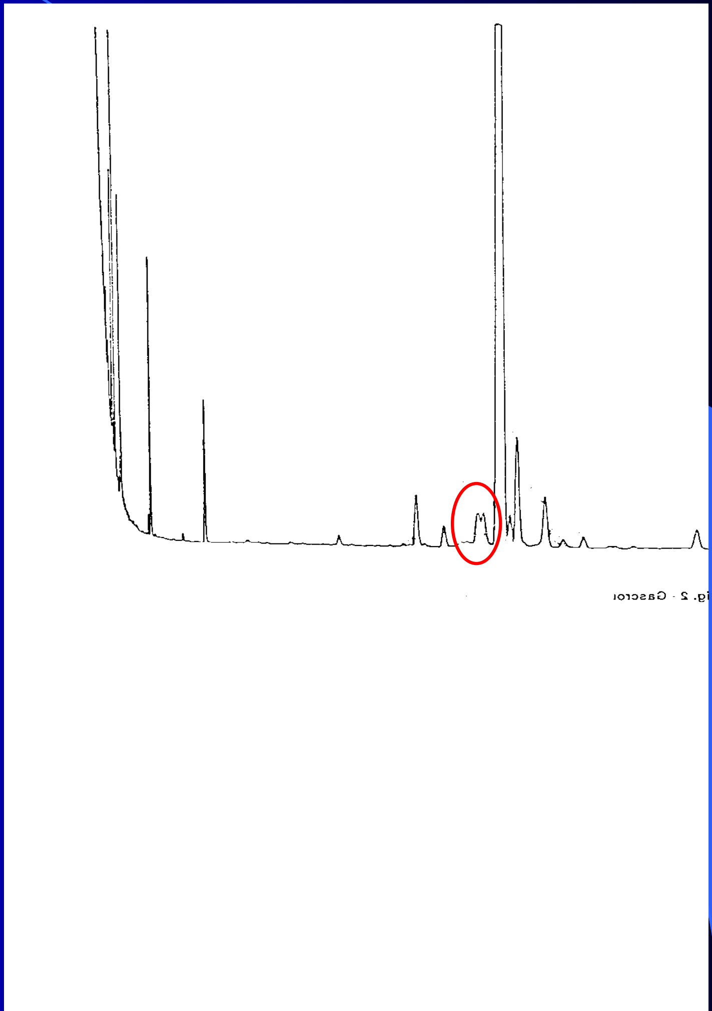


Comparison between GLC analysis of Sterols as performed by open tubular column (top) and packed column (bottom)

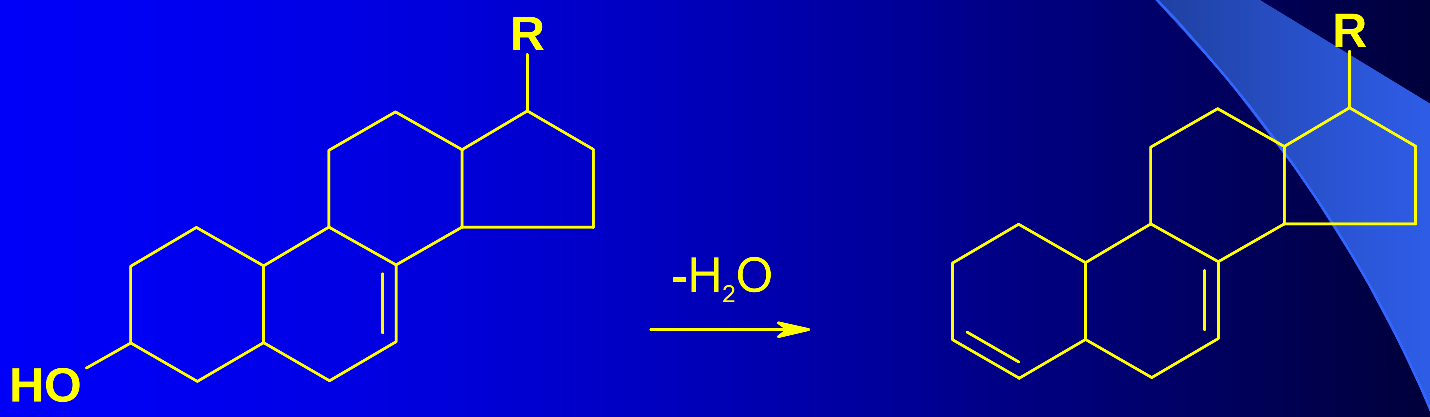
Capillary GLC analysis of sterols of virgin olive oil



Capillary GLC analysis of sterols of a refined olive oil



Sterenes formation



ALIPHATIC ALCOHOLS

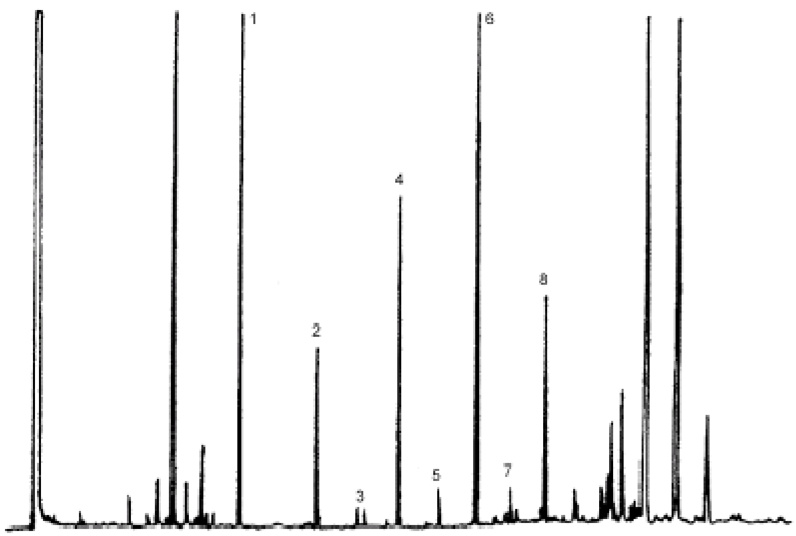


Figure 1 — Chromatogram of the alcoholic fraction of virgin oil

1 = Eicosanol	5 = Pentacosanol
2 = Decosanol	6 = Hexacosanol
3 = Tricosanol	7 = Heptacosanol
4 = Tetracosanol	8 = Octacosanol

WAXES

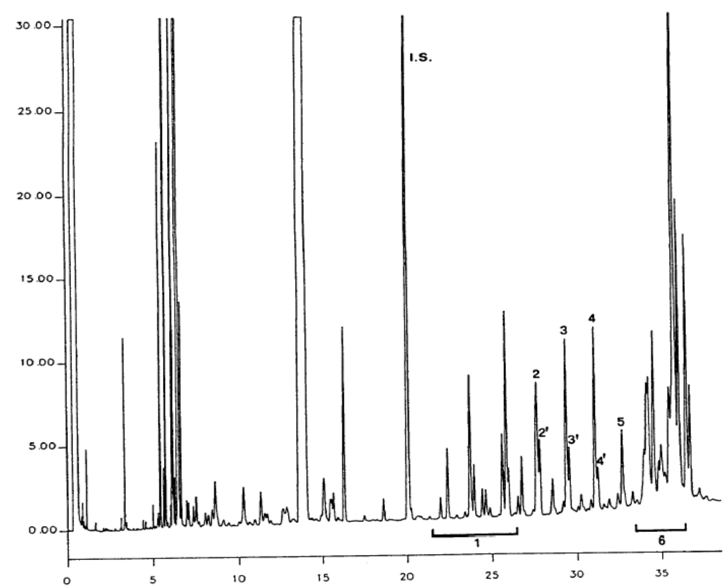


FIGURA -Esempio di gascromatogramma della frazione cere di un olio di oliva (*)

Legenda:

I.S. Lauril Arachidato

1	=	Esteri diterpenici
2+2'	=	Esteri C40
3+3'	=	Esteri C42
4+4'	=	Esteri C44
4	=	Esteri C46
6	=	Esteri steroli e Alcoli triterpenici

Figure 3: Soya oil/Olive oil 30/70

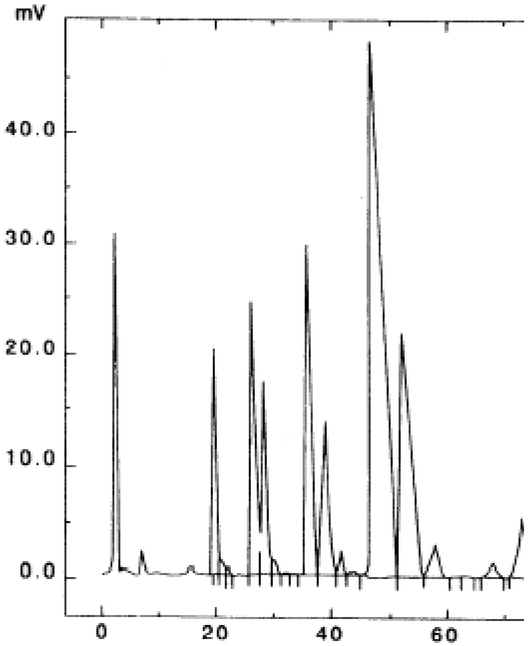
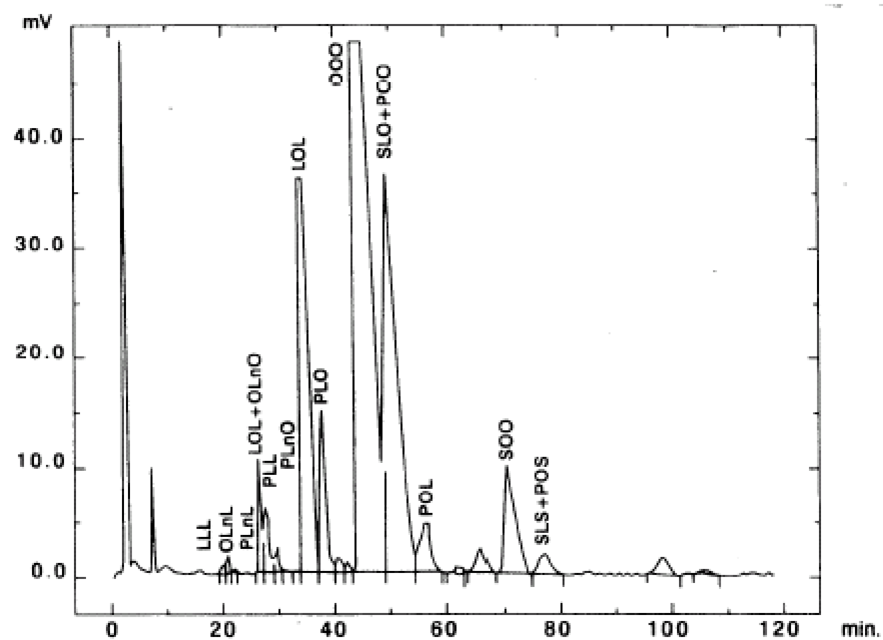


Figure 4: Olive oil



ECN = N_c-2D

ΔECN42 (HPLC – CALCULATED)

	EEC Limit	IOOC Limit
Extra virgin olive oil	≤ 0,2	≤ 0,2
Virgin olive oil	≤ 0,2	≤ 0,2
Lampante olive oil	≤ 0,3	≤ 0,3
Refined olive oil	≤ 0,3	≤ 0,3
Olive oil	≤ 0,3	≤ 0,3
Raw olive pomace oil	≤ 0,6	≤ 0,6
Refined olive pomace oil	≤ 0,5	≤ 0,5
Olive pomace oil	≤ 0,5	≤ 0,5

NEW EXPERIMENTAL APPROACHES

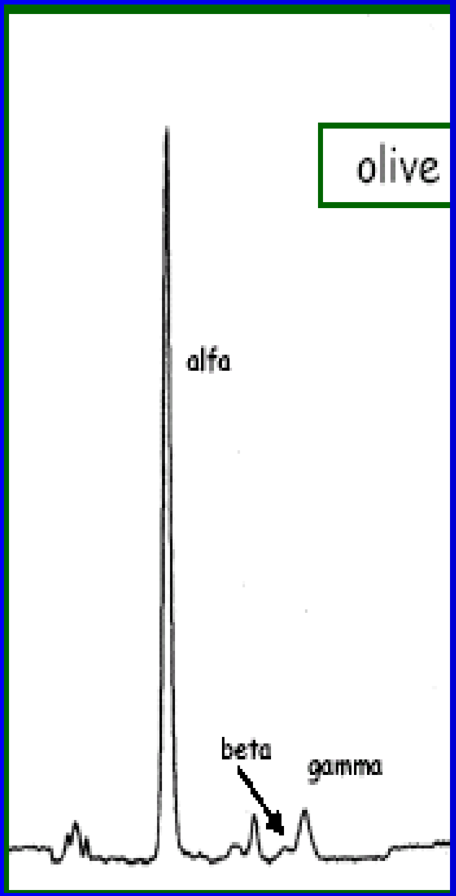
Open questions

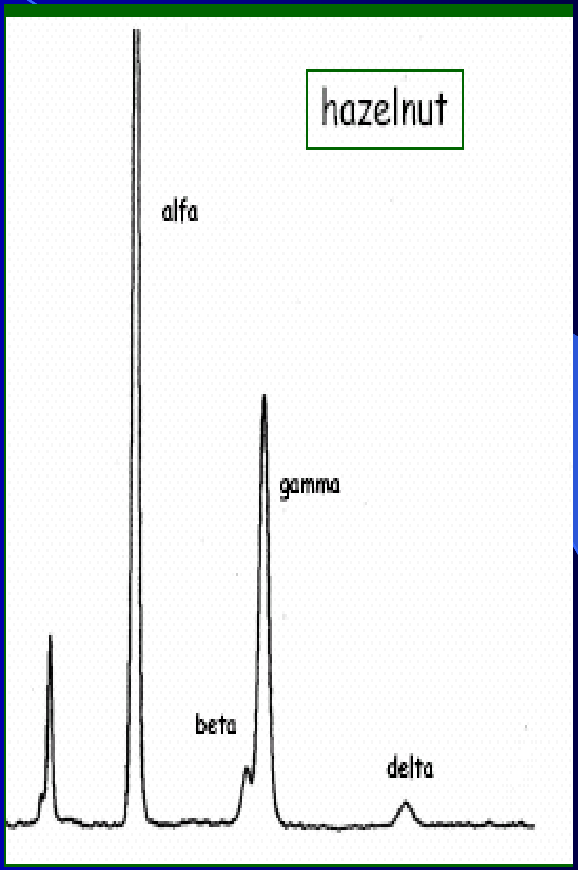
- Mixtures with hazelnut oils
- Mixtures with deodorised oils
- Second centrifugation oils (“remolido”)
- Oxidative status
- Further quality parameters
- Identification of origin:
  - Cultivar
  - Geographical

MIXTURES WITH HAZELNUT OILS
SEVERAL PROPOSES

- ❖Morchio, et al (1998): Analyses of tocopherols
- ❖ Mariani et al (1999) : Analyses of esterified sterols
- ❖ Fedeli, Cortesi et al (1998): Analyses of TAG
- ❖ Cert et al (1999): Analyses of TAG
- ❖ Vichi, Conte et al (2000): ECN 42-Free sterols
- ❖ Mannina et al (1999) : Filbertone
- ❖ Ruiz del Castillo et al. (1998) : NMR Filbertone [(E)-metilenepta-2-en-4-one]

Morchio, et al (1998): Analyses of tocopherols





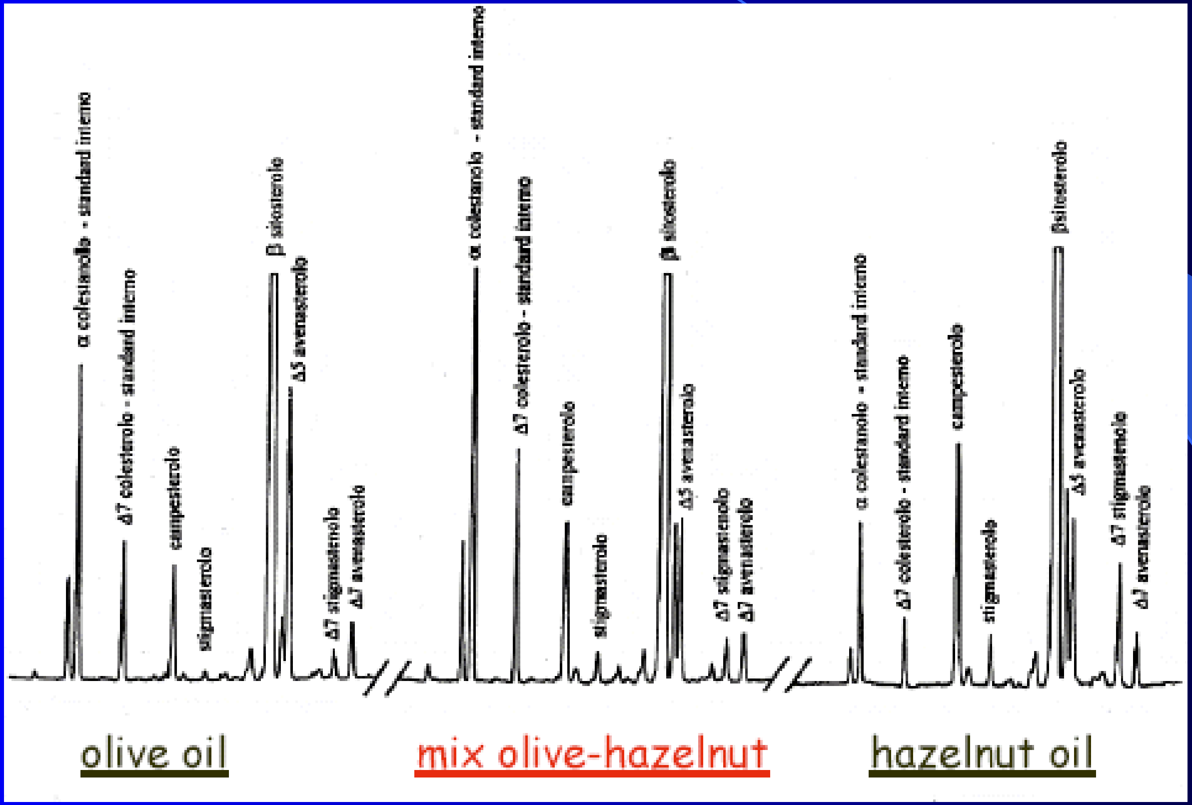
SSOG

TWO APPROACHES:

ANALYSIS OF STEROLS (ESTERIFIED AND FREE)

ANALYSIS OF TRIACYLGLYCEROLS

Mariani et al (1999) : Analyses of esterified sterols



C. Mariani, Seminar “Aggiornamento e prospettive della ricerca sull’olio di oliva”
Soc Italiana Studio Sostanze Grasse, Roma, 26-27 giugno 2003

Sterols esters

$$\text{Esterified campesterol} \times \frac{(\text{Esterified } \Delta 7 \text{ stigmastenol})^2}{\text{Esterified } \Delta 7 \text{ avenasterol}}$$

If sterols (from esters) ≤ 200 mg/kg

olive oil ≤ 1,5
olive blended with hazelnut > 1,5

If sterols (from esters) 200-600 mg/kg

olive oil ≤ 1
olive blended with hazelnut > 1

If sterols (from esters) > 600 mg/kg

olive oil ≤ 0.9
olive blended with hazelnut > 0.9

Free esters

$$\text{mg/kg Free } \Delta 7 \text{ stigmastenol} \times \frac{\text{\% Free } \Delta 7 \text{ stigmastenol}}{\text{\% Esterified } \Delta 7 \text{ stigmastenol}}$$

olive oil ≤ 0.5
olive blended with hazelnut > 0.5

Tne value of sterols must be exspressed as mg of sterols and not sterols esters per kg of oil

% Free Δ7 stigmastenol
olive oil ≤ 0,5
olive blended with hazelnut > 0,5

SSOG

Vichi, Conte et al (2000): ECN 42-Free sterols

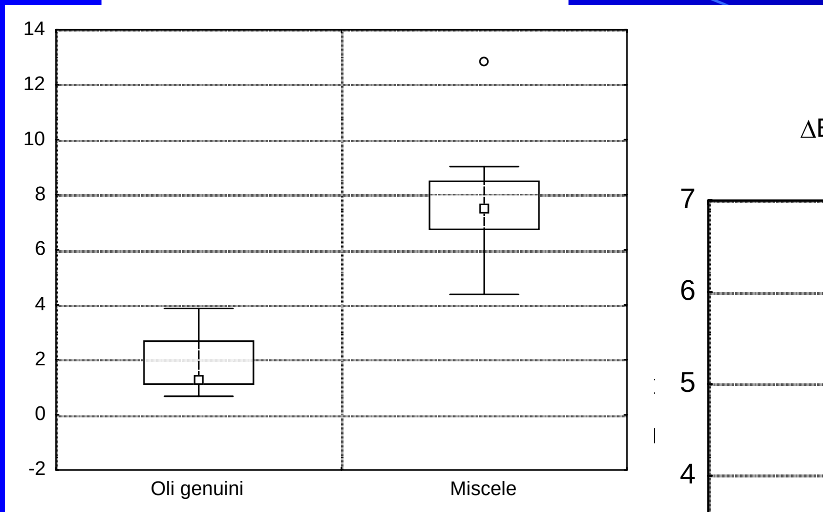
Sample	Esterified Sterols	ΔECN42
1	FP	G/N
2	N*	G/N
3	N*	N *
4	N/G	G/N
5	G*	G/N
6	N*	G/N

N: classified as not pure; G: classified as pure; G/N: not univoke classification; FP: false positive; *: correct classification.

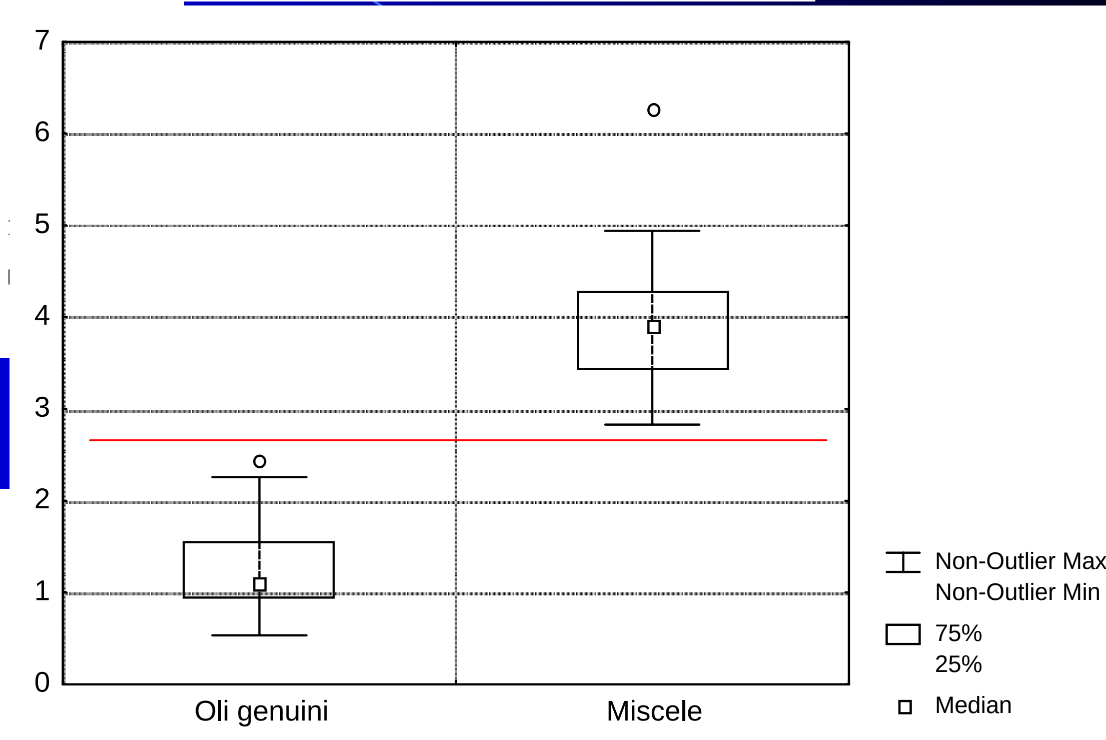
Vichi, Conte et al (2000): ECN 42-Free sterols

	Δ7-stigmastenol^3 /Δ7-avenasterol +ΔECN42 x 10	Δ7-stigmastenol^3 /Δ7-avenasterol +ΔECN42% x 10	Δ7-stigmastenol^2 /Δ7-avenasterol +ΔECN42 x 10	Δ7-stigmastenol$_2$ /Δ7-avenasterol +ΔECN42% x 20
1	10,57	122,36	4,00	90,19
2	20,32	242,03	6,82	158,10
3	16,09	211,33	6,24	152,50
4	3,68	75,58	2,42	76,07
5	1,15	12,11	0,75	15,32
6	8,40	97,49	5,06	82,18

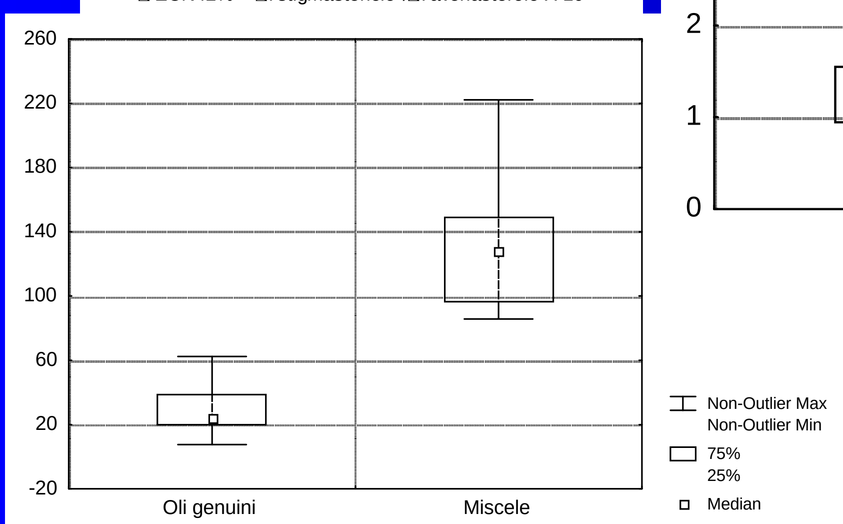
ΔECN42 X 10 + Δ7stigmastenolo^3/Δ7avenasterolo



ΔECN42 X 10 + Δ7stigmastenolo^2/Δ7avenasterolo



Δ ECN42% + Δ7stigmastenolo^3/Δ7avenasterolo X 10



Mixtures with deodorised oils

- A number of approaches, no results

- Serani's , method:
  evaluation of diacylglycerides + pheophytines

- Ring test in progress within
  Italian Technical Board for oils and fats

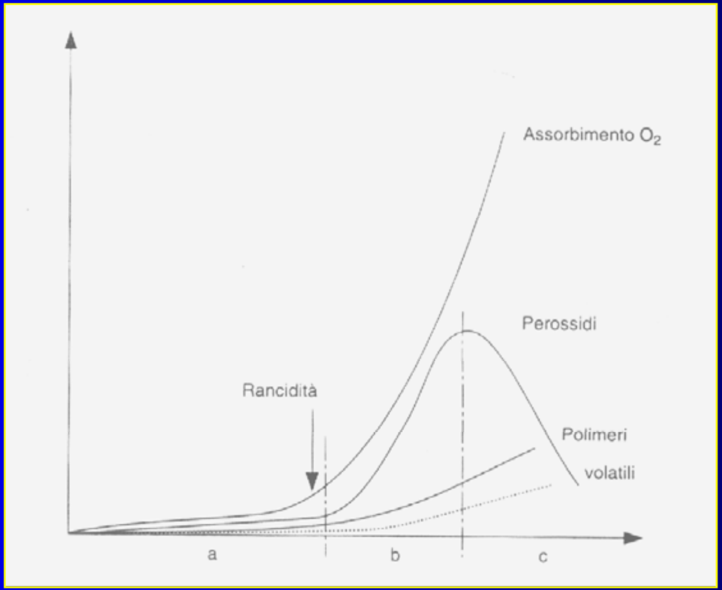
Second centrifugation oils (“remolido”)

Detection is useful and possible when pomaces storage time has been long : evaluation of ethyl esters in fatty acid composition

Sometimes, alipatic alcohols and waxes can result high

Oxidative status

Official parameter Peroxide value



Oxidative status

- Anisidine value
- Hexanal evaluation
- Nonanal evaluation
- HPLC evaluation of oxidation products
- Triglycerydes dimers

Anisidine value

Formerly Benzidine method (Holm, 1957)
List et al, 1974
Holm (1972)

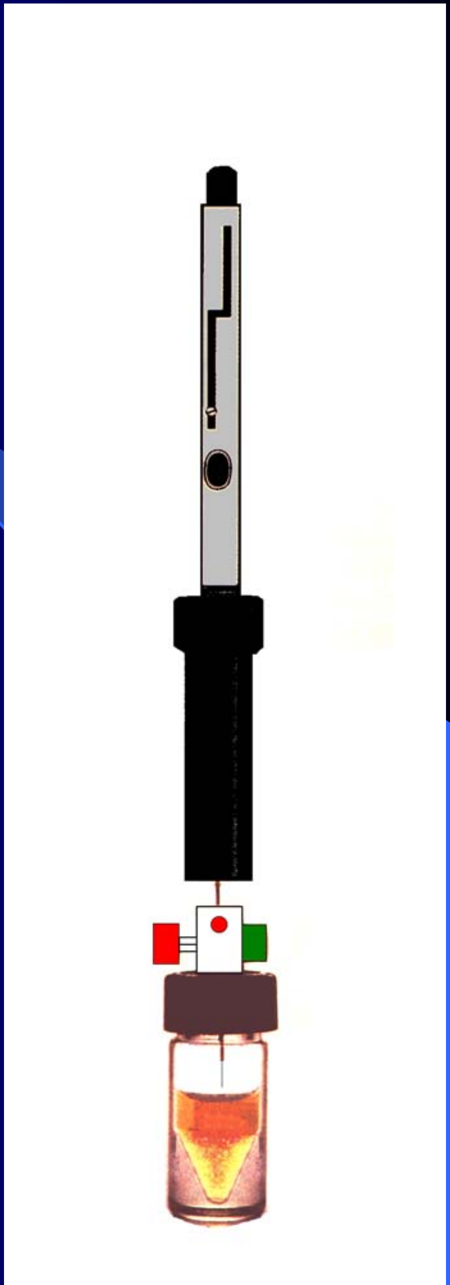
OXIDATION VALUE = ANISIDINE VALUE + 2 PEROXIDE VALUE

As for benzidine test, dat the same molar concentration,
different unsaturated aldehydes rise to different values of anisidine.

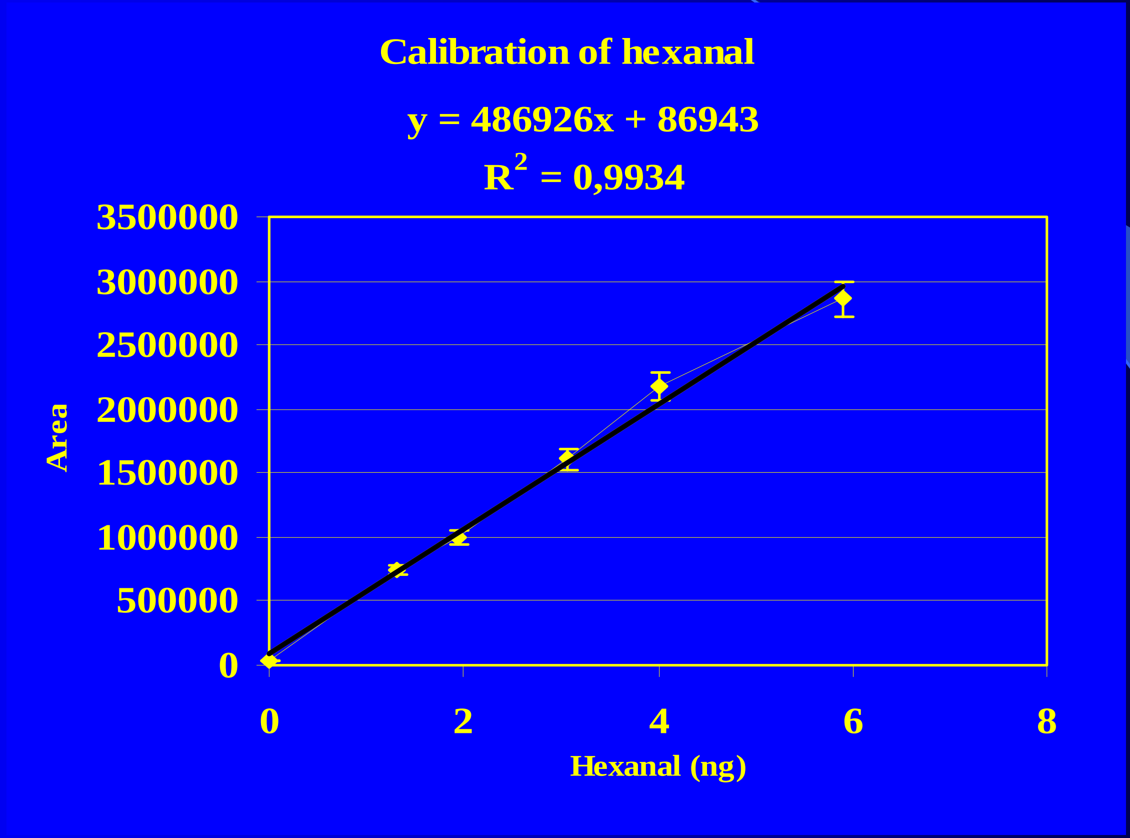
SPME
(Solid Phase Micro Extraction)

SPME is a technique of sample preparation that admit to get analytes purification and concentration by means of a thin film coating a fused silica fiber

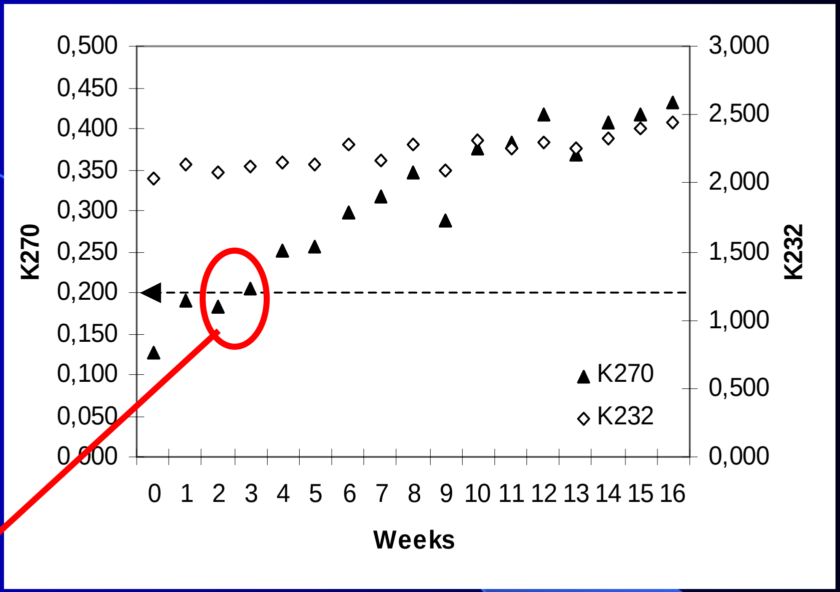
Figura da A. Verzera, Università di Messina

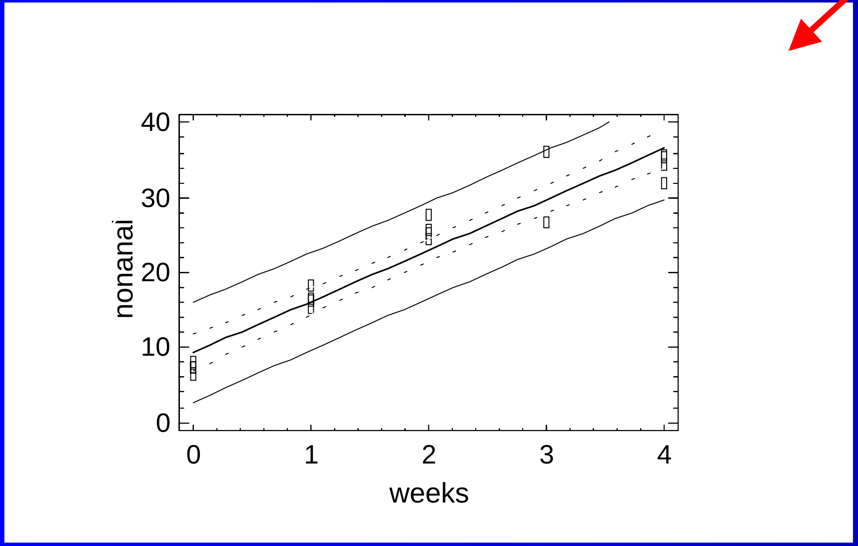


Hexanal evaluation SPME

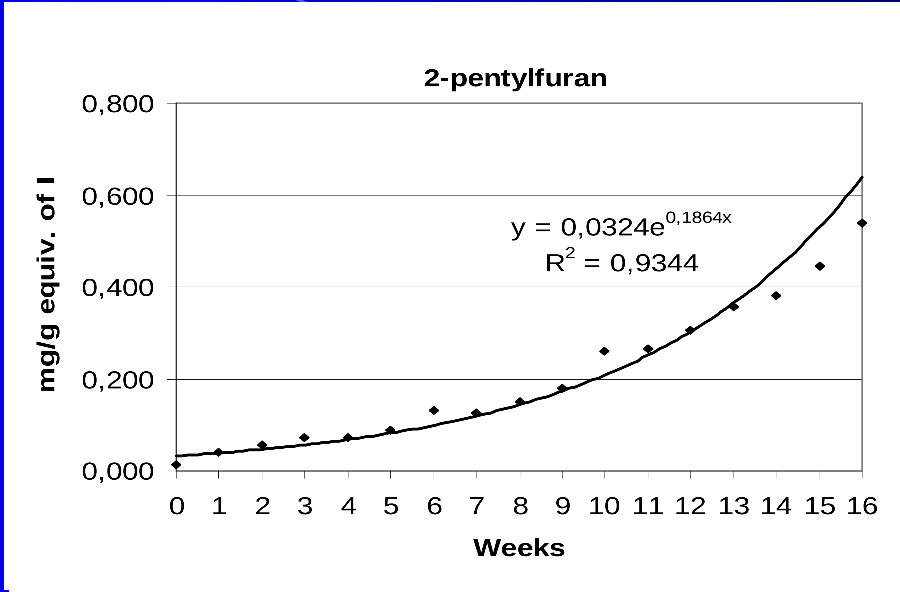


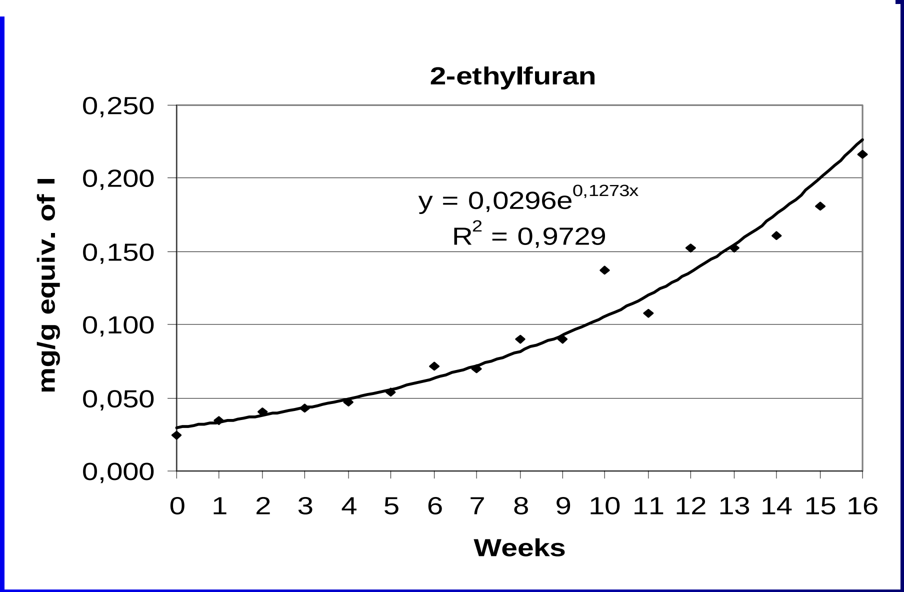
SPME NONANAL EVALUATION





Vichi S., Pizzale L., Conte L.S., Buxaderas S., Lopez-Tamames E.L. J. Agric. Food Chem., 51, 6564-6571, (2003).



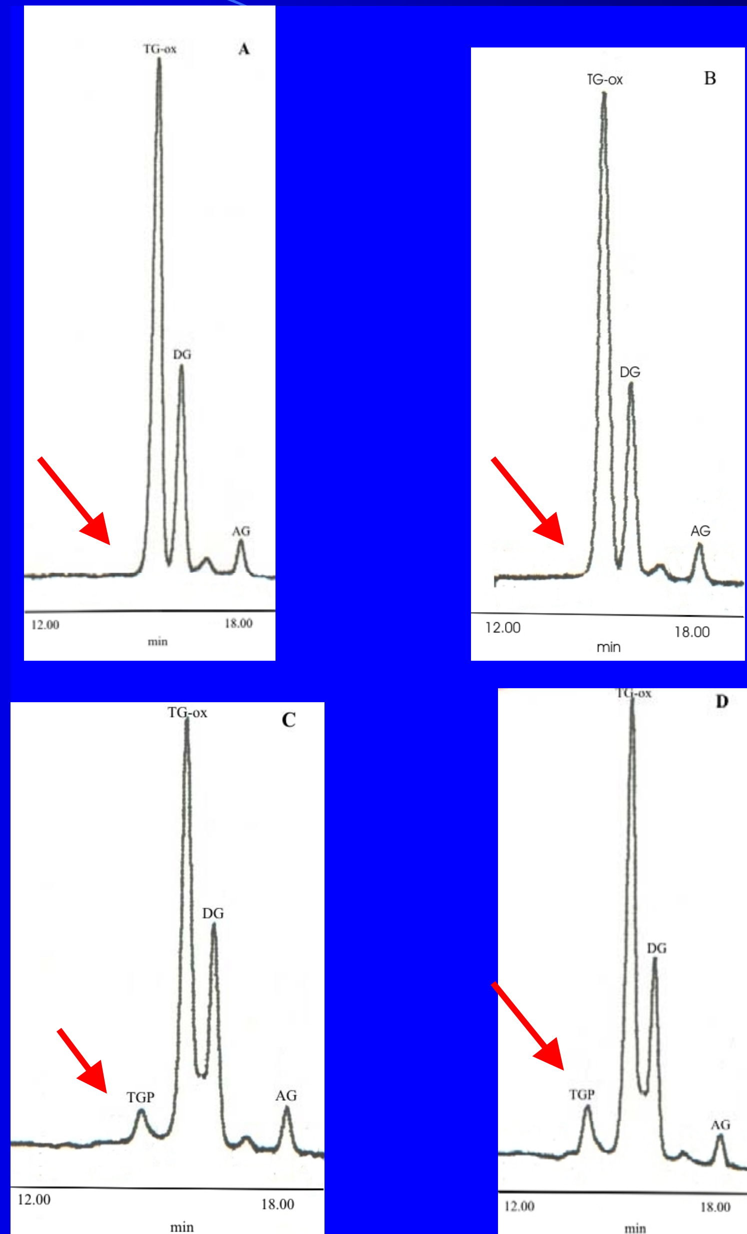


Vichi S., Pizzale L., Conte L.S., Buxaderas S., Lopez-Tamames E.L. J. Agric. Food Chem., 51, 6564-6571, (2003).

DIMERS TRIGLYCERYDES

Raw

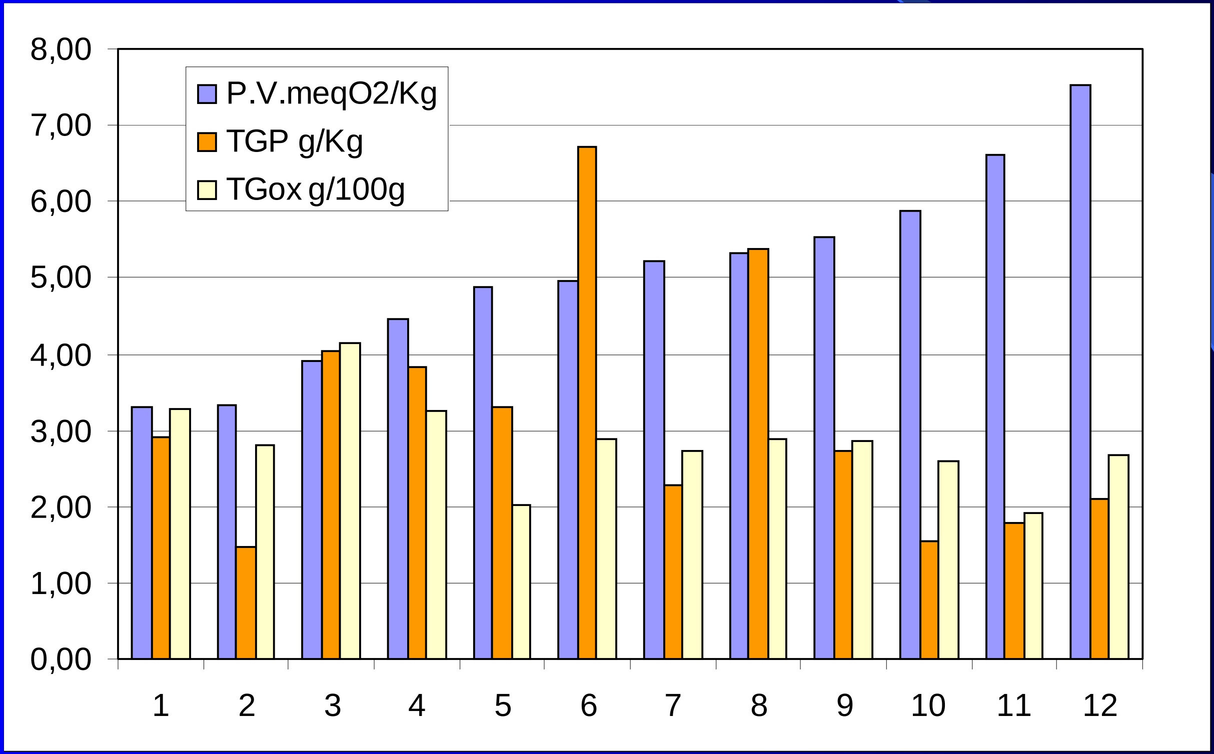
Neutralised

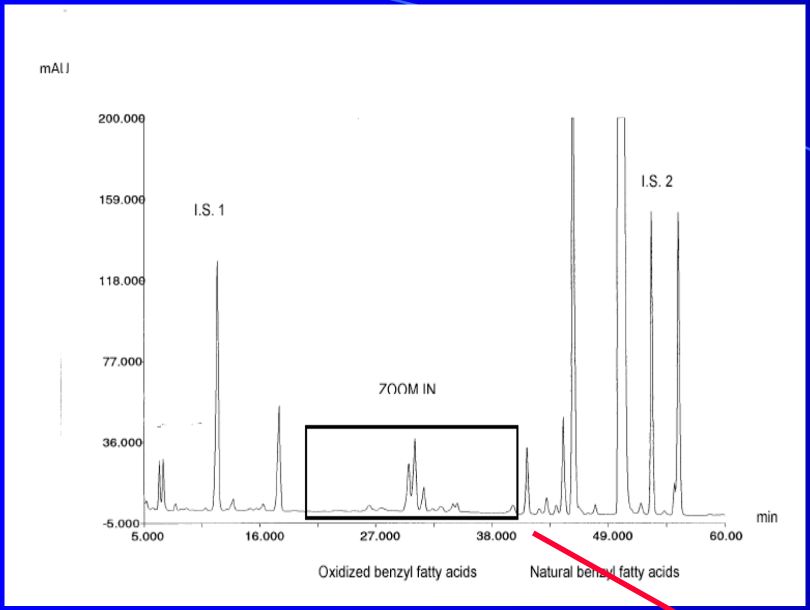


Bleached

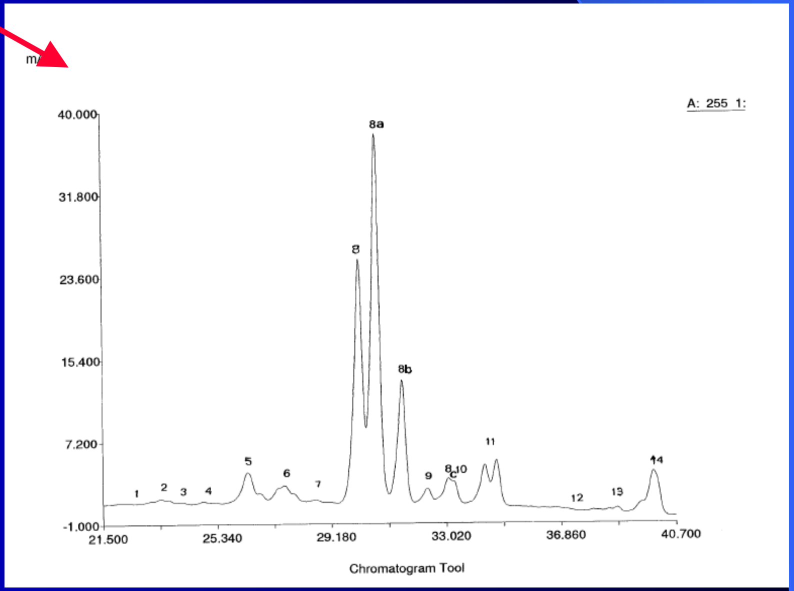
Deodorised

PEROXIDE VALUES VS DIMERS TRIGLYCERYDES

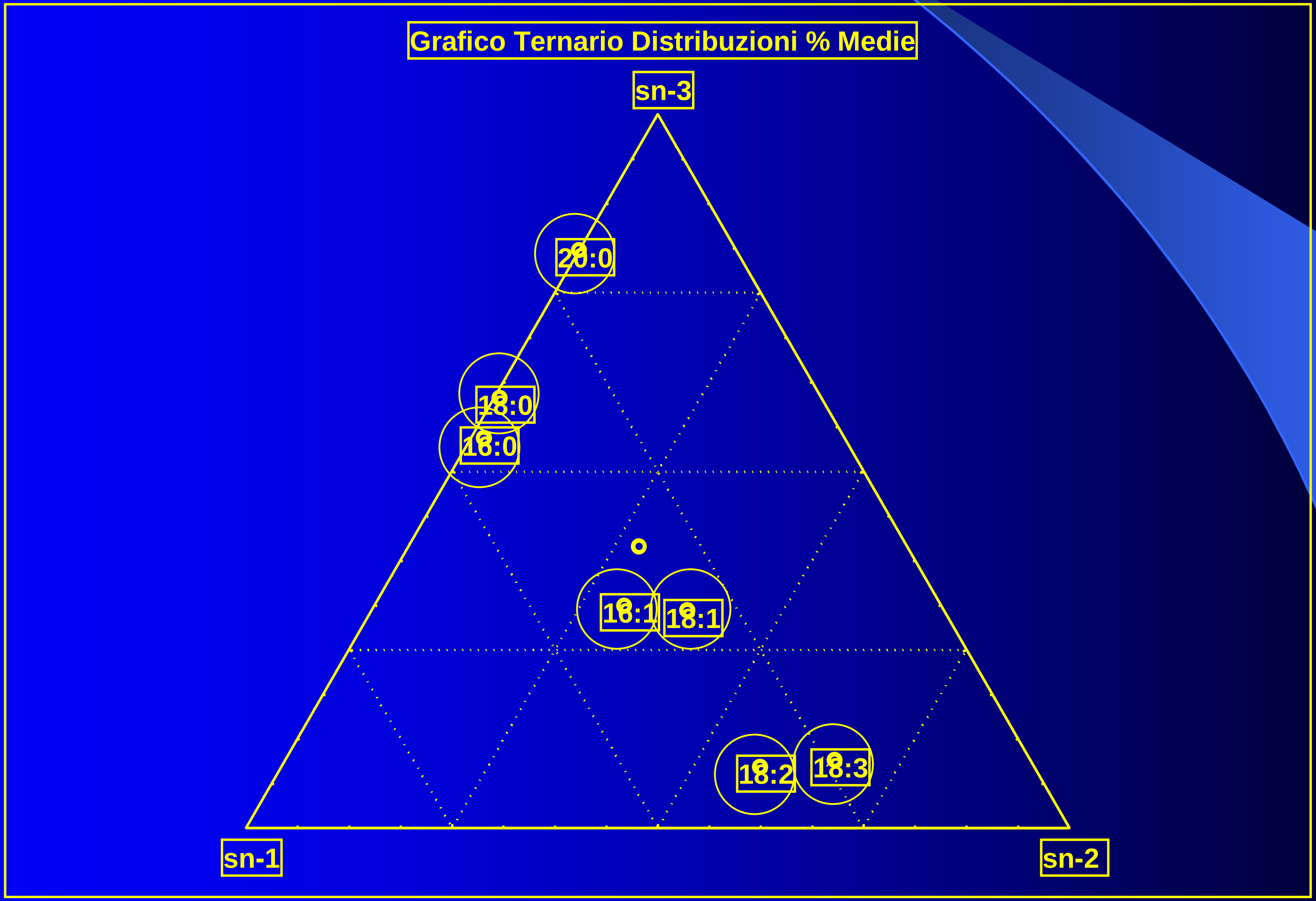




Peak N.	Benzyl ester derivatives		RRT	max UV abs. nm
1	di-Epoxy oleic acid	di -Epoxy - 18:1	0.31	256
2	Hydroxy linolenic acid	OH -18:3	0.40	225
3	di-Epoxy stearic acid	di -Epoxy - 18:0	0.42	256
4	Hydroperoxy linolenic acid	OOH -18:3	0.43	210-240
5	Keto linolenic acid	C=O -18:3	0.44	279
6	Hydroxy linoleic acid	OH -18:2	0.46	225
7	Hydroperoxy linoleic acid	OOH -18:2	0.48	210-240
8	Keto linoleic acid	C =O-18:2	0.51	277
8a	Keto linoleic acid	C=O -18:2	0.52	277
8b	Keto linoleic acid	C=O -18:2	0.53	277
9	Hydroxy oleic acid	OH -18:1	0.54	256
8c	Keto linoleic acid	C=O -18:2	0.56	279
10	Hydroperoxy oleic acid	OOH -18:1	0.57	210-230
11	Keto oleic acid	C=O -18:1	0.58	210-224
12	Epoxy oleic acid	Epoxy - 18:1	0.62	256
13	Epoxy stearic acid	Epoxy -18:0	0.69	256
14	Epidioxy oleic acid	Epidioxy -18:1	0.70	256



Distribution of fatty acids in position 1,2, 3 of triacylglycerol



		Garda Lake		Triest Gulfe		Emilia Romagna		
		Media	RSD	Media	RSD	Media	RSD	p
	sn-1	**43,8**	2,2	**43,7**	2,6	**44,1**	2,9	
C16:0	sn-2	**1,5**	0,2	**1,6**	0,2	**1,6**	0,8	
	sn-3	**54,7**	2,1	**54,7**	2,6	**54,3**	3,1	
	sn-1	**40,3**	5,6	**38,9**	6,9	**33,5**	5,0	**
C16:1	sn-2	**26,2**	6,2	**28,1**	6,5	**44,2**	3,9	***
	sn-3	**33,5**	7,5	**33,0**	9,3	**22,3**	4,6	***
	sn-1	**38,4**	4,9	**38,9**	3,9	**40,8**	10,2	
C18:0	sn-2	**0,7**	0,3	**0,5**	0,3	**0,7**	1,1	
	sn-3	**60,9**	5,0	**60,6**	3,9	**58,5**	10,3	
	sn-1	**31,0**	0,6	**31,2**	0,8	**31,2**	1,3	
C18:1	sn-2	**38,4**	0,8	**38,1**	1,1	**39,2**	1,8	
	sn-3	**30,6**	0,6	**30,6**	1,0	**29,6**	1,2	**
	sn-1	**37,3**	4,3	**34,2**	4,8	**22,3**	10,3	***
C18:2	sn-2	**52,1**	5,6	**55,6**	9,0	**76,9**	11,0	***
	sn-3	**10,7**	5,2	**10,2**	6,6	**0,7**	1,9	***
	sn-1	**28,2**	11,6	**23,6**	12,6	**14,4**	6,6	**
C18:3	sn-2	**59,9**	19,1	**65,2**	18,3	**84,8**	7,3	***
	sn-3	**11,9**	9,6	**11,2**	10,2	**0,8**	1,2	**
	sn-1	**12,8**	15,7	**21,4**	13,3	**26,9**	14,5	
C20:0	sn-2	**-**	-	**-**	-	**-**	-	
	sn-3	**87,2**	15,7	**78,6**	13,3	**73,1**	14,5	

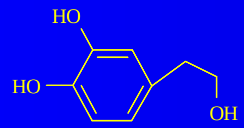
FURTHER QUALITY PARAMETERS

ANTIOXIDANT ACTIVITY:

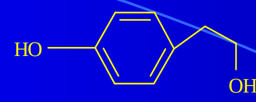
ANTIOXIDANTS IN DIET

SHELF LIFE

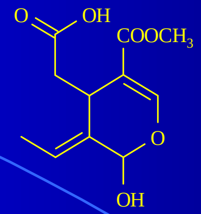
STRUCTURAL FORMULAS OF SOME POLYPHENOLS OF OLIVE FRUIT AND OIL



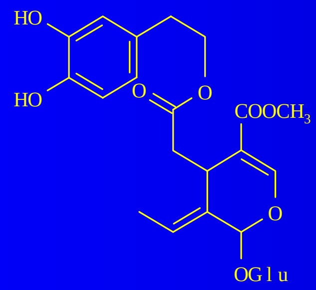
IDROSSITIROSOLO (HYTY)
3,4-DIIDROSSIFENETILALCOL



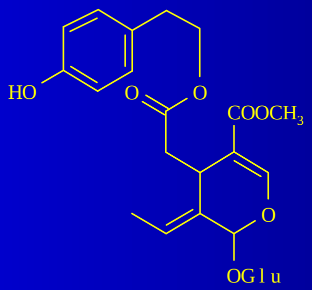
TIROSOLO (TY)
4-IDROSSIFENETILALCOL



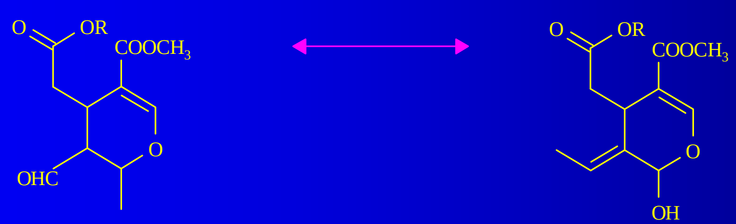
ACIDO ELENOLICO



OLEUROPEINA



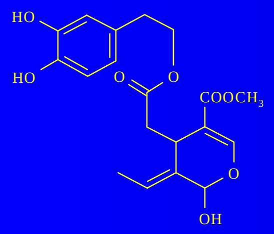
LIGUSTROSIDE



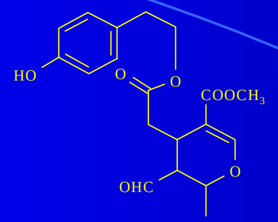
R=H AGLICONE OLEOSIDE n-METILESTERE

R=CH_3 AGLICONE OLEOSIDE DIMETILESTERE

STRUCTURAL FORMULAS OF SOME POLIPHENOLS OF OIL



AGLICONE OLEUROPEINA



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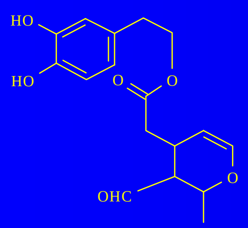
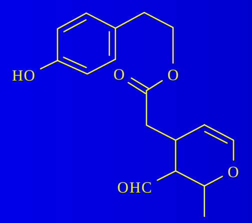


AGLICONE DEACETOSSIOLEUROPEINA



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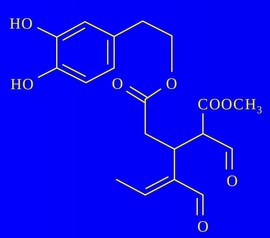
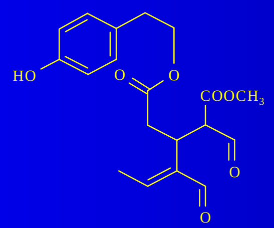
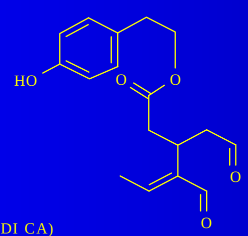


OLEUROPEINA (FORMA DIALDEIDICA)



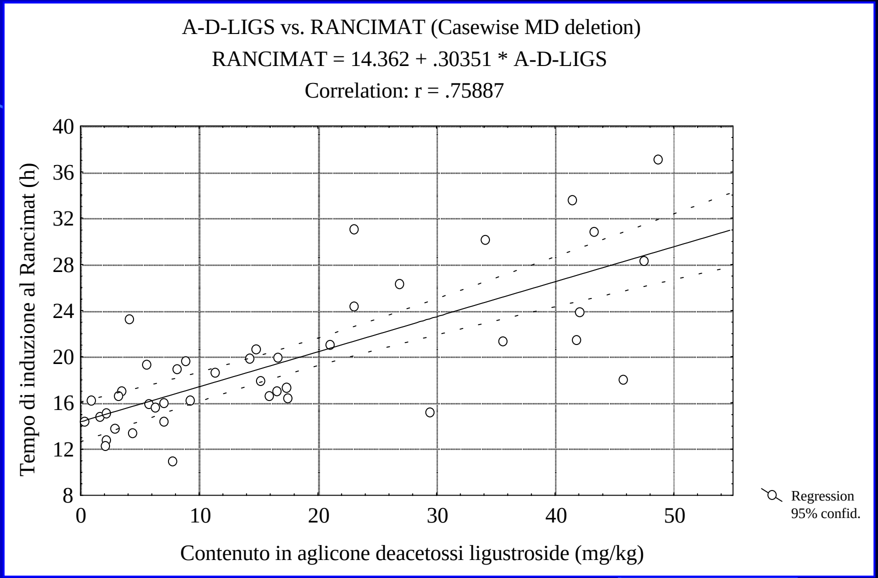
LIGUSTROSIDE (FORMA DIALDEIDICA)

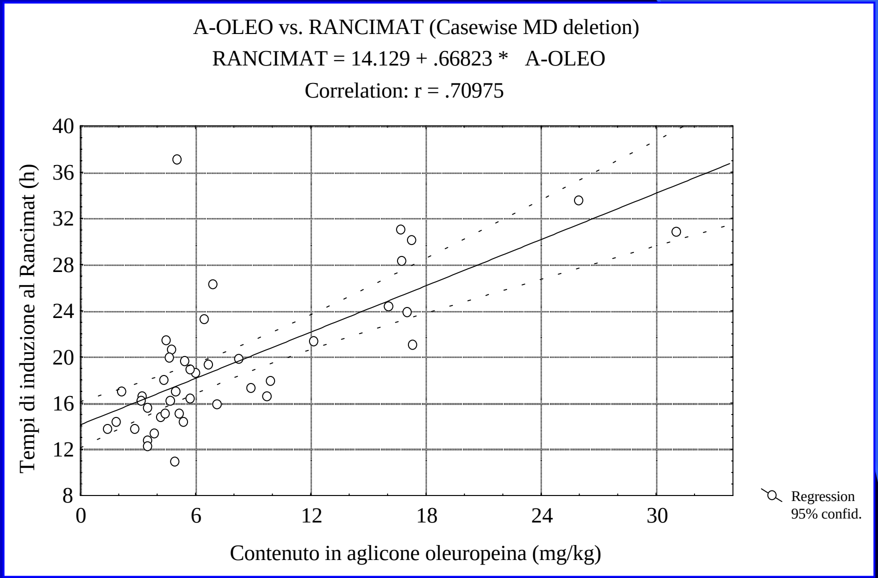
OLEUROPEINA(FORMA DEACETOSSI-DIALDEIDICA)



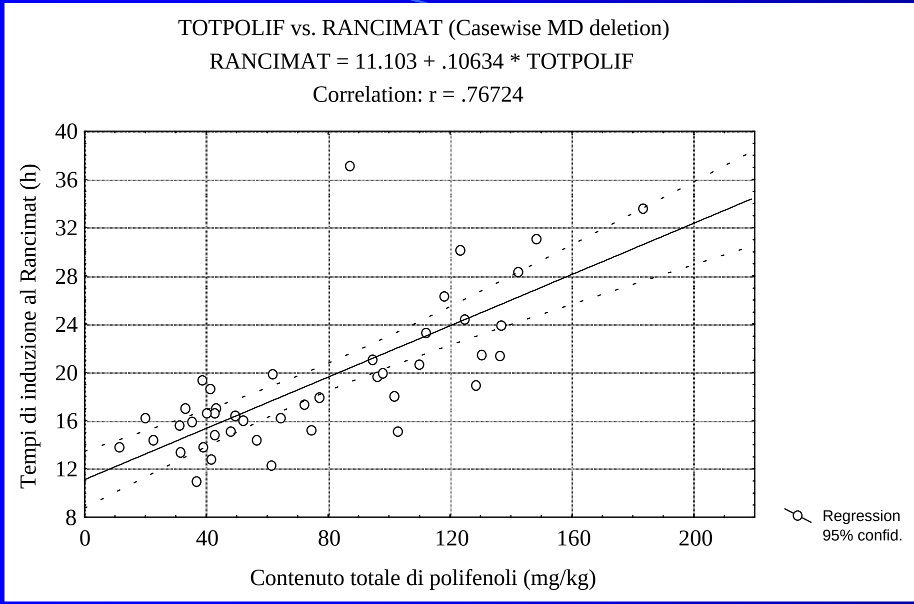
LIGUSTROSIDE(FORMA DEACETOSSI-DIALDEIDICA)

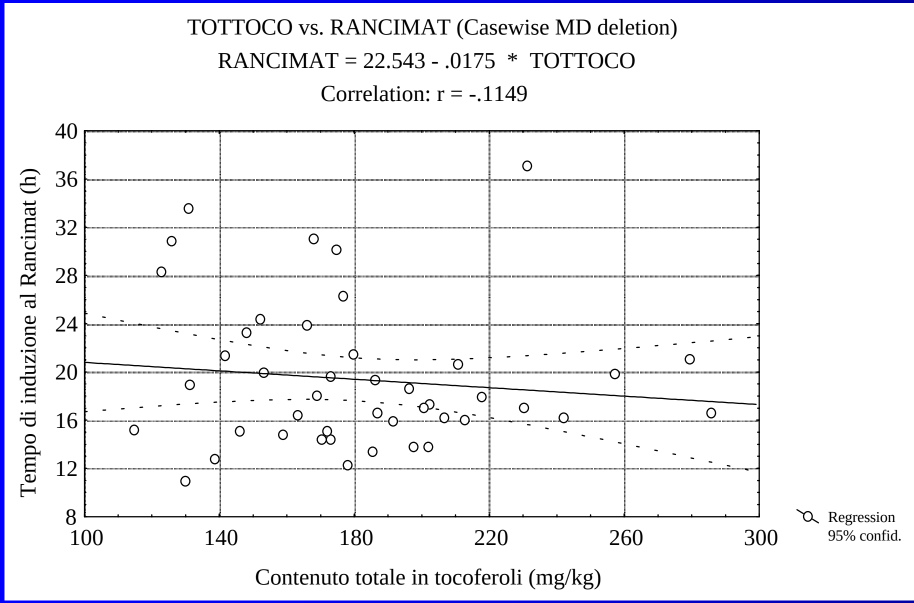
RESISTANCE TO OXIDATION: TEST RANCIMAT: INDUCTION TIME VS SOME POLYPHENOLS CONTENT



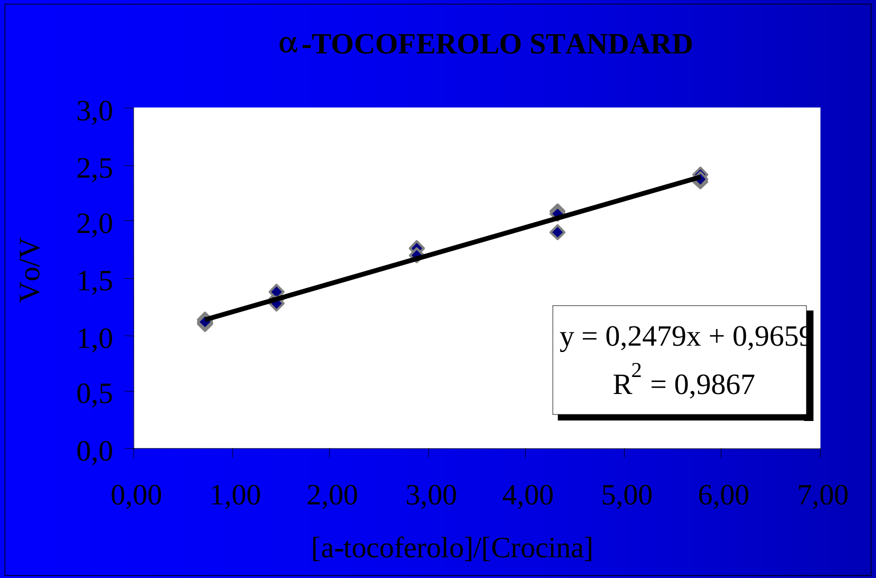


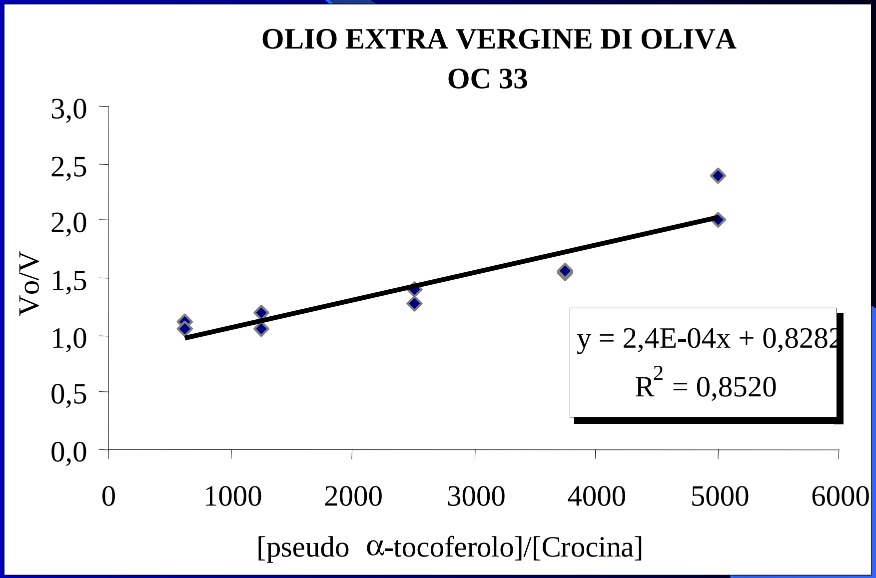
RESISTANCE TO OXIDATION: TEST RANCIMAT: INDUCTION TIME VS TOTAL AMOUNTS OF POLYPHENOLS AND TOCOPHEROLS





CHAIN BREAKING ACTIVITY: CROCIN BLEACHING TEST





Head space analysis: GC-MS with cold trapping and reverse of carrier flow rate in sampling step

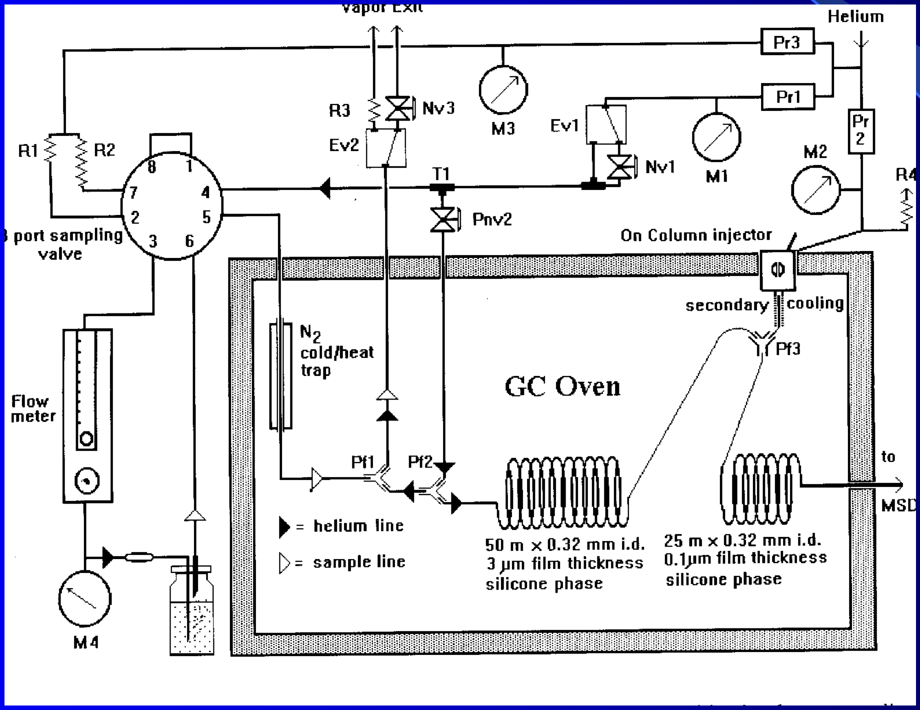
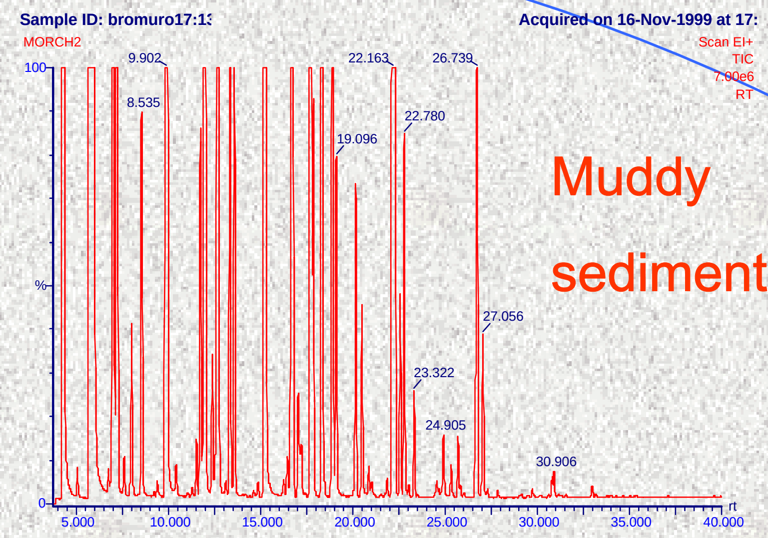
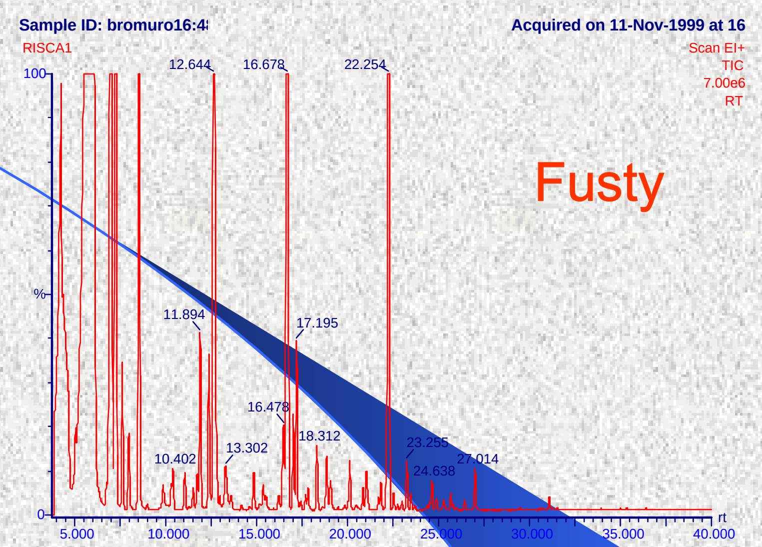
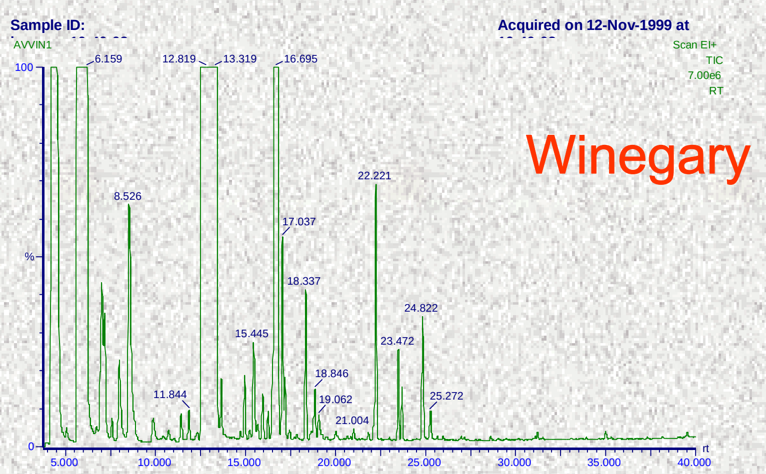
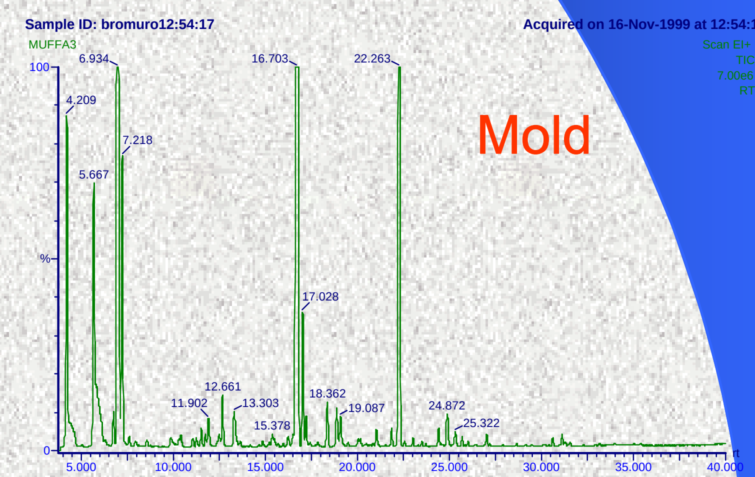


Figura di R. Barcarolo, Veneto Agricoltura









L.S. Conte G. Procida, A. Cichelli, A. Giomo, 2nd Euro Fed Lipid Symposium Strasbourg, 6-8 novembre 2002

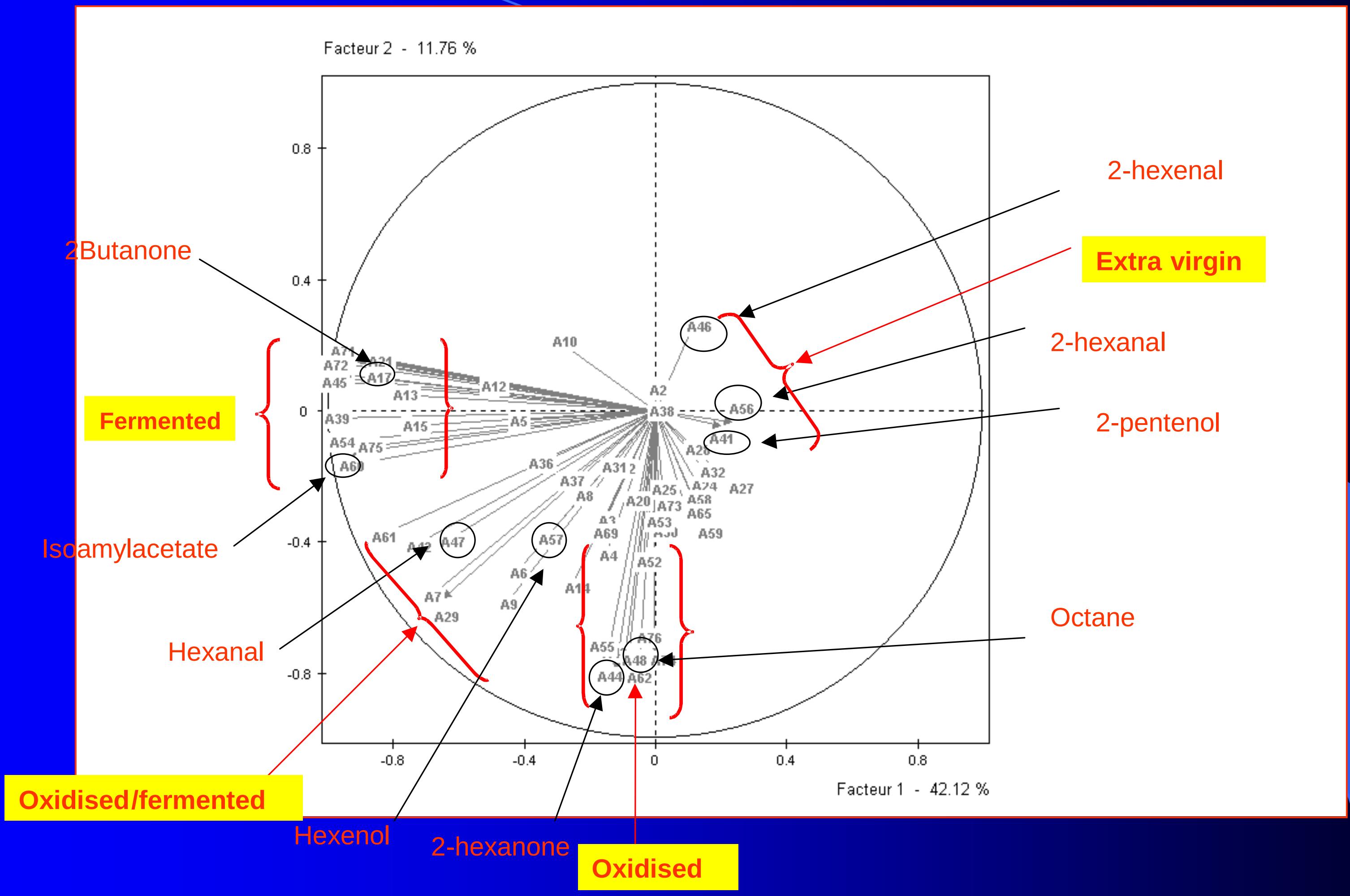
Rt (min)	Compound	Muddy	Mold	Fusty	Vinegary	Bitter	Rt (min)	Compound	Muddy	Mold	Fusty	Vinegary	Bitter
4.20	Methanol	80,4	340.6	1983.2	2968.2	2805.7	18.88	3-methyl-1-butanol	1145.6	39.5	129.5	85.4	33.4
4.28	Acethaldheyde	193.6	10.6	1.1	161.0	82.0	19.09	2-methyl-1-butanol	422.3	31.8	77.2	36.1	15.8
5.05	Methylformate	25.6	2.4	6.9	27.0	6.9	19.27	4-methyl-2-pentanone	n.d	n.d	n.d	n.d	4.3
5.89	Ethanol	9939.5	420.1	6892.7	10312.0	4546.6	20.15	Ethyisobutirrate	373.0	5.9	97.7	n.d	2.9
6.74	2-propenal	22.6	31.4	6.8	12.8	10.3	20.45	1-pentanol	215.9	n.d	10.1	1.3	n.d
7.00	Acetone	574.9	591.2	624.9	1022.4	113.2	20.66	2-pentenol	n.d	n.d	n.d	5.0	n.d
7.05	Propanal	556.4	235.1	623.0	60.0	143.1	20.86	Isobutylacetate	36.0	n.d	36.3	4.4	n.d
7.99	Ethylformate	206.0	n.d.	39.7	199.1	10.9	21.03	Methylisovaleriate	n.d	n.d	92.1	n.d	n.d
8.53	Methylacetate	486.4	6.2	642.2	687.2	343.9	21.70	2-hexanone	n.d	n.d	20.0	n.d	n.d
9.21	Dimethylsulfone	5.4	n.d	n.d	n.d	0.2	22.16	ethylbutirrate	409.8	n.d	38.0	n.d	n.d
9.37	2-propenol	20.0	n.d	n.d	n.d	n.d	22.19	3-hexenal	n.d	n.d	n.d	n.d	n.d
9.85	Isobutenal	140.1	1.4	8.4	30.3	27.9	22.26	hexanal	730.6	568.9	880.8	280.6	165.8
9.90	1-propanol	1716.4	9.1	30.5	1.5	7.4	22.31	octane	247.3	174.9	2466.6	70.7	29.9
10.41	2-butenal	41.0	10.7	38.2	32.3	14.7	22.56	Propylpropionate	215.8	n.d	n.d	n.d	n.d
11.27	2-butenone	10.6	8.4	4.9	n.d	1.1	22.78	Butylacetate	404.0	n.d	12.1	n.d	n.d
11.52	butanal	64.4	17.2	29.8	n.d	n.d	23.32	Methylvaleriate	117.5	n.d	n.d	n.d	n.d
11.72	2-butanone	465.0	10.3	53.8	n.d	3.9	23.47	Hexylmethylether	n.d	n.d	27.9	81.0	73.1
11.97	2-butanol	2317.6	n.d	n.d	n.d	n.d	23.68	3-methylexenylether	n.d	n.d	7.6	47.1	40.3
12.48	2-methyl-3-buten-2-ol	171.7	13.6	n.d	n.d	n.d	24.55	Ethyl-2-metilbutyrrate	25.5	n.d	9.4	n.d	n.d
12.67	Ethylacetate	2842.1	45.5	2094.5	45893.9	345.4	24.64	Ethylisovaleriate	n.d	n.d	51.4	n.d	n.d
13.10	Propylformate	15.2	n.d	n.d	n.d	n.d	24.82	2-hexenal	4.3	10.3	4.8	91.3	41.1
13.31	Isobutanol	607.6	48.5	136.1	59.0	23.5	24.92	3-hexenol	75.4	29.5	18.4	52.3	38.3
13.56	Methylpropionate	520.6	n.d	n.d	n.d	n.d	24.97	2-hexenol	19.3	4.3	2.9	16.2	5.1
14.85	3-metilbutanal	19.6	6.4	71.3	76.7	54.1	25.32	1-hexanol	21.1	13.5	20.7	25.6	18.1
15.20	Isopropylacetate	n.d	n.d	n.d	12.2	n.d	2570	Isoamylacetate	68.4	8.1	32.1	n.d	n.d
15.25	Butylformiate	3451.6	4.9	12.2	n.d	7.1	25.83	2-methylbutylacetate	13.3	n.d	10.7	n.d	n.d
15.35	2-metilbutanal	n.d	13.6	51.7	135.4	69.1	26.43	2-heptanone	n.d	n.d	21.2	n.d	n.d
16.21	1-penten-3-ol	21.8	9.8	35.0	31.0	12.8	26.63	Propylbutyrrate	76.2	n.d	n.d	n.d	n.d
16.44	Methylisobutirrate	50.6	2.9	81.5	n.d	n.d	26.74	Ethylvaleriate	599.9	n.d	n.d	n.d	n.d
16.49	2-pentanone	n.d	8.5	117.6	22.7	18.6	26.97	Heptanale	n.d	1.1	n.d	7.8	3.5
17.03	Pentanal	127.9	119.2	182.5	220.9	115.7	27.05	Buthylpropanoate	63.0	n.d	n.d	n.d	n.d
17.07	3-pentanone	n.d	n.d	n.d	n.d	n.d	27.29	Amylacetate	7.0	n.d	n.d	n.d	n.d
17.72	Ethylpropionate	1947.2	n.d	25.0	3.1	n.d	27.84	Methylexanoate	7.7	n.d	n.d	n.d	n.d
17.85	Propylacetate	421.1	n.d	43.1	n.d	n.d	29.50	2-heptenale	3.3	1.4	n.d	n.d	n.d

L.S. Conte G. Procida, A. Cichelli, A. Giomo, 2nd Euro Fed Lipid Symposium Strasbourg, 6-8 novembre 2002

Rt (min)	Compound	Muddy	Mold	Fusty	Vinegary	Bitter	Rt (min)	Compound	Muddy	Mold	Fusty	Vinegary	Bitter
18.33	Methylbutirrate	1266.2	n.d	116.3	n.d	n.d	24.92	3-hexenol	75.4	29.5	18.4	52.3	38.3
18.88	3-methyl-1-butanol	1145.6	39.5	129.5	85.4	33.4	24.97	2-hexenol	19.3	4.3	2.9	16.2	5.1
19.09	2-methyl-1-butanol	422.3	31.8	77.2	36.1	15.8	25.32	1-hexanol	21.1	13.5	20.7	25.6	18.1
19.27	4-methyl-2-pentanone	n.d	n.d	n.d	n.d	4.3	2570	Isoamylacetate	68.4	8.1	32.1	n.d	n.d
20.15	Ethyisobutirrate	373.0	5.9	97.7	n.d	2.9	25.83	2-methylbutylacetate	13.3	n.d	10.7	n.d	n.d
20.45	1-pentanol	215.9	n.d	10.1	1.3	n.d	26.43	2-heptanone	n.d	n.d	21.2	n.d	n.d
20.66	2-pentenol	n.d	n.d	n.d	5.0	n.d	26.63	Propylbutyrrate	76.2	n.d	n.d	n.d	n.d
20.86	Isobutylacetate	36.0	n.d	36.3	4.4	n.d	26.74	Ethylvaleriate	599.9	n.d	n.d	n.d	n.d
21.03	Methylisovaleriate	n.d	n.d	92.1	n.d	n.d	26.97	Heptanale	n.d	1.1	n.d	7.8	3.5
21.70	2-hexanone	n.d	n.d	20.0	n.d	n.d	27.05	Buthylpropanoate	63.0	n.d	n.d	n.d	n.d
22.16	ethylbutirrate	409.8	n.d	38.0	n.d	n.d	27.29	Amylacetate	7.0	n.d	n.d	n.d	n.d
22.19	3-hexenal	n.d	n.d	n.d	n.d	n.d	27.84	Methylexanoate	7.7	n.d	n.d	n.d	n.d
22.26	hexanal	730.6	568.9	880.8	280.6	165.8	29.50	2-heptenale	3.3	1.4	n.d	n.d	n.d
22.31	octane	247.3	174.9	2466.6	70.7	29.9	29.73	Pentylpropionate	9.9	n.d	n.d	n.d	n.d
22.56	Propylpropionate	215.8	n.d	n.d	n.d	n.d	30.80	Butylbutyrrate	24.1	n.d	n.d	n.d	n.d
22.78	Butylacetate	404.0	n.d	12.1	n.d	n.d	29.73	Pentylpropionate	9.9	n.d	n.d	n.d	n.d
23.32	Methylvaleriate	117.5	n.d	n.d	n.d	n.d	30.80	Butylbutyrrate	24.1	n.d	n.d	n.d	n.d
23.47	Hexylmethylether	n.d	n.d	27.9	81.0	73.1	30.90	Ethylhexanoate	32.3	n.d	n.d	n.d	n.d
23.68	3-methylexenylether	n.d	n.d	7.6	47.1	40.3	31.32	3-hexenylacetate	n.d	n.d	n.d	7.8	6.2
24.55	Ethyl-2-metilbutyrrate	25.5	n.d	9.4	n.d	n.d	31.35	Octanal	n.d	n.d	8.9	n.d	n.d
24.64	Ethylisovaleriate	n.d	n.d	51.4	n.d	n.d	32.97	Lymonene	14.1	1.7	2.3	1.7	n.d
24.82	2-hexenal	4.3	10.3	4.8	91.3	41.1	35.37	Nonanal	1.1	2.1	6.4	n.d	2.0

Olfactive threshold and sensory descriptors of volatile compounds at C5, C6 e C9

Composti Volatili	*Soglia (ug/kg)*	*Caratteristica sensoriale (olfativa)*
Esanale	*75*	*Verde-dolce*
Cis 3 esanale	*3*	*Verde*
Trans 2 esanale	*1125*	*Verde astringente*
Esil acetato	*1040*	*Verde*
Cis 3 esenilaceato	*750*	*Verde*
Esanolo	*400*	*Indesiderabile*
Cis 3 esenolo	*6000*	*Verde*
1-penten-3-one	*50*	*Verde*
Trans 2-pentenale	*300*	*Verde mandorla amara*
Pentanolo	*470*	*Fruttato*
Nonanale	*13500*	*Rancido*



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Thanks for your kind attention

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