2018 AOCS Annual Meeting & Expo
May 6-9th, Minneapolis, USA

Hot Topic 7
Olive Oil: Innovative Analytical Strategies to Guarantee Quality and Fight Fraud. Focus on the Advancements of the EU H2020 Project OLEUM

Oleum

Better solutions to protect olive oil quality and authenticity

Olive Oil Regulatory Framework Analysis, Update and Implementation.

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The Olive Oil Regulatory Framework

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Codex Alimentarius

Standard for olive oils and olive pomace oils
CODEX STAN 33-1981. Adopted in 1981.
Revisions: 1989, 2003, 2015.
Amendments: 2009, 2013.

1 Member Organization (The EU)
188 Member Countries

International Olive Council

International olive council. Trade standard for Olive oils and Olive Pomace Oils
COI/T.15/NC No 3/Rev. 11. July 2016

1 Member Organization (The EU)
13 Member Countries

~97% of the OO world production

The EU

European Commission, Reg (CEE) 2568/91
European Communities Official Journal L 248 5.9.1991 and further amendments

28 Member Countries

~75% of the OO world production

National standards

Argentina
Código alimentario argentino. Capitulo VII. Alimentos grasos. Aceites alimenticios. Artículos 535 y 536.

Australia
Australian standard. Olive oils and olive-pomace oils

California
State of California. Department of food and agriculture. Grade and Labeling Standards for Olive Oil, Refined-Olive Oil and Olive-Pomace Oil. Effective September 26, 2014.

Brazil
Ministério da agricultura, pecuária e abastecimento. Gabinete do ministro. Instrução normativa nº 1, de 30 de Janeiro de 2012.

China
General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China (AQSIQ) National Standard of the People's Republic of China ICS 67.200.10.

India
Draft Indian Standard olive oil — specification ICS No. 67.200 Doc No.: FAD 13 (2505).

South Africa
South African national standard. Olive oils and olive-pomace oils. SANS 1377:2015

USA
United States Standards for grades of olive oil and olive-pomace oil. Effective October 25, 2010.

The Olive Oil Regulatory Framework

OOs have to comply with different rules and standards depending on where they are traded

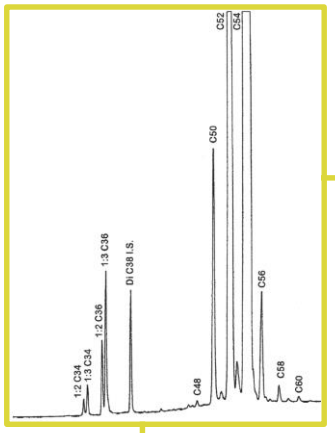
QUALITY CONTROL

International and National Standards

Harmonization ISO methods

- *Parameters*
- *Legal limits*
- *Analytical methods*

AUTHENTICITY CONTROL



DRAFT INTERNATIONAL STANDARD
ISO/DIS 21846

ISO/TC **34**/SC **11** Secretariat: **BSI**

Voting begins on: **2017-11-16** Voting terminates on: **2018-02-08**

Vegetable fats and oils — Determination of composition of triacylglycerols and composition and content of diacylglycerols by capillary gas chromatography

The Olive Oil Regulatory Framework

Dissimilarities that involve different commercial categories

	Virgin oils					Non-virgin oils		Olive pomace oils		
	Edible oils				Non-edible oil	Edible oils		Non-edible oil	Edible oils	
	EVOO	VOO	MGV	OOO*	LOO	ROO	OO	COPO	ROPO	OPO
EU	X	X	n.c.	n.c.	X	X	X	X	X	X
IOC	X	X	n.c.	X	X	X	X	X	X	X
CODEX	X	X	n.c.	X	n.c.	X	X	n.c.	X	X
Argentina	X	X	n.c.	X	X	X	X	n.c.	X	n.c.
USDA	X	X	n.c.	n.c.	X	X	X	X	X	X
Australia	X	X	n.c.	n.c.	X	X	X	X	X	X
South Africa	X	X	n.c.	n.c.	n.c.	X	X	n.c.	X	X
California	X	X	n.c.	n.c.	X**	X***	X	X	X°	X
China	X§	n.c.	X	n.c.	X	X	X	X	X	X
Brazil	X	X	n.c.	n.c.	X	X	X	n.c.	X	X
India	X	X	n.c.	X	X	X	n.c.	X	X	n.c.

EVOO = Extra Virgin Olive Oil, VOO= Virgin Olive Oil, MGV = Medium Grade Virgin Oil, OOO= Ordinary Olive Oil, LOO = Lampante Olive Oil, ROO = Refined Olive Oil, OO =Olive Oil, CPOO = Crude Olive Pomace Oil, RPOO = Refined Olive Pomace Oil, OPO = Olive Pomace Oil

*The category ordinary olive oil is going to be deleted by IOC and consequentely by Codex, too.

**is named Crude Olive Oil ***is named Refined olive oil blend °is named Refined olive-pomace blend oil §is named Premium virgin olive oil.

Categories of olive oils and olive pomace oils in international and national standards (n.c., category not considered)

Table from the D2.2

The Olive Oil Regulatory Framework

Dissimilarities that involve quality parameters

Limits for quality parameters for EVOO category (n.a., not applied)

	FA g oleic acid/100 g oil	PV meq O_2/Kg oil	K_{232}	K_{270}	FAEEs mg/kg oil	Md	Mf
EU	≤0.8	≤20	≤2.50	≤0.22	≤35	0	>0
IOC	≤0.8	≤20	≤2.50	≤0.22	≤35	0	>0
CODEX	≤0.8	≤20	≤2.50	≤0.22	n.a.	0	>0
Argentina	≤0.8	≤20	≤2.50	≤0.22	n.a.	n.a.	n.a.
USDA	≤0.8	≤20	≤2.50	≤0.22	n.a.	0	>0
Australia	≤0.8	≤20	≤2.50	≤0.22	n.a.	0	>0
South Africa	≤0.8	≤20	≤2.50	≤0.22	n.a.	0	>0
California	≤0.5	≤15	≤2.40	≤0.22	n.a.	0	>0
China	≤1.6*	≤10**	≤2.50	≤0.22	n.a.	0	>0
Brazil	≤0.8	≤20	≤2.50	≤0.22	FAME + FAEE< 75 mg/kg or > 150 mg/kg if FAEE/FAME > 1.5	0	>0
India	≤2.0*	≤20	n.a.	≤0.22	n.a.	n.a.	n.a.

*Expressed as mg KOH/g: 1.6 corresponds to 0.8%. **Expressed as mmol: 10 mmol correspond to 20 meq O_2/kg

The Olive Oil Regulatory Framework

Dissimilarities that involve purity parameters

Limits for purity parameters for EVOO category (n.a., not applied)

	Brassicasterol %	Campesterol %	Stigmasterol %	Apparent β-sitosterol %	Δ-7-stigmastenol %	Sterol content mg/kg oil
EU	≤0.1	≤4.0[a)]	<Campest.	≥93.0	≤0.5	≥1000
IOC	≤0.1	≤4.0[a)]	<Campest.	≥93.0	≤0.5	≥1000
CODEX	≤0.1	≤4.0[b)]	<Campest.	≥93.0	≤0.5	≥1000
Argentina	≤0.1	≤4.0[c)]	<Campest.	≥93.0	≤0.5	≥1000
USDA	≤0.1	≤4.5[d)]	<Campest.	≥93.0	≤0.5	≥1000
Australia	≤0.1	≤4.8	≤1.9	≥92.5	≤0.5	≥1000
South Africa	≤0.1	≤4.8	≤1.9	≥92.5	≤0.5	≥1000
California	≤0.1	n.a.	≤1.9	n.a.	n.a.	n.a.
China	n.a.	≤4.0	≤0.5	≥93.0	n.a.	≥1000
Brazil	≤0.1	≤4.0	<Campest.	≥93.0	n.a.	≥1000
India	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

a) When an authentic oil naturally has a campesterol level > 4.0 and ≤ 4.5, it is considered virgin or extra virgin olive oil if the stigmasterol level is ≤ 1.4%, the delta7 stigmastenol level is ≤ 0.3%. The other parameters shall meet the limits set out in the standard.
b) When an authentic oil naturally has a campesterol level > 4.0 and ≤ 4.5, it is considered virgin or extra virgin olive oil if the stigmasterol level Is ≤ 1.4%, the delta7 stigmastenol level is ≤ 0.3% and stigmastadienes is ≤ 0.05 mg/kg. The other parameters shall meet the limits set out in the standard.
c) When an authentic oil naturally has a campesterol level > 4.0 and ≤ 4.5, it is considered virgin or extra virgin olive oil if the delta7 stigmastenol level is ≤ 0,3% and the level of stigmasterol is ≤ 1.6%.
d) Campesterol values between 4.0 and 4.5 would be subject go further testing.

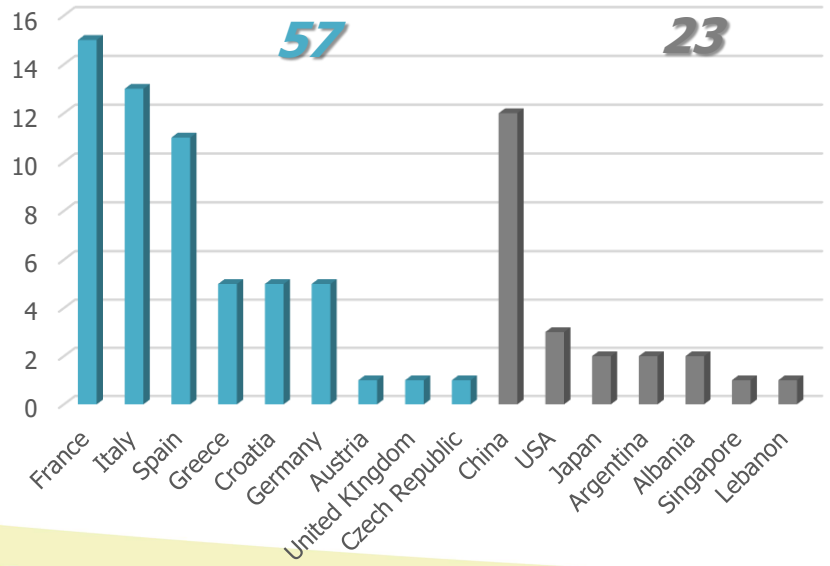
Some results from an on-line questionnaire:

feedback on analytical methods drawbacks, normative failures and inappropriateness, current and emerging frauds

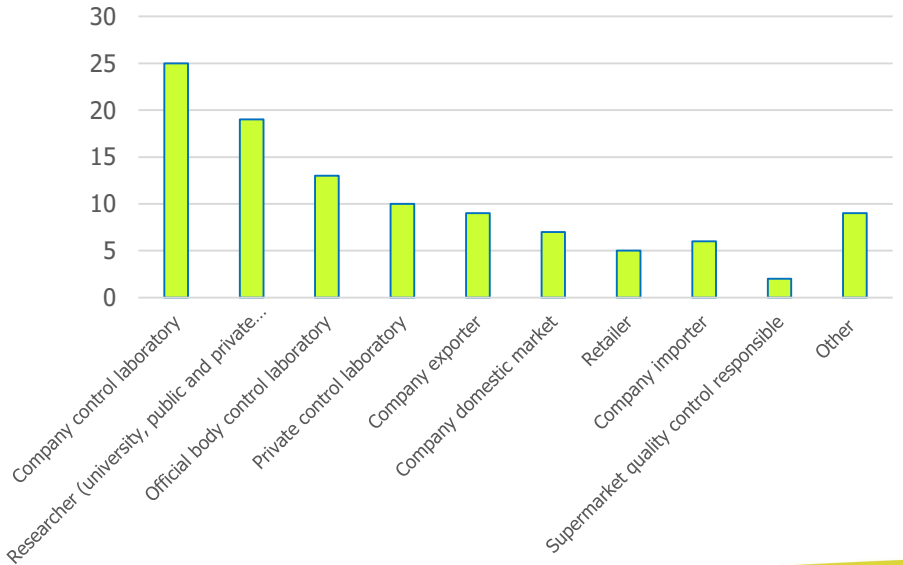
General information

[..] we need to collect information and opinions, so we need your co-operation in answering to some questions, in order to make us able to drive the following activities in the most productive way [..]
The questionnaire will take only about 5 minutes of your time [..]
The results will be available and elaborated in a completely anonymous way [..]

Please, indicate your Country



What is your professional area?

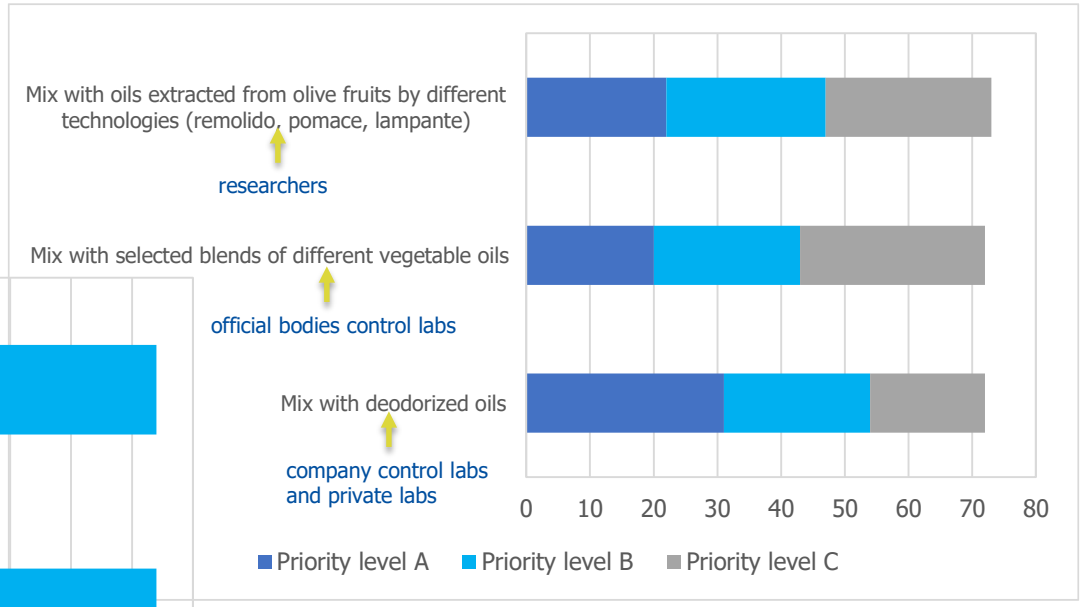


Some results from an on-line questionnaire:

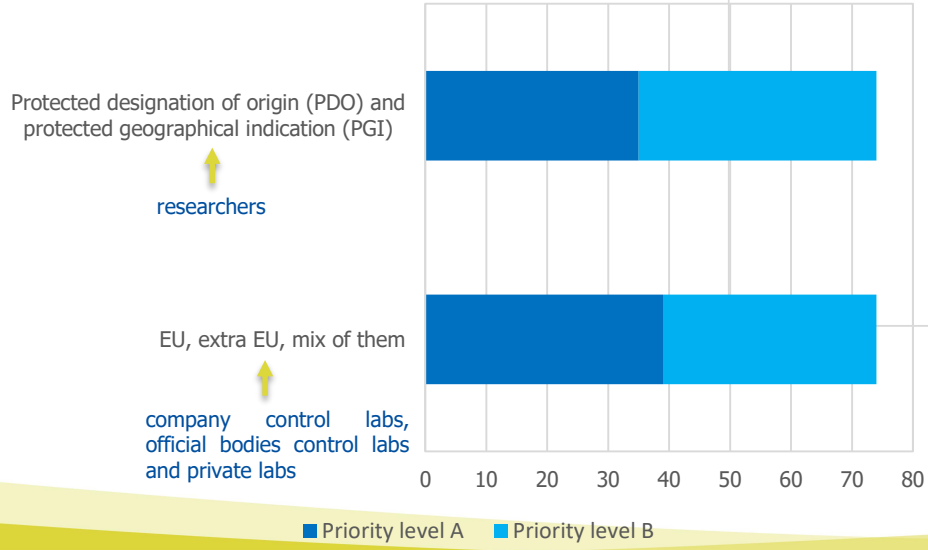
feedback on analytical methods drawbacks, normative failures and inappropriateness, current and emerging frauds

Please, answer the following questions to give information about common and emerging fraud landscape and trends

Olive oils obtained through illicit mixing



Faked declaration of origin



Other common and emerging frauds - Free text

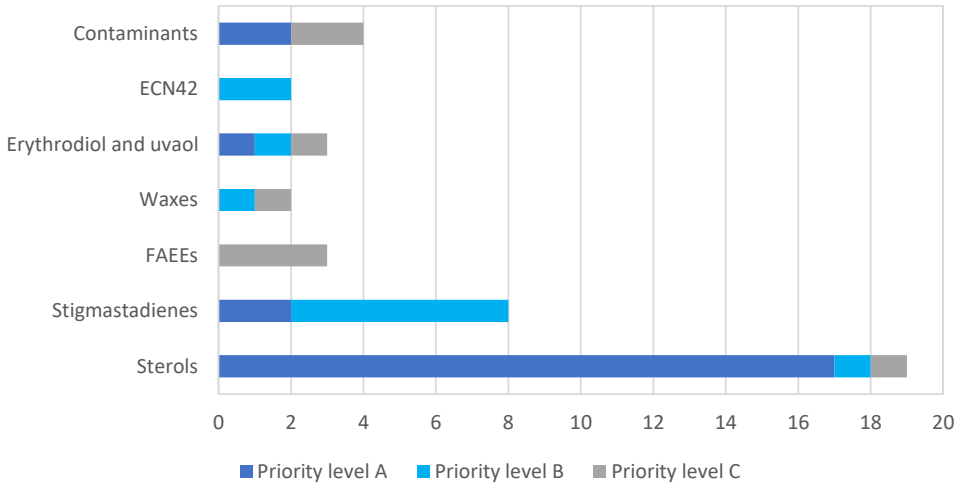
Modification of color and aroma (adding chemicals)

Some results from an on-line questionnaire:

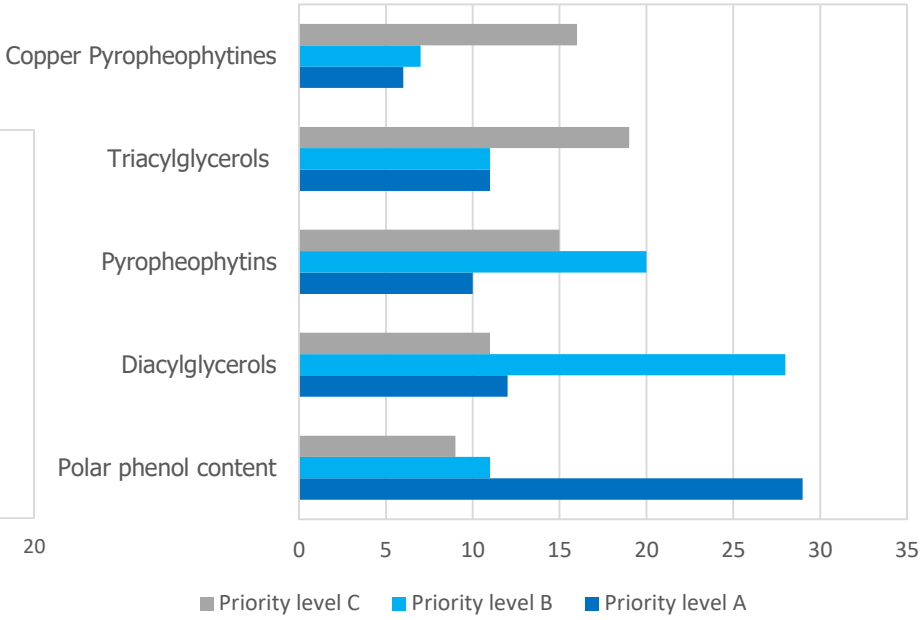
feedback on analytical methods drawbacks, normative failures and inappropriateness, current and emerging frauds

Please, answer the following questions to give your point of view about normative failures and inappropriateness

Methods are too time consuming, if this is the case, please indicate three, ranking them from A (highest priority) to C (lowest priority)



Analytical parameters used in domestic and international trade not supported by official methods within EU regulations



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Olive oil quality and authenticity: a review of current EU legislation, standards, relevant methods of analyses, their drawbacks and recommendations for the future

Lanfranco Conte[a], Alessandra Bendini[b*], Enrico Valli[b], Paolo Lucci[a], Sabrina Moret[a], Alain Maquet[c], Florence Lacoste[d], Paul Brereton[e], Diego Luis García-González[f], Wenceslao Moreda[f], Tullia Gallina Toschi[b]

Scope and Approach

This review will identify current gaps in EU legislation and discuss drawbacks of existing analytical methods with respect to OO. Suggestions for replacement of specific steps within the present EU methods with more efficient analytical solutions to reduce time and/or solvent consumption will be proposed.

Key Findings and Conclusions

This review critiques existing regulatory methods and standards, highlights weaknesses and proposes possible solutions to safeguard the consumer and protect the OO market.

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1 Suggestions for improvements of official analytical protocols

Determination of ethyl esters of fatty acids FAEEs

(adopted by the EU Reg. 61/2011, revised by the EU Reg. 1348/2013 and by the EU Reg. 2095/2016)

- Markers of the quality of olives

QUALITY CONTROL

Limitations and drawbacks of the official procedure

- ❑ Fractionation step by low-pressure column (silica gel)
  - time-consuming and laborious
  - large volumes of solvents (30 ml of hexane + 220 ml of hexane:ethyl ether 99:1) and quantity of silica gel (15 g, previously heated for 4 h in a muffle oven, then cooled, hydrated with 2% water and mantained for 12 h in a desiccator) are used
- ❑ on-column GC injector
  - type of GC injector not so widespread

Oleum

1 Suggestions for improvements of official analytical protocols

Determination of ethyl esters of fatty acids FAEEs

Possible analytical alternatives to be studied *(OLEUM, T4.2)*

Fractionation step by:

- ✓ SPE (1 g of silica gel cartridges)
- ✓ HPLC (silica column)
  - lower solvent volumes requested
  - possibility to mechanize the fractionation step
  - less time-consuming
- ✓ PTV (Programmed Temperature Vaporization) injector for GC

Oleum

2 Suggestions for improvements of official analytical protocols

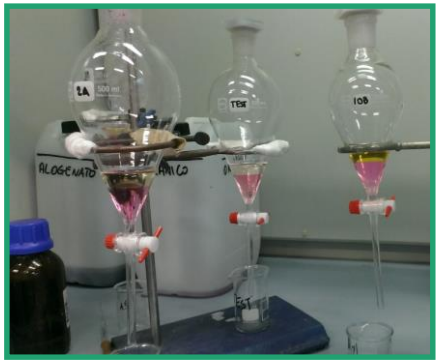
The Sterols composition

(adopted by the EEC Reg. 2568/91)

- Markers of the botanical origin

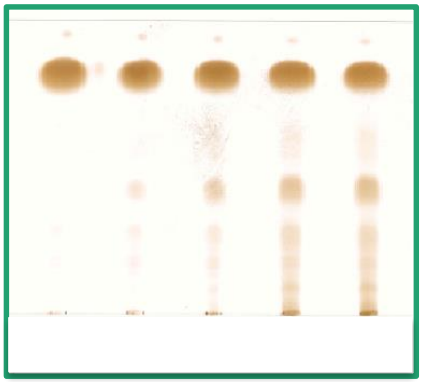
AUTHENTICITY CONTROL

Limitations and drawbacks of the official procedure



✓ Saponification protocol
(KOH in ethanol, extraction by diethyl ether)

- time-consuming and laborious



✓ Fractionation step by TLC

- time-consuming and laborious

Oleum

2 Suggestions for improvements of official analytical protocols

The Sterols composition

(adopted by the EEC Reg. 2568/91)

- Study of the ratio free/esterified sterols

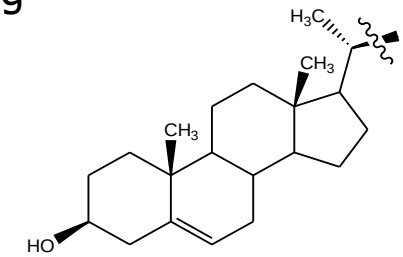
✓ Fractionation step by SPE or HPLC

- lower solvent volumes requested
- possibility to mechanize the fractionation step
- less time-consuming

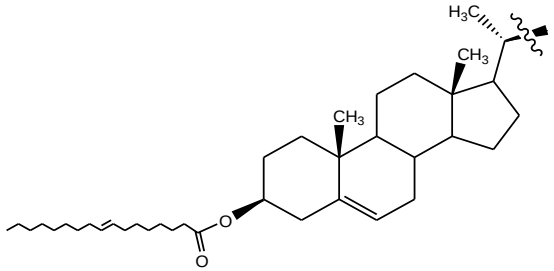
Possible analytical alternative to be studied (OLEUM, T4.3)

OILS	RATIO (Free/Esterified)
Genuine Olive Oils	2.9 - 3.5
Seed Oils	0.01 - 0.09

CSIC



Free Sterol



Esterified sterol (sterolic waxes)

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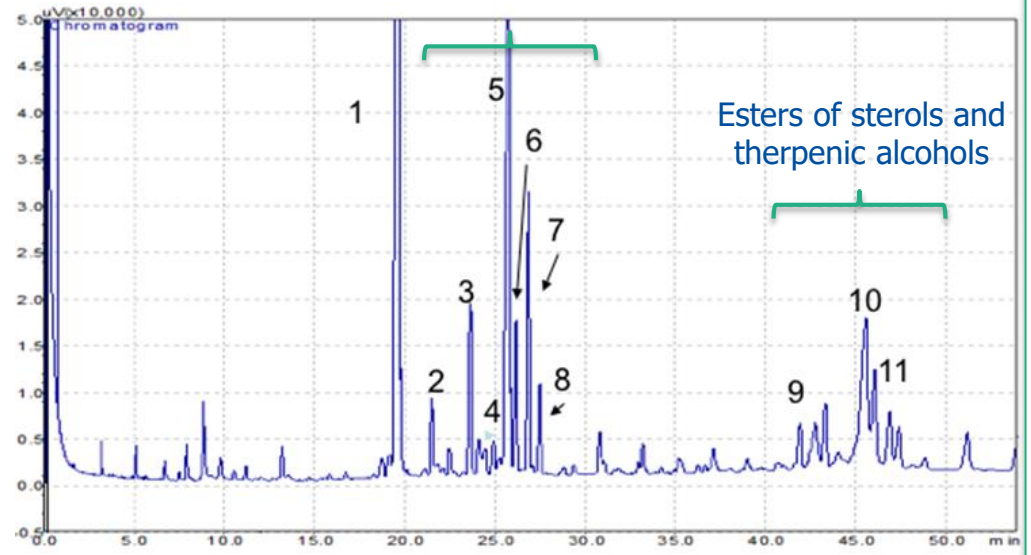
2 Suggestions for improvements of official analytical protocols

The Sterols composition

(adopted by the EEC Reg. 2568/91)

- Study of the ratio free/esterified sterols

Free sterols



GC trace of free and esterified minor components of refined olive oil mixed with high oleic sunflower oil (50/50 w/w) performed by UNIUD. Peak identification: **1**, Squalene; **2**, 5-alpha cholestanol (I.S.); **3**, Campesterol; **4**, Stigmasterol; **5**, β-sitosterol; **6**, Δ5-avenasterol; **7**, Δ7-stigmastenol, **8**, Erhytrodiol; **9**, Cholesteryl heptadecanoate (I.S.); **10**, β-sitosteryl oleate; **11**, Δ7-stigmastenol oleate.

Possible analytical alternative to be studied (OLEUM, T4.3)

Under finalization

Conclusions and future trends

This overview, developed within the context of the EU project H2020 OLEUM, highlights:

- need to harmonize the existing international and national technical norms to ensure the fair trade and the consumer protection in the olive oil sector
- need for a constant updating to effectively prevent emerging frauds (next OLEUM collaboration with EU FFN contact points)
- weak points of existing analytical methods adopted in the olive oil quality and authenticity control

Oleum

Thank you for your attention

http://www.oleumproject.eu/

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