European Commission
Agriculture and Rural Development

copa*cogeca
european farmers   european agri-cooperatives

Oleum

Better solutions to protect olive oil quality and authenticity

The OLEUM project results

Prof. Tullia Gallina Toschi

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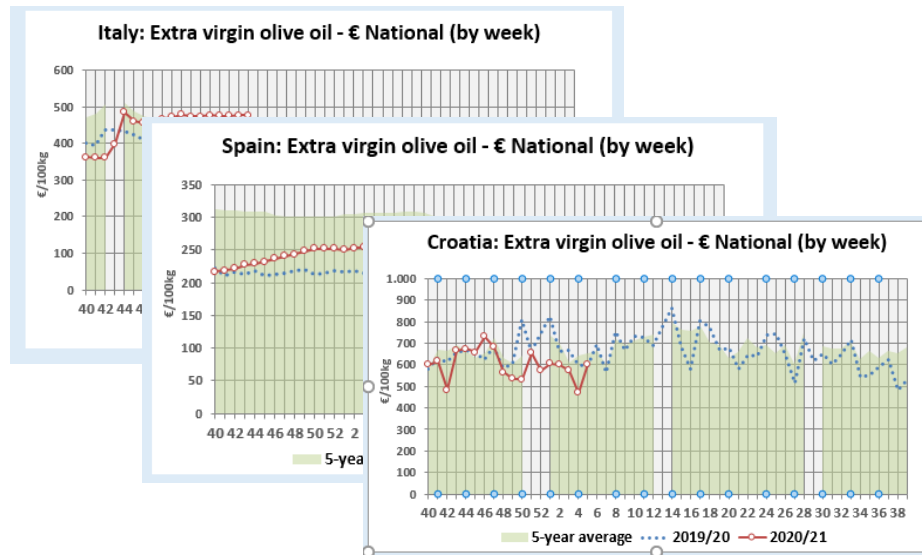
19 November - 2021

Olive oil always in the spotlight. Why?

- Being the **TOP HEALTHY OIL**, key of the Mediterranean diet, high value, mixable (as any liquid) → of interest for fraudsters (and of competitors..) → reputation

- Many categories, **not enough known by the consumer**, who finds on the market and consequently buys, for any use, (mainly) EVOO **paying** often **less than the correct** (and **sustainable**) **value**.

The European Commission's annual report on food fraud

✓ Every year, a **report describing the activities carried out by** the **EU Food Fraud Network** (FFN) and the **Administrative Assistance and Cooperation System** (AAC) is published.

✓ **The list** of cases **registered by AAC does not represent the totality of non-compliances and suspicions of food fraud** occurring throughout Europe, as it does not include suspected fraud cases that concern only the national level.

Food Fraud requests created in the AAC system per year

The number of requests for assistance and cooperation shared between Member States tends to increase over the years and this is a positive signal in terms of transparency and sharing of information.

https://ec.europa.eu/food/system/files/2021-09/ff_ffn_annual-report_2020_1.pdf

Olive oil always in the spotlight. Why?

Monthly prices of extra virgin, virgin and lampante olive oil (national average) € /100 kg.
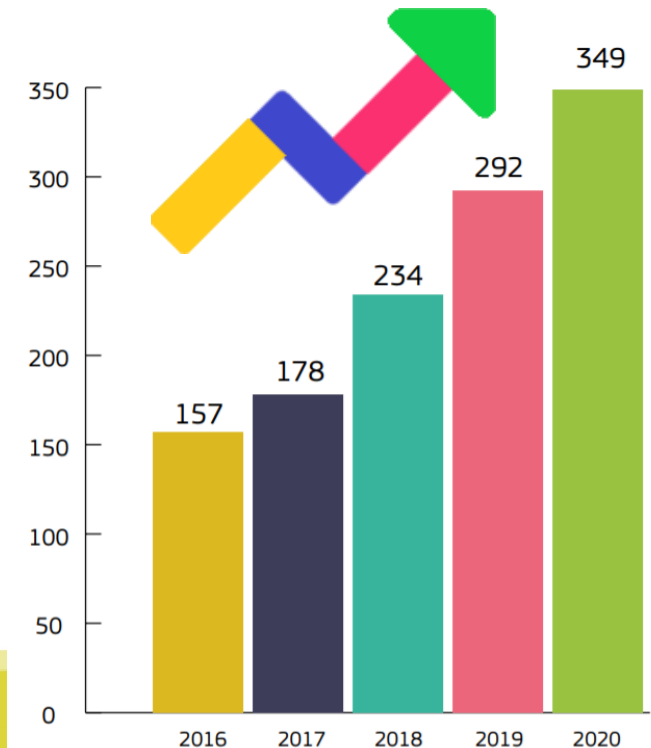
"Fats and oils" (**51**), where majority of cases and olive oil was the most notified product category in the AAC-FF system both in 2019 and in 2020.

Bar chart categories and values:
- fats and oils: 51
- fish and fish products: 34
- cats and dogs: 33
- poultry meat and poultry meat products: 28
- honey and royal jelly: 25
- meat and meat products (other than poultry): 25
- dietetic foods, food supplements, fortified foods: 22
- fruits and vegetables: 21
- horses: 17
- wine and alcoholic beverages: 12
- plant protection product: 11
- cephalopods, crustaceans and products thereof: 10
- herbs and spices: 9
- milk and milk products: 8
- Others: 43

EVOO remains **one** of the **most highly targeted products by fraudsters**.

This is also **due to**: 1) absence of **virgin olive oil** from the supermarkets and small shops (where does all the virgin go? Horeca?); 2) different values according with the different geographical origin and no documental or analytical method globally accepted to certify it.

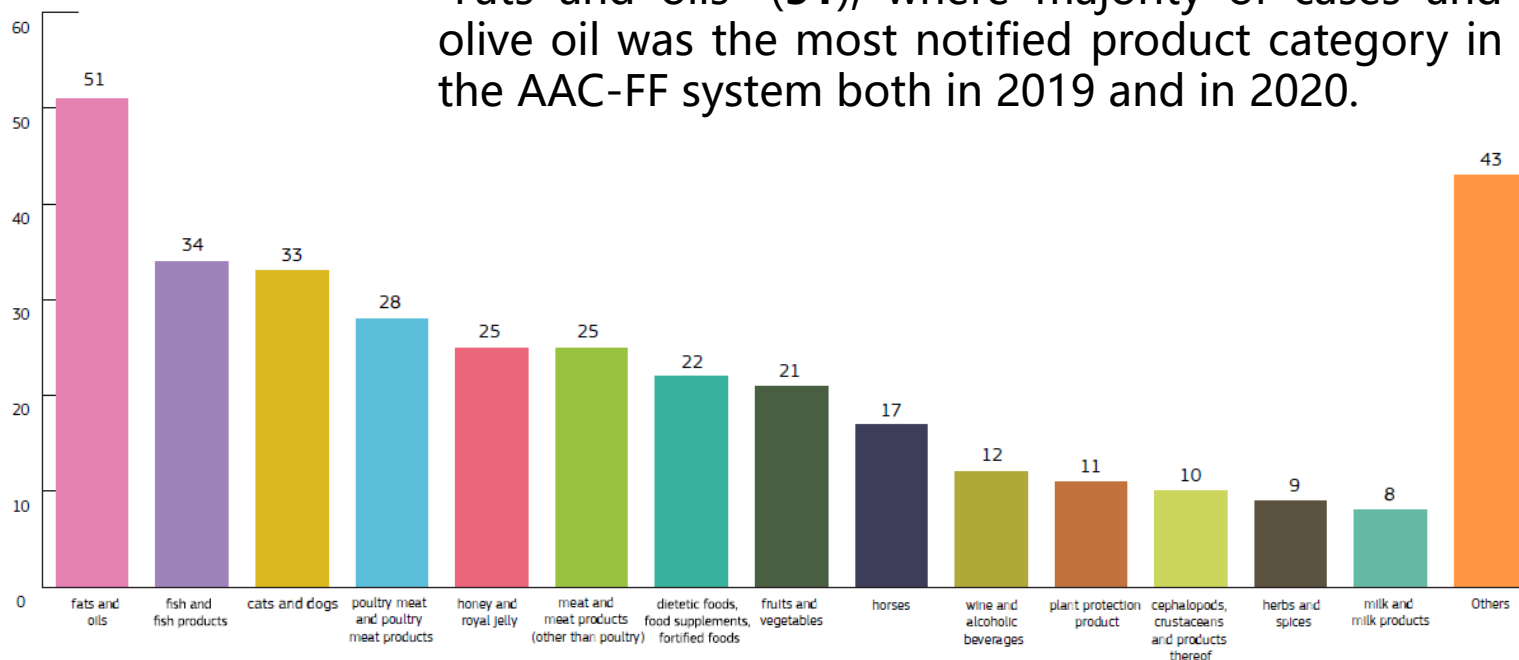**EXTRA VIRGIN** max. 0.8°
Sep-2021

			Evolution since last **month**	Evolution since last **year**
SPAIN	327.3	€/100 kg	1%	55%
ITALY	420.6	€/100 kg	-1%	13%
GREECE	320.0	€/100 kg	3%	48%

VIRGIN max. 2°
Sep-2021

			Evolution since last **month**	Evolution since last **year**
SPAIN	309.4	€/100 kg	2%	67%
ITALY	274.2	€/100 kg	-2%	41%
GREECE	246.0	€/100 kg	45%	54%

LAMPANTE > 2°
Sep-2021

			Evolution since last **month**	Evolution since last **year**
SPAIN	293.4	€/100 kg	3%	68%
ITALY	244.0	€/100 kg	1%	60%
GREECE	216.0	€/100 kg	8%	80%

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Most common infringements in the olive oil sector

Marketing of VOO as EVOO, <u>or</u> the **marketing as OOs of blends of other vegetable oils** (sunflower, corn, palm, rapeseed, etc.) **with OO**.

To ensure the health and protection of consumers, **JRC** publishes a **monthly summary** with **newspaper articles on food fraud**, with the aim of informing all the stakeholders and giving them the opportunity to act on these irregularities.

Study on the implementation of conformity checks in the olive oil sector throughout the European Union

Contract AGRI-2018-0485

Areté Research & Consulting in Economics (2020)
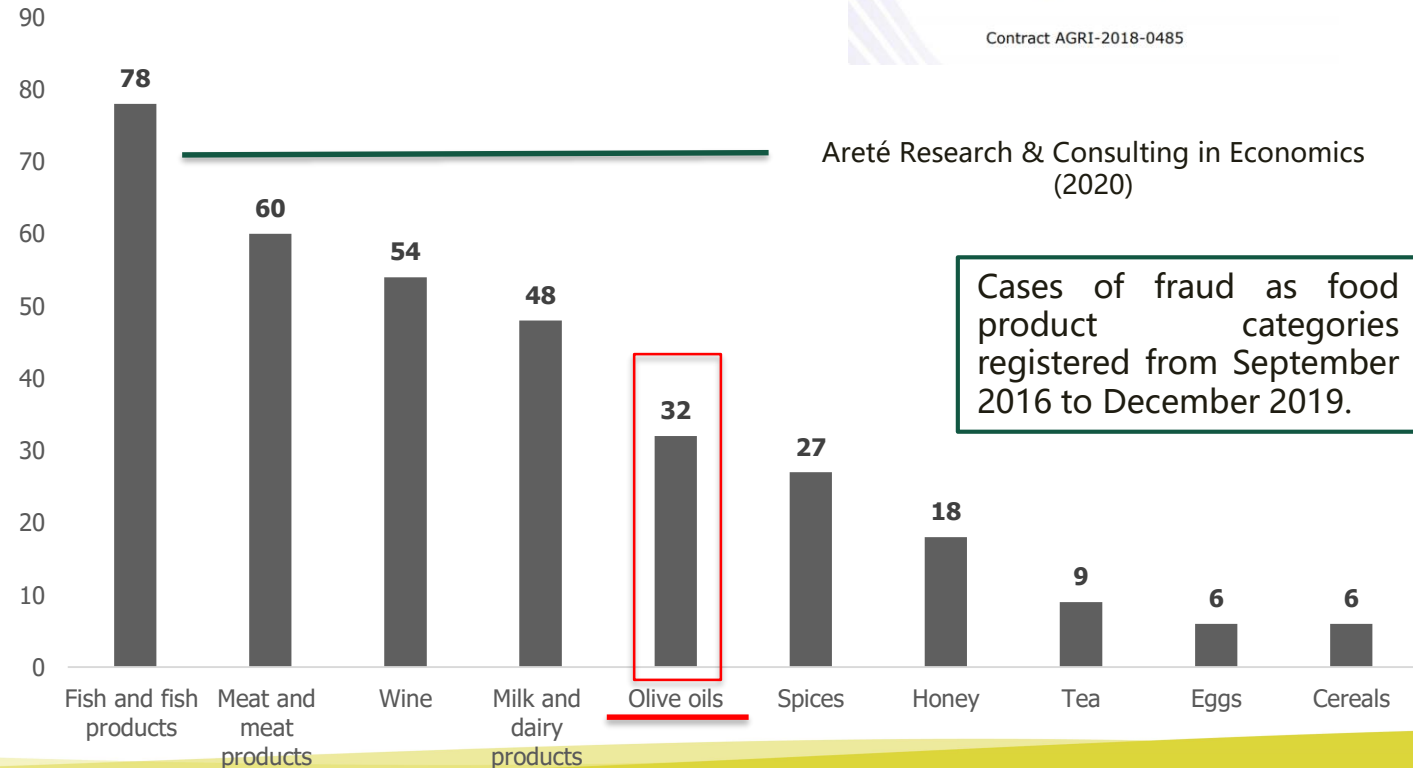
Cases of fraud as food product categories registered from September 2016 to December 2019.

Category	Value
Fish and fish products	78
Meat and meat products	60
Wine	54
Milk and dairy products	48
Olive oils	32
Spices	27
Honey	18
Tea	9
Eggs	6
Cereals	6

The OLEUM on-line survey

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Questionnaire Oleum project
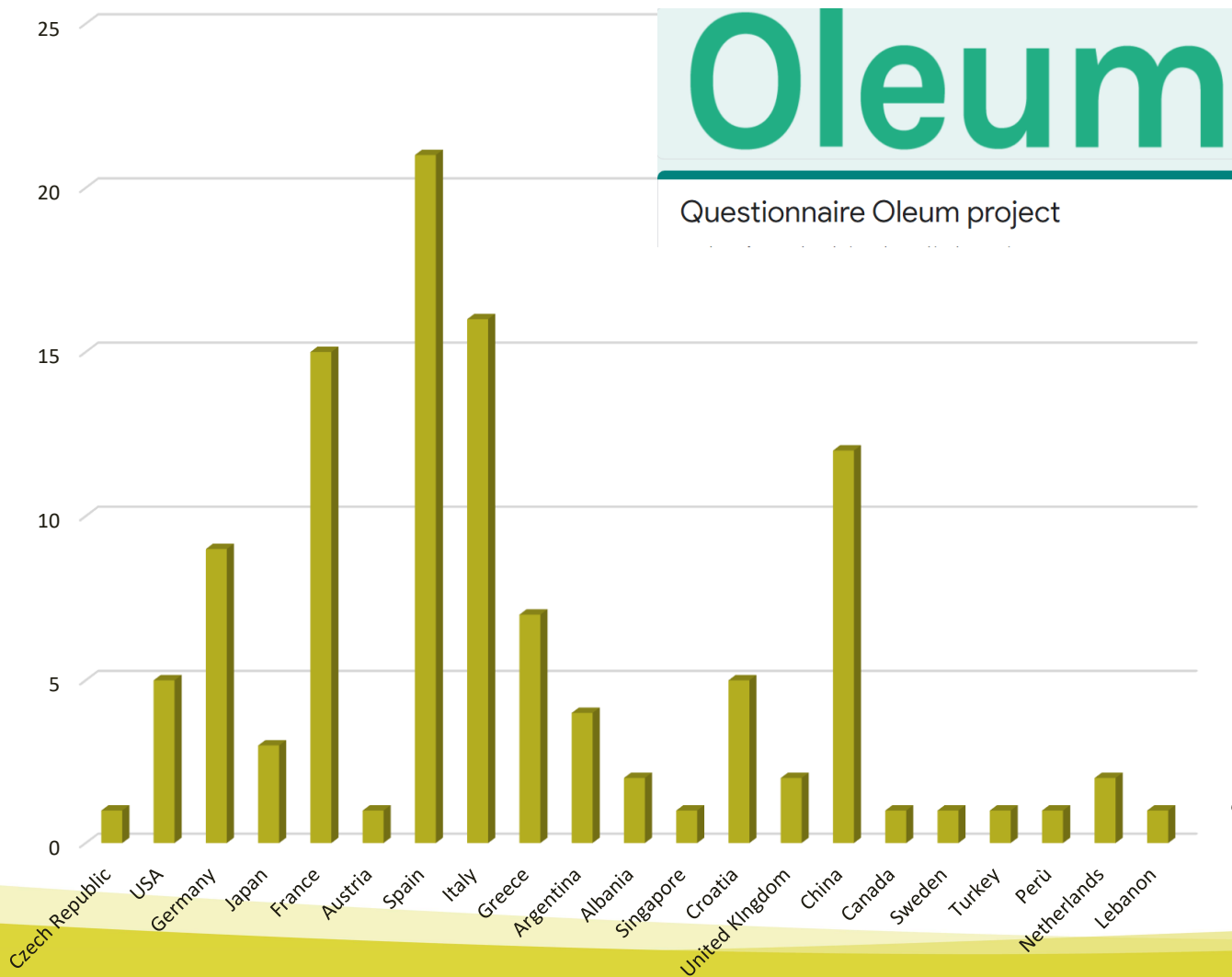
Sent by e-mail during 2018 **to over 200 stakeholders**. A total of **111 completed** questionnaires were returned **from** both **European** (87 questionnaires) **and non-European** (24 questionnaires) **countries**.
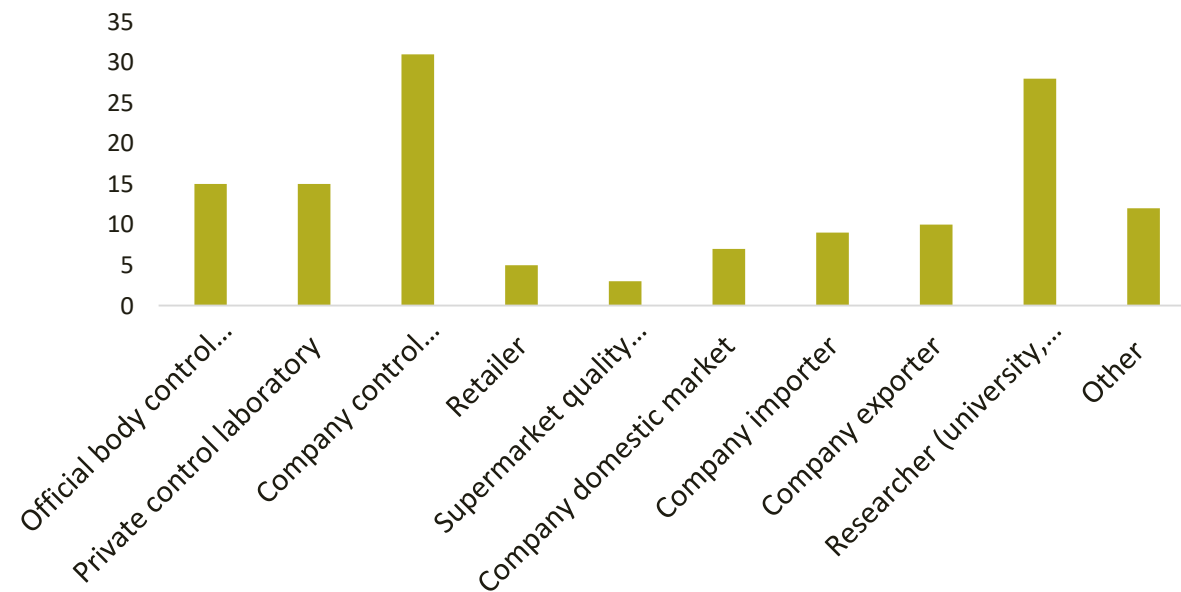
OLEUM questionnaire addressed to the EU FFN

✓ In order to support the olive oil sector, **under the guidance of the European Commission DG AGRI** (Unit G.4 – Arable crops and OO) **and DG SANTE** (Unit G.5 - Alerts, Traceability and Committees), **a questionnaire specifically addressed to the EU FFN** National Contact Points **has been developed** and sent during 2018.

✓ The **aim** was to **acquire consolidated reports by the control bodies on** the occurrence of **common and emerging fraud issues**.

✓ From the analysis of the responses received:

✓ most frequent fraudulent practice is **mixing with lower quality olive oil**;
✓ oils EU, non-EU and mix of EU and non-EU oils are the cases which need more control activities in relation to **false designations of origin**.
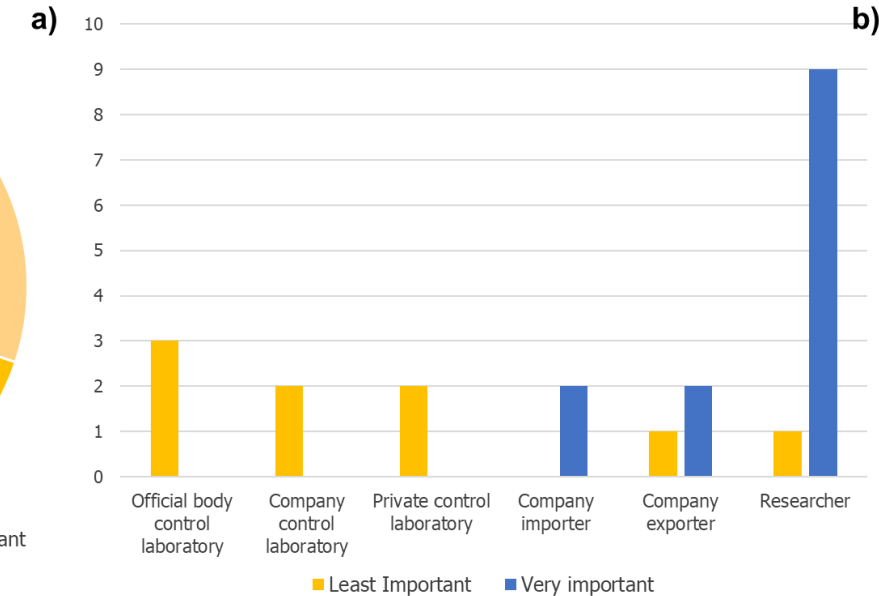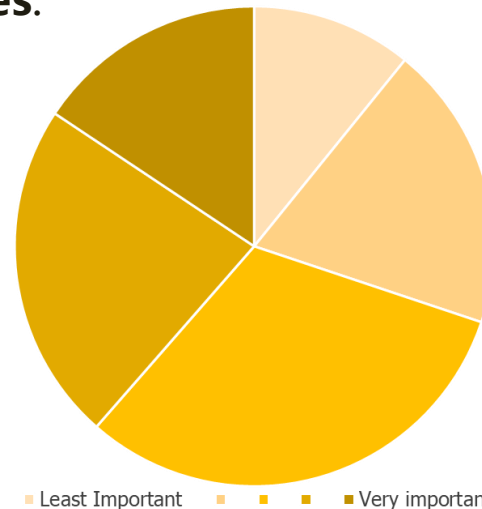
OLEUM questionnaire on the update and delivery of the appearance of common and emerging frauds

Name of the Food Fraud contact point:
Country:
Name(s) and role(s) of the person(s) who is (are) filling in the questionnaire:

a)

■ Least Important   ■ ■ ■ Very important

b)

■ Least Important   ■ Very important

Official body control laboratory | Company control laboratory | Private control laboratory | Company importer | Company exporter | Researcher

OLEUM related publications

Food Control 124 (2021) 107902

Contents lists available at ScienceDirect

Food Control

journal homepage: www.elsevier.com/locate/foodcont

Emerging trends in olive oil fraud and possible countermeasures

Enrico Casadei [a], Enrico Valli [a,*], Filippo Panni [a], James Donarski [b], Jordina Farrús Gubern [b], Paolo Lucci [c], Lanfranco Conte [c], Florence Lacoste [d], Alain Maquet [e], Paul Brereton [f], Alessandra Bendini [a], Tullia Gallina Toschi [a]

Trends in Food Science & Technology 105 (2020) 483–493

Contents lists available at ScienceDirect

Trends in Food Science & Technology

journal homepage: www.elsevier.com/locate/tifs

Review

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]

Authentication of olive oil
TOPIC ID: SFS-14a-2014

Programme
Horizon 2020 Framework Programme

Call
Sustainable Food Security (H2020-SFS-2014-2015)

Type of action
RIA Research and Innovation action

Topic description

Scope:

Specific challenge: the EU is the world largest producer, consumer and exporter of olive oil. Olive oil is normally sold at a higher price than other vegetable oils and fraudulent activities are tempting. To preserve the image of olive oil, it is necessary to guarantee its quality and authenticity. Olive oil characteristics are regulated at EU level by Regulation (EEC) N° 2568/91 which establishes a list of physical, chemical and organoleptic characteristics as well as methods for their analysis. The list and the methods are updated to include the existing scientific knowledge. Yet despite these regular revisions some issues have not yet found proper solutions. In particular there is a need for the development, validation and pre- as well as co-normative activities followed by the standardization of a method for the assessment of the organoleptic characteristics based on the existing methods, reference materials and already performed research and development work. The specific challenge consists in developing, validating and harmonising analytical methods and quality parameters that specifically address technical authenticity issues. These issues concern in particular 1) the blend of extra-virgin olive oil or virgin olive oil with soft deodorised olive oil, 2) the blend of extra-virgin olive oil or virgin olive oil with other vegetable oil. Beyond the case of olive oil, there is also a strong need for better coordination of research in the area of food authenticity, integrity and traceability across the food supply chain between Member States and Associated Countries.
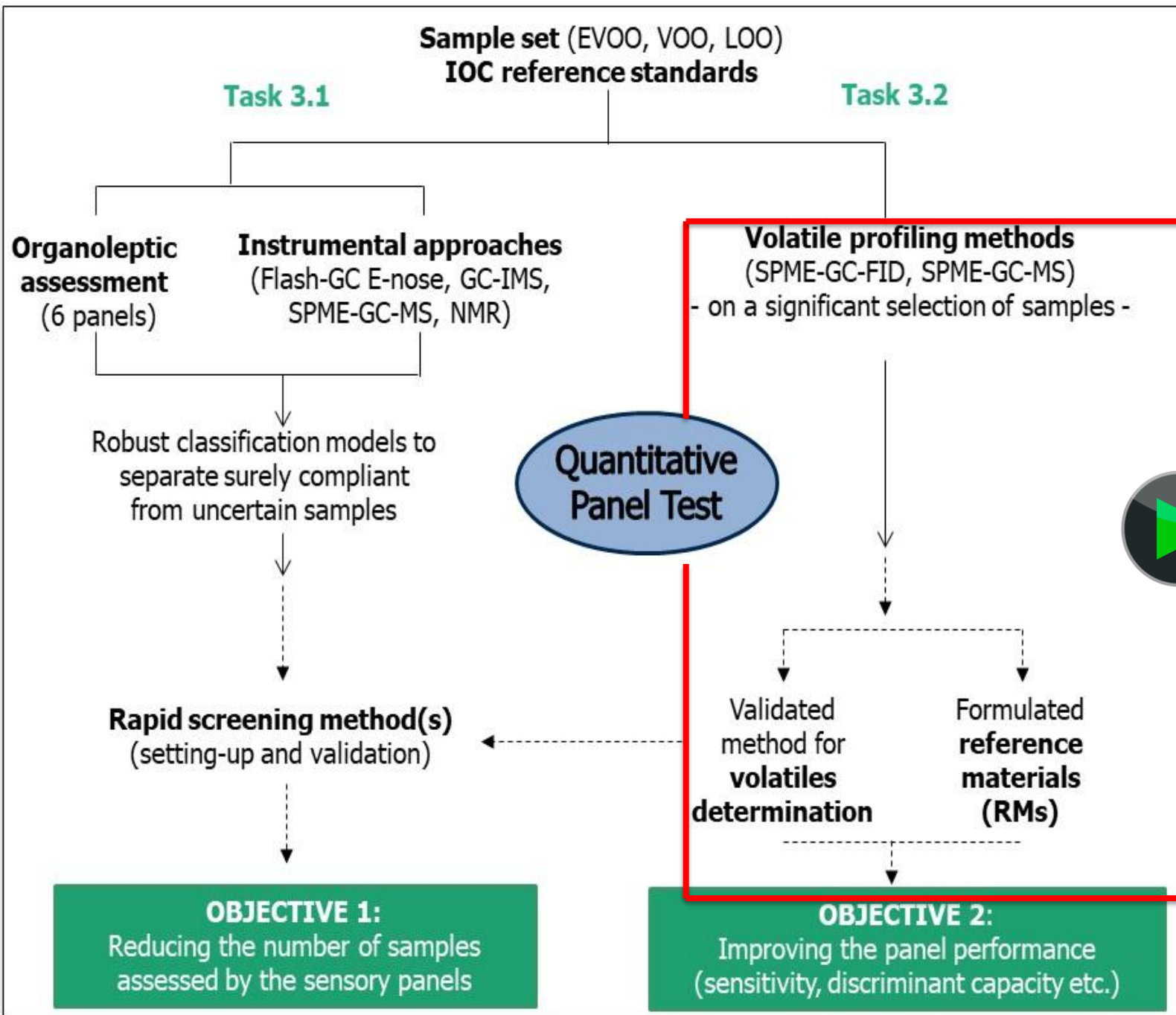
SPME-GC-FID/MS targeted methods for the analysis of selected volatile compounds in virgin olive oils

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Standard Operating Procedure

Method 4A:

Analysis of volatile compounds in virgin olive oil by Gas Chromatography

Two method versions included plus a guide on the preparation of the calibration curves

a. SOP for the SPME-GC-FID version
b. SOP for the SPME-GC-MS version
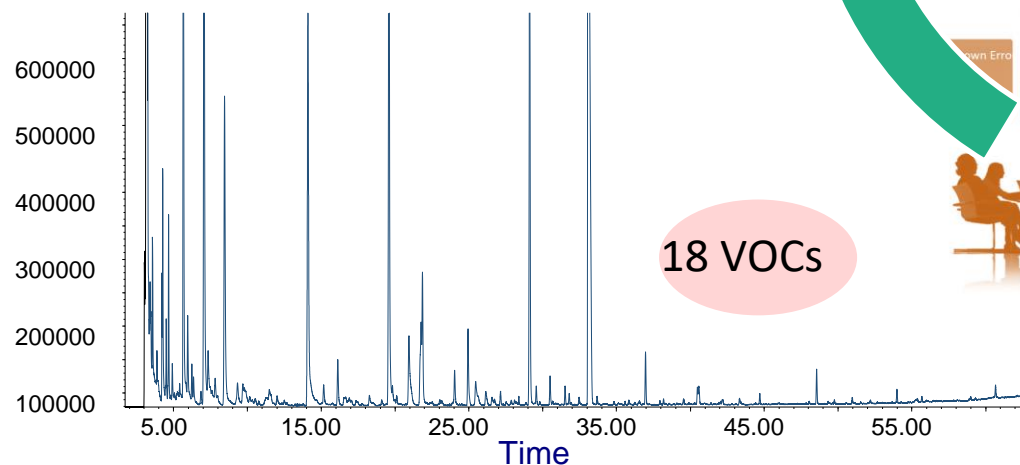c. Guide document on calibration curves

Targeted approach

a. SOP for the SPME-GC-**FID** version

b. SOP for the SPME-GC-**MS** version

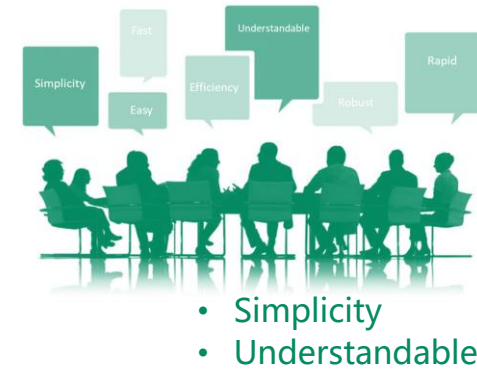c. Guide document on **calibration curves**

18 VOCs

Scientific community

- Extraction capacity
- Maximum information

Olive oil actors

- Simplicity
- Understandable

Regulation bodies

- Clear definition of purpose
- Internationally accepted

18 selected volatile compounds (the minimum number of highly diagnostic sensory markers)

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Negative attributes (defects)

Fusty/muddy sediment (Total: 5)
Octane
Ethanol
3-methyl-1-butanol
Propanoic acid
6-methyl-5-hepten-2-one

Winey-vinegary (Total: 3)
Acetic acid
Ethyl acetate
Ethanol

Musty-humid-earthy (Total: 3)
(E)-2-heptenal
1-octen-3-ol
Propanoic acid

Frostbitten olives (wet wood) (Total: 1)
Ethyl propanoate

Rancid (Total: 5)
Hexanal
Nonanal
(E,E)-2,4-hexadienal
(E)-2-decenal
Pentanoic acid

Positive attribute (fruity)

Fruity (green notes) (Total: 3)
(E)-2-hexenal
(Z)-3-hexenyl acetate
1-hexanol

1 procedure
2 detectors (FID and MS)

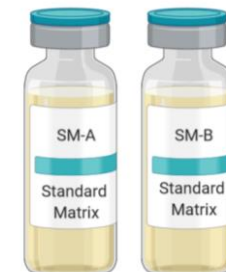**Measurand:** 18 selected volatile compounds (VOCs) in virgin olive oils (in **mg/kg**).
**Selection criteria:** Those VOCs with a demonstrated influence on aroma (sensory defects).

18 VOCs

Fermentative defects *(fusty/muddy, winey vinegary, musty)*
+ Damaged olives + Oxidation *(rancid)* + Positive attributes **(fruity)**

1. **Octane**
2. **Ethyl acetate**
3. **Ethanol**
4. **Ethyl propanoate**
5. **Hexanal**
6. **3-Methyl-1-butanol**
7. **(*E*)-2-Hexenal**
8. **(*Z*)-3-Hexenyl acetate**
9. **(*E*)-2-Heptenal**

10. **6-Methyl-5-hepten-2-one**
11. **1-Hexanol**
12. **Nonanal**
13. **1-Octen-3-ol**
14. **(*E,E*)-2,4-Hexadienal**
15. **Acetic acid**
16. **Propanoic acid**
17. **(*E*)-2-Decenal**
18. **Pentanoic acid**

***Internal standard: 4-methyl-2-pentanol**

2 Standard mixtures to simplify the analysis: SM A & SM B

SM-A   SM-B
Standard Matrix   Standard Matrix

Balance between overlapping at high concentrations, competition phenomena, and concentration ranges.

4-Methyl-2-pentanol

SM-A

SM-B
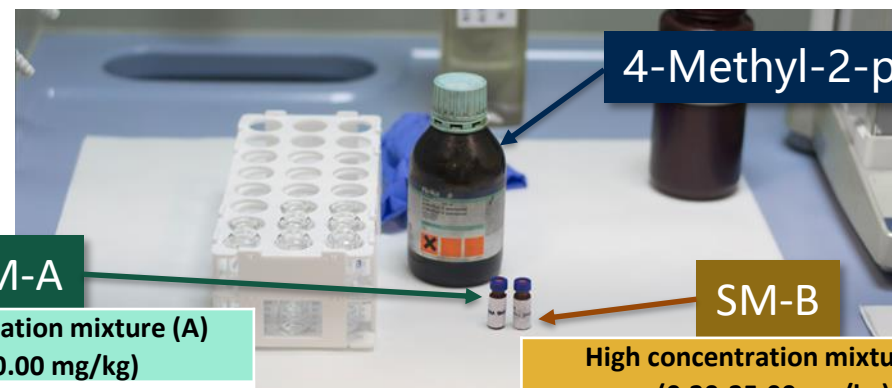
Low concentration mixture (A) (0.05-10.00 mg/kg)
Octane
Ethyl acetate
Ethyl propanoate
3-Methyl-1-butanol
(*E*)-2-Heptenal
6-Methyl-5-hepten-2-one
(*E,E*)-2,4-hexadienal
Propanoic acid
(*E*)-2-Decenal
Pentanoic acid

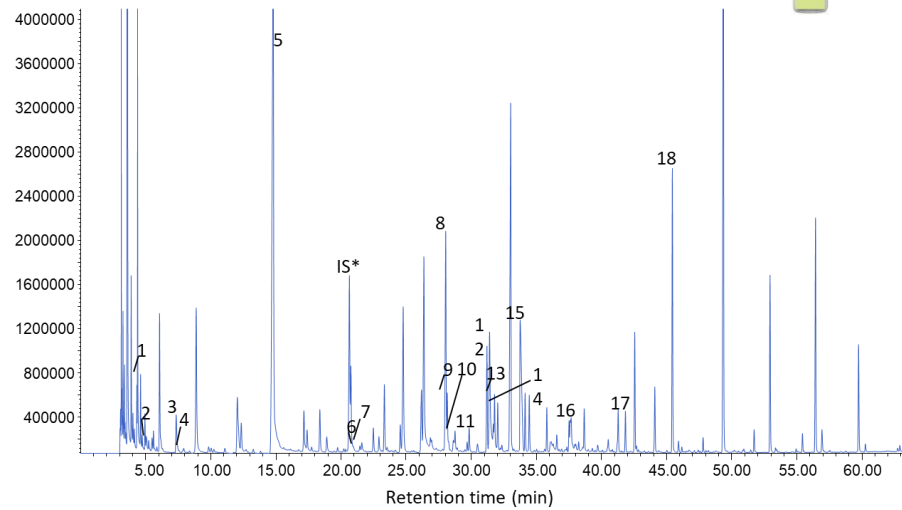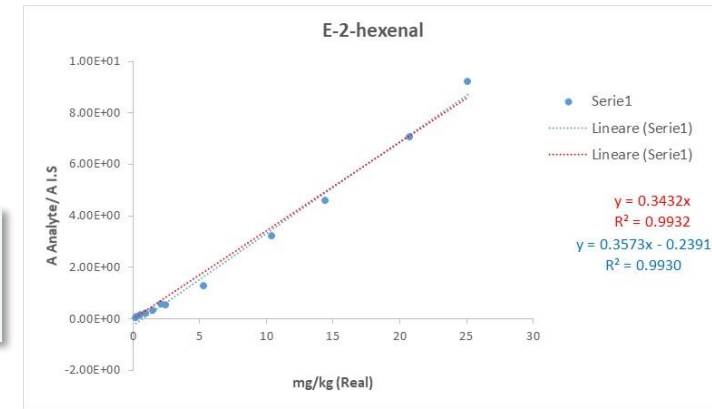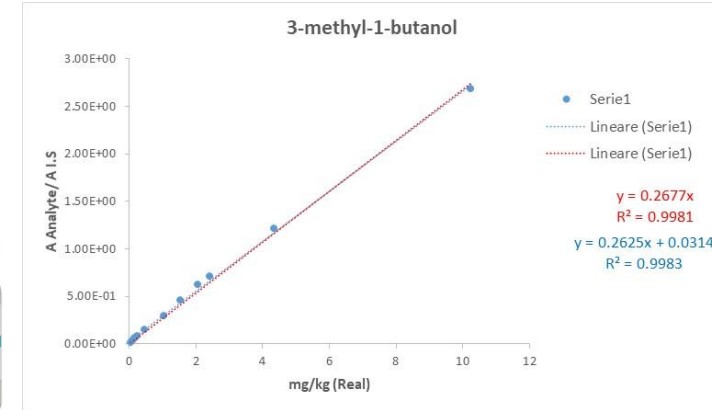| High concentration mixture (B) (0.20-25.00 mg/kg) |
| --- |
| Ethanol |
| Hexanal |
| (*E*)-2-Hexenal |
| (*Z*)-3-Hexenyl acetate |
| 1-Hexanol |
| Nonanal |
| 1-Octen-3-ol |
| Acetic acid |

SAMPLE PREPARATION

Virgin olive oil sample

I.S. 4-methyl-2-pentanol
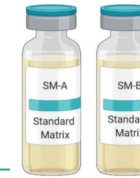
Calibration solutions **(18 VOCs)**

Chromatographic areas

Sample → Chromatographic areas → Quantification **(Calibration)** → Concentration (mg/kg)

I.S.

RESULTS

QM1

Linearity (R²)
- FID 0.96 (0.94- 0.99)
- MS 0.98 (0.94 0.99)

Repeatability (RSD%)
- FID 11.53 (6.50-15.60)
- MS 7.60 (3.89-17.23)

Reproducibility (RSD%)
- FID 39 (12-122)
- MS 31 (13-64) → Ethyl propanoate (below LOQ)

Recovery (%)
- FID 89 (50-160)
- MS 94 (72-106)

Precision IS (RSD%)
- FID 7.56
- MS 9.66

LOD (mg/kg)
- FID 0.08 (0.01-0.6)
- MS 0.03 (0.01-0.18) → (E)-2-decenal

LOQ (mg/kg)
- FID 0.246 (0.01-1.93)
- MS 0.08   (0.01-0.53)

QM2 may provide better results in repeatability, but worse in reproducibility, linearity and recovery.

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journal homepage: www.elsevier.com/locate/foodcont

ELSEVIER

Peer inter-laboratory validation study of a harmonized SPME-GC-FID method for the analysis of selected volatile compounds in virgin olive oils

Enrico Casadei [a], Enrico Valli [a], Ramón Aparicio-Ruiz [b, *], Clemente Ortiz-Romero [b], Diego L. García-González [b], Stefania Vichi [c], Beatriz Quintanilla-Casas [c], Alba Tres [c], Alessandra Bendini [a], Tullia Gallina Toschi [a]

Check for updates

SPME-GC-MS method in course of publication

Next phase: agreement on the limits and ranges

Collection of data in order to establish limits and ranges of volatile compounds.

Octane

Average: 0.09 ppm Dev.std: 0.07
Average: 0.14 ppm Dev.std: 0.08
Average: 0.46 ppm Dev.std: 0.51

(E)-2-hexenal

Average: 14.24 ppm Dev.std: 9.68
Average: 6.40 ppm Dev.std: 3.22
Average: 2.65 ppm Dev.std: 3.09
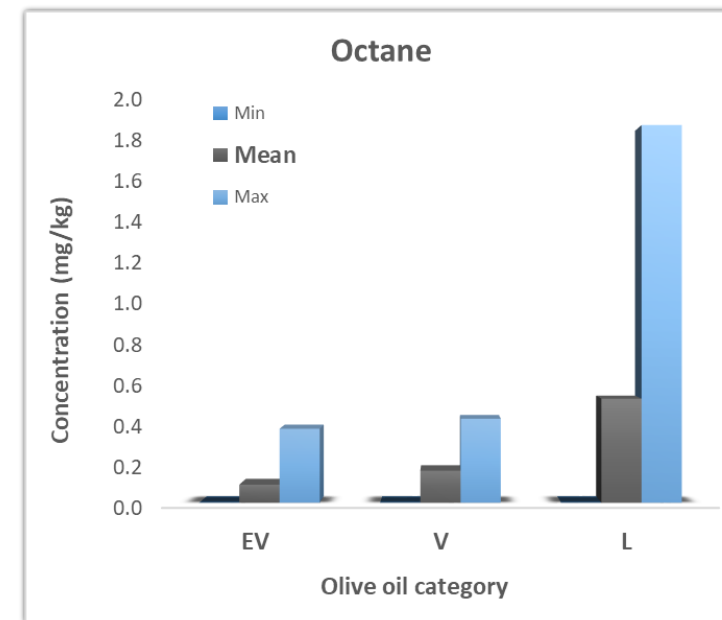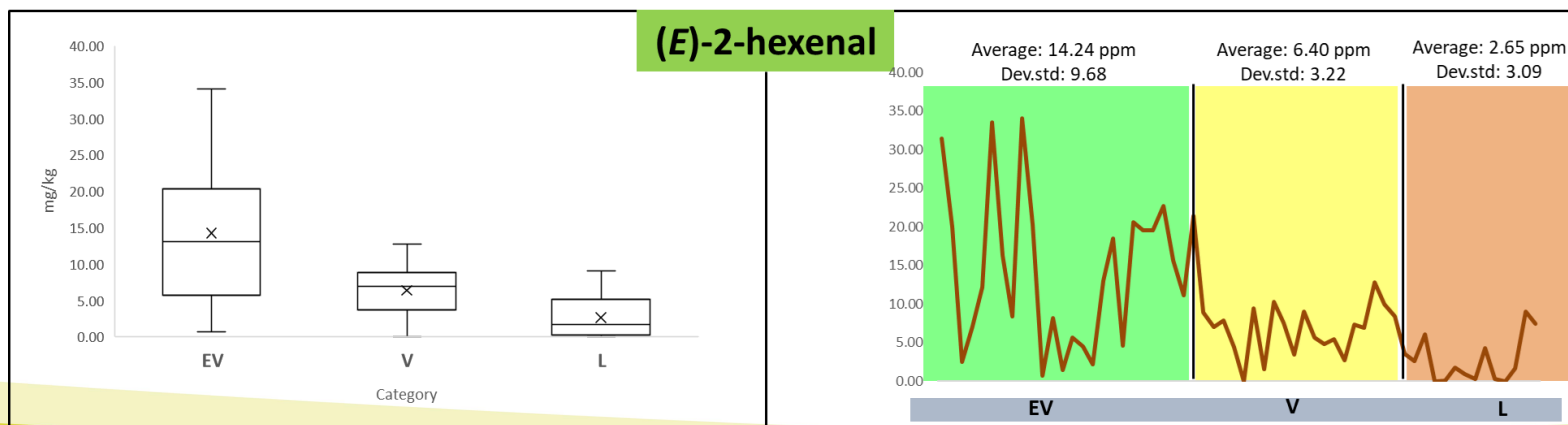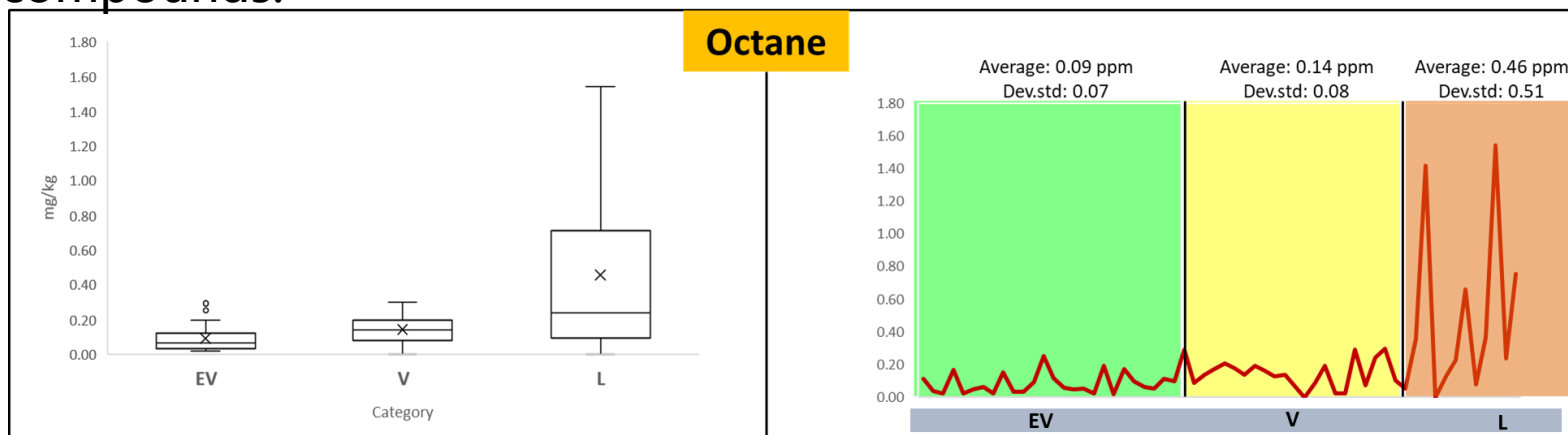
Definition of limits and ranges

- Reliable data on EVOO, VOO and LOO.
- Representative samples (well representative of categories and defects).
- Interlab perspective (NB reproducibility).

Panel 1

Panel 2

Classification Panel 1   Classification Panel 2

Final aim of the SPME/GC methods (targeted and suitable for regulatory adoption) is:

Panel 1 = Panel 2

Classification confirmed

Panel 1 ≠ Panel 2

Targeted instrumental approach

Limits and ranges of specific volatiles
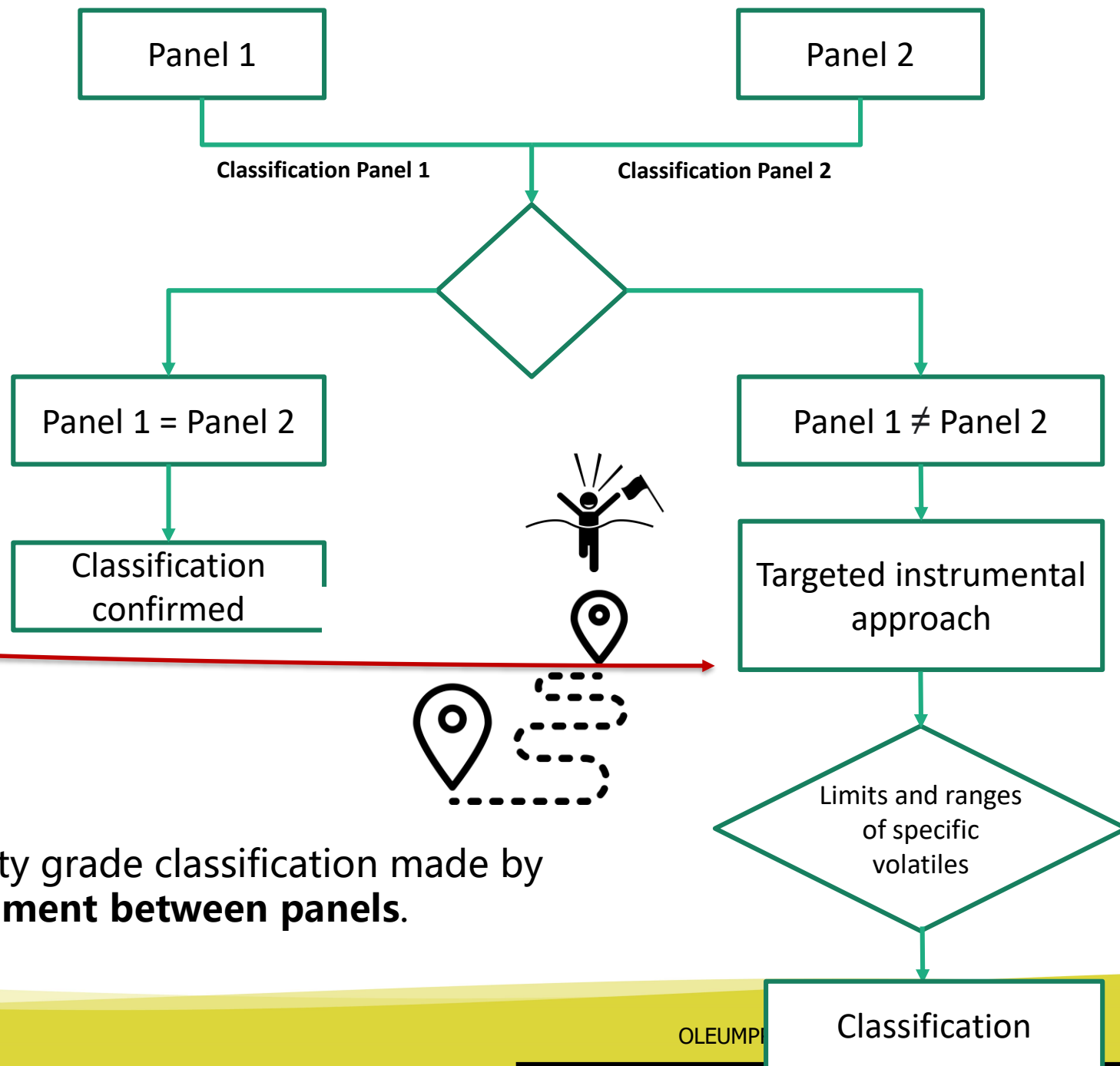
To confirm or disconfirm the quality grade classification made by the Panel test, **in case of disagreement between panels**.

Classification

Validated screening methods to support the Panel Test

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An HS-GC-IMS Method for the Quality Classification of Virgin Olive Oils as Screening Support for the Panel Test

Enrico Valli [1,2], Filippo Panni [1], Enrico Casadei [1,*], Sara Barbieri [3], Chiara Cevoli [1,2], Alessandra Bendini [1,2], Diego L. García-González [4] and Tullia Gallina Toschi [1,2]

Flash Gas Chromatography in Tandem with Chemometrics: A Rapid Screening Tool for Quality Grades of Virgin Olive Oils

Sara Barbieri [1], Chiara Cevoli [1], Alessandra Bendini [1,*], Beatriz Quintanilla-Casas [2,3], Diego Luis García-González [4] and Tullia Gallina Toschi [1]

Virgin olive oil volatile fingerprint and chemometrics: Towards an instrumental screening tool to grade the sensory quality

Beatriz Quintanilla-Casas [a,b], Julen Bustamante [a,b], Francesc Guardiola [a,b], Diego Luís García-González [c], Sara Barbieri [d], Alessandra Bendini [d], Tullia Gallina Toschi [d], Stefania Vichi [a,b,*], Alba Tres [a,b]

❑ HS-GC-IMS	73-94%
❑ FGC-E-nose untargeted approach	78-92%
❑ HS-SPME-GC/MS untargeted approach	93-100%
❑ ¹H-NMR untargeted approach	80-95%

Sequential model

1st step

EVOO vs. non-EVOO

EVOO

2nd step

VOO vs. LOO

LOO   VOO
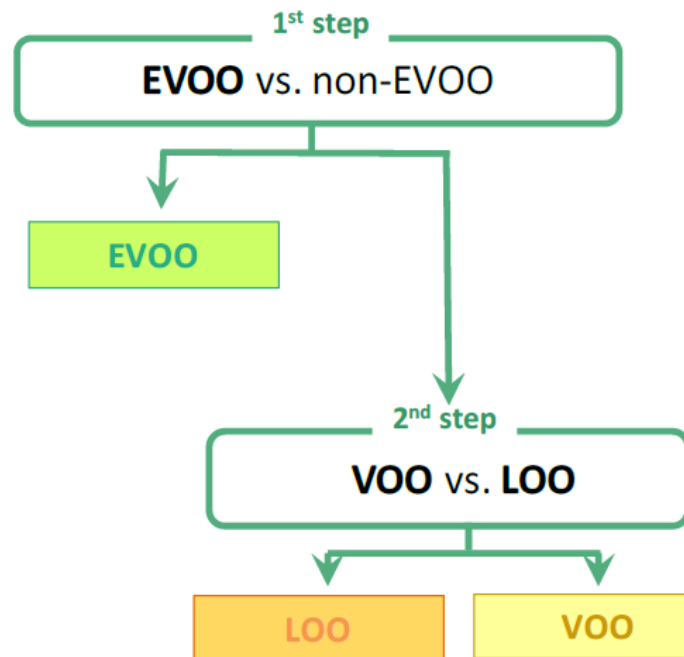
✓ Pre-classify samples.

✓ Support the work carried out by panel tests.

✓ Increasing the number of controls.

✓ Business-to-business.

HS-GC-IMS
semi-targeted approach

➢ Screening method to **pre-classify samples**, before the panel test, **into** different **clusters**:

   a) those with a probability of belonging to a commercial category greater than an established threshold;

   b) others (not reaching this threshold) that must be treated as insufficiently robustly classified.

➢ The developed models provided **percentages of correctly classified samples from 67% to 95%**, for the **quality grade prediction model**, and **from 48% to 80%,** for the **presence** of each **of the defects**. The reliabilit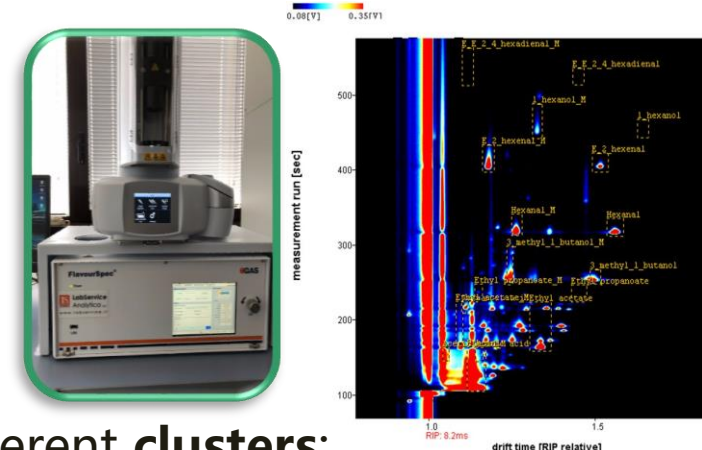y of the models can be improved upon by increasing the number of the samples to be included in the calibration as long as they are robustly classified sensorially.

➢ **Good results** in terms of **linearity** and **intra- and inter-day repeatability**; furthermore, to test the performance of this approach, inter-laboratory tests involving independent laboratories will be carried out in the future.

➢ **For routine quality control**, **firstly** clustering **LOO vs. no-LOO** to **identify non-edible samples** (LOO) before being assessed by panelists, **and then** classifying **EVOO vs. VOO**.

Sensory formulated reference materials (RMs)

➤ New **WINEY-VINEGARY** and **RANCID RMs** evaluated by 6 OLEUM panels.

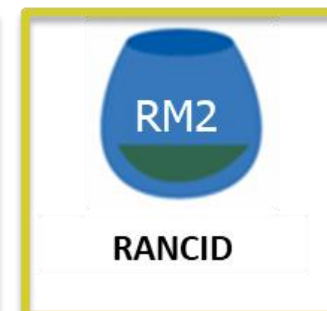➤ To be proposed for their adoption among tools for training tasters.

RMs evaluation sheet

RMs are only for smell, not for tasting

First part: **Evaluation of representativeness of RMs**

Second part: **determination of the detection threshold of the panels for RMs**

Third part: **shelf-life evaluation of RMs**

RM1

WINEY-VINEGARY

RM2

RANCID

OLEUM: Advanced solutions for assuring authenticity and quality of olive oil on a global scale

This project has received funding from the European Union's Horizon 2020 research and innovation programme under Grant Agreement No 635690. The views and opinions expressed in this document are the sole responsibility of the author and do not necessarily reflect the views of the European Commission.

DELIVERABLE D3.9

Title: Report on the suitability of Reference Materials in the training and monitoring of panellists according to the "IOC Guide for the selection, training and monitoring of skilled virgin olive oil tasters"

International Olive Council

http://www.internationaloliveoil.org/estaticos/view/224-testing-methods

ORGANOLEPTIC ASSESSMENT METHODS

COLLECTION OF METHODS AND STANDARDS ADOPTED BY THE INTERNATIONAL OLIVE COUNCIL FOR SENSORY ANALYSIS OF OLIVE OILS, OLIVE-POMACE OILS AND TABLE OLIVES

INTERNATIONAL OLIVE COUNCIL

COI/T.20/Doc. No 14/Rev. 5
June 2018

ENGLISH
Original: FRENCH

SENSORY ANALYSIS OF OLIVE OIL STANDARD

GUIDE FOR THE SELECTION, TRAINING AND QUALITY CONTROL OF VIRGIN OLIVE OIL TASTERS-QUALIFICATIONS OF TASTERS, PANEL LEADERS AND TRAINERS.

SPE/GC-FID to quantify ethyl esters of fatty acids

Purpose

The evaluation of is one of the (virgin, lampante or indirectly, some kind of sdOOs).
The official method requires high volume of solvents and a long and
complex preparative procedure therefore a **quicker
and more sustainable procedure** is proposed.

Principle

A 1 g in VOOs as rapid alternative to the preparative
phase defined by official method. The isolated fraction
is then analyzed by GC-FID using a low
polarity column and a cold on-column injector.

SOP changes after pre-trial

*Mandatory:

✓ To use silica cartridge with specific characteristics
✓ To include a blank extraction
✓ To use specific capillary low polarity GC column
✓ To adopt specific conditions for temperature program (oven, FID)

Conditioning: 10 mL hex:DEE, 99:1 (v/v) (*discard*)

6 mL hex (*discard*)

Loading: 100 mg sample + C17:0Me (+ Sudan I) (*discard*)

2 x 0.5 mL hex (use it to wash the sample vial) (*discard*)

Washing: 3 mL (*discard*)

Elution: 10 mL hex:DEE, 99:1 (v/v) (*collect!*)

Very good performance of the method
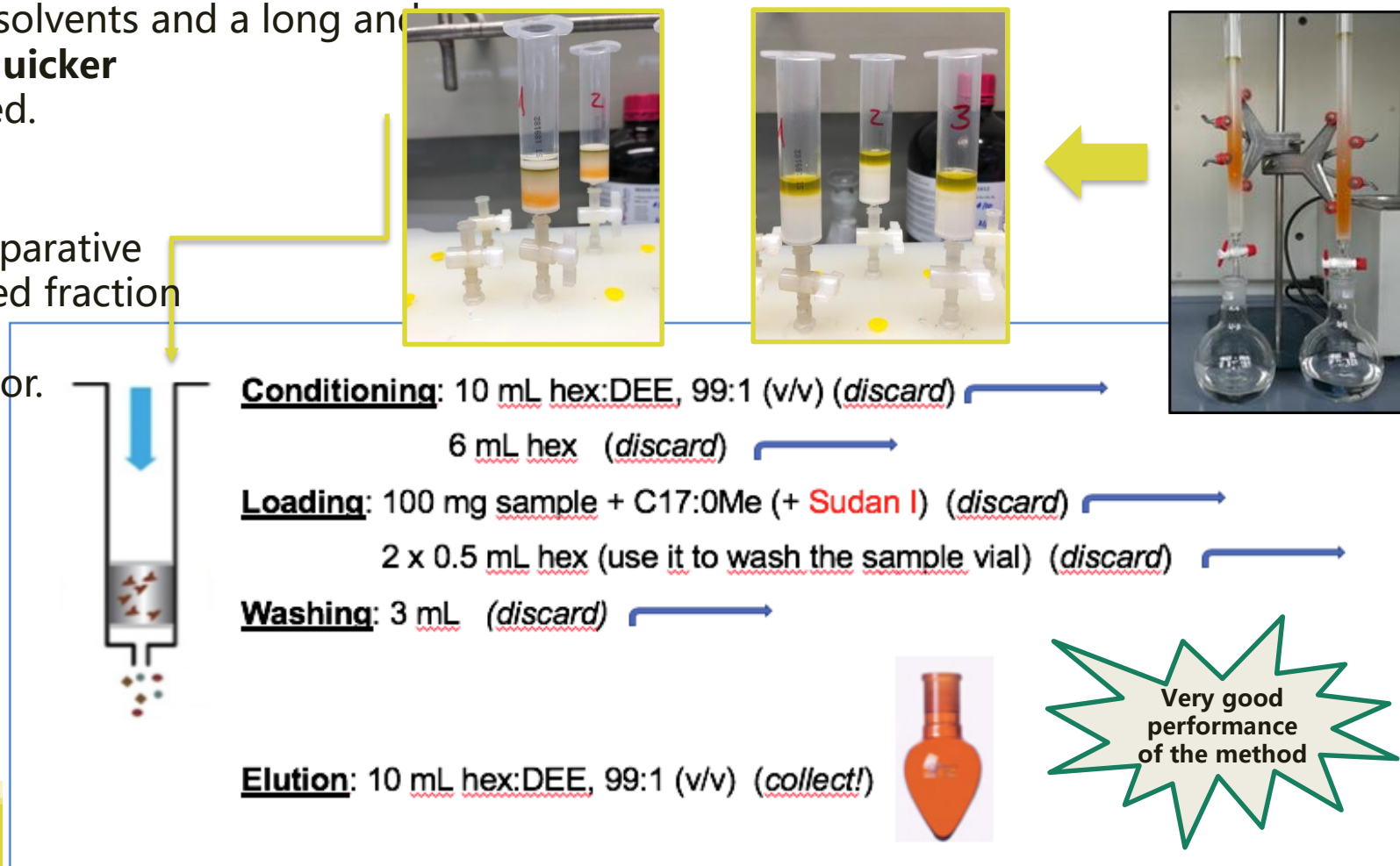
SPE/GC-FID method to detect both free and esterified hydroxylated minor compounds

Enrico Valli [1], Andrea Milani [2], Ana Srbinovska [2], Erica Moret [2], Sabrina Moret [2], Alessandra Bendini [1], Wenceslao Moreda [3], Tullia Gallina Toschi [1] and Paolo Lucci [2,*]

Purpose

The analytical evaluation of the composition of sterols is a well-established tool for assessing the purity of olive oils, as it depends on the botanical origin of oils. The methods that are available (ISO 12228, COI/T.20/Doc. No. 10, Reg. (CEE) 2568/1991, All. V) are suitable to determine the total composition of sterols after saponification, so not discriminating between the free and the esterified form. In different vegetable oils, sterols can be differently distributed between these two forms.

Principle

Free and esterified hydroxylated minor compounds (i.e. free and esterified sterols and triterpenic alcohols) **are converted into silyl derivatives**, in such a way, their polarity became the same of the corresponding esterified ones. **Oil is then fractioned by** solid phase extraction (**SPE**) **and** the fraction containing free and esterified hydroxylated minor compounds is **analysed by capillary GC with on column injection**.

Scope

This method determines the free and esterified hydroxylated minor compounds (free and esterified sterols, free and esterified triterpenic alcohols) of olive oils and seed oils and can be utilized as screening tool to detect adulteration by seed oils.

The proposed method **is not intended to replace the official method for total sterol**, but the two methods could be complementary and used together to obtain a higher degree of information regarding the nature of the oil sample and therefore to reinforce analytical methods available for the prevention of fraud.

Free Sterol

Esterified sterol
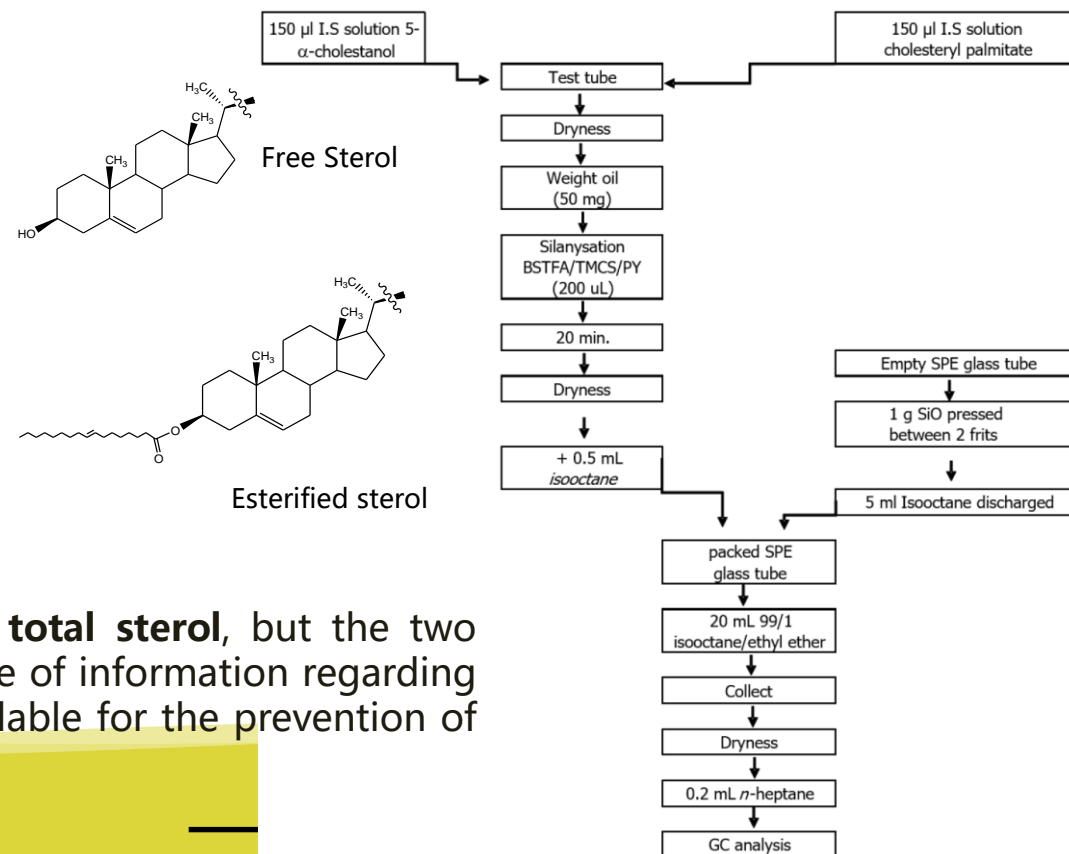
Detection of illegal processing (deodorization)

Olive oil mixtures. Part two: Detection of soft deodorized oil in extra virgin olive oil through diacylglycerol determination. Relationship with free acidity

Raquel B. Gómez-Coca[a,*], María de Carmen Pérez-Camino[a], Alessandra Bendini[b], Tullia Gallina Toschi[b], Wenceslao Moreda[a]

This method may be considered to assess product compliance.

○ Focus on the utility of two new indexes (R1 and R2) obtained combining the DAG concentration and the FA value (markers of lipid hydrolysis) of the samples under suspicious:

in genuine EVOO, **R1** > 0.23 and **R2** < 0

$$DAG_{theor} = 17.6 \times (\text{free acidity} - 0.10) + 10. \quad ^{e}R1 = 10 \times (\text{free acidity}/DAG_{exp}). \quad ^{f}R2 = DAG_{exp} - DAG_{theor}.$$

○ Useful approach to detect the presence of sdOOs when this is at least at 30% in the illegal mixture

		R1[e]	R2[f]
	EVOO_H	0.29	−3.52
	EVOO_H-2	0.25	−0.85
	EVOO_L	0.23	−2.19
Rancid	ROO_SD	0.15	7.77
Fusty	FOO_SD	0.16	14.02
Frostbitten	FBOO_SD	0.15	10.71
Brine	BOO_SD	0.15	5.25
Musty	MOO_SD	0.19	3.58
Winey	WOO_SD	0.19	2.60

Defective oil	% EVOO_H	% Soft deodorized oil [a]	R1[e]	R2[f]
ROO_SD	70	30	0.22	−0.09
	60	40	0.21	0.93
	50	50	0.19	2.13
	40	60	0.18	3.32
FOO_SD	70	30	0.21	1.80
	60	40	0.20	3.56
	50	50	0.19	5.17
	40	60	0.18	6.93
BOO_SD	70	30	0.22	−0.89
	60	40	0.21	−0.01
	50	50	0.20	0.87
	40	60	0.19	1.74

Proposal for a Protocol Fit for the Purpose of the EFSA Health Claim for Olive Oil 'Polyphenols'

Principle:

The UHPLC profile of the **extracted polar fraction** (PF) of the oil **before and after acid hydrolysis** is recorded by means of **diode array detection (280 nm)**. Acid hydrolysis of bound forms of **Hydroxytyrosol (Htyr)** and **Tyrosol (Tyr)** gives rise to free **Htyr** and **Tyr**, the content of which can then be accurately quantified using commercially available standards.
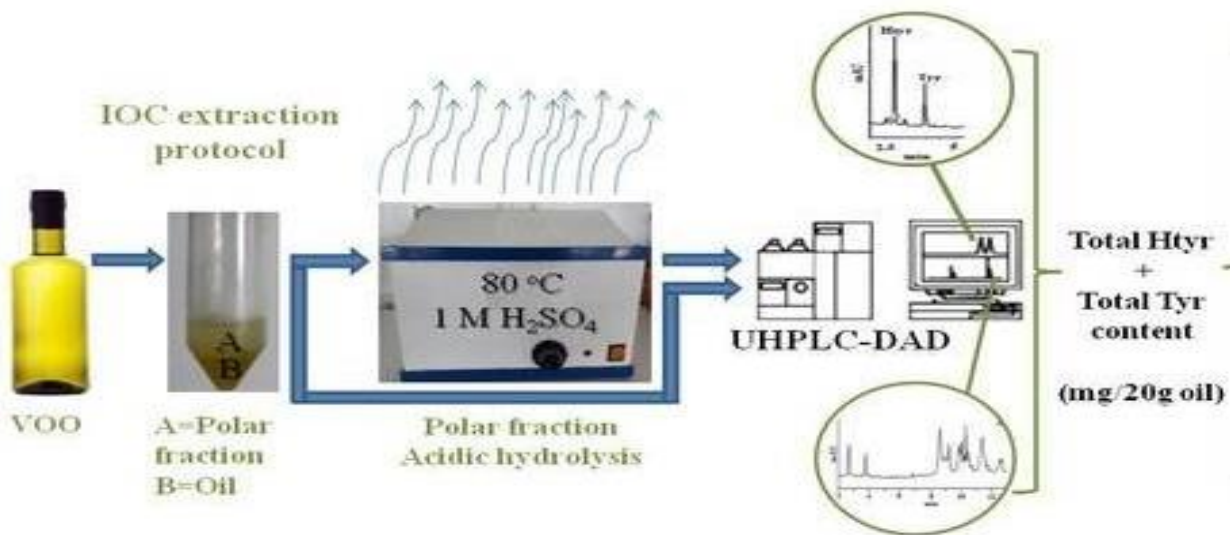
This content is expressed as total **Htyr** and **Tyr** (mg/20 g of oil) after correction for molecular weight differences between free and bound forms as follows:

$$\text{total Htyr and Tyr (mg/20g oil)} = [\text{Htyr}_{free}] + [\text{Tyr}_{free}] + \mathbf{2.2^*} \times [\text{Htyr}_{hydrolysate} - \text{Htyr}_{free}] + \mathbf{2.5^*} \times [\text{Tyr}_{hydrolysate} - \text{Tyr}_{free}]$$

*See Tsimidou et al., 2019, Molecules, 24(6), 1044 for explanation

Comparison of method performance

30 extra virgin olive oils
(**55**-**412** mg caffeic acid/kg oil, TPP, Folin Ciocalteu assay)

COI/T.20/Doc No 29
(ZRS Copper, Slovenia)*

UHPLC-DAD method
(AUTH)

Integrating only Htyr, Tyr free and bound forms

IOC accredited Laboratory on phenol analysis

Proposal for a Protocol Fit for the Purpose of the EFSA Health Claim for Olive Oil 'Polyphenols'

The data produced by both procedures **were statistically examined with various approaches** (Pearson correlation, Passing Bablok Bland Altman Analyses)...

molecules

MDPI

Article

Toward a Harmonized and Standardized Protocol for the Determination of Total Hydroxytyrosol and Tyrosol Content in Virgin Olive Oil (VOO). The Pros of a Fit for the Purpose Ultra High Performance Liquid Chromatography (UHPLC) Procedure

Maria Z. Tsimidou [1,*], Nikolaos Nenadis [1], Aspasia Mastralexi [1], Maurizio Servili [2], Bojan Butinar [3], Stefania Vichi [4], Ole Winkelmann [5], Diego Luis García-González [6] and Tullia Gallina Toschi [7]

SCAN ME

Only **8 (out of 30) samples** were **found with** a total Htyr Tyr content 5 mg/ 20 g oil by the **COI procedure** whereas **16 (out of 30) samples were compliant using the UHPLC DAD method**.

✓ **Software to estimate the EVOOs "best before date"**

✓ The availability of an instrument for the shelf-life prediction of VOO is worth of value since at the moment the sector operators haven't any method for defining the reliable "best before date" for their VOOs.

✓ The software developed has a **strong untapped potential on different market**. Two different exploitation opportunities (that are not discordant but complementary) are considered for the future of the project:

1) License the software to operators in the virgin olive oil market.

2) Selling the lab service for single sample analyses and prediction of its "best before date".

VITA OLEI

SOFTWARE «*USER FRIENDLY*»

Shelf-life prediction software prototype
(version 1.0)

OLEUM achievements and future impact

Oleum

✓ To **develop the OLEUM Databank**

✓ A **new and improved analytical tool**, which **facilitates the storage and retrieving of data** of heterogeneous nature.

✓ Data can be uploaded **with full descriptions of samples and analytical methods** used.

✓ If the Databank will be adopted by the National Control Bodies, **it will contribute to a reduction in litigations** (in case of doubt on the authenticity of an OO or atypical OOs in terms of limits/ranges/values of specific parameters).

✓ Will **permit a more effective collaboration of quality control laboratories** in Europe **and** it will foster the **use of harmonized methods** of olive oil analysis → **Reproducibility**.

✓ Having **a database with reference values**, which cover the existing variability including atypical olive oils**, is essential to confirm a fraud suspicion** in terms of limits / ranges / values of specific parameters.

OLEUM achievements and future impact
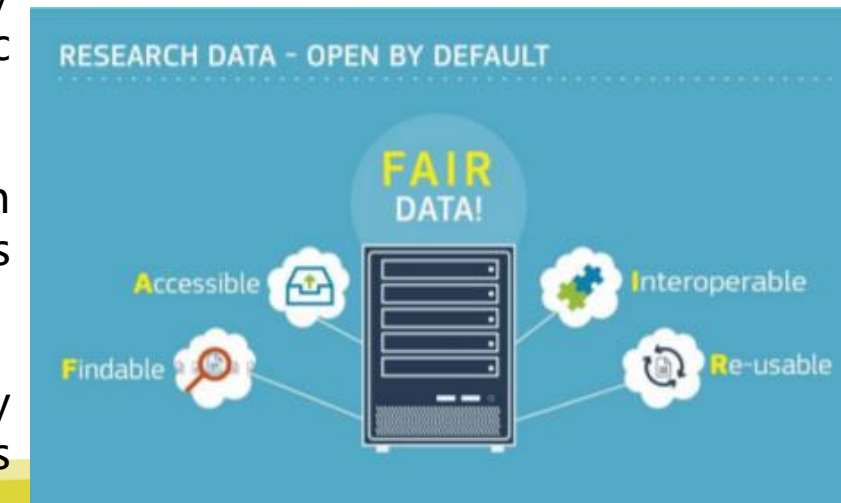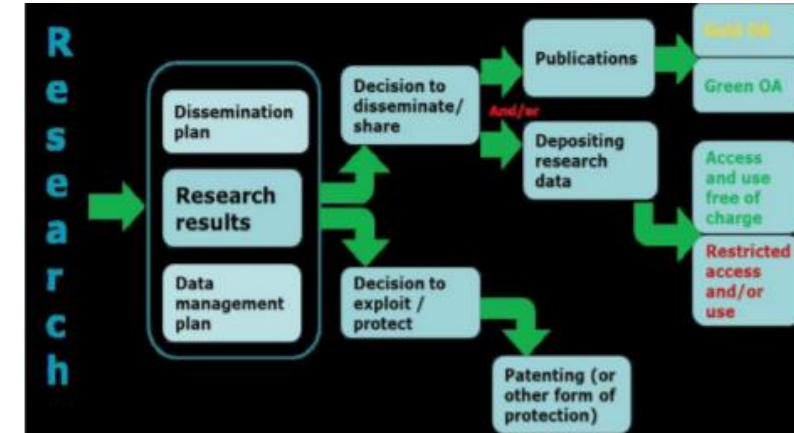
✓ Prosecute the experience of a wide operative **OLEUM Network**

✓ Getting to **know other stakeholders** in the olive oil sector
→ Technical and **technological transfer**.

✓ Yearly OLEUM Network **meetings → Collaborations** leading **to new method** and **standard operating procedure development**.

✓ **Exchange of knowledge** and news → **Contact with labs** and with **Universities**.

✓ **Participation in validation of new methods** – ring tests, including sharing of samples.

✓ **Exchange information** on analytical methods to learn from each other.

✓ **Solve problems** together arising in the olive oil field.

✓ **Communication to the outside world**: promotion of high-quality olive oil and its health benefits.

Who is the OLEUM Network?

OLEUM Network
- 90 members from 20 different countries.
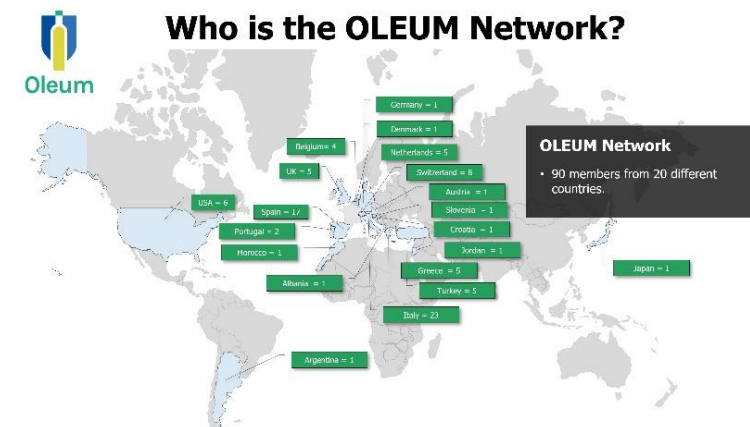
Future countermeasures

EU regulation dealing with OO, can be considered as **the most extensive and concrete.** The **analytical methodologies** to ensure OO quality and authenticity **are appropriate**, despite **some deficiencies**.

An information that is important to pass to the consumer is that **the level of attention and the high request in terms of conformity checks have currently improved the quality of the OO on the market in the last thirty years**.

To better guarantee OO quality and authenticity, **there is still the need to ameliorate conformity checks**, **reduce** the **cases of disagreement in the classifications**, **develop improved robust methods** and **supportive screening tools**, in an attempt to try to be one-step ahead of fraudsters.

Some strategies to beat fraud at a global level

A promising way that EU could take includes:

i)   a **joint strategy** able **to combine sensory and instrumental data** useful **in cases of disagreement between** two **panels**;

ii) an **improvement of the proficiency and alignment of the panels** by a mutual calibration achievable (e.g. by finding the same sensory **reproducible reference materials on the market**);

iii) **real and virtual compliant compositions** can be stored in **a repository of validated data** (e.g., **OLEUM Databank** under development within the OLEUM project) **and used as quality and authenticity references**;

iv) quality and authenticity information of a certain OOs could be put in relation with volumes produced and their geolocation; thus, the **intersection between official quality controls and traceability**, typical of a **blockchain scenario**, **could be the next fraud countermeasure**.

We should not forget the relevance of a formative role for the general public (seed of an effective "participative quality").
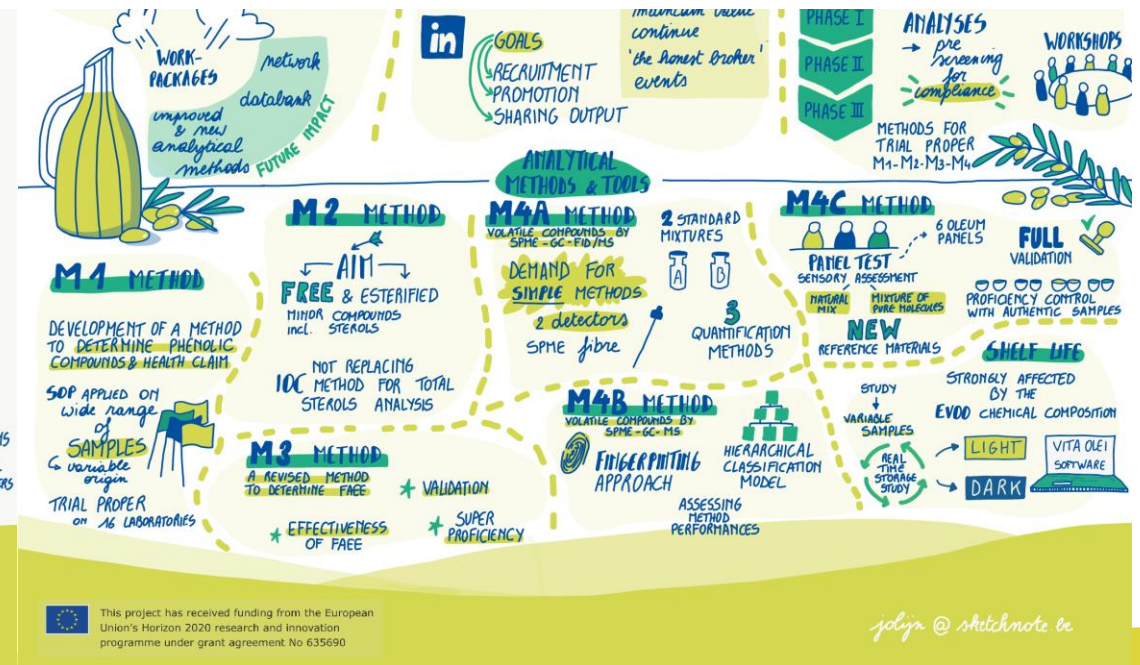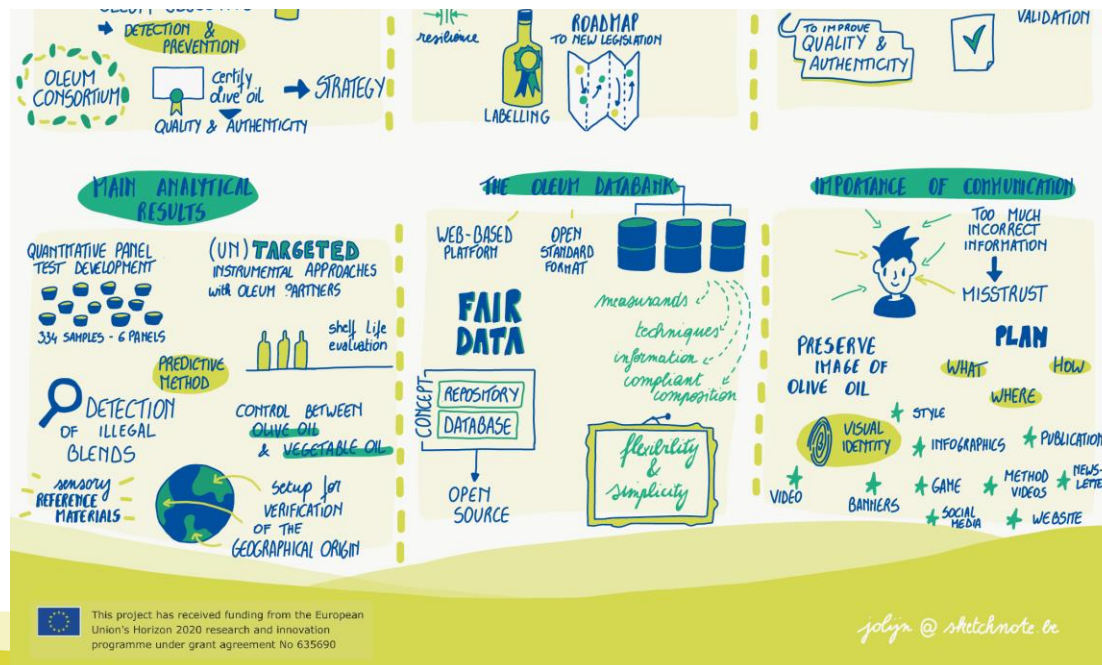

OLIVE OIL CATEGORIES
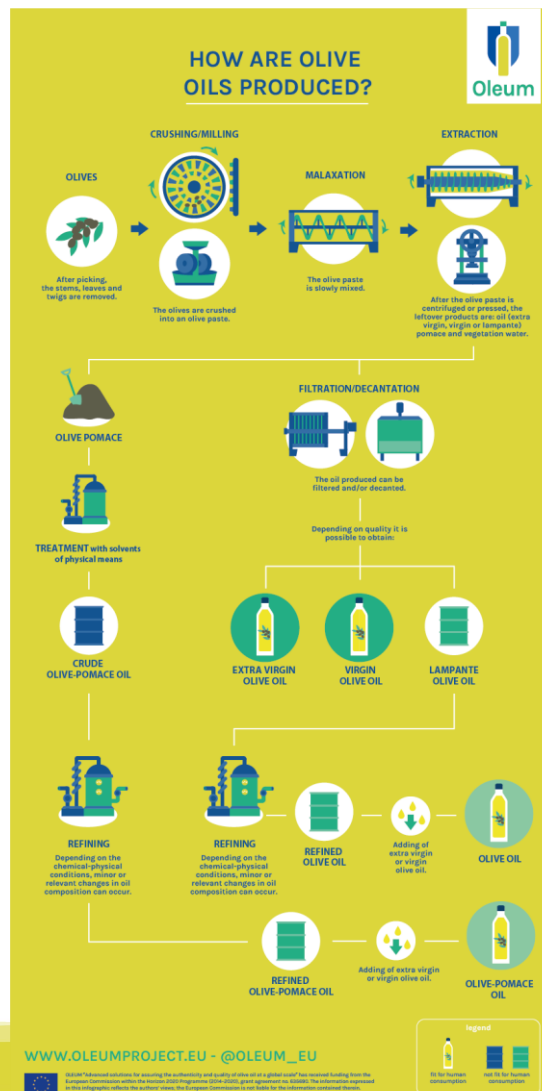
HOW ARE OLIVE OIL PRODUCED?

http://www.oleumproject.eu/publications

We need the courage to:

- Improve the knowledge of the top quality EVOO and its added value.

- Find a new space, visibility and right value for the VOO (sustainability? organic?).

- Adopt a new, effective, correct and disruptive communication.

Next calls to continue the work on olive oil and other food products with a similar strategy

HORIZON-CL6-2022-FARM2FORK-01-04: Innovative solutions to prevent adulteration of food bearing quality labels: focus on organic food and geographical indications (IA)
https://ec.europa.eu/info/funding-tenders/opportunities/portal/screen/opportunities/topic-details/horizon-cl6-2022-farm2fork-01-04
Deadline date: 15 February 2022 17:00:00 Brussels time

HORIZON-CL6-2022-FARM2FORK-01-11: Effective systems for authenticity and traceability in the food system (RIA)
https://ec.europa.eu/info/funding-tenders/opportunities/portal/screen/opportunities/topic-details/horizon-cl6-2022-farm2fork-01-11
Deadline date: 15 February 2022 17:00:00 Brussels time

http://www.oleumproject.eu

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