



copa***cogeca**
european farmers european agri-cooperatives



Oleum

Better solutions to
protect olive oil quality
and authenticity

«CDG HORTICULTURE, OLIVES AND SPIRITS – SUBGROUP
OLIVES»

Meeting via videoconference (Interactio)
on Friday 19 November 2021 from 14:00 to 17:30

The OLEUM project results

Department of Agricultural and Food Sciences,
Alma Mater Studiorum – Università di Bologna
EU H2020 OLEUM project Coordinator

19 November - 2021

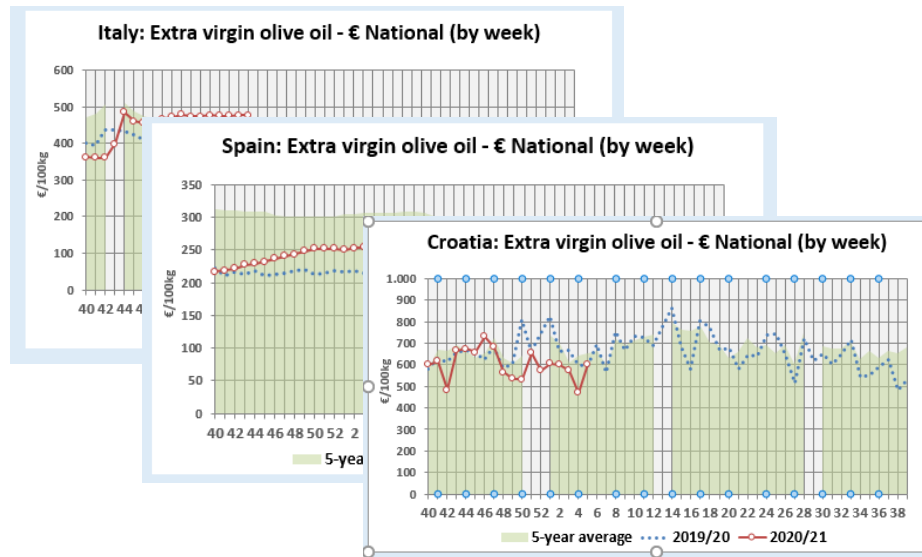


This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 635690.

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**.



OLIVE OIL CHARACTERISTICS

Extra virgin olive oils and virgin olive oils are obtained directly from olives and solely by mechanical means. The oils have a wide range of characteristics such as organoleptic properties (taste, colour, smell, feel) which depend on various factors such as the type of olive, soil, climatic conditions, harvest date etc.



Extra virgin olive oil is the category with the highest quality from an organoleptic point of view, it has no defects and is **fruity**. Its acidity level shall not exceed 0.8%.



Virgin olive oil is also fruity but has **some slight sensory defects**. Its acidity shall not exceed 2%.

Other categories of olive oils can also be sold directly to consumers



Olive oil composed of refined olive oil and virgin olive oils results of a mixture of refined olive oil with extra virgin and/or virgin olive oils. Its acidity degree shall not exceed 1%.



Olive-pomace oil results of the mixture of refined olive-pomace oil with extra virgin and/or virgin olive oils. It must have a degree of acidity not exceeding 1%.

Agriculture
and rural
development

OTHER CATEGORIES OF OLIVE OILS FOR REFINING OR INDUSTRIAL PURPOSES

Lampante olive oil

is the lowest quality virgin olive oil. It has substantial sensory defects (taste and/or smell) which can be the result of bad processing of the olives, or of weather incidents altering the olive fruits while on the trees. Lampante olive oil must be refined in order to remove its defects. As such, it cannot be sold to consumers. After refining, the oil obtained is known as 'refined olive oil'.

Refined olive oil

has little or no olive aroma, flavour, or colour. Refined olive oil cannot be sold to consumers. It is blended with extra virgin olive oil and/or virgin olive oil to make the "olive oil composed of..." category.

Crude olive-pomace oil

Olive-pomace is the residual paste obtained after the oil is extracted from the olives. The oil obtained out of this paste is the crude olive-pomace oil.

Refined olive-pomace oil

is obtained after the refining of crude olive-pomace oil. Blended with extra virgin and/or virgin olive oils, it can be marketed to consumers as olive-pomace oil.

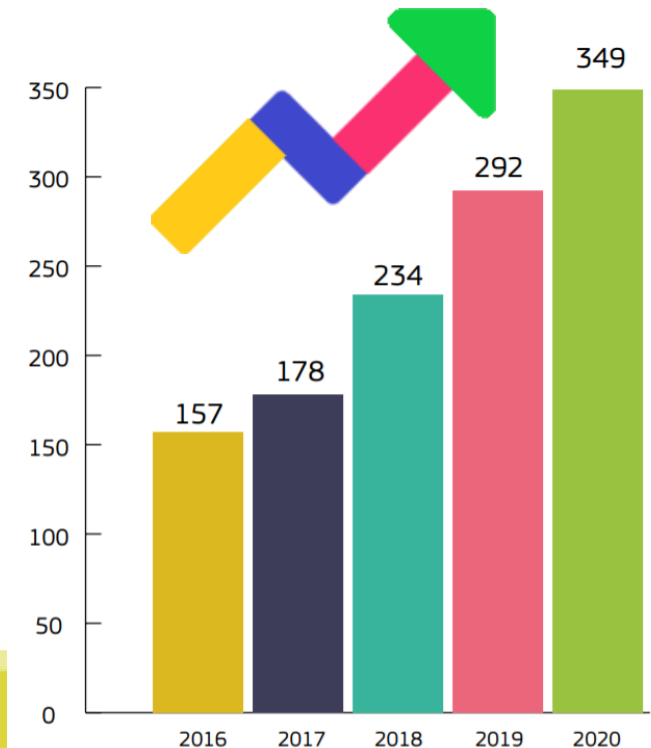
The European Commission's annual report on food fraud

- ✓ 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

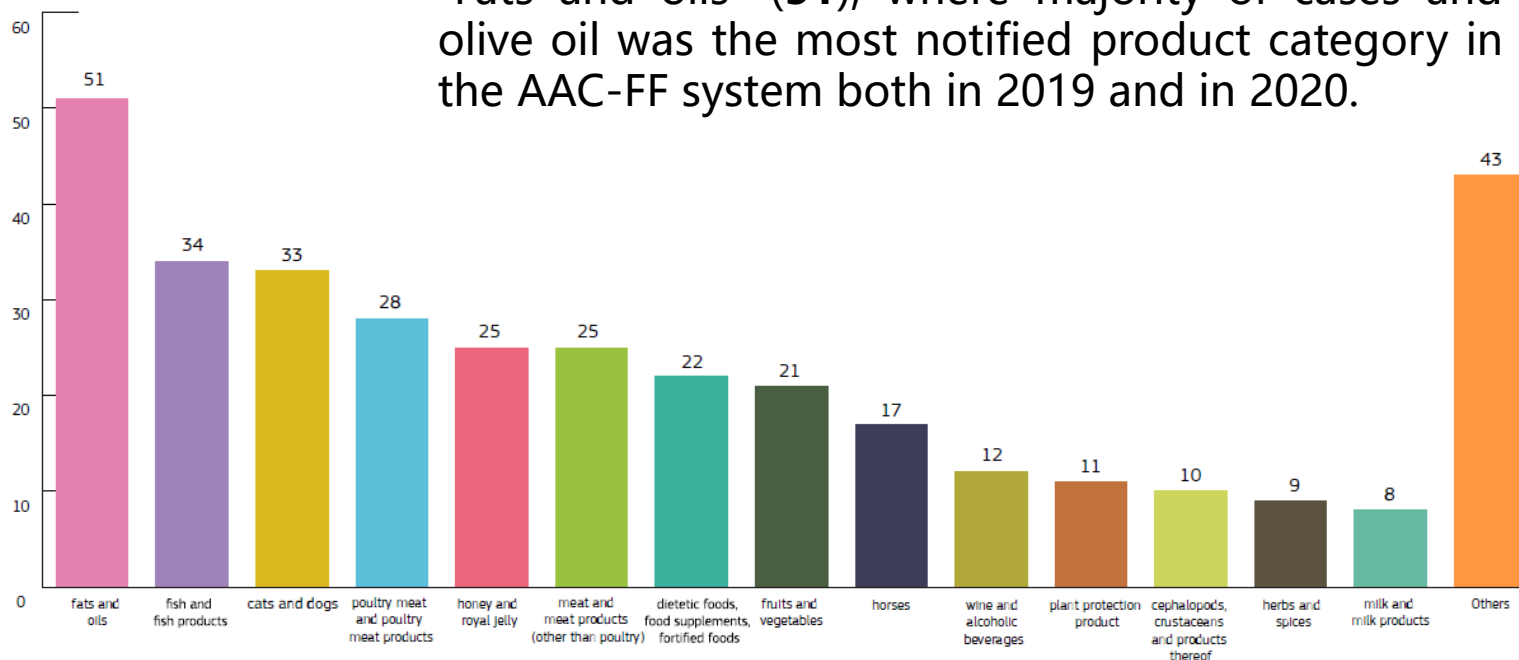
The number of requests for assistance and cooperation shared between Member States tends to increase over the years and this is a positive signal in terms of transparency and sharing of information.

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



Olive oil always in the spotlight. Why?

"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.



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.

Monthly prices of extra virgin, virgin and lampante olive oil (national average) € /100 kg.

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%

Most common infringements in the olive oil sector

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

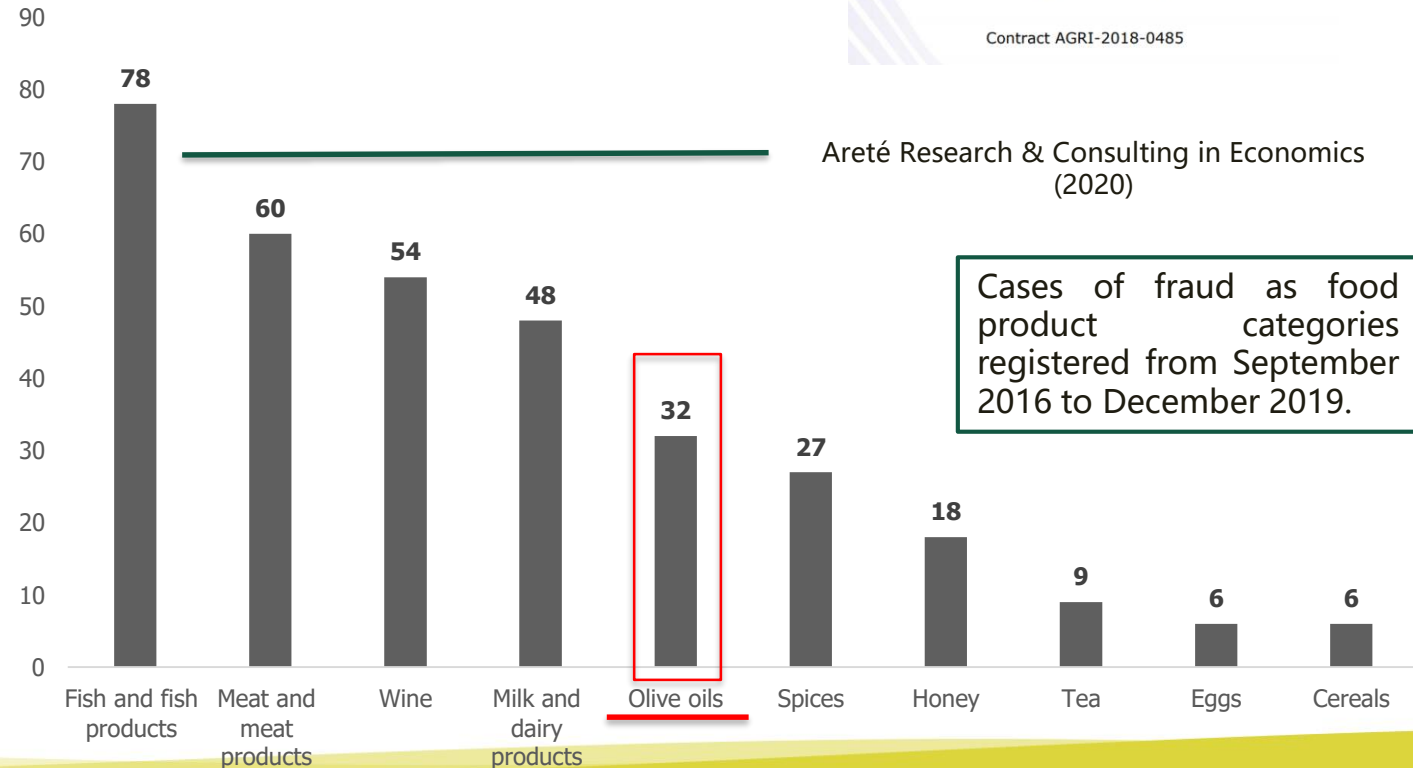
To ensure the health and protection of consumers, JRC publishes a **monthly summary** with **newspaper articles on food fraud**, with the aim of informing all the stakeholders and giving them the opportunity to act on these irregularities.



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

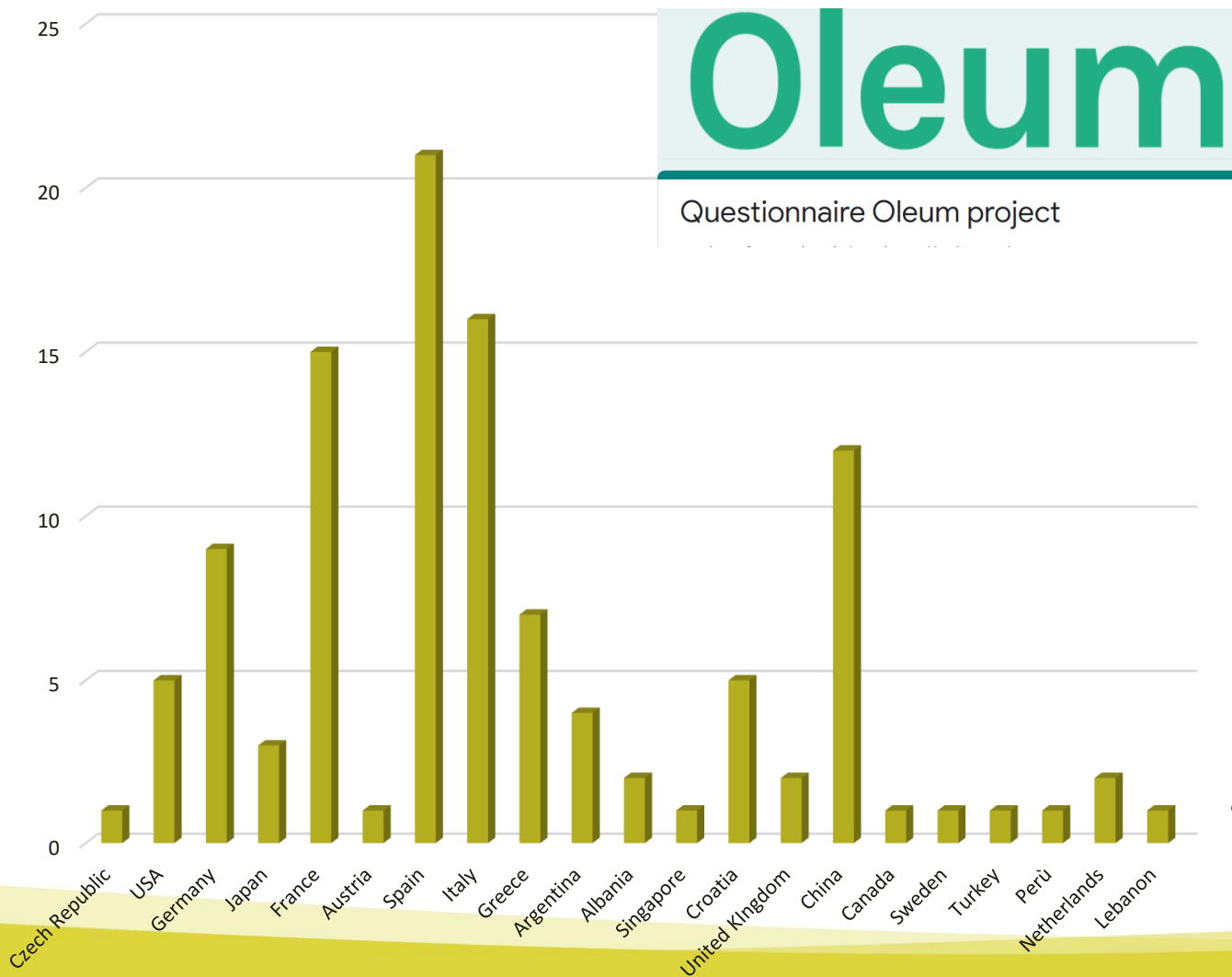
Contract AGRI-2018-0485

Areté Research & Consulting in Economics (2020)

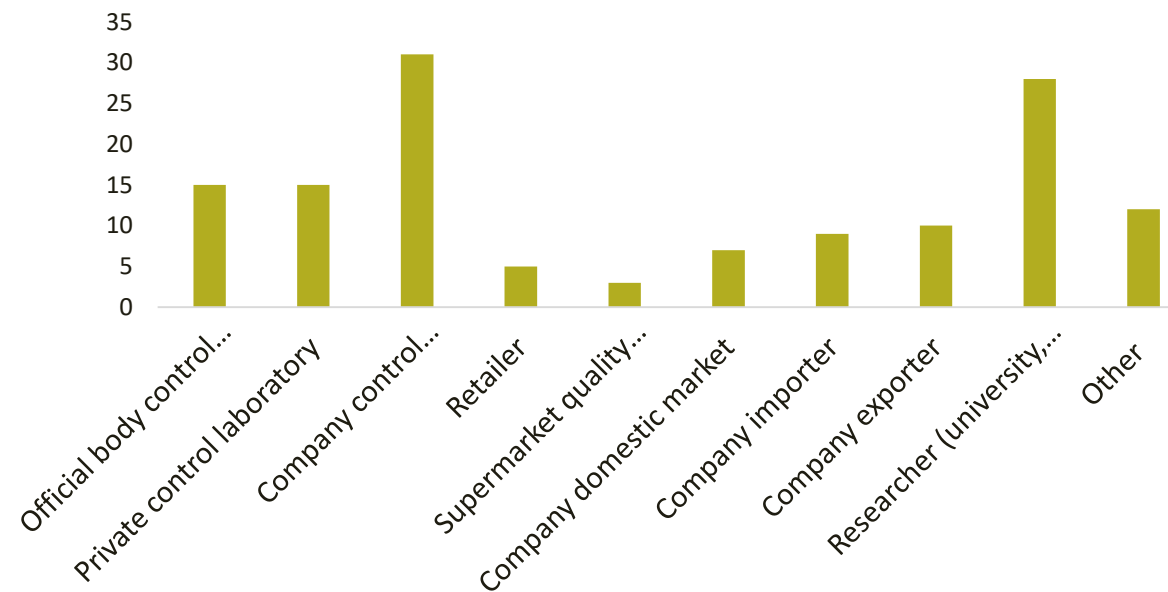


The OLEUM on-line survey

<https://docs.google.com/forms/d/e/1FAIpQLSdupgODpha7GqeKSR5rzLNdGWRQIV7q2r2XMcGQxVnPTvkAow/viewform?vc=0&c=0&w=1>



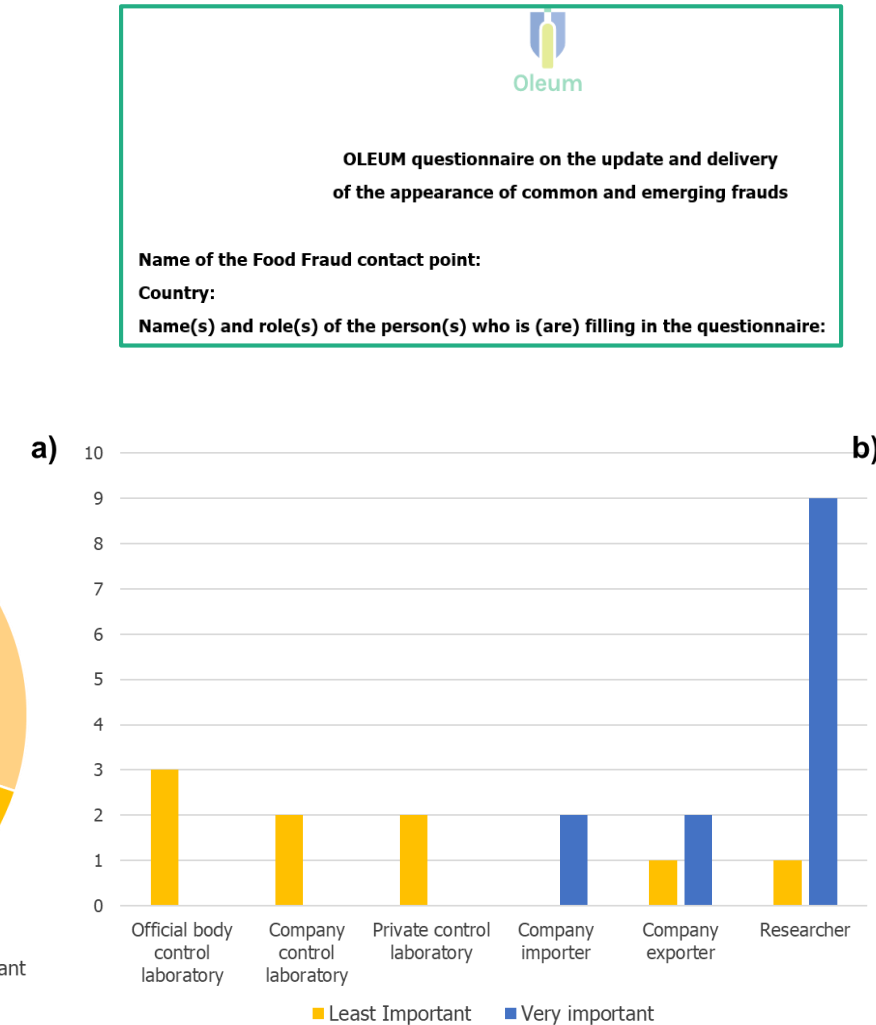
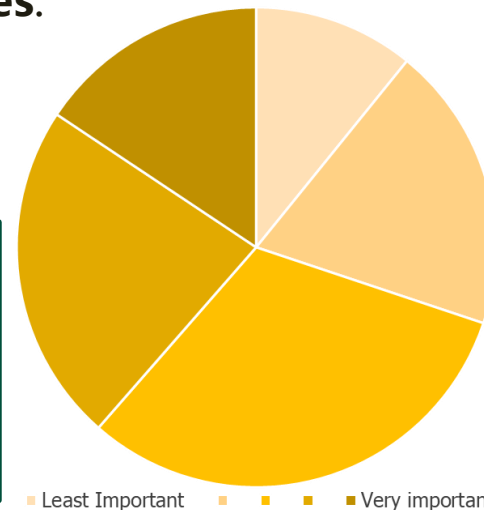
Sent by e-mail during 2018 to over 200 stakeholders. A total of 111 completed questionnaires were returned from both European (87 questionnaires) and non-European (24 questionnaires) countries.



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**.





SCAN ME



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

OLEUM related publications

SCAN ME



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

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.

Programme

Horizon 2020 Framework Programme

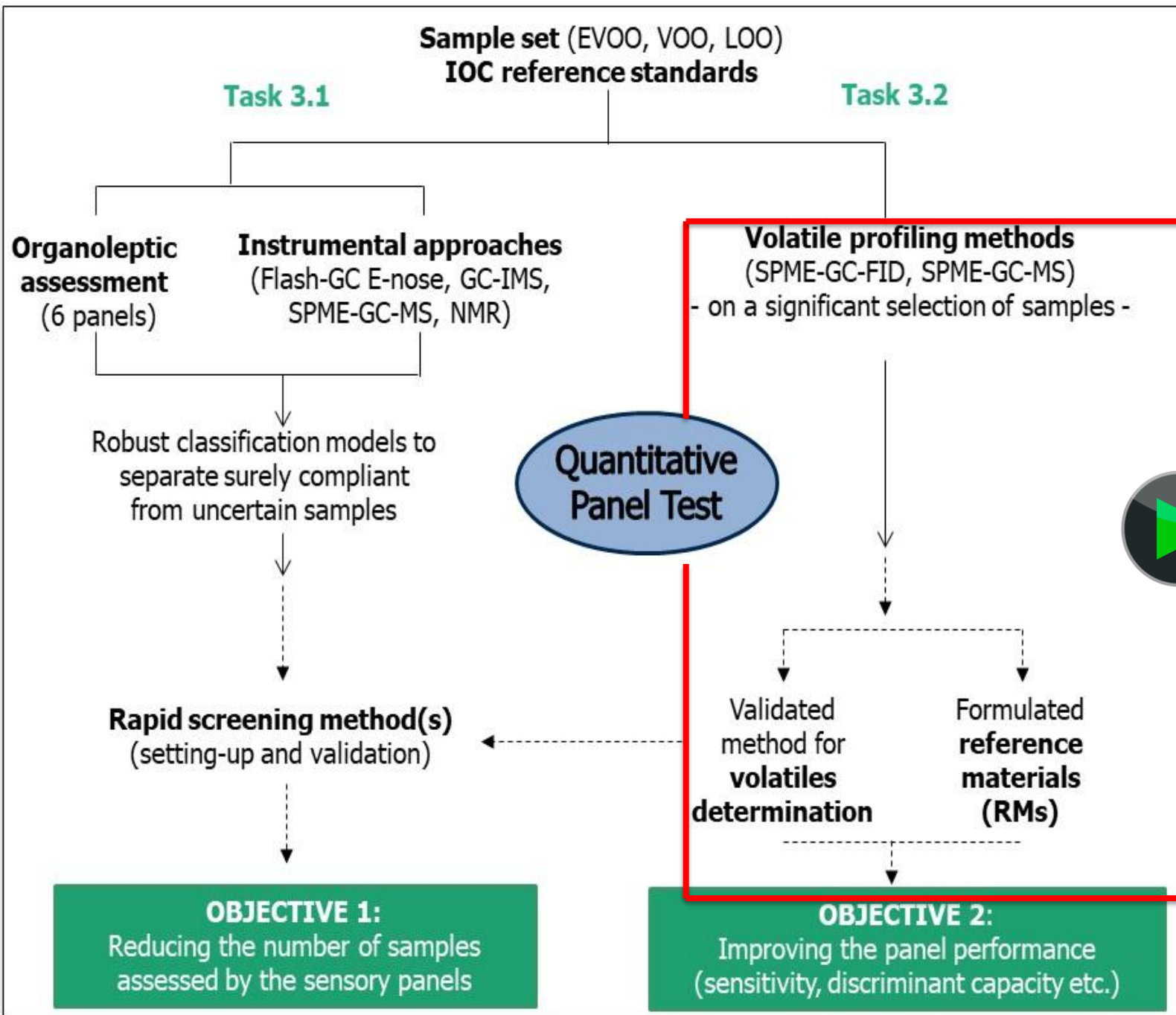
Call

[Sustainable Food Security \(H2020-SFS-2014-2015\)](#)

Type of action

RIA Research and Innovation action





Starting point



SPME-GC-FID/MS targeted methods for the analysis of selected volatile compounds in virgin olive oils



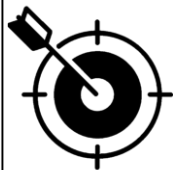
Standard Operating Procedure

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



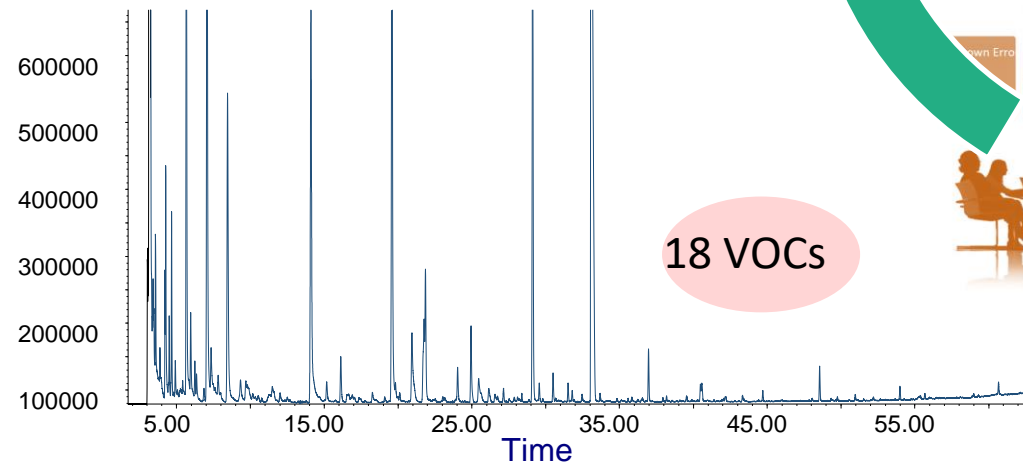
Targeted approach

1

a. SOP for the SPME-GC-FID version

b. SOP for the SPME-GC-MS version

c. Guide document on calibration curves



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)

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-earthly (Total: 3)
(<i>E</i>)-2-heptenal
1-octen-3-ol
Propanoic acid

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

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

Positive attribute (fruity)



Fruity (green notes) (Total: 3)
(<i>E</i>)-2-hexenal
(<i>Z</i>)-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

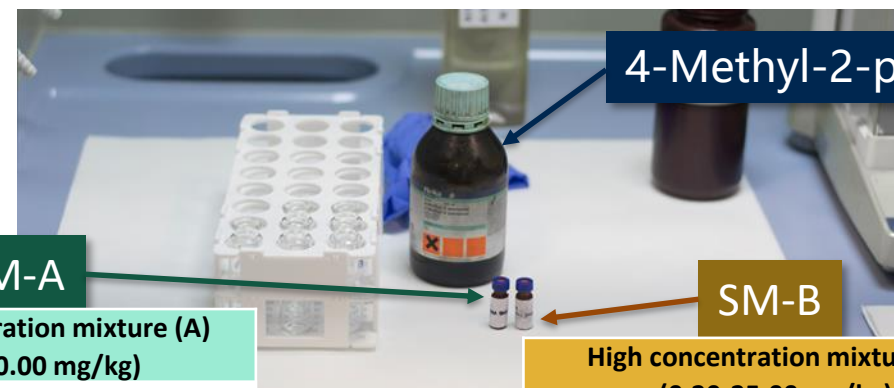
Fermentative defects (*fusty/muddy, winey vinegary, musty*)
+ **Damaged olives** + **Oxidation** (*rancid*) + **Positive attributes** (*fruity*)

- | | |
|---------------------------------|------------------------------------|
| 1. Octane | 10. 6-Methyl-5-hepten-2-one |
| 2. Ethyl acetate | 11. 1-Hexanol |
| 3. Ethanol | 12. Nonanal |
| 4. Ethyl propanoate | 13. 1-Octen-3-ol |
| 5. Hexanal | 14. (E,E)-2,4-Hexadienal |
| 6. 3-Methyl-1-butanol | 15. Acetic acid |
| 7. (E)-2-Hexenal | 16. Propanoic acid |
| 8. (Z)-3-Hexenyl acetate | 17. (E)-2-Decenal |
| 9. (E)-2-Heptenal | 18. Pentanoic acid |

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



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



SM-A

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

SM-B

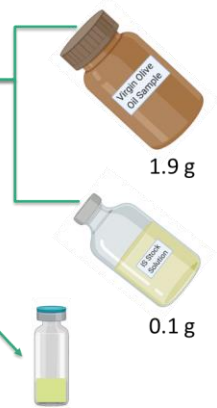
High concentration mixture (B)
(0.20-25.00 mg/kg)

Ethanol
Hexanal
(E)-2-Hexenal
(Z)-3-Hexenyl acetate
1-Hexanol
Nonanal
1-Octen-3-ol
Acetic acid

OLEUMPROJE

*Internal standard: 4-methyl-2-pentanol

SAMPLE PREPARATION



Virgin olive oil sample

I.S. 4-methyl-2-pentanol



Calibration solutions
(18 VOCs)

Chromatographic
areas

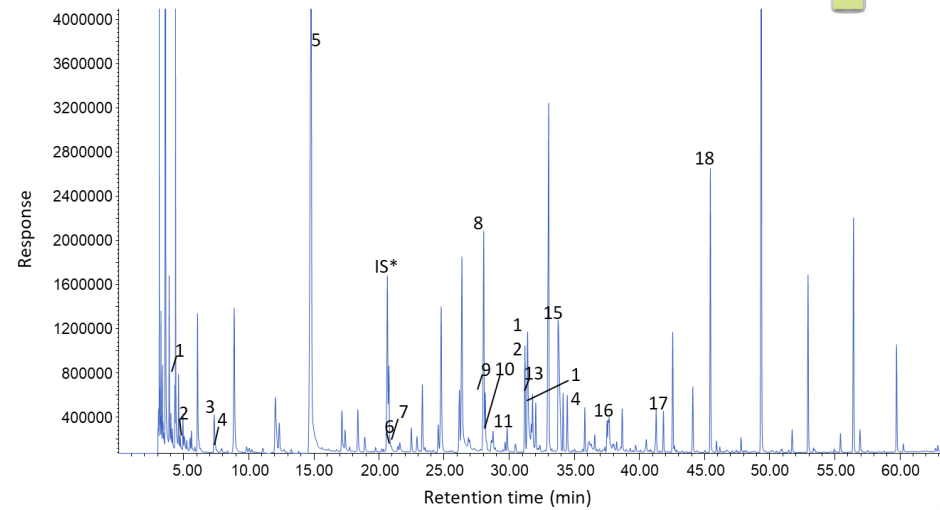
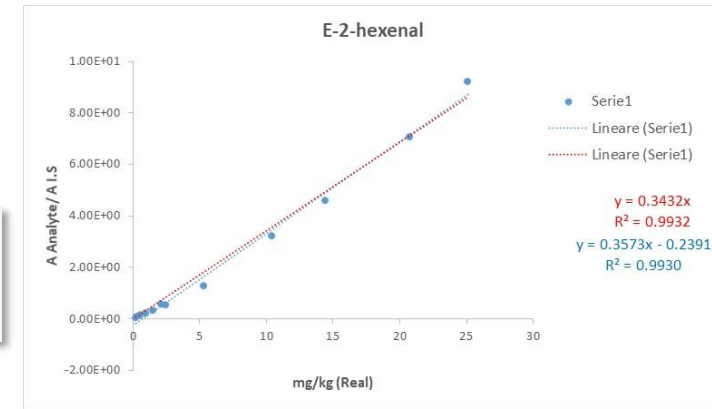
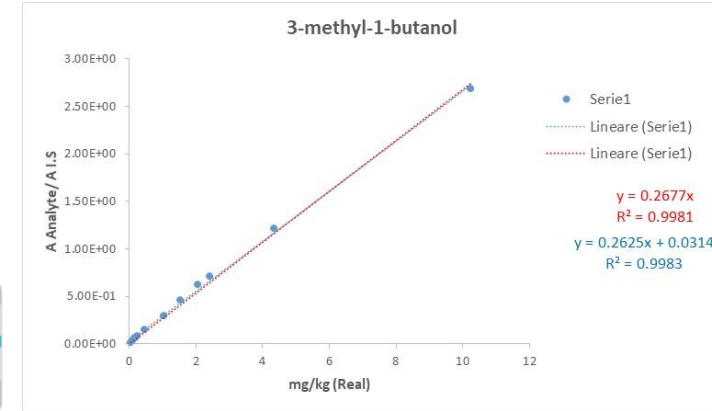
Quantification
(Calibration)

Concentratio
n (mg/kg)

Sample

Chromatographic
areas

I.S.



RESULTS

QM1

Linearity (R ²)	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.

SCAN ME



Food Control 123 (2021) 107823

Contents lists available at [ScienceDirect](https://www.sciencedirect.com)

Food Control

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

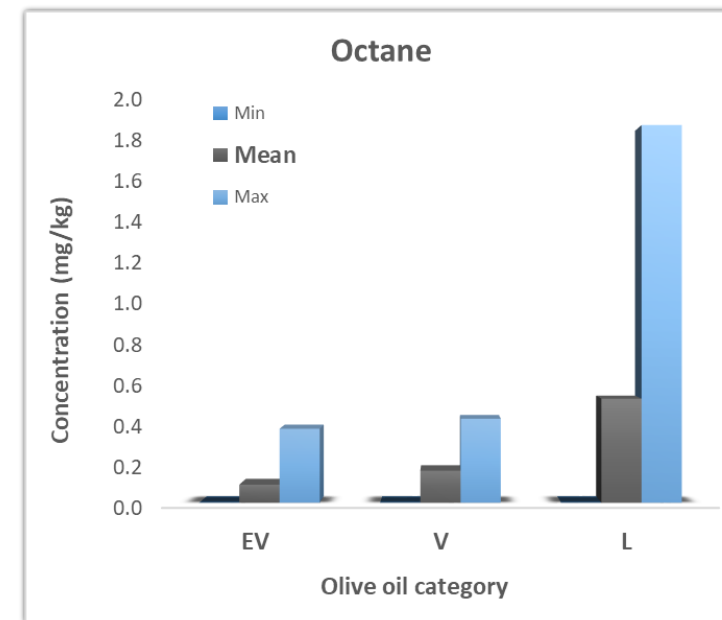
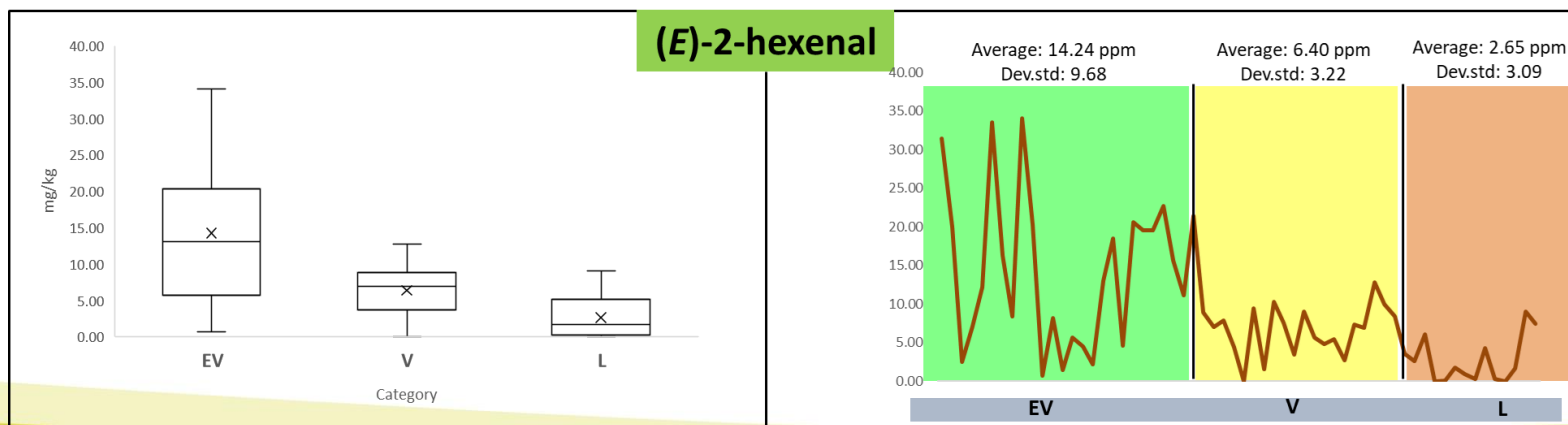
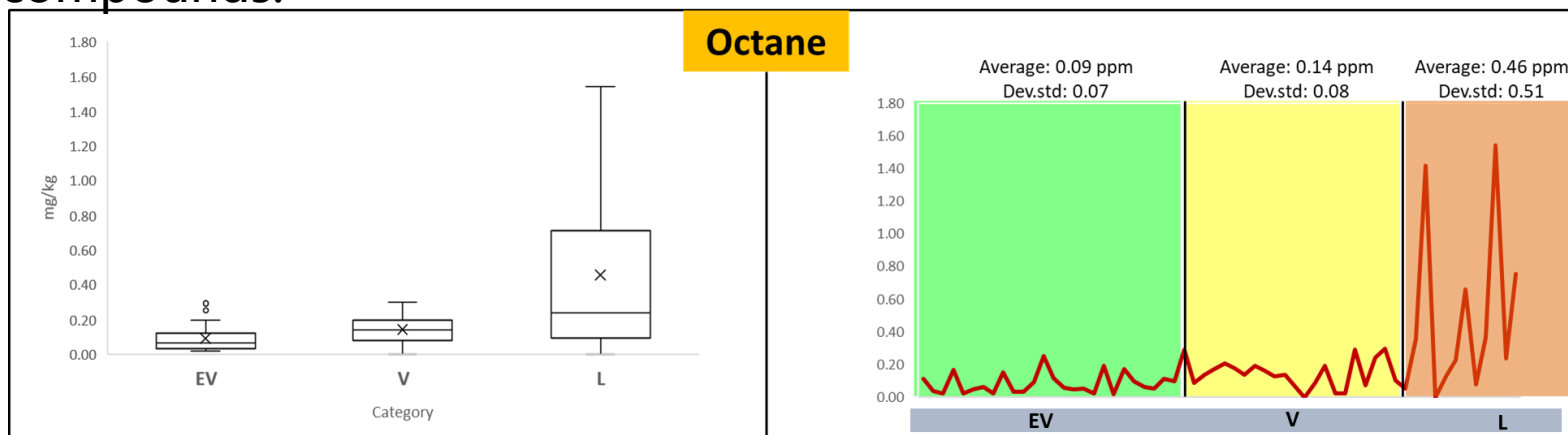
Peer inter-laboratory validation study of a harmonized SPME-GC-FID method for the analysis of selected volatile compounds in virgin olive oils

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

SPME-GC-MS method in course of publication

Next phase: agreement on the limits and ranges

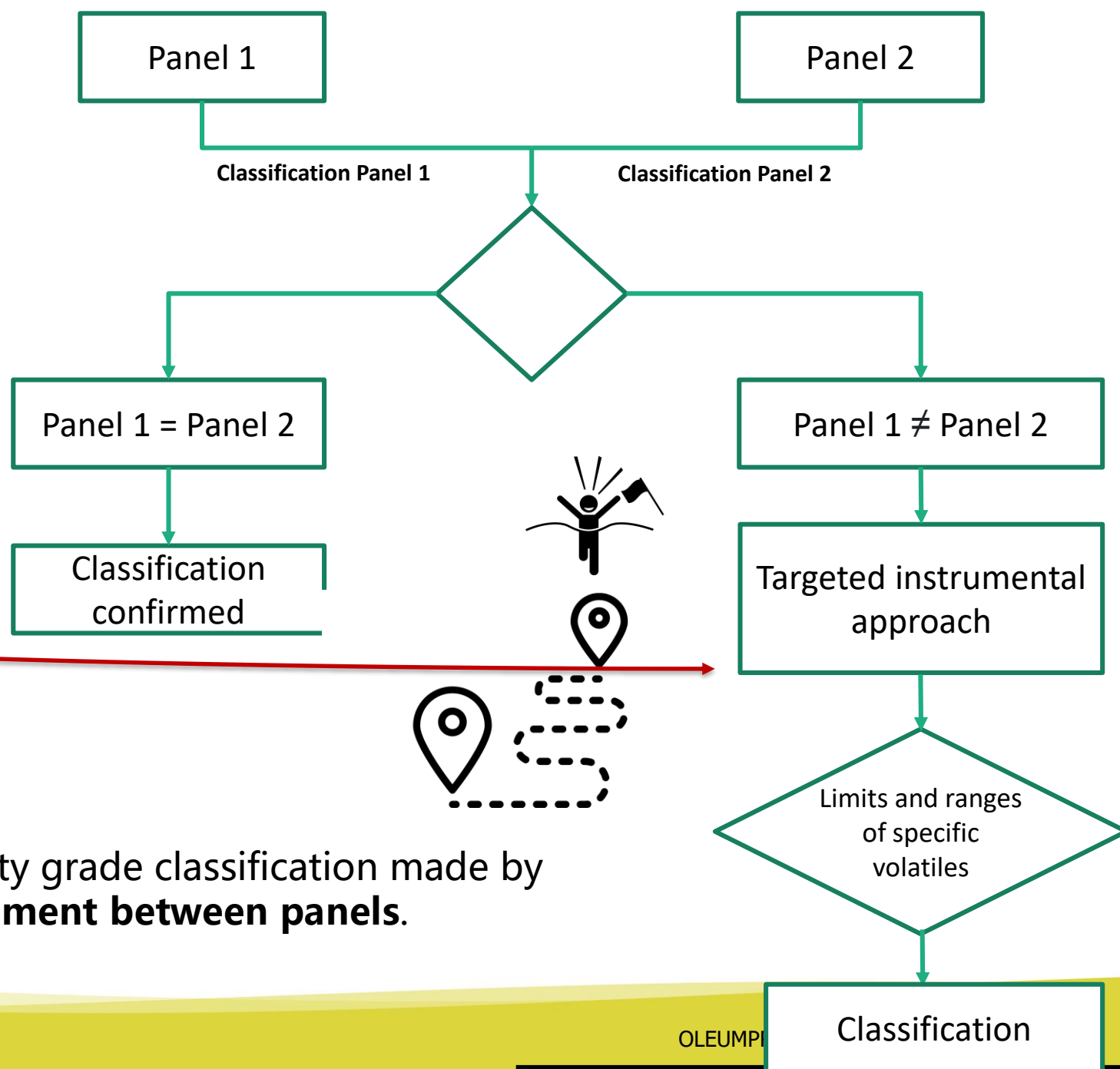
Collection of data in order to establish limits and ranges of volatile compounds.



Definition of limits and ranges

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

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



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

Validated screening methods to support the Panel Test



Article

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}



Article

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

- ☐ HS-GC-IMS
- ☐ FGC-E-nose untargeted approach
- ☐ HS-SPME-GC/MS untargeted approach
- ☐ ¹H-NMR untargeted approach

73-94%
78-92%
93-100%
80-95%

LWT - Food Science and Technology 121 (2020) 108936



Contents lists available at ScienceDirect

LWT - Food Science and Technology

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

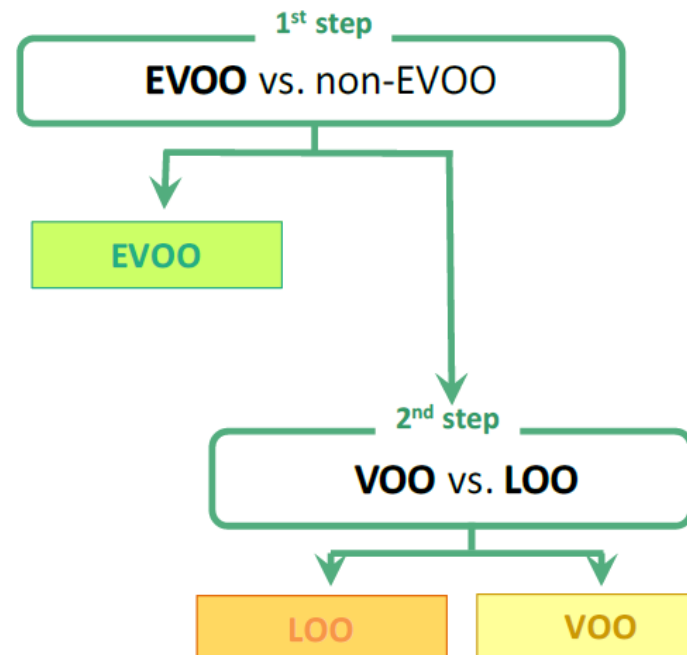


Virgin olive oil volatile fingerprint and chemometrics: Towards an instrumental screening tool to grade the sensory quality

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



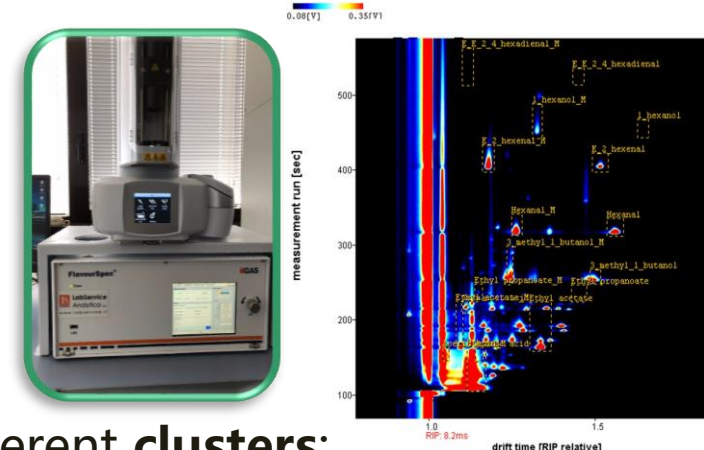
Sequential model



- ✓ 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 reliability 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

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

<https://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.

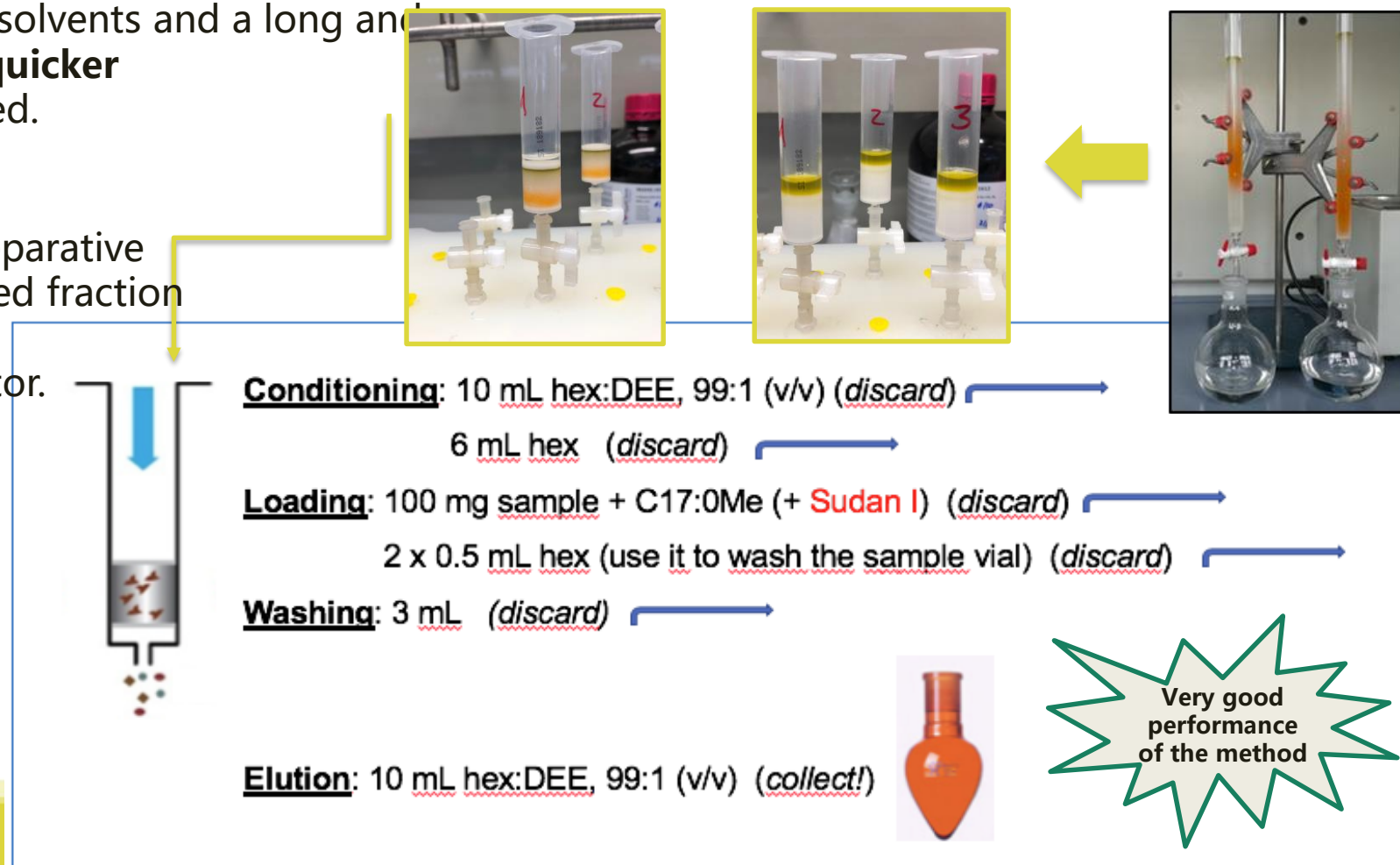
Principle

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)



SPE/GC-FID method to detect both free and esterified hydroxylated minor compounds

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 fractionated 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

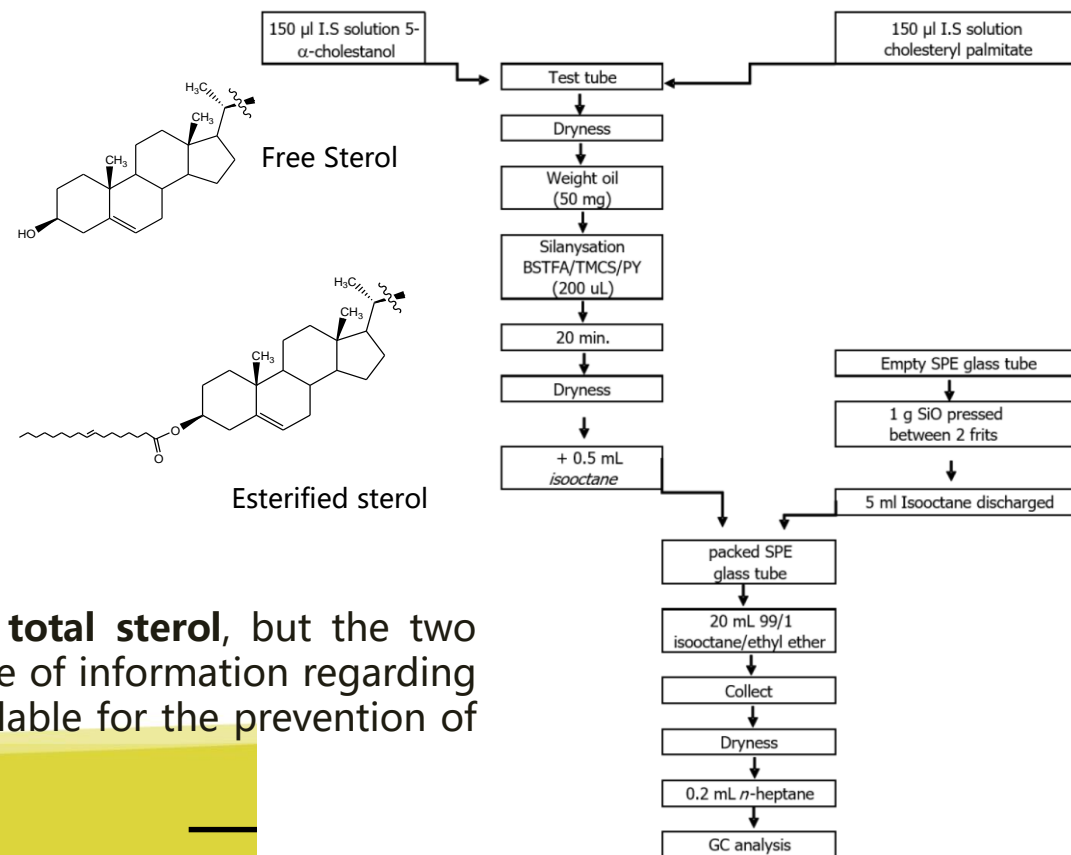
This method determines the free and esterified hydroxylated minor compounds (free and esterified sterols, free and esterified triterpenic alcohols) of olive oils and seed oils and can be utilized as screening tool to detect adulteration by seed oils.

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.

Article

In-House Validation of an SPE-GC-FID Method for the Detection of Free and Esterified Hydroxylated Minor Compounds in Virgin Olive Oils

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,*}



Detection of illegal processing (deodorization)

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:

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

- Useful approach to detect the presence of sdOOs when this is at least at 20% in the illegal mixture

		^e R1	^f R2
	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	^e R1	^f R2
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

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

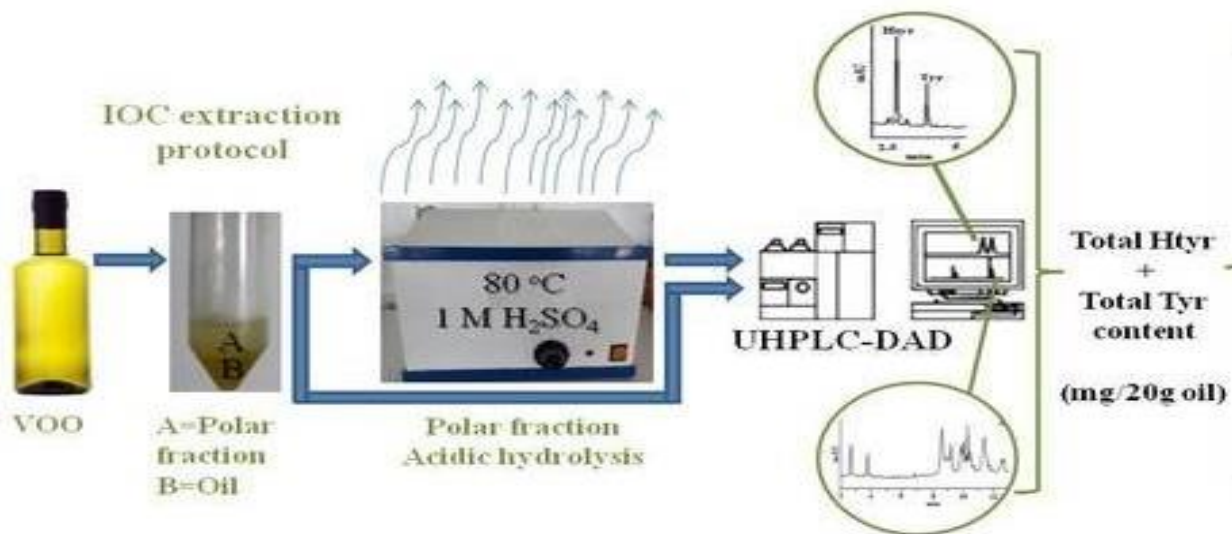
Raquel B. Gómez-Coca^{a,*}, María de Carmen Pérez-Camino^a, Alessandra Bendini^b, Tullia Gallina Toschi^b, Wenceslao Moreda^a



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:

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

*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)...



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

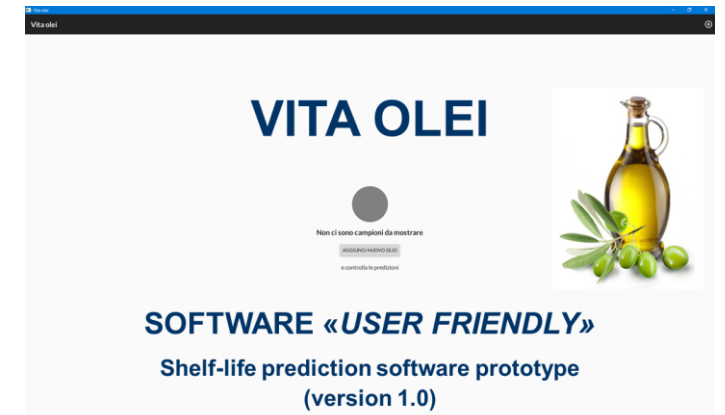


SCAN ME

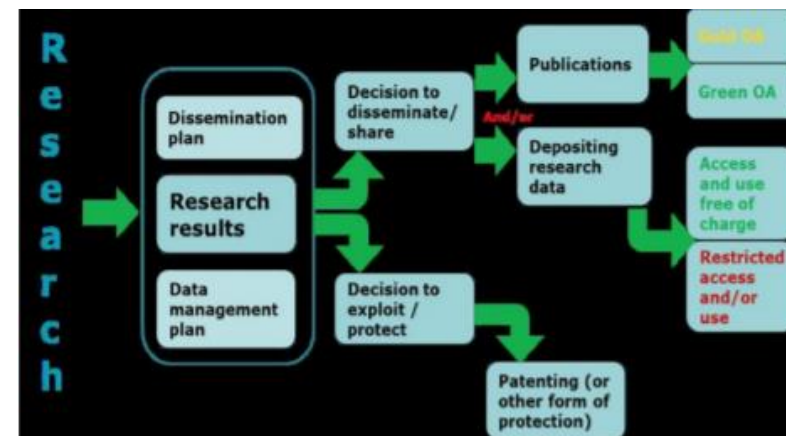


Only **8** (out of 30) samples were found with a total Htyr Tyr content 5 mg/ 20 g oil by the **COI procedure** whereas **16** (out of 30) samples were compliant using the **UHPLC DAD method**.

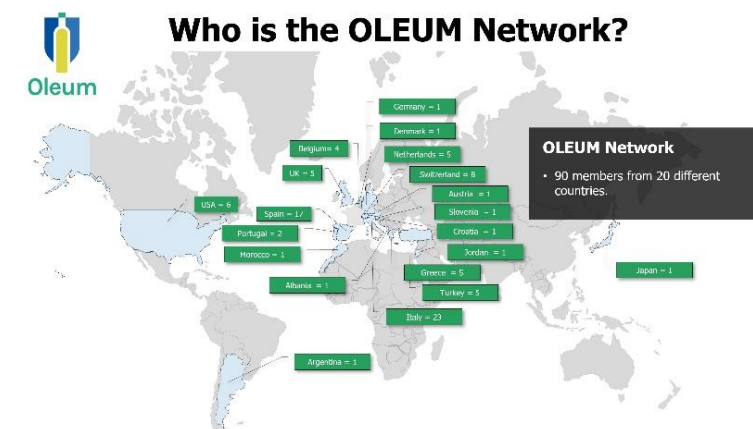
- ✓ **Software to estimate the EVOOs “best before date”**
- ✓ The availability of an instrument for the shelf-life prediction of VOO is worth of value since at the moment the sector operators haven't any method for defining the reliable “best before date” for their VOOs.
- ✓ The software developed has 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”.



- ✓ 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.



- ✓ 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.



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.

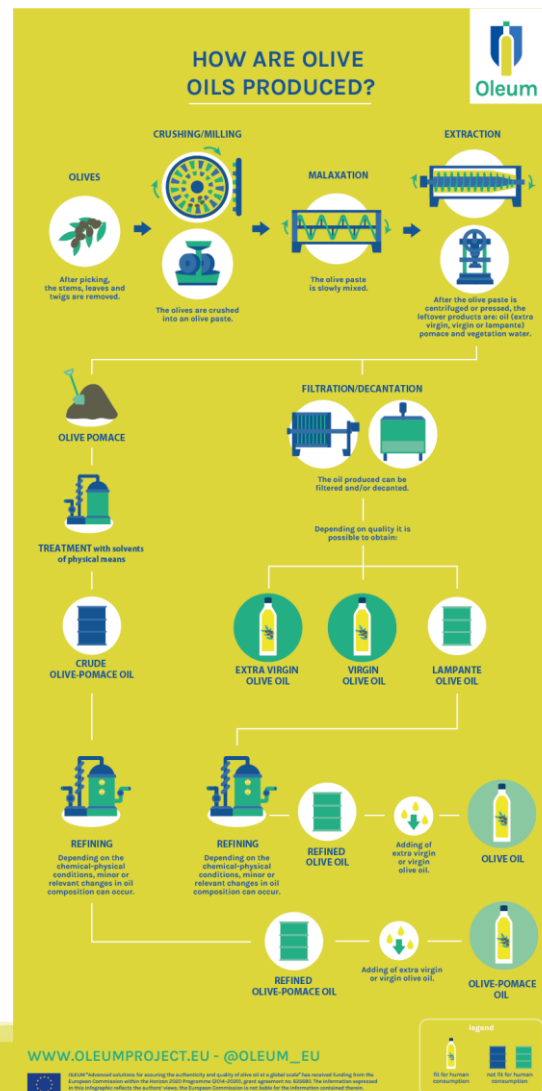
Some strategies to beat fraud at a global level

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

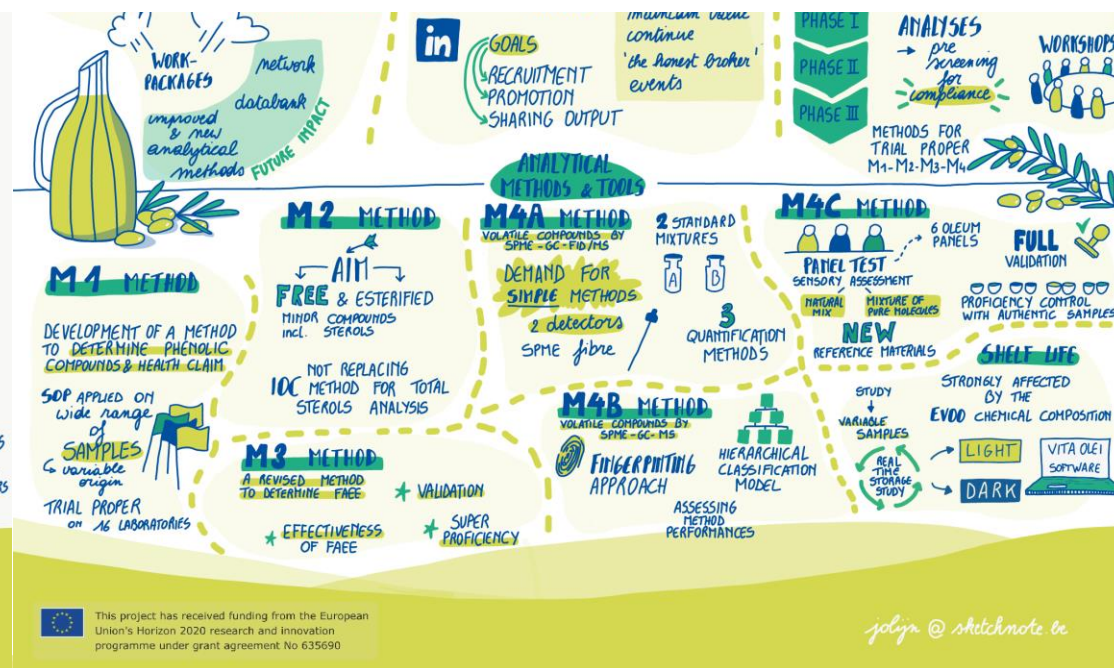
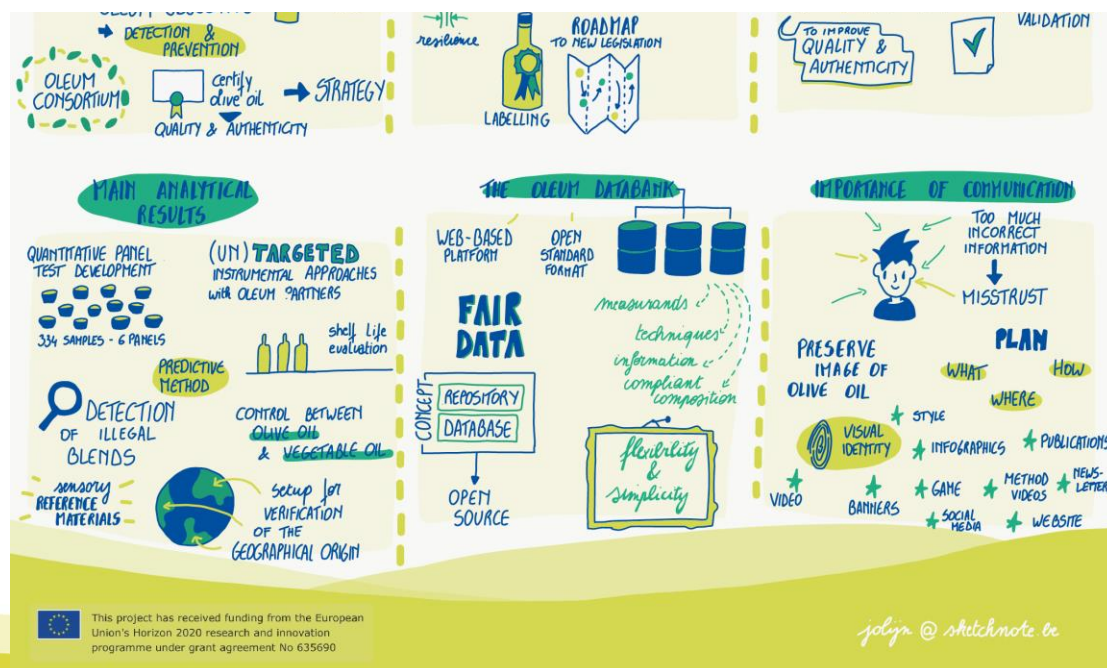


HOW ARE OLIVE OIL PRODUCED?

<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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EU H2020 OLEUM project Coordinator