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OLEUM Project: screening and confirmatory analytical techniques with respect to the sensory profile of virgin olive oils

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Oleum

KEY POINTS

1. **SPME-GC-FID/MS method (COI/T.20/Doc. No. 37)**
   The qualitative-quantitative (targeted) method COI/T.20/Doc. No. 37 currently represents the only validated approach; despite ongoing work within ISO, untargeted methods do not yet meet the requirements for official validation.

2. **Screening methods (HS-GC-IMS / Flash-GC)**

3. **Chemometric estimation approaches and AI**

4. **The future: reference materials**
   a) Cumulative standards of volatile compounds (SMA and SMB); **b)** synthetic or biotechnological sensory standards; and **c)** real samples robustly classified by sensory evaluation (EVOO, VOO, LOO), i.e., **ground-truth references for compliance**.

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1. SPME-GC-FID/MS method (COI/T.20/Doc. No. 37)

Panel disagreement…

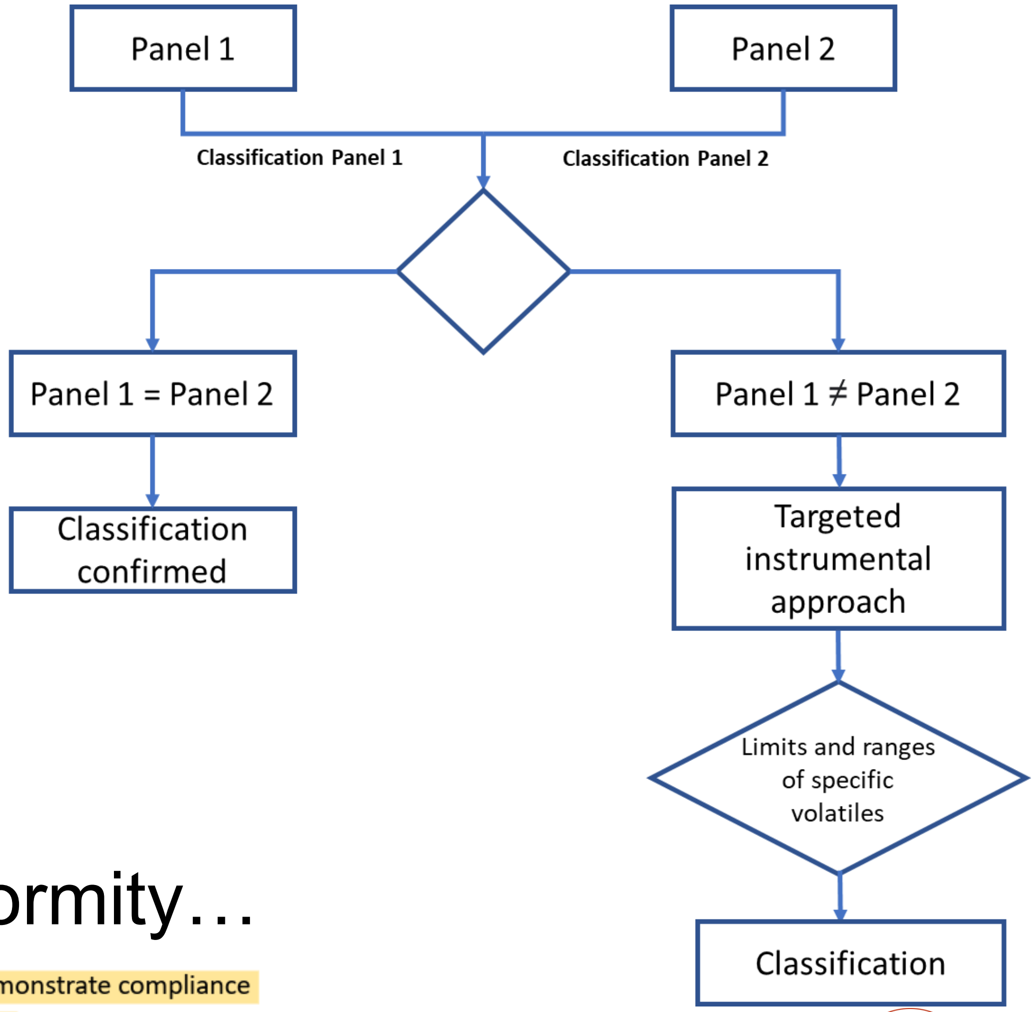
A possibility to strengthen the Panel test is to **quantify specific volatile compounds (as few as possible!) using an officially recognized targeted analysis, as simple as possible, to be applied in cases of disagreement between panels**.


SPECIAL REPORT 01/2026 EN

Control systems for olive oil in the EU

A comprehensive framework, but unevenly applied

EUROPEAN COURT OF AUDITORS



Non-conformity…

42 In the entire EU, the results of 93 % of physico-chemical analyses demonstrate compliance with the requirements, against only 68 % of organoleptic assessments. An organoleptic assessment has the same legal value as a physico-chemical analysis, but it remains subjective due to the inherent complexity of sensory perception. A system of counter-assessments has been set up to resolve controversial cases. An operator can thus request two additional assessments carried out by other tasting panels.

questionable →

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18 selected volatile compounds as the minimum number of 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-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 (Total: 3)
(*E*)-2-hexenal
(*Z*)-3-hexenyl acetate
1-hexanol

A single procedure
Two detectors (FID and MS)

Versatility and adaptability in the application of the method.

Measurands: Selected volatile compounds (VOCs) in virgin olive oils (in **mg/kg**).

Selection criteria: Those VOCs with a demonstrated influence on aroma (fruity and sensory defects).

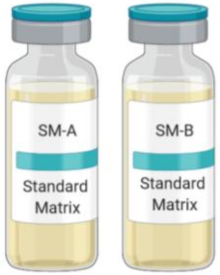
18 VOCs

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

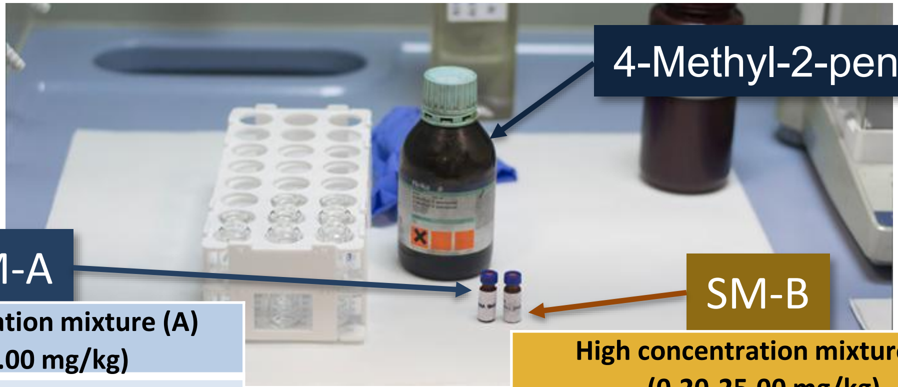
1. **Octane**
2. **Ethyl acetate**
3. **Ethanol**
4. **Ethyl propanoate**
5. **Hexanal**
6. **3-Methyl-1-butanol**
7. **(*E*)-2-Hexenal**
8. **(*Z*)-3-Hexenyl acetate**
9. **(*E*)-2-Heptenal**
10. **6-Methyl-5-hepten-2-one**
11. **1-Hexanol**
12. **Nonanal**
13. **1-Octen-3-ol**
14. **(*E,E*)-2,4-Hexadienal**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

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

2 Standard mixtures: SM A & SM B



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



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

MethodsX 10 (2023) 101972

Contents lists available at ScienceDirect

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

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Method Article

Method for the analysis of volatile compounds in virgin olive oil by SPME-GC-MS or SPME-GC-FID☆,☆☆

Ramón Aparicio-Ruiz[a], Enrico Casadei[b], Clemente Ortiz-Romero[a], Diego L. García-González[a,*], Maurizio Servili[c], Roberto Selvaggini[c], Florence Lacoste[d], Julien Escobessa[d], Stefania Vichi[e], Beatriz Quintanilla-Casas[e], Alba Tres[e], Pierre-Alain Golay[f], Paolo Lucci[g], Erica Moret[g], Enrico Valli[b,*], Alessandra Bendini[b], Tullia Gallina Toschi[b]

You can find the metod here!

https://doi.org/10.1016/j.mex.2022.101972

Food Control 123 (2021) 107823

Contents lists available at ScienceDirect

Food Control

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

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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]

You can find validations here!

https://doi.org/10.1016/j.foodcont.2020.107823

Food Control 135 (2022) 108756

Contents lists available at ScienceDirect

Food Control

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

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Collaborative peer validation of a harmonized SPME-GC-MS method for analysis of selected volatile compounds in virgin olive oils

Ramón Aparicio-Ruiz[a], Clemente Ortiz Romero[a], Enrico Casadei[b], Diego L. García-González[a], Maurizio Servili[c], Roberto Selvaggini[c], Florence Lacoste[d], Julien Escobessa[d], Stefania Vichi[e], Beatriz Quintanilla-Casas[e], Pierre-Alain Golay[f], Paolo Lucci[g], Erica Moret[g], Enrico Valli[b,*], Alessandra Bendini[b], Tullia Gallina Toschi[b]

https://doi.org/10.1016/j.foodcont.2021.108756

You can find full validation here!

European Journal of Lipid Science and Technology

Euro Fed Lipid

RESEARCH ARTICLE | Open Access

Multianalyte analysis of volatile compounds in virgin olive oils using SPME-GC with FID or MS detection: results of an international interlaboratory validation

Diego L. García-González, Enrico Casadei, Ramón Aparicio-Ruiz, Clemente Ortiz Romero, Enrico Valli, Paul Brereton, Anastasios Koidis, Martyna Korytkowska, Maurizio Servili, Roberto Selvaggini, Florence Lacoste, Julien Escobessa, Stefania Vichi, Beatriz Quintanilla-Casas, Alba Tres, Pierre-Alain Golay, Paolo Lucci, Erica Moret, Alessandra Bendini, Tullia Gallina Toschi ... See fewer authors

https://doi.org/10.1002/ejlt.202300079

SCAN ME

COI/T.20/Doc. No 37 – DETERMINATION OF VOLATILE COMPOUNDS IN VIRGIN OLIVE OIL BY SPME-GC-FID/MS

Method COI/T.20/Doc. No 37 /2024

Versions: English French

INTERNATIONAL OLIVE COUNCIL

✓ **The second inter-laboratory study (2025) has recently been completed**

METHOD OF ANALYSIS

ANALYSIS OF VOLATILE COMPOUNDS IN VIRGIN OLIVE OIL BY SPME-GC-FID/MS

SCOPE

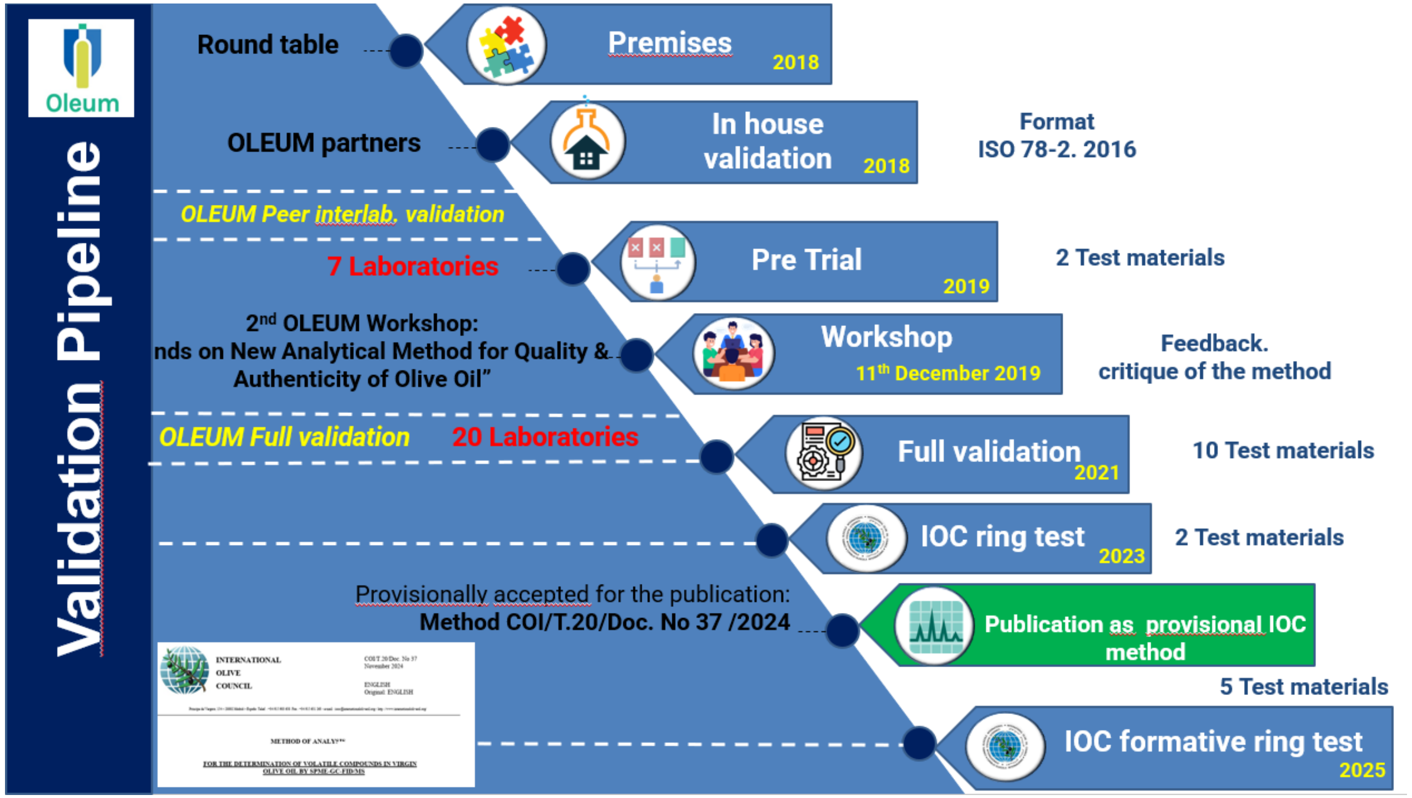
This method has been developed for the determination of the content of selected volatile compounds in the headspace of virgin olive oils. The volatile compounds are responsible for the aroma of virgin olive oils, and in particular for positive attributes or sensory defects. The selected volatile compounds represent the minimum number of diagnostic molecules responsible for fermentative and non-fermentative defects. Thus, fusty-muddy sediment defect is associated to octane, ethanol, 3-methyl-1-butanol, propanoic acid, 6-methyl-5-hepten-2-one, winey-vinegary defect to acetic acid, ethyl acetate, ethanol and musty-humid-earthy to (*E*)-2-heptenal, 1-octen-3-ol, propanoic acid), frostbitten olives defect to ethyl propanoate, and rancid defect to hexanal, nonanal, (*E*,*E*)-2,4-hexadienal, (*E*)-2-decenal, pentanoic acid. In addition, three compounds [(*E*)-2-hexenal, (*Z*)-3-hexenyl acetate, 1-hexanol] were included given their relationship with fruity attribute.

PRINCIPLE

This method is based on the isolation and preconcentration of volatile compounds present in the headspace of virgin olive oils and responsible for aroma by solid phase microextraction (SPME) and subsequent separation of the analytes by gas chromatography (GC) and quantification with a flame ionization detector (FID) or mass spectrometry (MS).

Differences

Both methods allow the quantification of volatile compounds by SPME-GC with two possible detectors, and the use of one detector or the other depends on the technical facilities of the laboratories applying this method; the double choice is designed to favor maximum application of the method.



The method has already been provisionally accepted by the IOC and will be officially published in June 2026.

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MINISTERIO DE CIENCIA E INNOVACIÓN

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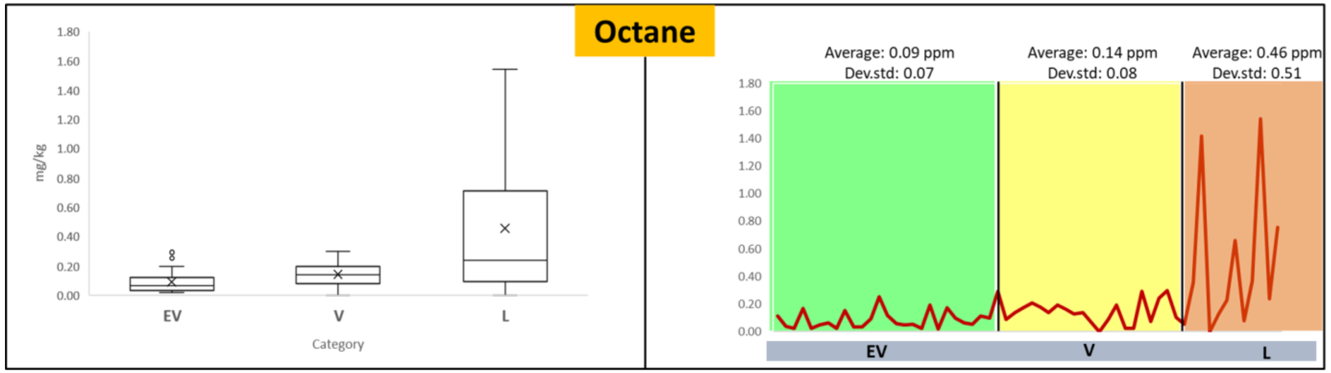
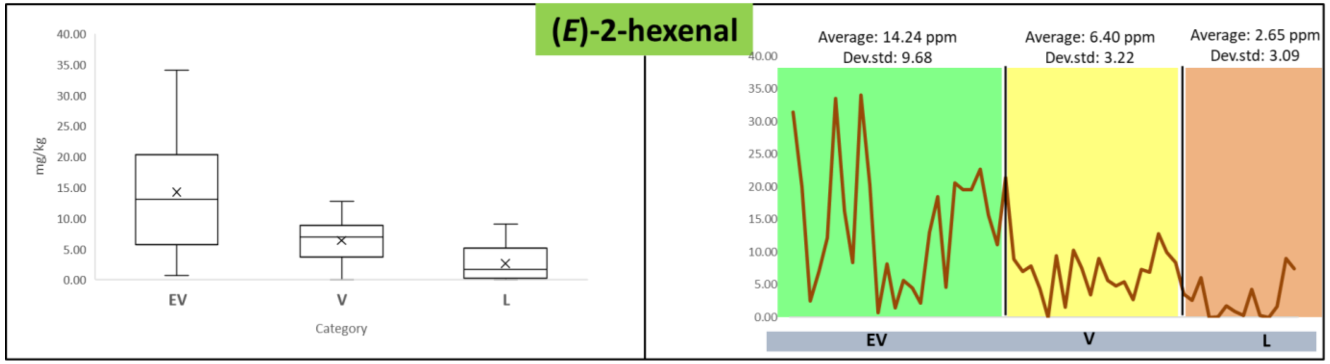
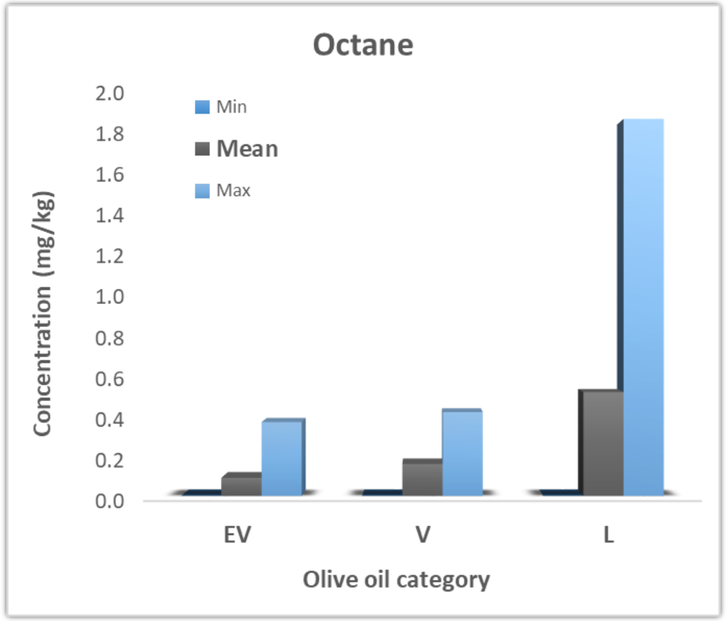
An imperative need for action!

Data collection aimed at establishing **limits and ranges** for volatile compounds.







- **OLEUM**: 60 virgin olive oils analyzed using SPME-GC-MS (ITERG-UNIUD-UNIPG) and SPME-GC-FID (CSIC-UNIBO).
- **Scientific collaboration** agreement between **UNIBO and ICQRF**: 53 virgin olive oils analyzed using SPME-GC-FID.

What is needed to define limits and ranges for volatile compounds within the SPME-GC-FID/MS method (COI/T.20/Doc. No. 37)?

- It is necessary to have a dataset of at least **200–300 real samples robustly classified by sensory evaluation (EVOO, VOO, LOO), i.e., ground-truth references for compliance, representative of the qualitative variability of oils on the market**.

- A **funded project** is therefore required.

Stakeholder engagement

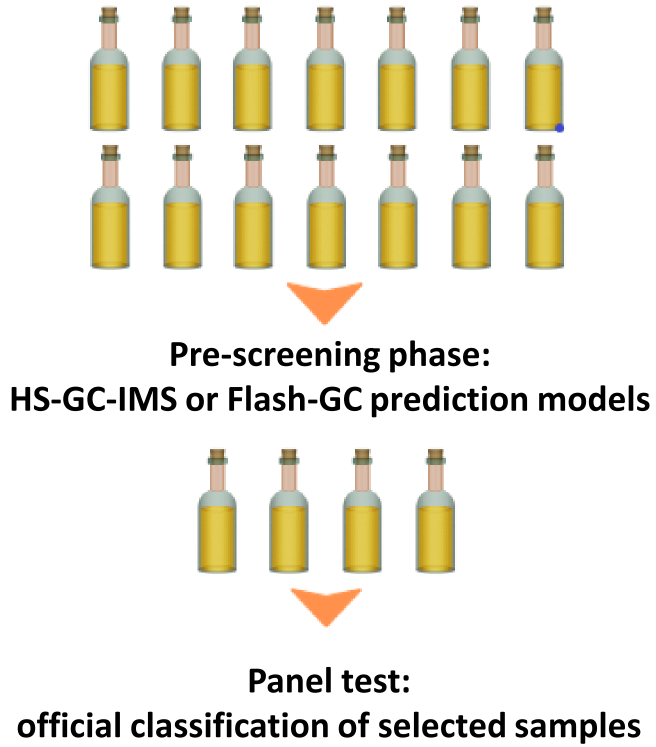
- Support from **industry associations**, **companies**, and **national and international bodies** (national, commercial and/or European funding is needed) will be essential.
- **Validated data** are needed in order to reach agreement and achieve harmonization of limits and ranges.

2. Screening methods (HS-GC-IMS / Flash-GC)

Laboratory analyses are costly and time-consuming…
Pre-screening is therefore necessary before official controls, as well as prior to bottling or blending.

The **Panel test** is **costly** and **time-consuming** to apply to thousands of samples; for this reason, **screening methods** for volatile compounds have been proposed, using instruments such as **GC-IMS** or **Flash-GC**, which are rapid and capable of performing a probabilistic classification of samples, thus **enabling selection across a much broader sample base**.

For example, out **of 100 samples** subjected to screening, **10 deemed of interest** (e.g., potentially misclassified and/or with specific sensory characteristics) can be selected for evaluation using the **official method**, namely the **Panel test**.



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Panel test expensive and time consuming….
screen before…
official controls, but also before botteling or blending.

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To provide support to the Panel test through **instrumental screening approaches** based on the analysis of volatile compounds (**HS-GC-IMS** Flavourspec and **FGC** Heracles), in order to pre/post categorize samples belonging with good probability (risk assessment) to a commercial category (extra virgin, virgin, lampante) to reduce or support, the daily work of the panels themselves.

foods

MDPI

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]

Foods **2020**, 9, 657; doi:10.3390/foods9050657

SCAN ME

foods

Foods **2020**, 9, 862; doi:10.3390/foods9070862

Article

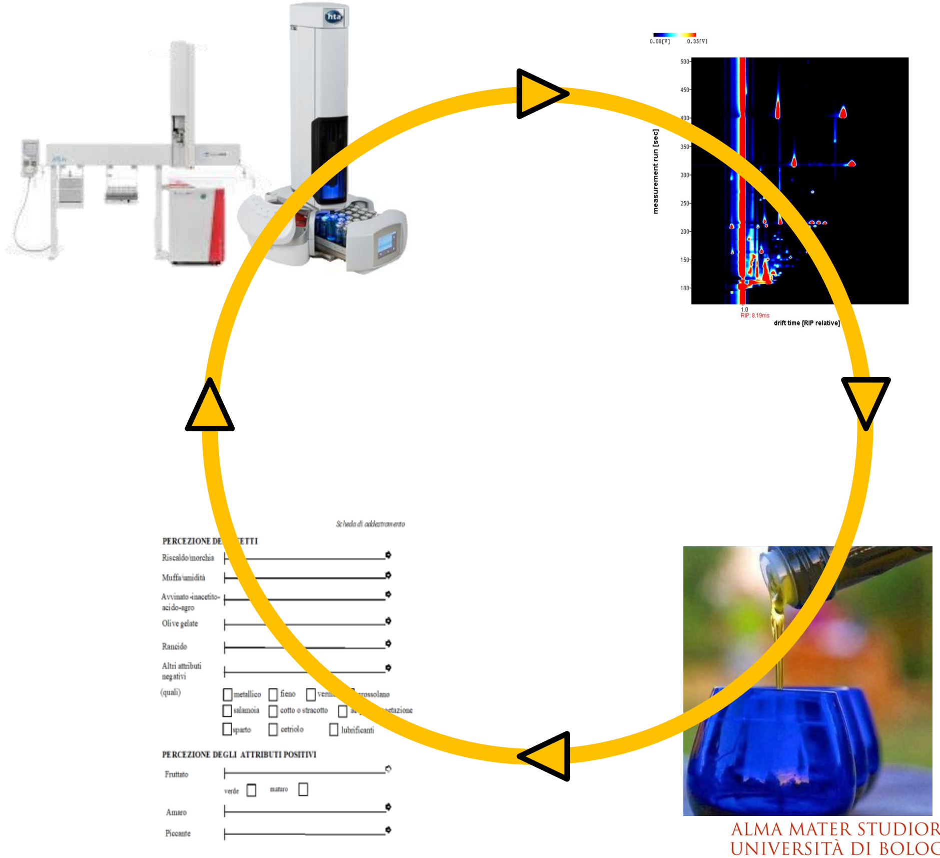
Flash Gas Chromatography in Tandem with Chemometrics: A Rapid Screening Tool for Quality Grades of Virgin Olive Oils

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

SCAN ME

Oleum

Better solutions to protect olive oil quality and authenticity



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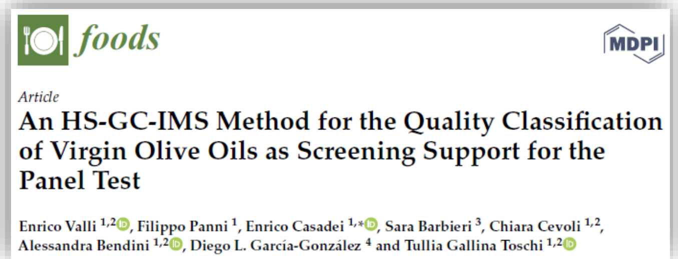
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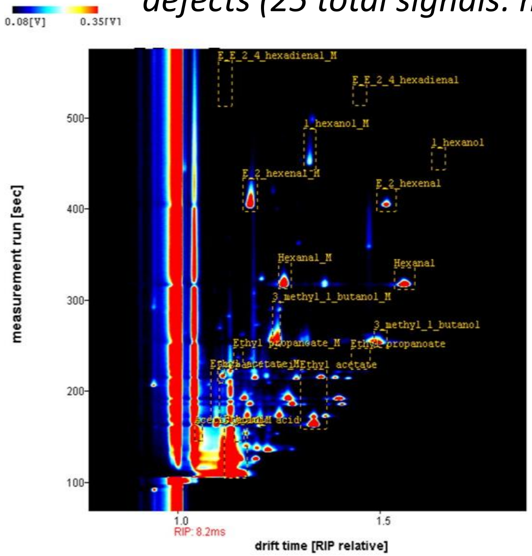
HS-GC-IMS analysis

sensory-classified virgin olive oils

→

Heatmap of 15 volatile compounds associated with fruity attributes and defects (25 total signals: monomers and dimers)



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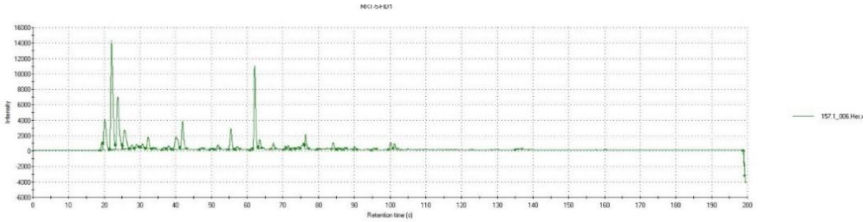
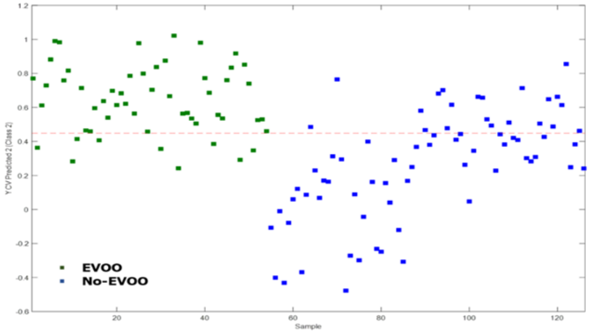
Flash-GC analysis

sensory-classified virgin olive oils

→



Chromatogram obtained from Flash-GC analysis



Chemometric analysis

PLS-DA models:

- EVOO *vs* no-EVOO
- LOO *vs* no-LOO
- EVOO *vs* VOO
- LOO *vs* VOO

→

Classification of samples

EVOO
VOO
LOO

Possible integration with AI systems

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Validation of chemometric models applied to screening methods

Oleum

HS-GC-IMS analysis

1st approach:
classification of samples based on commercial category

PLS-DA model	% external validation
EVOO	74
no-EVOO	77
LOO	73
no-LOO	95
EVOO	70
VOO	67
LOO	77
VOO	87

2nd approach:
samples correctly classified based on the presence of sensory defects

Defects	% external validation
Musty	60
no-musty	80
Rancid	62
no-rancid	64
Fusty/muddy	67
no-fusty/muddy	48

Flash-GC analysis

classification of samples based on commercial category

PLS-DA model	% external validation
EVOO	81
no-EVOO	77
LOO	85
no-LOO	85
EVOO	73
VOO	85
LOO	72
VOO	85

Satisfactory results in terms of the percentage of correctly classified samples *by the four PLS-DA prediction models for the classification of virgin olive oils (EVOO vs. no-EVOO; LOO vs. no-LOO; VOO vs. LOO; EVOO vs. VOO).*

Journal of Food Engineering 391 (2025) 112449

Contents lists available at ScienceDirect

Journal of Food Engineering

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

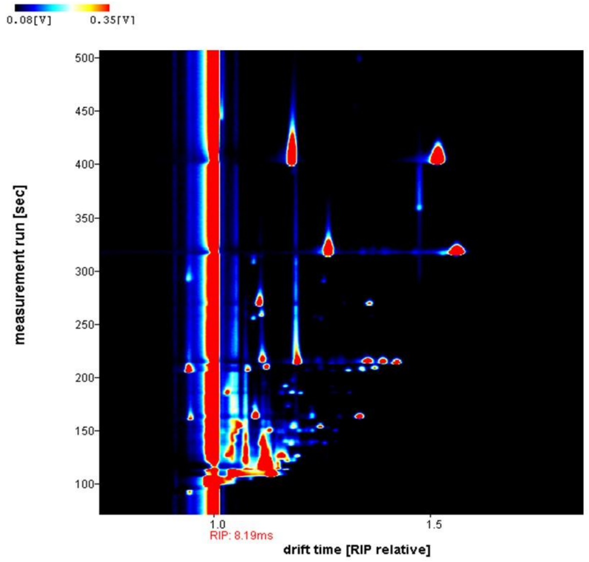
Data fusion of headspace gas-chromatography ion mobility spectrometry and flash gas-chromatography electronic nose volatile fingerprints to estimate the commercial categories of virgin olive oils

Chiara Cevoli[a,b], Ilaria Grigoletto[a], Enrico Casadei[a,b], Filippo Panni[c], Enrico Valli[a,b,*], Sara Barbieri[a], Alessandra Bendini[a,b], Francesca Focante[c], Angela Felicita Savino[c], Stefania Carpino[d], Angelo Fabbri[a,b], Tullia Gallina Toschi[b,e]

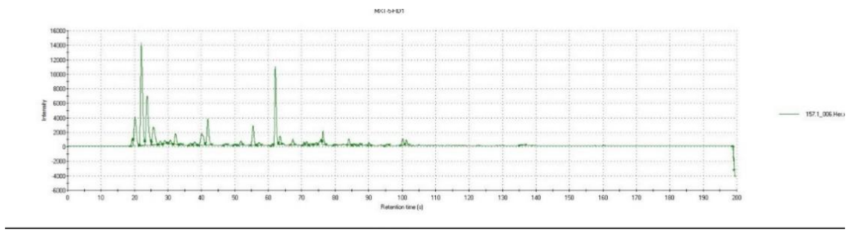
Comparison between the two emerging techniques using the previously developed calibrations

Set consisting of 49 commercial virgin olive oil samples

HS-GC-IMS



Flash-GC



COMMERCIAL CATEGORY	CORRECTLY CLASSIFIED SAMPLES
EVOO	100%
VOO	> 90%
LOO	> 89%

Comparable results between the two techniques, with **satisfactory** performance in terms of correctly classified samples

⇩

Confirmation of the **effectiveness** of both methods and the **robustness** of the estimation models

⇩

Reliability of this approach for r**apid sample screening**, as a valid solution to **support sensory panels**, thereby **increasing the efficiency of controls.**

Which screening method is (or will be) the most widely used?

A screening method becomes widely adopted when the instrumentation provides:

1. Manufacturer support
A dedicated company that supports users (training, updates, documentation)

2. Reliable technical support
Widespread network and rapid response times

3. Data stability and reproducibility
Robust, repeatable and operator-independent analyses

4. Data transferability across instruments
Possibility of calibration / harmonization and comparison between different instruments

STANDARDIZATION = WIDESPREAD ADOPTION

Without standardization there is no confidence in the data
↓
no adoption on a large scale

EXAMPLE: Why has USB-C become the standard?

It has prevailed because it guarantees:

- Reversible and easy to use
- High speed and power capability
- Compatibility across different devices
- Open standard and supported by the industry
- One single cable for data, video and charging

THE WINNER

USB-C

- ✓ Reversible (any orientation)
- ✓ Universal
- ✓ High speed (up to 40 Gbps)
- ✓ High power (up to 240 W)
- ✓ Data, video and audio
- ✓ Global industry adoption

OTHER PROPOSALS (that did not prevail)

USB-A

- ✗ Not reversible
- ✗ Bulky
- ✗ Only USB 2.0/3.x
- ✗ Limited power
- ✗ Being phased out

USB-B

- ✗ Bulky
- ✗ Not practical
- ✗ Specific use (printers, etc.)
- ✗ Not universal

Micro-USB

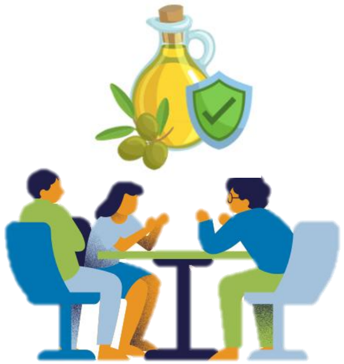
- ✗ Not reversible
- ✗ Fragile
- ✗ Limited speed and power
- ✗ Outdated

Lightning (Apple)

- ✗ Proprietary
- ✗ Works only with Apple devices
- ✗ No standard openness

For screening methods too: the one that is standardizable, transferable and well supported wins.

Application context of screening methods



So how can they be used in a **company**?

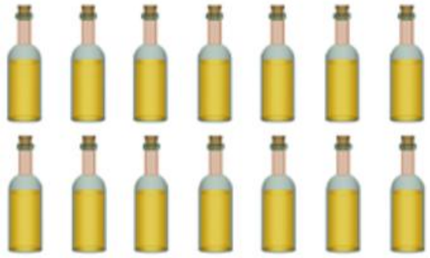
screening

pre-classification

Panel test

Safety in bottling

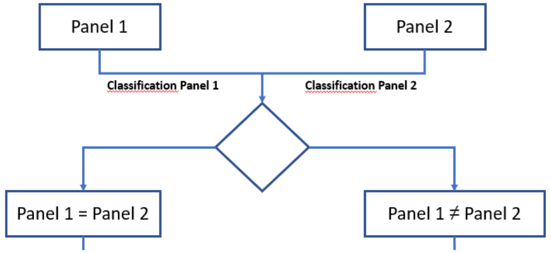
… and for the **control laboratories**?



screening

pre-classification

Panel test



SPME-GC-MS/FID
validated IOC method

3. Chemometric prediction methods and AI

Inter-laboratory comparison → PLS-DA classification models

171 samples

1st STRATEGY

Models developed using data from four laboratories (instrumental and sensory) combined into a **single dataset** (calibration and cross-validation)

2 classification approaches (EV vs noEV and V vs noV) for a total of **2 PLS-DA models**

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	noV	noV	noV	noV
99	EV	noV	noV	noV	noV

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	EV	EV	EV	EV
99	EV	EV	noEV	noEV	EV

2nd STRATEGY
Dependent laboratory

Models developed using data from four laboratories (instrumental and sensory) in **separate datasets** (calibration and cross-validation)

2 classification approaches (EV vs noEV and V vs noV) for each dataset (for each laboratory)
Total of **8 PLS-DA models (4 datasets × 2 approaches)**

4 PLS-DA models V *vs* noV

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	noV	noV	noV	noV
99	EV	noV	noV	noV	noV

4 PLS-DA models EV vs noEV

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	EV	EV	noEV	EV
99	EV	noEV	noEV	noEV	noEV

Statistical significance was tested for all models (Permutation test, Wilcoxon, Sign Test, and Rand t-test)

For all models, classification rule based on the highest probability

1st strategy → Models developed using data from the four laboratories combined into a **single dataset** (cross-validation results)

One PLS-DA model (V *vs* noV)

88% of samples are classified in the same way by the four datasets from the four laboratories.
91% of samples are classified in the same way by at least three of the four laboratory datasets.

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	noV	noV	noV	noV
99	EV	noV	noV	noV	noV
100	BL	noV	noV	noV	noV
101	BL	noV	noV	noV	noV
102	V	V	V	V	V
103	BL	V	V	V	V
104	V	V	V	V	V
105	EV	noV	noV	noV	noV
106	V	noV	noV	V	V
107	V	noV	noV	noV	noV
108	BL	noV	noV	noV	noV
109	BL	noV	noV	noV	noV
110	V	noV	noV	noV	noV
111	EV	noV	noV	noV	noV
112	V	noV	V	V	V
113	V	V	V	V	V
114	V	V	V	V	V
115	EV	noV	noV	noV	noV
116	BL	noV	noV	noV	noV
117	V	V	V	V	V
118	V	noV	V	noV	noV
119	V	V	V	V	V

Classification example

Level of agreement among laboratories

Both models are statistically significant

One PLS-DA model (EV *vs* noEV)

84% of samples are classified in the same way by the four datasets from the four laboratories.
89% of samples are classified in the same way by at least three of the four laboratory datasets.

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	EV	EV	EV	EV
99	EV	EV	noEV	noEV	EV
100	BL	EV	noEV	EV	EV
101	BL	noEV	noEV	noEV	noEV
102	V	noEV	noEV	noEV	noEV
103	BL	noEV	noEV	noEV	noEV
104	V	noEV	noEV	noEV	noEV
105	EV	EV	EV	EV	EV
106	V	noEV	noEV	noEV	noEV
107	V	noEV	noEV	noEV	noEV
108	BL	EV	EV	EV	EV
109	BL	noEV	noEV	noEV	noEV
110	V	noEV	noEV	noEV	noEV
111	EV	noEV	noEV	noEV	noEV
112	V	noEV	noEV	noEV	noEV
113	V	noEV	noEV	noEV	noEV
114	V	noEV	noEV	noEV	noEV
115	EV	EV	noEV	EV	EV
116	BL	noEV	noEV	noEV	noEV
117	V	noEV	noEV	noEV	noEV
118	V	noEV	noEV	noEV	noEV
119	V	noEV	noEV	noEV	noEV

2nd strategy (dependent laboratory)→ Models developed using data from four laboratories in **separate datasets** (cross-validation results)

4 PLS-DA models (V *vs* noV)

80% of samples are classified in the same way by the four models.
84% of samples are classified in the same way by at least three models.

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	noV	noV	noV	noV
99	EV	noV	noV	noV	noV
100	BL	noV	noV	noV	noV
101	BL	noV	noV	noV	noV
102	V	V	V	V	V
103	BL	V	V	V	V
104	V	V	V	V	V
105	EV	noV	noV	noV	noV
106	V	noV	noV	V	noV
107	V	noV	noV	noV	V
108	BL	noV	noV	noV	noV
109	BL	noV	noV	noV	noV
110	V	noV	noV	noV	noV
111	EV	noV	noV	noV	noV
112	V	noV	noV	V	V
113	V	V	V	V	V
114	V	V	V	V	V
115	EV	noV	noV	noV	noV
116	BL	noV	noV	noV	noV
117	V	V	V	V	V
118	V	V	V	noV	noV
119	V	V	V	V	V

Classification example

EV *vs* noEV

```
Probability of Model Insignificance vs. Permuted Samples
For model with 2 component(s)

Y-column:  1
                    Wilcoxon      Sign Test     Rand t-test
Self-Prediction:    0.180         0.222         0.022
Cross-Validated:    0.175         0.212         0.020

Y-column:  2
                    Wilcoxon      Sign Test     Rand t-test
Self-Prediction:    0.180         0.222         0.023
Cross-Validated:    0.175         0.212         0.018

Values less than 0.05 indicate the model is
significant at the 95% confidence level.
```

The four EV vs noEV models are not yet statistically significant → EV samples account for 18% of the dataset.

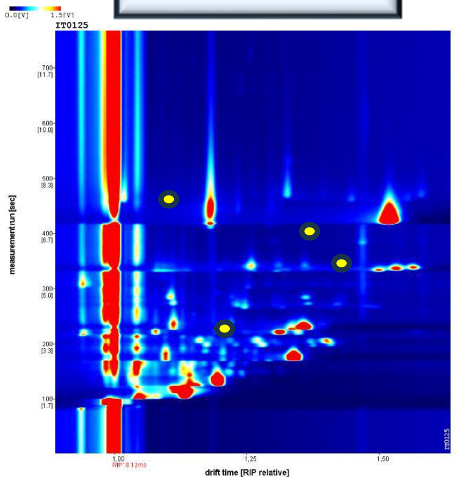
4 PLS-DA models (EV *vs* noEV)

80% of samples are classified in the same way by the four models.
85% of samples are classified in the same way by at least three models

Codice campione	Classificazione sensoriale	O	Z	I	C
98	EV	EV	EV	noEV	EV
99	EV	noEV	noEV	noEV	noEV
100	BL	EV	EV	noEV	EV
101	BL	noEV	noEV	noEV	noEV
102	V	noEV	noEV	noEV	noEV
103	BL	noEV	noEV	noEV	noEV
104	V	noEV	noEV	noEV	noEV
105	EV	EV	EV	EV	EV
106	V	noEV	noEV	noEV	noEV
107	V	noEV	noEV	noEV	noEV
108	BL	noEV	noEV	noEV	EV
109	BL	EV	noEV	noEV	EV
110	V	noEV	noEV	noEV	noEV
111	EV	noEV	noEV	noEV	EV
112	V	noEV	noEV	noEV	noEV
113	V	noEV	noEV	noEV	noEV
114	V	noEV	noEV	noEV	noEV
115	EV	EV	EV	EV	EV
116	BL	noEV	noEV	noEV	EV
117	V	noEV	noEV	noEV	noEV
118	V	noEV	noEV	noEV	noEV
119	V	noEV	noEV	noEV	noEV

Alternative approaches to HS-GC-IMS data analysis

All pixels



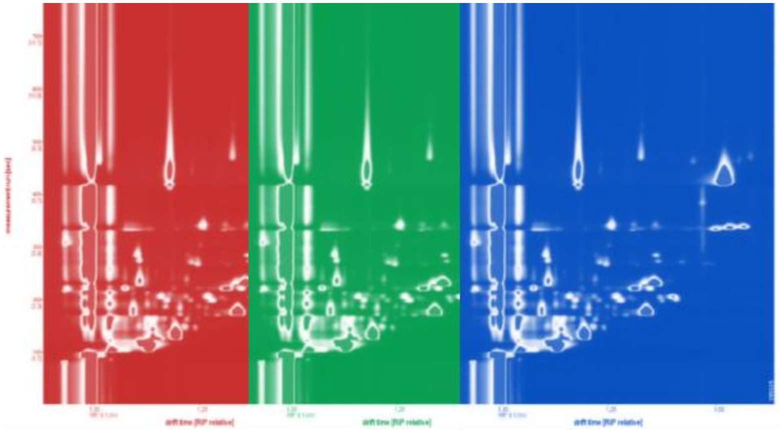
3D to 2D data transformation

90 MB,
4500x5230 pixel

- Very informative.

- Analyzing every single pixel requires a lot of computing power.
- Including all pixels can result in the collection of an excessive amount of data that is unnecessary for the purposes of the analysis.

Image analysis (.png)



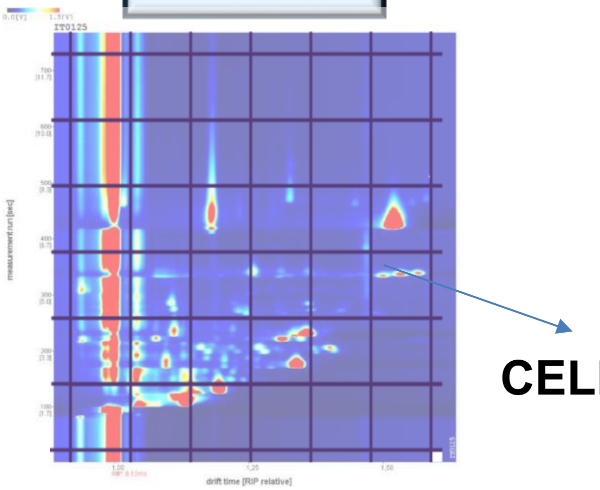
RGB CHANNELS

9 MB,
4500x5230 pixel

- "Lighter" data, since it is derived from the three RGB (Red-Green-Blue) channels.

- We are working with an indirect parameter (color variation) that is correlated with changes in intensity.

Matrix area



CELLS

30 KB,
20x19 cells (editable)

- Very «light» data.
- Model training is much faster; we work with numerical data .

- Set an appropriate number of cells; otherwise, you risk losing important information.

Application of AI to HS-GC-IMS data processing

- HS-GC-IMS generates complex fingerprints (multidimensional heatmaps)
- This requires **advanced data processing** for **rapid and robust interpretation**

- Limitations of traditional approaches (e.g., PLS-DA):

a) Limited transferability across instruments

b) Relatively high operational complexity (*requiring both analytical and chemometric expertise*)

The Role of AI

- **Rapid, automated analysis** of **GC-IMS** and **sensory data**
- **Direct integration** of **heatmaps/sensory data as input**
- **Output**: probabilistic classification (EVOO vs VOO)
- Robust and reproducible predictions (given sufficient data)

Note: The system requires continuous monitoring and supervision.

4. The future: reference materials

Both instrumental and sensory analyses require reference materials.

→ **SPME-GC-FID/MS method (COI/T.20/Doc. No. 37)**

COI/T.20/Doc. No 37
page 4

3.1. PROTOCOL FOR THE PREPARATION OF CALIBRATION CURVES FOR VOLATILE ANALYSIS (SPME-GC-MS AND SPME-GC-FID)

The calibration curves are prepared by using two standard mixtures (SM)*, named SM A and SM B, each containing different analytes as described below (Table 1):

Table 1. Standards of volatile compounds included in the two standard mixtures.

Low concentration mixture (SM A) (0.05-10.00 mg/kg)	High concentration mixture (SM B) (0.20-25.00 mg/kg)
Octane	Ethanol
Ethyl acetate	Hexanal
Ethyl propanoate	(*E*)-2-Hexenal
3-Methyl-1-butanol	(*Z*)-3-Hexenyl acetate
(*E*)-2-Heptenal	1-Hexanol
6-Methyl-5-hepten-2-one	Nonanal
(*E,E*)-2,4-Hexadienal	1-Octen-3-ol
Propanoic acid	Acetic acid
(*E*)-2-Decenal	
Pentanoic acid	

*In order to minimize competition phenomena between volatile compounds, the standards are divided into two mixtures (SM A and SM B).

Commercial certified reference materials (e.g., **SM A, SM B** for the COI/T.20/Doc. No. 37 method) to support instrumental methods

→ **IMPACT**

- Greater data reliability
- Harmonization of analytical data
- Reduction of non-compliance

IS analytica

Advantages:

- ✓ Ready-to-use: no weighing or mixing required
- ✓ Certified (ISO 17034 supplier)
- ✓ Shelf life: 6 months
- ✓ Packaging: sealed amber glass vials

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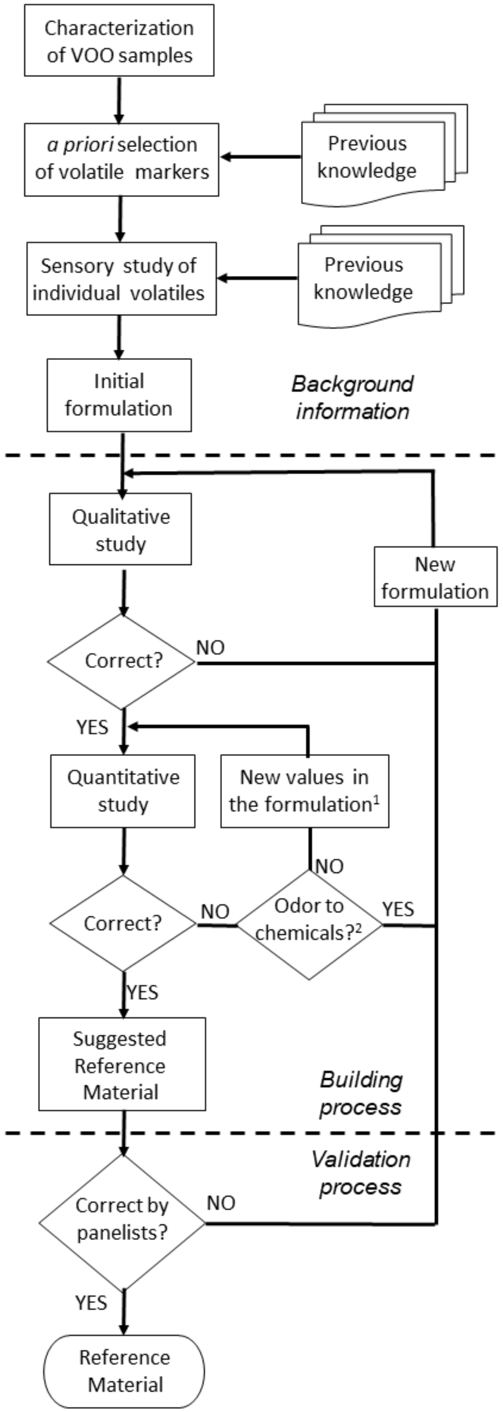
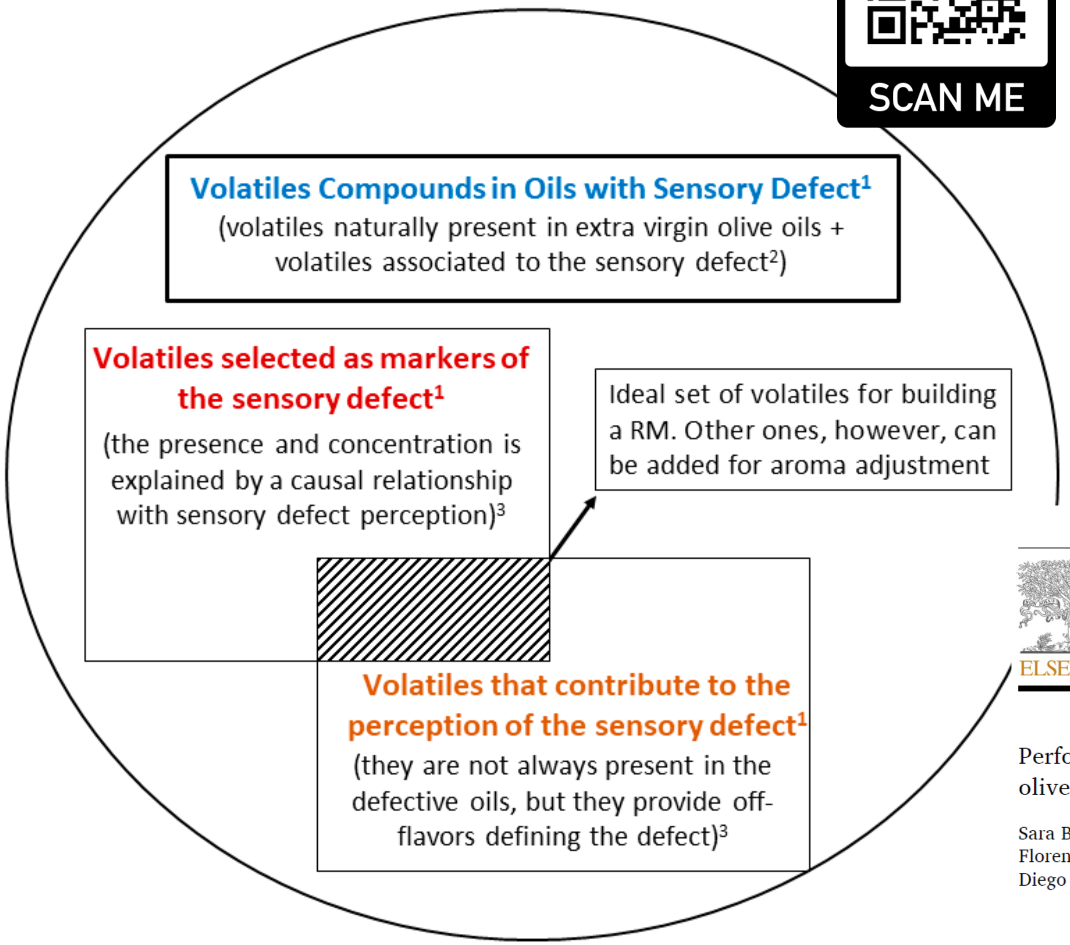
Formulation of two olfactory sensory reference materials

Flow diagram for formulating RMs





Scheme to select volatile compounds as candidates to formulate RMs of aroma sensory defects detected in VOOs.

SCAN ME

foods

Article

Formulations of Rancid and Winey-Vinegary Artificial Olfactory Reference Materials (AORMs) for Virgin Olive Oil Sensory Evaluation

Ramón Aparicio-Ruiz [1,2,*], Sara Barbieri [3,*], Tullia Gallina Toschi [4] and Diego L. García-González [1]

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ELSEVIER

Performance testing of new artificial olfactory reference materials in virgin olive oil sensory assessment

Sara Barbieri [a], Ramon Aparicio-Ruiz [b,c], Karolina Brkic Bubola [d], Milena Bucar-Miklavcic [e], Florence Lacoste [f], Ummuhan Tibet [g], Ole Winkelmann [h], Alessandra Bendini [i,*], Diego Luis Garcia-Gonzalez [b], Tullia Gallina Toschi [i]

SCAN ME

Oleum

The possibility of panel disagreement is the starting point → real samples robustly classified by sensory evaluation (EVOO, VOO, LOO) as ground-truth references for compliance

SCAN ME

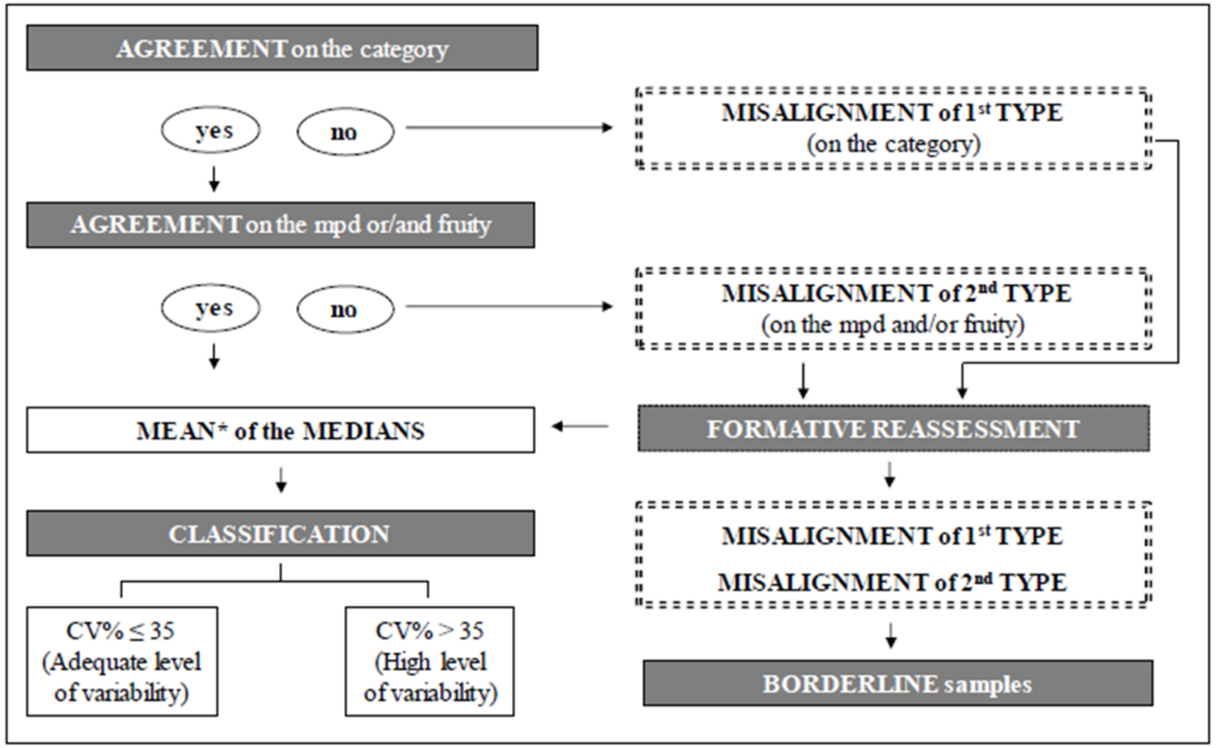
foods

MDPI

Article

Alignment and Proficiency of Virgin Olive Oil Sensory Panels: The OLEUM Approach

Sara Barbieri [1], Karolina Brkić Bubola [2], Alessandra Bendini [1,*], Milena Bučar-Miklavčič [3], Florence Lacoste [4], Ummuhan Tibet [5], Ole Winkelmann [6], Diego Luis García-González [7] and Tullia Gallina Toschi [1]



Majority on 6 panels in agreement on the category and on the mpd or/and fruity
to robustly sensory classify samples

A mistake in the classification will be a mistake in the instrumental calibration!

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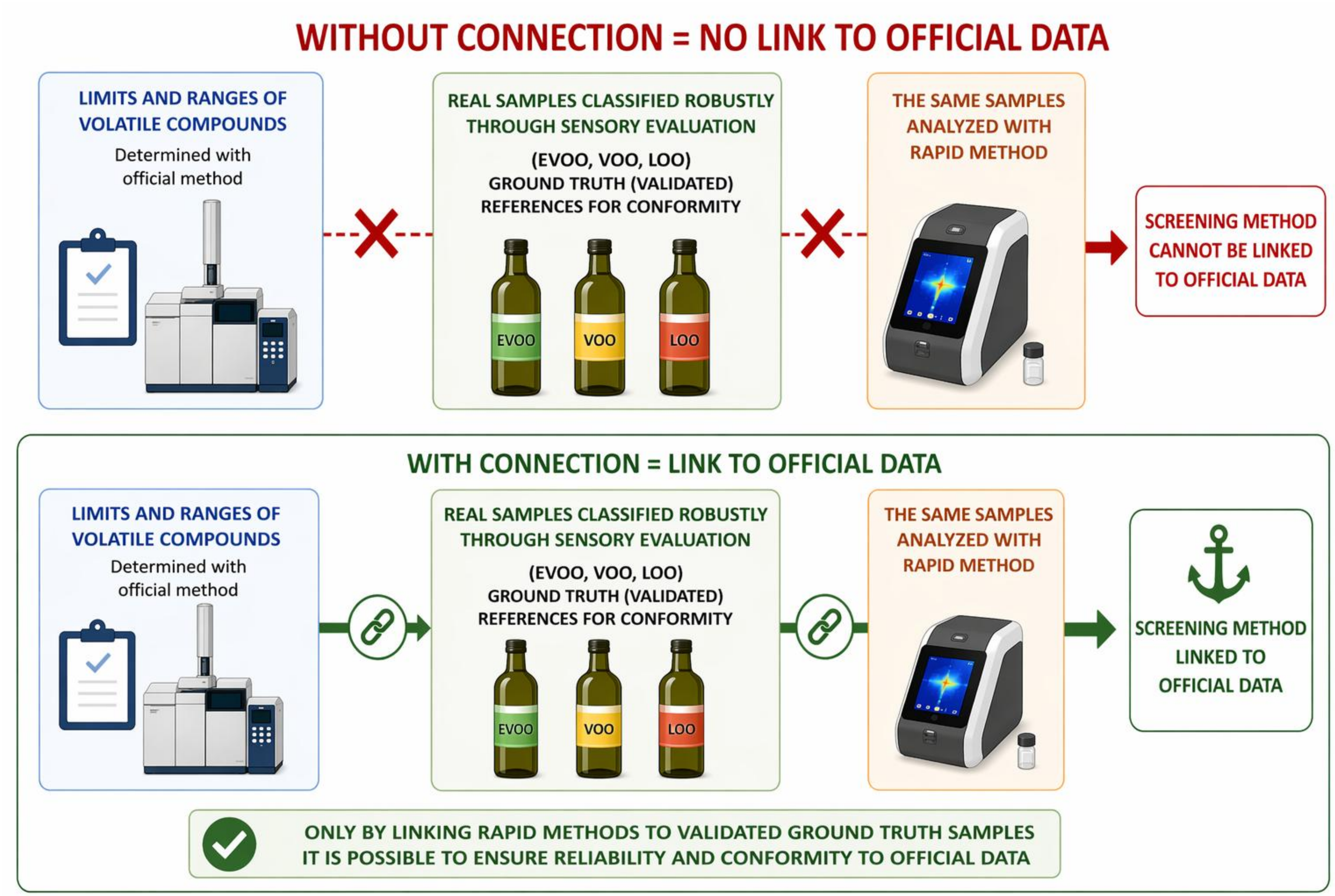

WITHOUT CONNECTION = NO LINK TO OFFICIAL DATA
LIMITS AND RANGES OF VOLATILE COMPOUNDS
Determined with official method
REAL SAMPLES CLASSIFIED ROBUSTLY THROUGH SENSORY EVALUATION
(EVOO, VOO, LOO)
GROUND TRUTH (VALIDATED) REFERENCES FOR CONFORMITY
EVOO
VOO
LOO
THE SAME SAMPLES ANALYZED WITH RAPID METHOD
SCREENING METHOD CANNOT BE LINKED TO OFFICIAL DATA
WITH CONNECTION = LINK TO OFFICIAL DATA
LIMITS AND RANGES OF VOLATILE COMPOUNDS
Determined with official method
REAL SAMPLES CLASSIFIED ROBUSTLY THROUGH SENSORY EVALUATION
(EVOO, VOO, LOO)
GROUND TRUTH (VALIDATED) REFERENCES FOR CONFORMITY
EVOO
VOO
LOO
THE SAME SAMPLES ANALYZED WITH RAPID METHOD
SCREENING METHOD LINKED TO OFFICIAL DATA
ONLY BY LINKING RAPID METHODS TO VALIDATED GROUND TRUTH SAMPLES
IT IS POSSIBLE TO ENSURE RELIABILITY AND CONFORMITY TO OFFICIAL DATA

What we have

- ✓ **Validated and official SPME-GC-FID/MS (COI/T.20/Doc. No. 37) method → achieved**

- ✓ **Limits and ranges for volatile compounds → absolutely needed**

- ✓ **Screening methods (HS-GC-IMS / Flash-GC) → in use for estimation purposes, anchoring to the official method is required**

- ✓ **Reference materials →** Available: a) SMA, SMB, b) two olfactory sensory reference materials, and c) **real samples robustly classified by sensory evaluation** (EVOO, VOO, LOO) **as ground-truth references for compliance** (spin-off?)

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Recommendations and Future Directions

- Develop and commercialize **standards** and **reference materials** → **proficiency**

- Strengthen **international collaboration**, including the application of **screening methods** (calibration standards, shared **robustly sensory-classified samples**, **instrumental reliability and signal stability**, **harmonized analytical conditions**) → **harmonized models**

- Promote the use of **untargeted methods**, including through **open-access data processing (prompting) with sustainable AI**, while maintaining **robust quality control based on specific measurands**, with shared **limits and ranges** (**data quality oversight**).

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