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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☆,☆☆

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REVIEW HIGHLIGHTS

- A SPME-GC method to determine selected volatile compounds in virgin olive oils was developed and validated.
- The method can support the official sensory evaluation of virgin olive oils (Panel test).
- Two detectors can be used depending on the technical facilities of the laboratory.

ARTICLE INFO

Method name:
Analysis of selected volatile compounds in virgin olive oil by SPME-GC-MS or SPME-GC-FID

Keywords:
Olive oil
Volatiles
Gas chromatography

ABSTRACT

During the course of the EU H2020 OLEUM project, a harmonized method was developed to quantify volatile markers of the aroma of virgin olive oil with the aim to support the work of sensory panel test to assess the quality grade. A peer validation of this method has been carried out, with good results in terms of analytical quality parameters. The method allows the quantification of volatile compounds by SPME-GC with two possible detectors, flame ionization detector and mass spectrometry, depending on the technical facilities of the labs applying this method. The method was optimized for the quantification of 18 volatile compounds that were selected as being markers responsible for positive attributes (e.g. fruity) and sensory defects (e.g. rancid

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and winey-vinegary). The quantification is carried out with calibration curves corrected by the internal standards. Additionally, a protocol is provided to prepare the calibration samples. This procedure enhances reproducibility between labs since one of the main sources of errors is the application of different procedures in calibration.

Specifications table

Subject area:	Agricultural and Biological Sciences
More specific subject area:	Food Science
Name of your method:	Analysis of selected volatile compounds in virgin olive oil by SPME-GC-MS or SPME-GC-FID
Name and reference of original method:	Aparicio-Ruiz, R., Ortiz Romero, C., Casadei, E., García González, D.L., Servili, M., Selvaggini, R., Lacoste, F., Escobessa, J., Vichi, S., Quintanilla-Casas, B., Golay, P.A., Lucci, P., Moret, E., Valli, E., Bendini, A., Gallina Toschi, T. (2022) Collaborative peer validation of a harmonized SPME-GC-MS method for analysis of selected volatile compounds in virgin olive oils. Food Control, 135, 108756. doi:10.1016/j.foodcont.2021.108756. Casadei, E., Valli, E., Aparicio-Ruiz, R., Ortiz Romero, C. García- González, D.L., Vichi, S. Quintanilla-Casas, B., Tres, A., Bendini, A. Gallina Toschi, T. (2021). Peer inter-laboratory validation study of a harmonized SPME-GC-FID method for the analysis of selected volatile compounds in virgin olive oils. Food Control, 123, 107823. doi:10.1016/j.foodcont.2020.107823.
Resource availability:	N.A.

Method details

This method is based on the isolation and preconcentration of volatiles by solid phase microextraction (SPME) and subsequent separation of analytes by gas chromatography and quantification with a flame ionization detector (FID) and mass spectrometry (MS), depending on the technical facilities of the labs applying this method.

Apparatus

- Headspace glass vial, 20 mL.
- Septum and aluminum seals or seals adaptable to the autosampler if the latter is used.
- Gas chromatograph (GC), suitable for use with a capillary column, equipped with a split/splitless injector and a mass spectrometry detector (MS)/flame ionization detector (FID). Depending on the detector available in the laboratory, the gas chromatographic analysis can be carried out with one of the two detectors (see the section "Gas chromatography analysis (choose one of two options)" below).

  - **SPME injector liner**, 0.75 mm ID.
  - **Capillary column, fused silica,** a polar phase based on polyethylene glycol (PEG) (e.g. ZB-WAX or TR-WAX), length 60 m, internal diameter 0.25 mm and coating 0.25 – 0.50 μm.

- **SPME fiber**, length 1 cm, 50/30 μm film thickness and endowed with the Stable Flex stationary phase of divinylbenzene/carboxen/polydimethylsiloxane. The fiber should be previously conditioned by following the instructions of the supplier.
- **Analytical balance** for weighing to an accuracy of ± 0.1 mg.

Reagents

- **4-methyl-2-pentanol,** chromatography grade, with a purity higher than 95%.
- **Analytical standards**
  - Octane: CAS Number 111-65-9; assay ≥ 99.7%; density 0.703 g/mL at 25˚C;
  - Ethanol: CAS Number 64-17-5; assay ≥ 99.9%; density 0.7893-0.7899 at 20˚C;
  - 3-Methyl-1-butanol: CAS Number 123-51-3; assay ≥ 98.5%; density 0.809 g/mL at 25˚C;
  - Propanoic acid: CAS Number 79-09-4; assay ≥ 99.8%; density 0.993 g/mL at 25˚C;
  - 6-methyl-5-hepten-2-one: CAS Number 110-93-0; assay ≥ 97.0%; density 0.855 g/mL at 25˚C;
  - Acetic acid: CAS Number 64-19-7; assay ≥ 99.8% (GC); density 1.049 g/mL at 25˚C;
  - Ethyl acetate: CAS Number 141-78-6; assay ≥ 99.8%; density 0.902 g/mL at 25˚C;
  - (*E*)-2-heptenal: CAS Number 18829-55-5; assay ≥ 95%; density 0.857 g/mL at 25˚C;
  - 1-octen-3-ol: CAS Number 3391-86-4; assay ≥ 98.0%; density 0.83 g/mL at 25˚C;
  - Ethyl propanoate: CAS Number 105-37-3; assay ≥ 99.7%; density 0.888 g/mL at 25˚C;
  - Hexanal: CAS Number 66-25-1; assay 98 %; density 0.834 g/mL at 25˚C;
  - Nonanal: CAS Number 124-19-6; assay ≥ 95%; density 0.827 g/mL at 25˚C;
  - (*E,E*)-2,4-hexadienal: CAS Number 142-83-6; assay ≥ 95.0%; density 0.871 g/mL at 25˚C;
  - (*E*)-2-decenal: CAS Number 3913-81-3; assay ≥ 95.0%; density 0.841 g/mL at 25˚C;
  - Pentanoic acid: CAS Number 109-52-4; assay ≥ 99.8%; density 0.939 g/mL at 25˚C;
  - (*E*)-2-hexenal: CAS Number 6728-26-3; assay ≥ 97.0%; density 0.846 g/mL at 25˚C;

- (*Z*)-3-hexenyl acetate: CAS Number 3681-71-8; assay ≥ 98.0%; density 0.897 g/mL at 25°C;
- 1-hexanol: CAS Number 111-27-3; assay ≥ 99.9% (GC); density 0.814 g/mL at 25°C.

- *n*-Alkanes, from 8 to 20 carbon atoms, custom blend standard (~40 mg/L each, in hexane).
- Carrier gas: helium (or hydrogen if the equipment and installation are adapted and allow for safety procedure; attention should be paid to eventual formation of interferents), pure, gas chromatography grade.
- Auxiliary gases: in the case of using nitrogen for conditioning/cleaning the fiber with an autosampler, use gas chromatography grade.

Procedure

Before starting the analysis of the samples, build the external calibration curves following the protocol below.

The integration method for obtaining the chromatographic areas should be the same in both the calibration curves and the analytes in the sample.

Protocol for the preparation of calibration curves for volatile analysis (SPME-GC-MS and SPME-GC-FID)

Caution: SM A and SM B must be stored at -18°C and, for their subsequent use, some precautions must be followed: leave the two mixtures for an adequate time at room temperature (never heating), shake carefully before use and return them to the freezer. Moreover, it is very important to never leave the vials open and never keep them at room temperature for an extended period of time. Work at controlled room temperature (T = 20-25°C) due to the volatility of the standards. A rapid preparation of the solutions is advisable to avoid evaporation of compounds.

The calibration curves are prepared by using two mixtures*, each containing different analytes as described below:

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 volatiles compounds it was decided to divide the stock solution in two mixtures (SM A and SM B).

Note: For calculation of the concentrations, use the exact concentration of SM A and SM B.

Preparation of Standard Mixtures (SM) at 10000 mg/kg

For the low concentration mixture (SM A):

1. Put an empty vial of 20 mL in the analytical balance and tare it.
2. Weigh 5.000 ± 0.001 g of refined olive oil in the vial.
3. Put approx. 0.100 ± 0.001 g for each standard, exactly weighed, for the mixture A (low concentration, 10 compounds) in the vial.
4. Add refined olive oil up to reach approx. 10.000 ± 0.001 g, exactly weighed.
5. Close the vial (cap + septum).
6. Shake for 30 seconds on the agitator (e.g. vortex).

Note: Do not forget to write down the weights for concentration calculation.

For the high concentration mixture (SM B):

1. Put an empty vial of 20 mL in the analytical balance and tare it.
2. Weigh 5.000 ± 0.001 g of refined olive oil in the vial.
3. Put approx. 0.100 ± 0.001 g for each standard, exactly weighed, for the mixture B (high concentration, 8 compounds) in the vial.
4. Add refined olive oil up to reach approx. 10.000 ± 0.001 g, exactly weighed.
5. Close the vial (cap + septum).
6. Shake for 30 seconds on the agitator (e.g. vortex).

Note: Do not forget to write down the weights for the concentration calculation.

Preparation of dilutions from the two SM (A and B)

Note: the following procedure has to be applied in the same way, both from SM A and SM B

SM1 (200 mg/kg):

1. Place a vial (labelled as SM1) of 20 mL on the analytical balance and tare it.
2. Weigh 5.000 ± 0.001 g of refined olive oil.
3. Put approx. 0.200 ± 0.001 g of SM, exactly weighed, in the vial.
4. Add refined olive oil up to reach approx. 10.000 ± 0.001 g, exactly weighed.
5. Close the vial (cap + septum).
6. Shake for 30 seconds on the agitator (e.g. vortex).

Note: Do not forget to write down the weights for the concentration calculation.
SM2 (20 mg/kg):

1. Place a vial (labelled as SM2) of 20 mL on the analytical balance and tare it.
2. Weigh 5.000 ± 0.001 g of refined olive oil.
3. Put approx. 1.000 ± 0.001 g of SM1, exactly weighed, in the vial.
4. Add refined olive oil up to reach approx. 10.000 ± 0.001 g, exactly weighed.
5. Close the vial (cap + septum). Shake for 30 seconds on the agitator (e.g. vortex).

Note: Do not forget to write down the weights for the concentration calculation.
SM3 (2 mg/kg):

1. Place a vial (labelled as SM3) of 20 mL on the analytical balance and tare it.
2. Weigh 5.000 ± 0.001 g of refined olive oil.
3. Put approx. 1.000 ± 0.001 g of SM2, exactly weighed, in the vial.
4. Add refined olive oil up to reach approx. 10.000 ± 0.001 g, exactly weighed.
5. Close the vial (cap + septum).
6. Shake for 30 seconds on the agitator (e.g. vortex).

Note: Do not forget to write down the weights for the concentration calculation.

Preparation of dilutions for building the calibration curves

For the low concentration mixture (SM A), it is necessary to prepare the following 12 dilutions starting from SM1, SM2 or SM3 (see the procedure and Table 2): 0.05, 0.10, 0.15, 0.20, 0.25, 0.50, 1.00, 1.50, 2.00, 2.50, 5.00, and 10.00 mg/kg. For the high concentration mixture (SM B), it is necessary to prepare 12 dilutions starting from SM1, SM2 or SM3 (see the procedure and Table 2): 0.20, 0.25, 0.50, 1.00, 1.50, 2.00, 2.50, 5.00, 10.00, 15.00, 20.00, and 25.00 mg/kg.

1. Place each vial (20 mL) on the analytical balance and tare it.
2. Weigh 1.000 ± 0.001 g of refined olive oil.
3. Add 0.100 ± 0.001 g of IS dilution[1].
4. Add the appropriate weight (Column 5 of Table 2) of SMx (Column 1 of Table 2) in the vial. The approximate obtained concentration is indicated in the column 6 of Table 2, although the exact concentration needs to be calculated.
5. Add refined olive oil up to reach approx. 2.000 ± 0.001 g, exactly weighed (Column 3 of Table 2 shows the amount of refined olive oil approximately to be added).
6. Close the vial quickly with cap and septum.
7. Shake the vials gently and softly (never spread the oil through the vial walls or the septum).

Note: Do not forget to write down the weights for the concentration calculation; [1], Preparation of IS dilution explained in the section "Standard solution and sample preparation (SPME-GC-MS and SPME-GC-FID)", IS in refined olive oil at a concentration of about 50 mg/kg. Fig. 1 shows the chromatograms of SM A and SM B, added with IS and both SM at 10.00 mg/kg, analysed by SPME-GC-MS.

Sequence of the GCanalysis for building the calibration curves

In the sequence of the chromatographic analyses, set the higher concentrations at the end of the sequence, and analyse the blank samples (empty vials) and blank refined oil (oil without compounds added). The following example is for the low concentration mixture (SM A):

1. Blank (empty vial).
2. Blank of the matrix (refined olive oil: 2.0 g).
3. Blank of the matrix + IS (refined olive oil: 1.9 g + IS: 0.1 g).
4. Blank (empty vial).
5. 0.05 mg/kg vial.
6. 0.10 mg/kg vial.
7. 0.15 mg/kg vial.
8. 0.20 mg/kg vial.
9. Blank (empty vial).
10. 0.25 mg/kg vial.

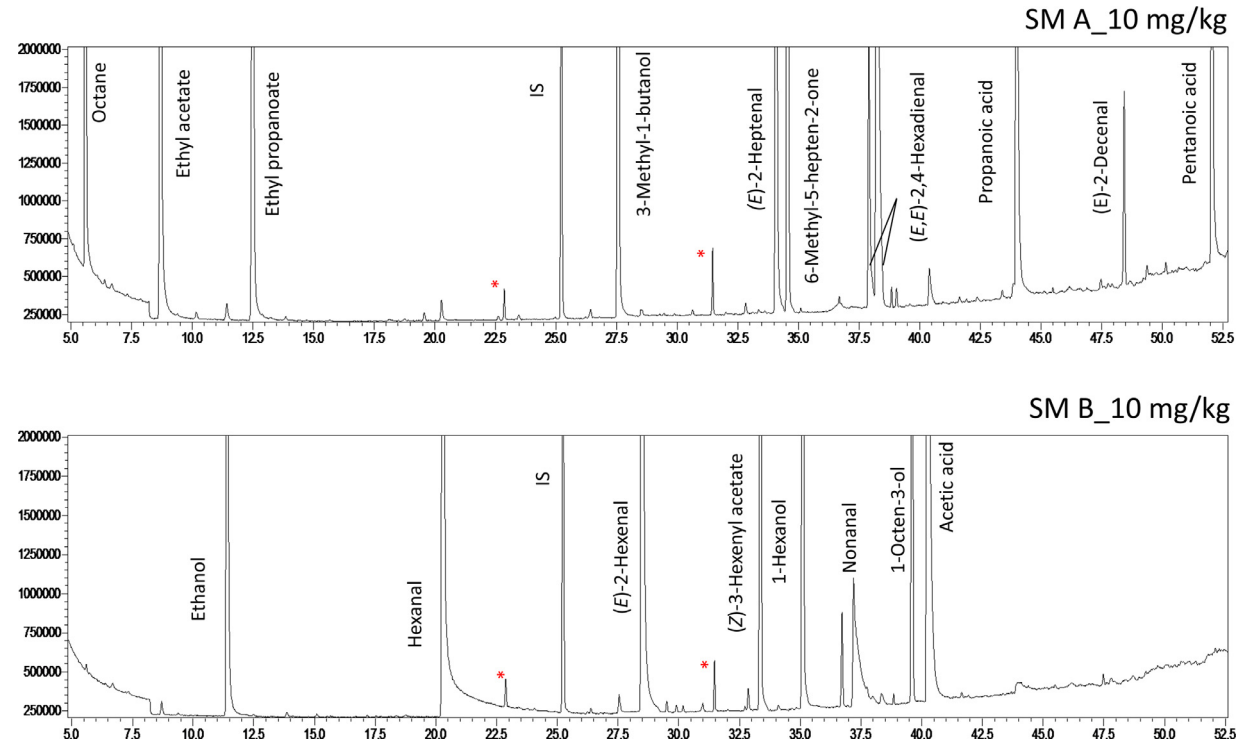

Fig. 1. Chromatograms of SM A and SM B, added with IS, at 10 mg/kg (SPME-GC-MS). *Compounds deriving from the SPME Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) fiber.

11. 0.50 mg/kg vial.
12. 1.00 mg/kg vial.
13. 1.50 mg/kg vial.
14. Blank (empty vial).
15. 2.00 mg/kg vial.
16. 2.50 mg/kg vial.
17. 5.00 mg/kg vial.
18. 10.00 mg/kg vial.
19. Blank (empty vial).

Standard solution and sample preparation (SPME-GC-MS and SPME-GC-FID)

Caution: work at controlled room temperature (T = 20-25°C) due to the volatility of the standards. Samples must be at room temperature before sample preparation.

○ **Preparation of the stock standard solution**

Weigh 15.000 ± 0.001 g of refined olive oil in a vial, then add 0.100 ± 0.001 g, exactly weighed, of 4-methyl-2-pentanol and add refined olive oil up to reach 20.000 ± 0.001 g, exactly weighed (approximate concentration of 5000 mg/kg). Take note of exact weights (± 0.001 g) for calculation of concentration. A rapid preparation of the stock standard solution is advisable to avoid evaporation of internal standard.

○ **Preparation of standard solution**

Weigh 5.000 ± 0.001 g of refined olive oil in a vial, then add 0.100 ± 0.001 g of the stock standard solution of 4-methyl-2-pentanol, exactly weighed, and add refined olive oil up to reach 10.000 ± 0.001 g, exactly weighed (approximate concentration of 50 mg/kg). Take note of exact weights (± 0.001 g) for calculation of concentration. A rapid preparation is advisable to avoid evaporation of internal standard.

○ **Sample preparation**

Weight exactly 1.900± 0.001 g of virgin olive oil (sample) in a 20 mL glass vial and add 0.100 ± 0.001 g of 4-methyl-2-pentanol standard solution (5.1.2) as internal standard (IS approximate concentration = 2.5 mg/kg). Take note of exact weights (± 0.001 g) for

Table 1
Characteristic relative m/z of the 18 selected compounds used to build the calibration curves.

Compound	Characteristic m/z
Octane	85
Ethyl acetate	43
Ethanol	31, 45
Ethyl propanoate	57
Hexanal	44
3-Methyl-1-butanol	55, 70
(*E*)-2-Hexenal	69, 83
(*Z*)-3-Hexenyl acetate	67, 82
(*E*)-2-Heptenal	83
6-Methyl-5-hepten-2-one	108
1-Hexanol	56
Nonanal	98
(*E*,*E*)-2,4-Hexadienal	81
1-Octen-3-ol	57
Acetic acid	60
Propanoic acid	74
(*E*)-2-Decenal	70
Pentanoic acid	60, 73

calculation of concentration. Hermetically close the vial with polytetrafluoroethylene septum (PTFE). Shake the vial manually, but very gently and softly (never spread the oil through the vial walls or the septum). Leave for 10 min at 40˚C under agitation (250 rpm in continuous is suggested) to allow for the equilibration of the volatiles in the headspace. After the equilibration time, the septum covering each vial is pierced with a SPME needle and the fiber exposed to the headspace for 40 min.

- **Gas chromatography analysis (choose one of two options):**
- **With FID:** The volatiles adsorbed by the fiber are thermally desorbed in the hot injection port of a GC for 5 min at 250˚C with the purge valve off (splitless mode) and deposited onto a capillary column of a GC with a FID at 260ºC. The carrier gas is helium (or hydrogen if the equipment and installation are adapted and allow for safety procedure), at a flow rate of 1.5 mL/min (this is a suggestion, the flow rate must be adjusted to optimize the separation of peaks depending on the use of helium or hydrogen as carrier gas). The oven temperature is held at 40˚C for 10 min and then programmed to increase by 3˚C/min to a final temperature of 200˚C. A cleaning step can be added (20˚C/min to 250˚C and hold the temperature for 5 min).
- **With MS:** The volatiles adsorbed by the fiber are thermally desorbed in the hot injection port of a GC for 5 min at 250˚C with the purge valve off (splitless mode) and deposit onto a capillary column of a GC with a mass spectrometry (MS) detector. The transfer line temperature is set at 260 ºC. The temperature of the ion source is set according to the technical specifications of the instrument. The carrier gas is helium (or hydrogen if the equipment and installation are adapted and allow for safety procedure), at a flow rate of 1.5 mL/min (this is a suggestion, the flow rate must be adjusted to optimize the separation of peaks depending on the use of helium or hydrogen as carrier gas). The oven temperature is held at 40˚C for 10 min and then programmed to increase by 3˚C/min to a final temperature of 200˚C. A cleaning step can be added (20˚C/min to 250˚C and hold the temperature for 5 min).
- **Peak identification**

The identification of the volatile compounds is carried out by MS (Table 1) if available, checked with standards and by comparison with the linear retention indexes (LRI).

○ **Calculation of linear retention indexes**

To identify with greater certainty each extracted compound, the LRI are determined. The mixture of *n*-alkanes is injected in the GC; the retention times of the alkanes are used in the following equation, obtaining the LRI of each analyte extracted.

$$LRI = 100 \times z + 100 \times \left(\frac{(RT_{analyte} - RT_z)}{(RT_{z+1} - RT_z)} \right)$$

z is the number of carbon atoms of the alkane that elutes before the molecule; the $RT_{analyte}$, the RT_z and the RT_{z+1} are the retention times of the analyte of interest, of the alkane that elutes before it and the one that elutes after it.

- **Quantitative analysis**

The quantification of selected volatile compounds is carried out by calibration based on the internal standard (IS) and the external calibration curve. The IS is also added in the sample because it is necessary for evaluating the goodness of the fiber sampling and the whole analytical procedure. Thus, an abrupt change in the peak area assigned to IS, or in its usual RT, would mean an issue that needs to be checked.

Table 2
Weights of the refined oil, internal standard (IS) and standard mixtures (SMx) for preparing the calibration curves.

SMx	Conc.[1] (mg/kg)	Weight of refined oil (g)	Weight of IS dilution[2] (g) (2.5 mg/kg)	Weight of SMx (g)	Final conc. of volatile (mg/kg)
SM3	2 mg/kg	0.85	0.1	0.05	0.05
		0.80		0.10	0.10
		0.75		0.15	0.15
		0.70		0.20	0.20
		0.65		0.25	0.25
SM2	20 mg/kg	0.85		0.05	0.5
		0.80		0.10	1.00
		0.75		0.15	1.50
		0.70		0.20	2.00
		0.65		0.25	2.50
SM1	200 mg/kg	0.85		0.05	5.00
		0.80		0.10	10.00
		0.75		0.15	15.00
		0.70		0.20	20.00
		0.65		0.25	25.00

Note:
[1] Conc., concentration;
[2] Preparation of IS dilution explained in the joint protocols for GC-FID/MS, IS in refined olive oil at a concentration of approximately 50 mg/kg (final concentration about 2.5 mg/kg).

External calibration curve ($A_{Analyte}/A_{IS}$ *vs.* $C_{Analyte}$):
$A_{Analyte}/A_{IS} = m \cdot C_{Analyte}$, $(Y = m \cdot x)$
$\mathbf{C_{Analyte} = (A_{Analyte}/A_{IS})/m}$
Where:
$C_{Analyte}$ is the concentration of the analyte.
$A_{Analyte}$ is the area corresponding to the analyte.
A_{IS} is the area corresponding to the IS; m, is the slope of the calibration curve (built for the selected analyte).

Note: Analysis should be carried out in triplicate. The means will be calculated from the areas of those triplicates.

Note: An abrupt reduction of the IS area can be observed in the case of lampante olive oils with a high median level of main defect.

Data and results about the validation of the method is reported in Aparicio Ruiz et al., 2022 [1] and Casadei et al., 2021 [2] (see References).

Ethics statements

Authors declare to comply with the Journal ethical guidelines.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Ramón Aparicio-Ruiz: Conceptualization, Methodology, Formal analysis, Data curation, Writing – original draft, Writing – review & editing. **Enrico Casadei:** Conceptualization, Methodology, Validation, Formal analysis, Writing – original draft, Writing – review & editing. **Clemente Ortiz-Romero:** Formal analysis, Methodology, Validation, Writing – review & editing, Data curation. **Diego L. García-González:** Conceptualization, Methodology, Validation, Data curation, Writing – original draft, Writing – review & editing. **Maurizio Servili:** Conceptualization, Methodology, Validation, Data curation, Writing – review & editing, Supervision. **Roberto Selvaggini:** Conceptualization, Methodology, Formal analysis, Data curation, Writing – review & editing. **Florence Lacoste:** Conceptualization, Methodology, Validation, Data curation, Writing – review & editing, Supervision. **Julien Escobessa:** Methodology, Formal analysis, Data curation, Writing – review & editing. **Stefania Vichi:** Formal analysis, Methodology, Validation, Data curation, Writing – review & editing, Supervision. **Beatriz Quintanilla-Casas:** Formal analysis, Methodology, Data curation, Writing – review & editing. **Alba Tres:** Formal analysis, Methodology, Validation, Data curation, Writing – review & editing, Supervision. **Pierre-Alain Golay:** Conceptualization, Methodology, Formal analysis, Data curation, Writing – review & editing. **Paolo Lucci:** Conceptualization, Methodology, Validation, Data curation, Writing – review & editing, Supervision. **Erica Moret:** Conceptualization, Methodology, Formal analysis, Data curation, Writing – review & editing. **Enrico Valli:** Conceptualization, Methodology, Validation, Data curation, Writing – original draft, Writing – review & editing, Supervision. **Alessandra Bendini:** Conceptualization, Methodology, Validation,

Data curation, Writing – review & editing, Supervision. **Tullia Gallina Toschi:** Conceptualization, Methodology, Validation, Project administration, Writing – review & editing, Supervision.

Data availability

Data will be made available on request.

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