AOAC SMPR® 2020.010

Standard Method Performance Requirements (SMPRs®) for Targeted Testing (TT) of Other Vegetable Oils and Low-Quality Olive Oils as Adulterants for Evaluation of Extra Virgin Olive Oil (EVOO)

Intended Use:

AOAC SMPRs® describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, a single-laboratory validation (SLV), or a multi-site collaborative study. SMPRs are written and adopted by AOAC using the consensus of stakeholders representing industry, government, academic, and/or research institutions. AOAC SMPRs are used by AOAC expert review panels (ERPs) in their evaluation of validation study data for method being considered for *Performance Tested Methods*℠ or AOAC *Official Methods of Analysis*℠ and can be used as acceptance criteria for verification at user laboratories.

1 Applicability

Targeted testing (TT) methods to monitor extra virgin olive oil (EVOO) for the detected presence of economically motivated adulterants (EMAs), such as other vegetable oils and low-quality olive oils.

2 Analytical Technique

A targeted method to be used to detect, identify, and quantify EVOO for possible EMAs, such as other vegetable oils and low-quality olive oils.

Any method capable of detecting and identifying the presence of the defined adulterants and quantifying the amount (proportion/concentration) present in the food item will be considered.

The scope of the TT method will be defined by the authentic samples and/or reference standard material (if available) used in validating the method.

3 Definitions

Authentic EVOO.—Type(s) of EVOO used to validate the method. The method's scope of authenticity is defined by the EVOO(s) used in validating the methods.

Authentic samples.—Samples representative of the genuine commodity. These samples should represent the food's or ingredient's variability seen naturally in the commodity. Type(s) of EVOO used to validate the method will also be used to define the method's scope of authenticity the authentic samples and/or reference standard materials used to validate the method will be used to properly define the TT method testing scope.

Economically motivated adulteration (EMA).—Fraudulent addition of nonauthentic substances or removal or replacement of authentic substances without the purchaser's knowledge for economic gain of the seller.

False origin.—EVOO containing mislabeled geographic and botanical sources.

Multilaboratory validation (MLV).—Demonstration between laboratories using adulterated samples created by a third-party group and supplied blindly to the participating laboratories according to guidelines described in the AOAC Appendix D: "Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Study" be considered for classification as AOAC Final Action Method; Horwitz, W. (1995) "Protocol for the design, conduct, and interpretation of method performance studies," *Pure Appl. Chem.* **67**, 331–343; CAC/GL 27-1997: "Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control of Food"; CAC/GL 37-2001: "Harmonized IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement;" and "Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement"

The predicted ($PRSD_R$) of reproducibility is calculated from the Horwitz equation:

$$PRSD_R = 2C^{-0.15}$$

where C is expressed as a mass fraction.

For quantitative methods undergoing MLV, 10–12 laboratories must be recruited to provide at least eight valid data sets; two blind duplicate replicates at five concentration levels for each analyte/matrix combination to each collaborator.

$$\text{HorRat (repeatability, r)} = RSD_r/PRSD_R$$

$$\text{HorRat (reproducibility, R)} = RSD_R/PRSD_R$$

For interlaboratory studies: acceptable HorRat (R) of 1 with limits of acceptability of 0.5–2; for within-laboratory studies: acceptable HorRat (r) of 0.3–1.3.

Single-laboratory validation (SLV).—Demonstration by one laboratory of method performance on samples described according to internationally accepted validation guidelines contained in guidance documents, such as AOAC'S Appendix D: "Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Study;" ISO/IEC 17025:2017 Document: *"*General requirements for the competence of testing and calibration laboratories;" Codex Alimentarius Committee Guidance Document CAC/GL 71-2009: "Guidelines for the Design and Implementation of National Regulatory Food Safety Assurance Program Associated with the Use of Veterinary Drugs in Food Producing Animals*"* (Adopted 2009. Revision 2012, 2014); Harmonized ISO/IUPAC/AOAC Guidelines for Single-Laboratory Validation of Methods of Analysis CAC/GL-49-2003: "Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis;" CAC/GL 56-2005: "Guidelines on the Use of Mass Spectrometry (MS) for Identification, Confirmation, and Quantitative Analysis of Residues"; "Establishing the Fitness for Purpose of Mass Spectrometric Methods;" and SANTE/12682/2019: "Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed" (A Guidance document on analytical quality control and method validation procedures for pesticide residues analysis in food and feed).

Once the method has been demonstrated to meet the minimum requirements for validation and fit for purpose criteria, the method can be reviewed and considered by AOAC for classification as First Action *Official Method of Analysis*℠.

4 Method Performance Requirements

See Tables 1 and 2.

Table 1. Method performance requirements for other vegetable oils in EVOO

Analytical parameter	Acceptance criteria
Analytical range, %	10–50
LOQ, %	≤10
Recovery, %	80–120
Accuracy, %	±20
Precision (repeatability) RSD_r	10
Precision (reproducibility) RSD_R	18

Table 2. Method performance requirements for low-quality olive oils in EVOO

Analytical parameter	Acceptance criteria
Analytical range, %	10–50
LOQ, %	≤10
Recovery, %	80–120
Accuracy, %	±20
Precision (repeatability) RSD_r	10
Precision (reproducibility) RSD_R	18

5 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blanks and appropriate check standards.

6 Reference Materials

A detailed description of the process used to obtain and evaluate authentic/reference standard materials (sources), and of the test protocol used for validating the method must be provided.

7 Validation Guidance

(a) Data demonstrating method performance is required.

(b) *Samples*.—Complete documentation for the collection and use of authentic samples must be supplied by the method authors. The scope of "authentic" samples used to validate the method must be applicable to the defined scope of the TT method. Expansion of the scope is possible with the inclusion of additional authentic samples and abbreviated validation using the protocol listed in this SMPR.

(c) For SLV studies, the method will be evaluated using prescribed adulterated materials as shown in Tables 1 and 2. Methods approved at this level will proceed to a second level of evaluation (MLV), where blinded samples containing unknown adulterants will be sent to participating laboratories.

(d) Statistical analysis of interlaboratory studies. Sample size needed to meet performance requirement on proportion.

8 Maximum Time-to-Results

None

References

CAC/GL 27-1997: "Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control of Food" http://www.fao.org/input/download/standards/355/CXG_027e.pdf

CAC/GL 37-2001: "Harmonized IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement" http://www.fao.org/input/download/standards/376/CXG_037e.pdf

CAC/GL-49-2003: "Harmonized ISO/IUPAC/AOAC Guidelines for Single-Laboratory Validation of Methods of Analysis" http://www.fao.org/fao-who-codexalimentarius/codex-texts/guidelines/en/

CAC/GL 56-2005–CXG 56: "Guidelines on the Use of Mass Spectrometry (MS) for Identification, Confirmation, and Quantitative Analysis of Residues" http://www.fao.org/fao-who-codexalimentarius/codex-texts/guidelines/en/

Codex Alimentarius Committee Guidance Document CAC/GL 71-2009: "Guidelines for the Design and Implementation of National Regulatory Food Safety Assurance Program Associated with the Use of Veterinary Drugs in Food Producing Animals" (Adopted 2009. Revision 2012, 2014) http://www.fao.org/fao-who-codexalimentarius/codex-texts/guidelines/en/

ISO/IEC 17025:2017 Guideline Document: "General requirements for the competence of testing and calibration laboratories" (Codex Alimentarius Committee Guidance Document) https://www.iso.org/obp/ui/#iso:std:iso-iec:17025:en

SANTE/12682/2019: "Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed" (A Guidance document on analytical quality control and method validation procedures for pesticide residues analysis in food and feed) https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkdoc_2019-12682.pdf

Appendix D: "Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Study" (1989) *J. Assoc. Off. Anal. Chem.* **72**, 694–704

Appendix F: "Guidelines for *Standard Method Performance Requirements"* (2016) *Official Methods of Analysis of AOAC INTERNATIONAL*, AOAC INTERNATIONAL, Rockville, MD, USA, www.eoma.aoac.org

Thompson, M., Ellison, S.L.R., & Wood, R. (2002) "Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis," *Pure Appl. Chem.* **74**, 835–855, http://publications.iupac.org/pac/2002/pdf/7405x0835.pdf

Bethem, R., Boison, J.O., Gale, J., Heller, D., Lehotay, S., Loo, J., Musser, S., Price, P., & Stein, S. (2003) "Establishing the Fitness for Purpose of Mass Spectrometric Methods," *J. Am. Soc. Mass Spectrom*. **14**, 528–541

Horwitz, W. (1995) "Protocol for the Design, Conduct, and Interpretation of Method Performance Studies," *Pure Appl. Chem.* **67**, 331–343

Thompson, M., Ellison, S., Fajgelj, A., Willetts, P., & Wood, R. (1999) "Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement," *Pure Appl. Chem.* **71**, 337–348

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