AOAC SMPR 2020.XXX; Draft AOAC 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); Version 3; February 13, 2020

Intended Use

AOACI *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, or a multi-site collaborative study.

SMPRs are written and adopted by AOACI using the consensus of stakeholders representing the industry, government, and academic and/or research institutions. AOACI SMPRs are used by AOACI expert review panels (ERPs) in their evaluation of validation study data for method being considered for *Performance Tested Methods*^{SM} or AOACI *Official Methods of Analysis*^{SM} and can be used as acceptance criteria for verification at user laboratories.

1. Applicability

This document contains assessment parameters on the performance of 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, 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

Applicability Statement — a general statement about the intended purpose and scope of the method entailing key aspects of expected achievements for the specific situation and circumstances. Key points to cover are the intended matrix, the purpose, and an indication of sensitivity, selectivity, enforcement and action levels where available.

Authentic Samples — Samples representative of the genuine commodity. Ideally these samples should represent the food's or ingredient's variability seen naturally in the commodity. 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 —The fraudulent addition of non-authentic substances or removal or replacement of authentic substances without the purchaser's knowledge for economic gain of the seller.

False Origin — Extra Virgin Olive Oil containing mislabelled geographic and botanical sources.

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

Single Laboratory Validation (SLV) — Demonstration by one laboratory of method performance on samples described in Table 1.

Multi-laboratory Validation (MLV) — Demonstration between laboratories using adulterated samples created by a third-party group and supplied blindly to the participating laboratories.

4. Method Performance Requirements

(Table 1: Method Performance Requirements for Other Vegetable Oils in EVOO)

Analytical Parameter	Acceptance Criteria
Analytical Range (%)	10 – 50% OF EVOO
LOQ	≥ 10 %
Recovery	80 – 120 %
Accuracy	± 20%

NOTES:

At least 2 recent publications report fortifying EVOO with between 10 and 50 % of other vegetable oils and demonstrating by Raman, UV-VIS and IMS Technology that it is possible to detect adulteration and quantify them to well characterized % of adulteration. For soybean oil adulteration, it was demonstrated through ESI-MS that adulterations over the 1% to 90% range were easily detectable and quantifiable to as low as 1% fortification

In one study for soybean adulteration, soybean edible oil in different concentrations (10, 50, 100, 150, 200, 250 and 300 g/kg) were added to EVOO corresponding to 1, 5, 10, 15, 20, 25 and 30 % adulteration, resp.

Detection of adulteration (sunflower oil) in extra virgin olive oils by using UV-IMS and chemometric analysis. Food Control, Volume 85, March 2018, Pages 292-299. Rocío Garrido-Delgado, Ma. Eugenia Muñoz-Pérez, Lourdes Arce. https://doi.org/10.1016/j.foodcont.2017.10.012

Continuous statistical modelling for rapid detection of adulteration of extra virgin olive oil (hazelnut oil) using mid infrared and Raman spectroscopic data. Food Chemistry, Volume 217, 15 February 2017, Pages 735-742. Konstantia Georgouli, Jesus Martinez Del Rincon, Anastasios Koidis. https://doi.org/10.1016/j.foodchem.2016.09.011

Rapid methodology via mass spectrometry to quantify addition of soybean oil in extra virgin olive oil: A comparison with traditional methods adopted by food industry to identify fraud
Food Research International, Volume 102, December 2017,
Pages 43-50. Roberta da Silveira, Julianna Matias Vágula, Ingrid de Lima Figueiredo, Thiago Claus, … Jesui Vergilio Visentainer https://doi.org/10.1016/j.foodres.2017.09.076

Comparative chemometric analysis of fluorescence and near infrared spectroscopies for authenticity confirmation and geographical origin of Argentinean extra virgin olive oils. Food Control, Volume 96, February 2019, Pages 22-28. Ana M. Jiménez-Carvelo, Valeria A. Lozano, Alejandro C. Olivieri. https://doi.org/10.1016/j.foodcont.2018.08.024

Multivariate modeling for detecting adulteration of extra virgin olive oil with soybean oil using fluorescence and UV–Vis spectroscopies: A preliminary approach. LWT - Food Science and Technology, Volume 85, Part A, November 2017, Pages 9-15. Karla Danielle Tavares Melo Milanez, Thiago César Araújo Nóbrega, Danielle Silva Nascimento, Matías Insausti, … Márcio José Coelho Pontes https://doi.org/10.1016/j.lwt.2017.06.060

Detection of olive oil adulteration with waste cooking oil via Raman spectroscopy combined with iPLS and SiPLS. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, Volume 189, 15 January 2018, Pages 37-43. Yuanpeng Li, Tao Fang, Siqi Zhu, Furong Huang, … Yong Wang. https://doi.org/10.1016/j.saa.2017.06.049

(Table 2: Method Performance Requirements for Low-Quality Olive Oils in EVOO)

Analytical Parameter	Acceptance Criteria
Analytical Range (%)	10 – 50 % of EVOO
LOQ	≥ 10%
Recovery	80 – 120 %
Accuracy	± 20%

NOTES:

Recommend we use the same analytical ranges in terms of % EVOO for low quality olive oils
A comparative study of mid-infrared, UV–Visible and fluorescence spectroscopy in combination with chemometrics for the detection of adulteration of fresh olive oils with old olive oils. Food Control, Volume 105, November 2019, Pages 209-218. Oguz Uncu, Banu Ozen https://doi.org/10.1016/j.foodcont.2019.06.013

Chaotic parameters from fluorescence spectra to resolve fraudulent mixtures of fresh and expired protected designation of origin extra virgin olive oils. Talanta, Volume 195, 1 April 2019, Pages 1-7
Miguel Lastra-Mejías, Regina Aroca-Santos, Manuel Izquierdo, John C. Cancilla, … José S. Torrecilla.
https://doi.org/10.1016/j.talanta.2018.10.102

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 single lab validation studies, the method will be evaluated using prescribed adulterated materials as shown in Table 1. Methods approved at this level will proceed to a second level of evaluation (multi-laboratory) 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.