AOAC SMPR 2021.XXX; Draft AOAC Standard Method Performance Requirements (SMPRs) for Non-Targeted Testing (NTT) of Ingredients for Food Authenticity Methods Evaluation of Vanilla Powder and Extracts; 10/19/21

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, or a multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholder panels composed of representatives from the industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels in their evaluation of validation study data for method being considered for *Performance Tested MethodsSM* or AOAC *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 Non-Targeted Testing methods to monitor vanilla powder and extract for the probable presence of Economically Motivated Adulterants (EMA).

This SMPR was designed to evaluate Non-Targeted Testing (NTT) methods developed to assess potential economic adulteration in vanilla powder and extract. The SMPR was purposely designed with general descriptions to be applicable to a broad range of innovative analytical platforms and chemometric approaches. Binary analytical results of "Authentic" or "Not Authentic" on defined samples from the performance of the method will be used to perform the evaluations by the Expert Review Panel.

Complete documentation of the collection and use of authentic samples is to be supplied by the method authors. The scope of authentic samples will be the applicable scope of the NTT method and expansion of the scope is possible with the inclusion of additional authentic samples into the baseline calibration, and validation using the protocol listed in this SMPR.

2. Analytical Technique

A non-targeted method to be used to evaluate foods and ingredients for possible EMAs. Any method generating a baseline fingerprint of the authentic material and comparing test sample fingerprints to assess differences will be considered. The final binary result identifies test samples as either authentic or potentially adulterated. This method demonstrates reliability using the requirements listed in this SMPR.

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 (i.e., multi-laboratory validation) where blinded samples containing unknown adulterants will be sent to laboratories participating in the ensuing multi-laboratory validation.

The scope of the NTT method will be defined by the authentic samples used in generating the baseline fingerprint.

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 scope, the purpose, and an indication of sensitivity, specificity, and significance (USP Appendix XVIII).

Authentic Samples – Samples representative of the genuine commodity. These samples should represent the food's or ingredient's variability seen naturally in the commodity. The authentic samples used to generate the product fingerprint will be used to properly define the NTT method testing scope.

Baseline Fingerprint – A food-specific model created by software evaluation of collected analytical data.

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 (USP Appendix XVIII).

Single Laboratory Validation – Demonstration by one laboratory of method performance on samples described in Table 1.

Multilaboratory Validation – Demonstration between laboratories using adulterated samples created by a third-party group and supplied blindly to the participating laboratories.

Vanilla – For this SMPR, "vanilla" is defined as the powder and extract forms.

4. Method Performance Requirements

(Table 1: Method Performance Requirements for Vanilla Non-Targeted Testing)

Test	Adulterant	% Adulterant in Test Materials	Number of Samples to be Tested[1]	Number of Test Results Qualified as Adulterated
Baseline	None (Authentic Vanilla)	0%	Establish Baseline Fingerprint[2]	
Validation using Authentic Samples[3]	None	0%	30	0
Validation[4]	Tonka Bean Extract	10%	30	30
Validation[4]	Coumarin	500 ppm	30	30
Validation[4]	Artificial Vanillin	1000 ppm	30	30
Validation[4]	Ethyl Vanillin	1000 ppm	30	30
Validation[4]	Guaiacol	1000 ppm	30	30
Validation[4]	Vanillic Acid[5]	1000 ppm	30	30

1. Multiple samples from the same batch of adulterated material can be used for method evaluation. Each sample must be analyzed separately.
2. Full details on protocol used to establish an authentic fingerprint must be supplied.
3. Samples used for this step must be independent than those used to create the baseline and must cover the entire scope of the method.
4. Method validation using adulterated samples shall cover the entire scope used in creating the baseline fingerprint.
5. Vanillic acid as an adulterant is not applicable to vanilla extracts that contain ~0.2% vanillic acid.

5. System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blanks, and appropriate check standards.

6. Reference Materials

Detailed protocols used to identify reference materials as authentic and to create adulterated samples must be supplied.

7. Validation Guidance

a) Data demonstrating method performance is required.

b) Available guidance documents:

a. AOAC INTERNATIONAL Guidelines for Validation of Botanical Identification Methods, Journal of AOAC International Vol. 95, No. 1, 2012

b. Statistical analysis of interlaboratory studies. LII. Sample size needed to meet performance requirement on proportion. http://lcfltd.com/AOAC/tr347-SAIS-LII-sample-size-needed-for-PR-for-proportion.pdf

c. United States Pharmacopeia (USP). Appendix XVIII: Guidance on Developing and Validating Non-targeted Methods for Adulteration Detection. Food Chemicals Codex, 3rd supplement to 11th ed.; USP: Rockville, MD, 2019

8. Maximum Time-to-Results

None.