**AOAC SMPR® 2021.013**

## *Standard Method Performance Requirements* (SMPRs®) for Nontargeted Testing (NTT) of Ingredients for Food Authenticity/Fraud Evaluation of Vanilla Powder and Extracts

Intended Use: Surveillance and Monitoring by Trained Analysts

### *1 Purpose*

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, single-laboratory validation (SLV), or multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholders composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC method review experts, including expert review panels (ERPs), in their evaluation of validation study data for methods being considered for AOAC *Performance Tested Methods*℠*, Reviewed and Recognized*℠, or AOAC *Official Methods of Analysis*℠, and can be used as acceptance criteria for verification at user laboratories.

### *2 Applicability*

The document contains assessment parameters on the performance of nontargeted testing (NTT) methods to monitor vanilla powder and extract for the probable presence of economically motivated adulterants (EMA).

The SMPR was designed to evaluate 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 an ERP.

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 the SMPR.

### *3 Analytical Technique*

NTT 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. The method demonstrates reliability using the requirements listed in the SMPR.

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

### *4 Definitions*

*Applicability statement.*—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 intended matrix scope, purpose, and 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.*—Food-specific model created by software evaluation of collected analytical data.

*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 (USP Appendix XVIII).

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

*Single-laboratory validation (SLV).*—Demonstration by one laboratory of method performance on samples described in Table 1.

*Vanilla.*—For this SMPR, defined as the powder and extract forms.

### *5 Method Performance Requirements*

*See* Table 1.

### *6 System Suitability Tests and/or Analytical Quality Control*

Suitable methods will include blanks and appropriate check standards.

### *7 Reference Materials*

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

### *8 Validation Guidance*

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

**(b)** For SLV 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., MLV), where blinded samples containing unknown adulterants will be sent to laboratories participating in the ensuing MLV.

**(c)** Data demonstrating method performance is required.

**(d)** Available guidance documents:

(*1*) *AOAC INTERNATIONAL Guidelines for Validation of Botanical Identification Methods* (2012) *J. AOAC Int*. **95**, 268–272(2012); DOI: 10.5740/jaoacint.11-447

(*2*) *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

(*3*) U.S. Pharmacopeia (USP) (2019) *Appendix XVIII: Guidance on Developing and Validating Nontargeted Methods for Adulteration Detection. Food Chemicals Codex*, 3rd Supplement to 11th Ed., USP, Rockville, MD, USA

### *9 Maximum Time-to-Results*

None.

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**Table 1. Method performance requirements for vanilla nontargeted testing**

| Test | Adulterant | Adulterant in test materials, % | No. of samples to be tested[a] | No. of test results qualified as adulterated |
|---|---|---|---|---|
| Baseline | None (authentic vanilla) | 0 | Establish baseline fingerprint[b] | |
| Validation using authentic samples[c] | None | 0 | 30 | 0 |
| Validation[d] | Tonka bean extract | 10 | 30 | 30 |
| Validation[d] | Coumarin | 500 ppm | 30 | 30 |
| Validation[d] | Artificial vanillin | 1000 ppm | 30 | 30 |
| Validation[d] | Ethyl vanillin | 1000 ppm | 30 | 30 |
| Validation[d] | Guaiacol | 1000 ppm | 30 | 30 |
| Validation[d] | Vanillic acid[e] | 1000 ppm | 30 | 30 |

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