

Standard Method Performance Requirements (SMPRs®) for Nontargeted Testing (NTT) of Ingredients for Food Authenticity/Fraud Evaluation of Honey

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 stakeholders composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels (ERPs) in their evaluation of validation study data for methods being considered for *Performance Tested Methods*SM 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 nontargeted testing (NTT) methods to monitor honey for the probable presence of economically motivated adulterants (EMA).

The SMPR was designed to evaluate NTT methods developed to assess potential economic adulteration in defined commodities. 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 performance of the method will be used to perform evaluations by the ERP.

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

2 Analytical Technique

A nontargeted 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 requirements in this SMPR.

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

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

3 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 an indication of sensitivity, specificity, and significance (USP Appendix XVIII).

Authentic honey.—Type(s) of honey used to generate baseline fingerprint. The method’s scope of authenticity is defined by the honey(s) used in generating the baseline fingerprint.

Authentic samples.—Samples representative of the genuine commodity. These samples should represent the food’s or ingredient’s variability seen naturally in the commodity. Authentic samples used to generate the product baseline fingerprint will be used to properly define the NTT method testing scope. Documentation for authentic honey samples must contain any feeding protocols used in the production of the honey to properly authenticate the material.

Authentic sample documentation will be reviewed by the ERP to verify method scope. Suggested parameters include country of origin, feed, and applicable analytical tests such as sugar profile, C3/C4 sugars, and water. Additional information can be included.

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).

False origin.—Honeys 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 participating laboratories.

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

Sugars.—Intentionally-added sugars to be included in a method’s evaluation include high-fructose corn syrup, sucrose, fructose, glucose, beet sugar, cane sugar, and invert sugar.

4 Method Performance Requirements

See Table 1.

Samples used for this step must be independent than those used to create the baseline and must cover the entire scope of the method.

5 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include authentic and adulterated material check standards.

6 Reference Materials

Detailed description of the process used to obtain and evaluate authentic samples and of the test protocol establishing the baseline fingerprint must be supplied.

7 Validation Guidance

(a) Data demonstrating method performance are required.

(b) Available guidance documents:

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

(2) Statistical analysis of interlaboratory studies. LII. Sample size needed to meet performance requirement on proportion. <http://>

Table 1. Method performance requirements

Test	Adulterant	Adulterant in test materials, %	No. samples to be tested ^a	No. test results qualified as adulterated
Baseline	None (authentic honey)	0	Establish baseline fingerprint ^b	
Validation using authentic samples ^c	None	0	30	0
Validation ^d	Sugars	5	30	30
Validation ^d	Molasses	5	30	30

^a Multiple samples from the same batch of adulterated material can be used for method evaluation.

^b Full details on protocol used to establish an authentic fingerprint must be supplied.

^c Samples used must be independent than those used to create the baseline and must cover entire scope of method.

^d Method validation using adulterated samples shall cover entire scope used in creating baseline fingerprint.

lcfld.com/AOAC/tr347-SAIS-LII-sample-size-needed-for-PR-for-proportion.pdf

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

8 Maximum Time-to-Results

None.

Developed by the AOAC Food Authenticity Methods (FAM) Working Group on Nontargeted Testing. Approved by stakeholders of AOAC FAM on June 30, 2020.

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