AOAC SMPR® 2023.001

Standard Method Performance Requirements **(SMPRs) for Determination of Pesticides in Cannabis-Containing Beverages**

Intended Use: Surveillance Methods for Routine Monitoring

Purpose:

What: AOAC *Standard Method Performance Requirements* (SMPRs®) are voluntary consensus standards developed in accordance with the AOAC policy, "AOAC Due Process for Development of AOAC Non-Method Consensus Standards and Documents." SMPRs describe a scientific community's recommended minimum method performance characteristics and analytical requirements for a specific method related intended use.

Who: Drafted by AOAC working groups, SMPRs are adopted by AOAC by a consensus of stakeholders affiliated with its integrated science programs and projects which are composed of volunteer subject matter experts representing academia, government, industry, and nonprofit sectors from around the world.

Uses: AOAC uses SMPRs in its core science programs in which they are a resource for AOAC method experts, including expert review panels, in the evaluation of validation study data for methods submitted to the AOAC *Official Methods of Analysis*SM and AOAC *Performance Tested Methods*SM programs. Additionally, AOAC SMPRs may be used to provide acceptance criteria for the verification of methods and serve as a resource to guide method development and optimization.

1 Applicability

Determination of pesticides in cannabis-containing beverages. The method will be able to identify and quantify target pesticide residues in specified cannabis-containing beverages.

2 Analytical Technique

Method(s) able to identify and quantify pesticide residues in cannabis-containing beverage matrices. Method(s) must include detailed sample preparation for all matrices evaluated.

3 Definitions

Cannabis-containing beverages.—Refer to section *7. Validation Guidance and References, Matrices* for detailed information on matrix categories for cannabis-containing beverages.

Limit of quantitation (LOQ).—Minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result.

Measurement uncertainty.—Non-negative parameter characterizing the dispersion of the values being attributed to the measured value.

Pesticide residues.—Refer to section *7. Validation Guidance and References, Analytes and target levels* for

detailed information on pesticides residue analytes.

Recovery.—Fraction or percentage of spiked analyte that is recovered when test sample is analyzed using the entire method.

Repeatability.—Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation ($\%RSD_r$).

Reproducibility.—Standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation ($\%RSD_R$).

Target level.—For the purposes of this SMPR, the concentration of pesticide residue that should be used by method developers. Target level should be considered equivalent to regulatory limit, maximum residue limit (MRL), and action level. The term 'target level' is used here because no other single term is used by stakeholders and regulatory bodies.

4 Method Performance Requirements

See Table 1.

5 System Suitability Tests and/or Analytical Quality Control

System suitability and analytical quality control measures must be used. Method developers may choose the most appropriate suitability and quality control procedures based on their method(s). Examples include blanks, check standards at the lowest point and midrange point of the analytical range, recovery check samples, and duplicate sample analysis.

6 Reference Material(s)

Certified reference material should be used when available. Internally produced well-characterized materials may be used for a variety of cannabis-containing beverages until appropriate certified reference material is made available by an internationally recognized organization, such as Institute for Reference Materials and Measurements (IRMM) or United States National Institute of Standards and Technology (NIST).

7 Validation Guidance and References

"Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., Latimer, G.W., Jr. (Ed.), Oxford Academic, New York, NY, USA, https://doi.org/10.1093/9780197610145.005.004 (accessed October 19, 2023)

"Guidelines for Dietary Supplements and Botanicals" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., Latimer, G.W., Jr. (Ed.), Oxford Academic, New York, NY, USA, https://doi.org/10.1093/9780197610145.005.011 (accessed October 19, 2023)

Guidelines for the Validation of Chemical Methods for the FDA Foods Program (2019) 3rd Ed., U.S. Food

and Drug Administration, Silver Spring, MD, USA, https://www.fda.gov/media/81810/download

ICH Topic Q2 (R1), Validation of Analytical Procedures: Text and Methodology (1995) International Council for Harmonization, Geneva, Switzerland, https://www.ema.europa.eu/en/documents/scientific-guideline/ich-q-2-r1-validation- analytical-procedures-text-methodology-step-5_en.pdf

SANTE 11312/2021, Analytical quality control and method validation procedures for pesticide residues analysis in food and feed, https://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?CntID=727

Matrices

Eight matrix categories, A-H, for cannabis-containing beverages are listed in Table 2. Each method submission must address one or more matrix category. Detailed and complete procedures for preparation of test samples of cannabis containing beverage matrices must be addressed during method validation and those data must be included in the method validation submission.

Method Matrix Claim

The number of matrix categories (Table 2) that a method developer chooses for validation will correspond to a matrix claim. Method developers must submit a method matrix claim. The matrix claim is based on the number of matrix categories for which validation data is submitted. The matrix claim and corresponding number of required matrix categories are shown in Table 3.

Each matrix category should be validated using a minimum of three matrix examples. For example, if validating matrix category B (Carbonated), then three matrices could include sparkling water, soft drink, and sparkling lemonade. Examples of matrices for each category are provided in Table 4. This is not intended to be an exhaustive list of all possible matrices. Preferred matrix examples are recommended/desired matrices due to presence in the current marketplace.

Cannabis-containing beverage matrices may be limited in diversity or availability for specific matrix categories. Method developers should reference Table 1 and associated table notes in *Guidelines for the Validation of Chemical Methods for the FDA Foods Program* (2019) 3rd Ed., U.S. Food and Drug Administration, for guidance in selecting alternative matrices that best meet the needs and requirements for method validation.

Alternative matrices must be evaluated for characteristics that define the matrix category and should be selected based on those characteristics that are most similar to the target matrix category. Reference preferred matrix example and other matrix examples in Table 4 for typical chemical composition of matrix categories. For example, category C (High-Sugar) would be similar to sugar content found in typical soft drink, fruit juice, or lemonade.

Matrix examples may fall into more than one matrix category. For example, tea and coffee are listed in both categories A (High-Water Content) and F (Polyphenol-Rich). Matrices that are present in more than one category can be used to satisfy the requirements of each applicable category with a single validation. Table 4 is constructed to assist method developers in coordinating matrix categories with matrix examples and test samples for validation studies. Samples that fall into categories A (High-Water Content) and E (High-Fat) can contain additional components that make their analyses more challenging. For instance, soft drinks have high-water content as well as being carbonated and containing high amounts of sugar. Because these additional components add complexity to the analysis, a method that

successfully addresses soft drinks can reasonably be assumed to also work for samples that only contain high-water content and carbonation, or only high-water content. Therefore, a method developer could address three matrix categories using a single complex matrix such as soft drinks, satisfying categories A (High-Water Content), B (Carbonated), and C (High-Sugar).

Analytes and Target Levels

Method developers must choose a set of analytes from the following options:

Option 1: Analytes listed in Table 1 of AOAC SMPR 2018.011 (*see* Appendix A). Target levels specific to beverage regulations may be substituted.

Option 2: Analytes and target levels listed in Table 5. *Note*: This list contains required analytes and optional analytes.

Option 3: Analytes required by a state or government. Regulatory limits are the target levels. The specific regulation must be submitted with method validation documents and referenced in the method. The method submission should include the words "as required by [regulation] as of [regulation version date]."

8 Maximum Time-to-Result

None.

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Table 1. Specific method performance requirements	
Parameter	Requirement
Method matrix claim	Must be stated[a]
Analyte identification	Criteria must be specified[b]
Limit of quantitation (LOQ), mg/kg	Less than target level[c]
Applicable range, mg/kg	Lowest and highest concentration must be stated and supporting data provided for each analyte
Mean recovery, %	70–120[d-g]
Repeatability (RSD_r), %	≤20
Reproducibility (RSD_R), %	≤44
Matrix effects	Must be evaluated and method of evaluation detailed
Measurement uncertainty	Must be determined and method of determination detailed

[a] *See* section *7. Validation Guidance and References, Method Matrix Claim.*
[b] *See* SANTE 11312/2021.
[c] *See* section *7. Validation Guidance and References, Analytes and Target Levels* for target levels.
[d] Correction may be permitted with appropriate justification.
[e] In exceptional circumstances, lower recoveries <70% are acceptable if the repeatability requirement is met.
[f] Appropriate justification must be provided.
[g] *See* SANTE 11312/2021, section *Correction for method bias, E4* and *Appendix E (An overview of the options to account for method bias and use of recovery correction factors).*

Table 2. Cannabis-containing beverages matrix categories	
A. High-water content	E. High-fat
B. Carbonated	F. Polyphenol-rich
C. High-sugar	G. Contains alcohol
D. High-acid	H. Other

Table 3. Number of matrix categories required for each matrix claim	
Method matrix claim	No. required matrix categories
Broad range of cannabis-containing beverages	6-8
Variety of cannabis-containing beverages	4-5
Select cannabis-containing beverages	1-3

Table 4. Matrix categories and matrix examples organized to assist method developers					
Matrix categories			No. matrix categories satisfied	Preferred matrix example	Other matrix examples
A. High-water content			1	Bottled water	Tea/coffee; herbal blends
	B. Carbonated		2	Sparkling water	Soft drinks, sparkling lemonade, flavored seltzer
		C. High-sugar	3	Soft drink	Fruit juice, lemonade soft drinks, sparkling lemonade, flavored seltzer
D. High-acid			1	Citrus-flavored beverage	Lemonade, fruit juice, soft drink
E. High-fat			1	Any dairy-containing drink	Milkshakes
	F. Polyphenol-rich		2	Coffee with dairy	Coffees with dairy, tea with dairy
F. Polyphenol-rich			1	Coffee or tea	Coffee, tea, kombucha, green tea, ginger-turmeric tea (multiherb blends)
G. Contains alcohol			1	Spirits	Wine, hard cider, hard seltzer, spirits
H. Other			1		Smoothies/shakes, sports drinks, dry powder mixes, protein drinks

Table 5. Analyte list includes required and optional pesticides for Option 2 listed in section Analytes and target levels above		
Required compounds		
Compound[a]	CAS No.	Target level, mg/kg
Abamectin (avermectins B1a and B1b)	71751-41-2	0.3
Acephate	30560-19-1	0.4
Acequinocyl	57960-19-7	2
Acetamiprid	135410-20-7	0.2
Aldicarb	116-06-3	0.1
Azoxystrobin	131860-33-8	0.2
Bifenazate	149877-41-8	0.2
Bifenthrin	82657-04-3	0.2
Boscalid	188425-85-6	0.4
Carbaryl	63-25-2	0.2
Carbofuran	1563-66-2	0.1
Chlorantraniliprole	500008-45-7	0.2
Chlorfenapyr	122453-73-0	0.2
Chlorpyrifos	2921-88-2	0.1
Clofentezine	74115-24-5	0.2
Cyfluthrin (mix of isomers)	68359-37-5	1
Cypermethrin (mix of isomers)	52315-07-8	1
Daminozide	1596-84-5	0.1
Diazinon	333-41-5	0.2
Dichlorvos	62-73-7	0.1
Dimethoate	60-51-5	0.1
Ethoprophos	13194-48-4	0.1
Etofenprox	80844-07-1	0.1
Etoxazole	153233-91-1	0.2
Fenoxycarb	72490-01-8	0.1
Fenpyroximate (mix of isomers)	134098-61-6	0.4
Fipronil	120068-37-3	0.1
Flonicamid	158062-67-0	1
Fludioxonil	131341-86-1	0.4
[a] Multicomponent compounds and compounds with isomers are listed to help method development but should not be considered an exhaustive list. Specific isomer choices and multicomponent compounds are left to the discretion of the method developer and must be addressed in method submission.		

Appendix A. Pesticides listed in Table 1 of AOAC SMPR 2018.011 [parts per million (ppm) are equivalent to mg/kg; lowest action level is the target level)			
Compound	CAS No.	Lowest action level, ppm	Target LOQ, ppm[a]
Abamectin (avermectins B1a and B1b)	71751-41-2	0.05	0.025
Acephate	30560-19-1	0.1	0.05
Acequinocyl	57960-19-7	0.1	0.05
Acetamiprid	135410-20-7	0.1	0.05
Aldicarb	116-06-3	0.1	0.05
Allethrin	584-79-2	0.1	0.05
Ancymidol	12771-68-5	0.1	0.05
Azadirachtin	108168-76-9	0.1	0.05
Azoxystrobin	131860-33-8	0.02	0.01
Benzovindiflupyr	1072957-71-1	0.1	0.05
Bifenazate	149877-41-8	0.01	0.005
Bifenthrin	82657-04-3	0.01	0.005
Boscalid	188425-85-6	0.1	0.05
Buprofezin	69327-76-0	0.1	0.05
Captan	133-06-2	0.05	0.025
Carbaryl	63-25-2	0.2	0.1
Carbofuran	1563-66-2	0.1	0.05
Chlorantraniliprole	500008-45-7	0.2	0.1
Chlordane	57-74-9	0.1	0.05
Chlorfenapyr	122453-73-0	0.1	0.05
Chlormequat chloride	999-81-5	0	0.005[b]
Chlorpyrifos	2921-88-2	0.1	0.05
Clofentezine	74115-24-5	0.1	0.05
Clothianidin	21088-92-5	0	0.005[b]
Coumaphos	56-72-4	0.1	0.05
Cyantraniliprole	736994-63-1	0	0.005[b]
Cyfluthrin (Baythroid)	68359-37-5	0.01	0.005
Cypermethrin	52315-07-8	0.05	0.025
Cyprodinil	121552-61-2	0	0.005[b]
Daminozide	1596-84-5	0.05	0.025
Deltamethrin	52918-63-5	0	0.005[b]
Diazinon	333-41-5	0.1	0.05
Dichlorvos	62-73-7	0.1	0.05
Dimethoate	60-51-5	0.1	0.05
Dimethomorph	110488-70-5	2	1
Dinotefuran	165252-70-0	0	0.005[b]
Dodemorph	1593-77-7	0	0.005[b]
Endosulfan I (alpha)	959-98-8	0	0.005[b]

Endosulfan II (beta)	33213-65-9	0	0.005[b]
Endosulfan sulfate	1031-07-8	0	0.005[b]
Ethephon	16672-87-0	0	0.005[b]
[a] AOAC expert review panel can consider LOQs that are higher than the target LOQ based on its judgement.			
[b] The AOAC Cannabis Working Group recommended an LOQ of 0.005 ppm for pesticides that do not have a regulatory imposed action level.			