AOAC SMPR® 2022.001

Standard Method Performance Requirements (SMPRs®) for Quantitation of Cannabinoids in Beverages

Intended Use: Consensus-Based Reference Method

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, a single-laboratory validation, or a multi-site collaborative study. SMPRs are written and adopted by AOAC through its stakeholder-based integrated science programs and projects, which are composed of representatives and experts from the academic, government, industry, and nonprofit sectors. AOAC SMPRs are used by AOAC method review experts, including expert review panels, 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 method will be able to identify and quantify individual cannabinoids (as listed in Tables 1 and 2) in at least four beverage matrices (as listed in Table 3). The method must include detailed sample preparation procedures for each individual matrix evaluated.

3 Analytical Technique

Any analytical technique(s) that measures the analytes of interest and meets the following method performance requirements is/are acceptable.

4 Definitions

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.

Recovery.—Fraction or percentage of spiked analyte that is recovered when the 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).

Quantitative method.—Method of analysis in which response is the amount of the analyte measured either directly (enumeration in a mass or a volume) or indirectly (color, absorbance, impedance, etc.) in a certain amount of sample.

5 Method Performance Requirements

See Tables 4 and 5.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blank check samples, and check standards at the lowest point and midrange point of the analytical range.

7 Reference Material(s)

See Table 1 (required analytes) and Table 2 (additional optional analytes) for sources of reference materials.

Refer to "Annex F: Development and Use of In-House Reference Materials" in "Appendix F: Guidelines for Standard Method Performance Requirements," current edition of the *Official Methods of Analysis of AOAC INTERNATIONAL*. Available at: http://www.eoma.aoac.org/app_f.pdf

8 Validation Guidance

Detailed and complete procedures for reproducible preparation of test samples of each beverage matrix must be addressed during method validation and those data must be included in the method validation submission. Required matrix categories are listed in Table 3; method developers must include validation data and detailed sample preparation procedures for at least one beverage from each matrix category, for a minimum of four beverages included in the validation submission package. Describe each beverage sample in detail including any known specifics about the product or manufacturing such as the presence of fats/dairy, nanoemulsions, or microencapsulated ingredients.

Measurement uncertainty must be determined, and the method of determination detailed.

"Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis," current edition of the *Official Methods of Analysis of AOAC INTERNATIONAL*. Available at: http://www.eoma.aoac.org/app_d.pdf

"Appendix F: Guidelines for *Standard Method Performance Requirements*," current edition of the *Official Methods of Analysis of AOAC INTERNATIONAL*. Available at: http://www.eoma.aoac.org/app_f.pdf

"Appendix K: Guidelines for Dietary Supplements and Botanicals," current edition of the *Official Methods of Analysis of AOAC INTERNATIONAL*. Available on line at: http://www.eoma.aoac.org/app_k.pdf

Guidelines for the Validation of Chemical Methods for the FDA Foods Program (2019) 3rd Ed., U.S. Food and Drug Administration, Siliver Spring, MD, USA

9 Maximum Time-to-Result

None.

Final version: March 4, 2022. Approved by: Cannabis Analytical Science Program. Effective date: May 1, 2022.

Table 1. Required cannabinoids

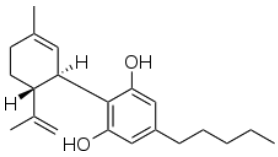
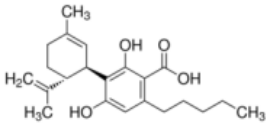
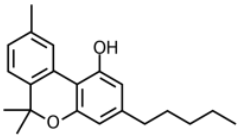
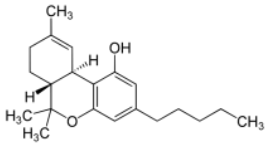
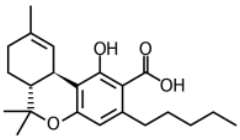
Common name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
Cannabidiol	CBD	2-[(1*R*,6*R*)-6-isopropenyl-3-methylcyclohex-2-en-1-yl]-5-pentylbenzene-1,3-diol	13956-29-1		Restek Cerilliant Sigma-Aldrich API Standards Echo Pharm Lipomed AG
Cannabidiolic acid	CBDA	2,4-Dihydroxy-3-[(1*R*,6*R*)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-pentylbenzoic acid	1244-58-2		Cerilliant USP Restek Lipomed AG Echo Pharmaceutical
Cannabinol	CBN	6,6,9-Trimethyl-3-pentyl-benzo[*c*]chromen-1-ol	521-35-7		Cerilliant Restek
Tetrahydro-cannabinol	THC	(−)-(6*aR*,10*aR*)-6,6,9-trimethyl-3-pentyl-6*a*,7,8,10*a*-tetrahydro-6*H*-benzo[*c*]chromen-1-ol	1972-08-3		Cerilliant USP Echo Pharmaceuticals
Tetrahydro-cannabinolic acid	THCA	(6aR,10aR)-1-hydroxy-6,6,9-trimethyl-3-pentyl-6a,7,8,10a-tetrahydro-6h-benzo[c]chromene-2-carboxylic acid	23978-85-0		Cerilliant USP Echo Pharmaceuticals

Table 2. Additional cannabinoids (not required for this SMPR)

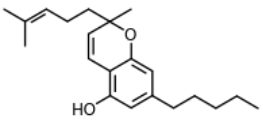
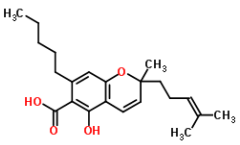
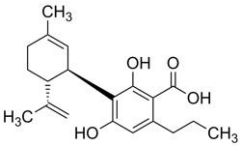
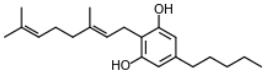
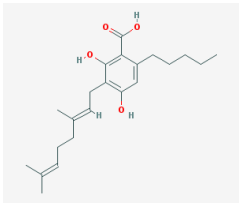
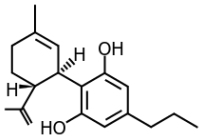
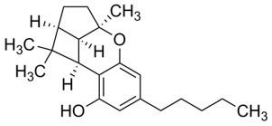
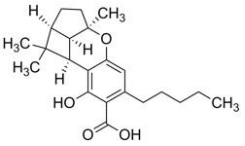
Name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
Cannabichromene	CBC	2-Methyl-2-(4-methylpent-3-enyl)-7-pentyl-5-chromenol	20675-51-8		Cerilliant Sigma-Aldrich Echo Pharmaceuticals
Cannabichromenic acid	CBCA	5-Hydroxy-2-methyl-2-(4-methyl-3-penten-1-yl)-7-pentyl-2*H*-chromene-6-carboxylic acid	20408-52-0		Cerilliant
Cannabidivarinic acid	CBDVA	2,4-Dihydroxy-3-[(1*R*,6*R*)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-propylbenzoic acid	31932-13-5		Cerilliant
Cannabigerol	CBG	2-[(2*E*)-3,7-dimethylocta-2,6-dienyl]-5-pentyl-benzene-1,3-diol NIST: 1,3-Benzenediol, 2-(3,7-dimethyl-2,6-octadienyl)-5-pentyl	25654-31-3; NIST: 2808-33-5		Cerilliant Lipomed AG Echo Pharmaceuticals SPEX Certiprep Tocris (UK)
Cannabigerolic acid	CBGA	3-[(2*E*)-3,7-dimethylocta-2,6-dienyl]-2,4-dihydroxy-6-pentylbenzoic acid	25555-57-1		Cerilliant Echo Pharmaceuticals SPEX Certiprep
Cannabidivarin	CBDV	2-((1*S*,6*S*)-3-methyl-6-(prop-1-en-2-yl) cyclohex-2-enyl)-5-propylbenzene-1,3-diol	24274-48-4		Cerilliant SPEX Certiprep
Cannabicyclol	CBL	(1*aS*,3*aR*,8*bR*,8*cR*)-1*a*,2,3,3*a*,8*b*,8*c*-hexahydro-1,1,3*a*-trimethyl-6-pentyl-1*H*-4-oxabenzo[*f*] cyclobut[*cd*]inden-8-ol	21366-63-2		Cerilliant
Cannabicyclolic acid	CBLA	(±)-1a,2,3,3a,8b,8c-hexahydro-8-hydroxy-1,1,3a-trimethyl-6-pentyl-1H- 4-Oxabenzo[f] cyclobut[cd]indene-7-carboxylic acid	40524-99-0		Cerilliant

Table 2. (*continued*)

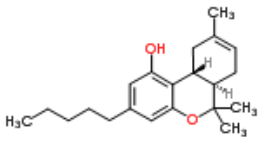
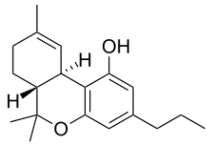
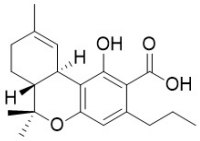
Name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
$^{\Delta 8}$Tetrahydrocannabinol	$^{\Delta 8}$THC	6,6,9-Trimethyl-3-pentyl-6*a*,7,10,10*a*-tetrahydrobenzo[*c*]chromen-1-ol	5957-75-5		Cerilliant SPEX Certiprep
Tetrahydrocannabivarin	THCV	6,6,9-Trimethyl-3-propyl-6*a*,7,8,10*a*-tetrahydro-6*H*-benzo[*c*]chromen-1-ol	28172-17-0		Cerilliant USP
Tetrahydrocannabivaric acid	THCVA		39986-26-0		Cerilliant

Table 3. Matrix categories

Carbonated beverages	Coffees	Teas and multiherb blends	Other
Sodas	With and without dairy/fats	Kombucha	Fruit juices
Sparkling water		Green tea	Smoothies/shakes
		Ginger-turmeric	Sports drinks
			Dry powder mixes
			Wine
			Beer
			etc.

Table 4. Method performance requirements for cannabinoids (part 1)

Parameter	Requirement
Limit of quantitation, %	≤0.0002
Analytical range, %	0.002–10

Table 5. Method performance requirements for cannabinoids (part 2)

	Ranges, %				
Parameter	0.0002–0.01	0.01–0.05	0.05–0.5	0.5–5	5–10
Recovery, %	70–130	80–120	85–118	90–111	95–105
RSD_r, %	8	≤5	≤5	≤3	≤2
RSD_R, %	12	≤10	≤10	≤8	≤6