AOAC SMPR® 2019.003

Standard Method Performance Requirements (SMPRs®) for Quantitation of Cannabinoids in Plant Materials of Hemp (Low THC Varieties *Cannabis* sp.)

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 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 methods being considered for *Performance Tested Methods*℠ certification or AOAC *Official Methods of Analysis*℠ adoption 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 plant materials expressed on a dry weight basis. The method must be able to report total THC (as defined in this SMPR), regardless of how it is measured.

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

Hemp plant materials.—Fresh or dried, whole or milled plant material of low THC cultivars of *Cannabis* spp.

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

Quantitative method.—Method of analysis 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.

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 ($\%RSD_R$).

Total THC.—Maximum potential percentage w/w delta-9-tetrahydrocannabinol that the test sample could yield on a dry weight basis, including delta-9-THC and delta-9-THCA.

5 Method Performance Requirements

See Tables 3 and 4.

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.

A detailed description of the method's dry weight procedures and calculations must be included.

7 Reference Material(s)

See Tables 1 and 2 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*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA. Available at: http://www.eoma.aoac.org/app_f.pdf

8 Validation Guidance

An initial and important stage in cannabis testing is preparation of a homogeneous sample of the plant material. Detailed and complete procedures for reproducible preparation of test samples from the plant material must be addressed during method validation and those data must be included in the method validation submission. A detailed description of the method's dry weight procedures and calculations must be included in the submission.

Appendix D: *Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis, Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA. Available at: http://www.eoma.aoac.org/app_d.pdf

Appendix F: *Guidelines for Standard Method Performance Requirements, Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA. Available at: http://www.eoma.aoac.org/app_f.pdf

Appendix K: *Guidelines for Dietary Supplements and Botanicals, Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA. Available at: http://www.eoma.aoac.org/app_k.pdf

9 Maximum Time-to-Result

None

Approved by attending stakeholders of the AOAC Cannabis Analytical Science Program (CASP) meeting on September 7, 2019. Final Version Date: October 3, 2019.

Posted: October 9, 2019

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Table 1. Required cannabinoids

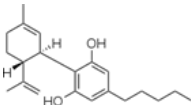
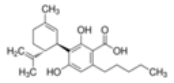
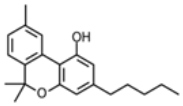
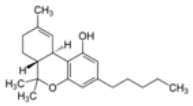
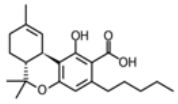
Common name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
Cannabidiol	CBD	2-[(1*R*,6*R*)-6-isopropenyl-3-methylcyclohex-2-en-1-yl]-5-pentylbenzene-1,3-diol	13956-29-1		Restek Cerilliant Sigma-Aldrich API Standards Echo Pharm Lipomed AG
Cannabidiolic acid	CBDA	2,4-Dihydroxy-3-[(1*R*,6*R*)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-pen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

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Table 2. Additional, desirable cannabinoids

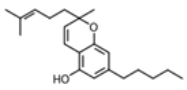
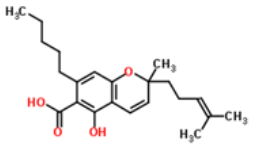
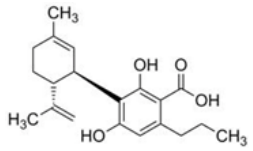
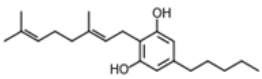
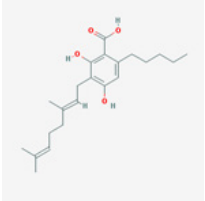
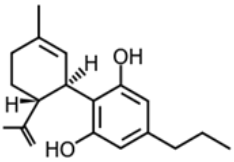
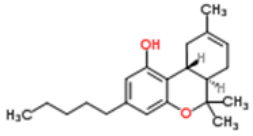
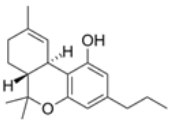
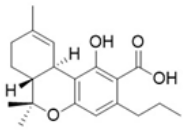
Common name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
Cannabichromene	CBC	2-Methyl-2-(4-methylpent-3-enyl)-7-pentyl-5-chromenol	20675-51-8		Cerilliant Sigma Aldrich Echo Pharmaceuticals
Cannabichromenic acid	CBCA	5-Hydroxy-2-methyl-2-(4-methyl-3-penten-1-yl)-7-pentyl-2*H*-chromene-6-carboxylic 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
$^{\Delta 8}$Tetrahydro-cannabinol	$^{\Delta 8}$THC	6,6,9-Trimethyl-3-pentyl-6a,7,10,10a-tetrahydrobenzo[c]chromen-1-ol	5957-75-5		Cerilliant SPEX Certiprep
Tetrahydro-cannabivarin	THCV	6,6,9-Trimethyl-3-propyl-6a,7,8,10a-tetrahydro-6H-benzo[c]chromen-1-ol	28172-17-0		Cerilliant USP
Tetrahydrocannabivaric acid	THCVA		39986-26-0		Cerilliant

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Table 3. Method performance requirements (part 1) for cannabinoids

Parameter	Requirement[a]
Limit of quantitation (LOQ), %	≤0.05
Analytical range (CBD and CBDA), %	0.05–35
Analytical range (others), %	0.05–5

[a] All calculated on dry weight basis.

Table 4. Method performance requirements (part 2) for cannabinoids

	Analytical range, %[a]		
Parameter	0.05–0.5	>0.5–5	5–35[b]
Recovery, %	85–118	90–111	95–105
RSD_r, %	≤5	≤3	≤2
RSD_R, %	≤10	≤8	≤6

[a] All calculated on dry weight basis; observed values to be compared to indicated limits for acceptability.

[b] Only applicable to CBD and CBDA.

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