DRAFT AOAC SMPR 2016.XXX; Version 2; 21 December 2016

Method Name: **Quantitation of cannabinoids in cannabis concentrates**

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 Stakeholder Panels 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 method being considered for *Performance Tested Methods* 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 Table 1a and Table 1b) present in cannabis concentrates.

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**:

Cannabis Concentrates
A product resulting from chemical or physical processing of *cannabis sativa* or any of its hybrids, largely free of solvents with cannabinoid content higher than the starting material.

Limit of Quantitation (LOQ)
The 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.

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
The 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).

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

5. Method Performance Requirements:

See table 2 and 3.

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 tables 1A and 1B 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*, 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

8. Validation Guidance:

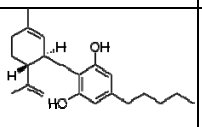
Appendix D: Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method of Analysis; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_d.pdf

Appendix F: Guidelines for Standard Method Performance Requirements; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

Appendix K: Guidelines for Dietary Supplements and Botanicals; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available on line at: http://www.eoma.aoac.org/app_k.pdf

9. Maximum Time-To-Result: None

Table 1A: Required Cannabinoids

Common Name	Abbrev-iation	IUPAC Name	CAS Number	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

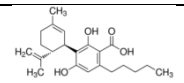
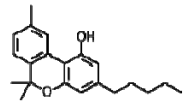
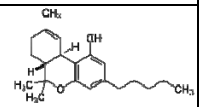
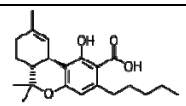
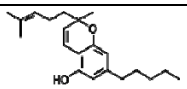
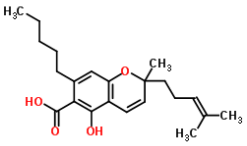
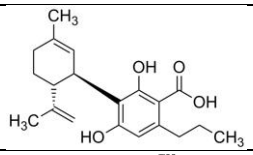
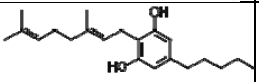
Cannabidiolic Acid	CBDA	2,4-dihydroxy-3-[(1R,6R)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-pentylbenzoic acid [SGC: name corrected]	1244-58-2		Cerilliant USP Restek Lipomed AG Echo Pharmaceutical
Cannabinol	CBN	6,6,9-Trimethyl-3-pentyl-benzo[c]chromen-1-ol	521-35-7		Cerilliant Restek
Tetrahydro-cannabinol	THC	(−)-(6aR,10aR)-6,6,9-Trimethyl-3-pentyl-6a,7,8,10a-tetrahydro-6H-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 1B: Additional, Desirable Cannabinoids

Name	Abbreviation	IUPAC Name	CAS Number	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-2H-chromene-6-carboxylic acid	20408-52-0		no reference material
Cannabidivarinic acid	CBDVA	2,4-dihydroxy-3-[(1R,6R)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-propylbenzoic acid	31932-13-5		Cerilliant
Cannabigerol	CBG	2-[(2E)-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)

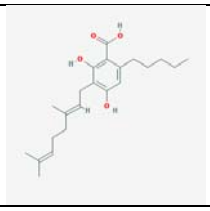
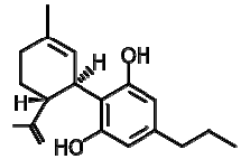
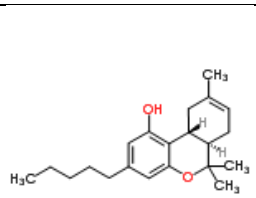
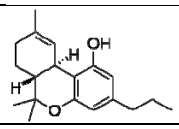
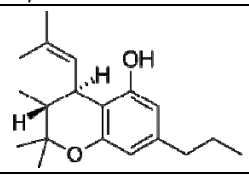
Cannabigerolic - acid	CBGA	3-[(2E)-3,7-dimethylocta-2,6-dienyl]-2,4-dihydroxy-6-pentylbenzoic acid	25555-57-1		Cerilliant Echo Pharmaceuticals SPEX Certiprep
Cannabidivarin	CBDV	2-((1*S*,6*S*)-3-methyl-6-(prop-1-en-2-yl) cyclohex-2-enyl)-5-propylbenzene-1,3-diol	24274-48-4		Cerilliant SPEX Certiprep
$^{\Delta 8}$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-6*H*-benzo[c]chromen-1-ol	28172-17-0		Cerilliant USP
Tetrahydrocannabivarin - acid	THCVA		28172-17-0		No reference material

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Table 2: Method performance requirements (part 1).

Parameter	Requirement THC, THCA, CBDA, CBD, Individually Reported	Requirement (Additional, Desirable Cannabinoids (Table 1b), including CBN)
Limit of Quantitation (LOQ) (% w/w)	≤ 0.3	≤ 0.3
Analytical Range (% w/w)	≤ 0.3 – ca. 100	≤ 0.3 – ca. 50

Reported as individual cannabinoids

Table 3: Method performance requirements (part 2).

Parameters	Ranges (% w/w)		
	≤ 0.3 – 1	> 1 - 10	> 10 – ca. 100
Recovery (% w/w)	95 – 105	97 - 103	98 - 102
% RSD_r	≤ 5	≤ 4	≤ 2
% RSD_R	≤ 7	≤ 5	≤ 3