2.3 SAMPLING AND HANDLING HEMP FOR THC & CBD[3] ANALYSIS

2.3.1 Introduction

Refer to the jurisdiction having authority for any cannabinoid analysis sampling and handling regulations or guidance applicable to your license. In the absence of such cannabinoid analysis sampling and handling regulations or guidance by the jurisdiction having authority, use these Guidance Procedures.

2.3.2 Sampling Timeline and Grower Responsibilities

a. The grower shall refer to the jurisdiction having authority to determine a timeline.
b. During the sampling, the grower or an authorized representative shall be present at the growing site.
c. Floral materials harvested for phytocannabinoid extraction shall not be moved beyond the processor, nor commingled, nor extracted, until test results are complete.

2.3.3 Pre-Harvest Sampling Procedure

a. Adequate personal protective equipment shall be used.
b. Proper equipment shall be used to prevent cross contamination.
c. The material selected for pre-harvest sampling will be determined by the grower. Cuttings will be collected to make one representative sample.
d. Refer to the authority having jurisdiction to determine adequate number of samples and proper locations. In the absence of jurisdictional requirements, the following guidance is given.
    i. Clip the top 12 inches of hemp plant's primary stem, including female floral material.
    ii. Take cuttings from at least five (5) hemp plants within the plot.
    iii. Place the complete sample in a clean paper bag.
    iv. Seal the bag by folding over the top once and staple the bag shut.
    v. A separate sample must be taken from each non-contiguous plot of a given variety.
    vi. A separate sample must be taken for each variety.
    vii. Samples shall be secured in a paper bag (to allow for air-drying during transport).
    viii. Label the sample container with a sample ID.

2.3.4 Handling Procedures of Pre-Harvest Samples

a. Samples will be taken for drying and storage.
b. Samples should be arranged in a single layer for drying.
c. Drying oven will be used when possible.
d. Samples in the oven will be left in the labeled sample bag.
e. If selected for testing, the entire sample will be sent to a testing lab for analysis.

2.3.5 Post-Harvest Sampling Procedures for Floral Material

a. Refer to the authority having jurisdiction to determine adequate number of samples and proper locations. In the absence of jurisdictional requirements, the following guidance is

[3] Cannabidiol.

given.

b. Adequate personal protective equipment shall be used.
c. Proper equipment shall be used to prevent cross contamination.
d. The plot selected for sampling shall be designated by the pre-harvest sample results. The material selected for post-harvest sampling from this plot will be determined by the grower. All post-harvest samples of floral material shall be taken from the designated harvested plot materials in the form (intact-plant, flowers, ground materials, etc.) in which the material will be sent to the processor.
e. Grower must inventory the entire harvest to determine the form in which it exists and follow the protocol as appropriate in parts (a), (b), or (c) below.
    i. If, upon inventory, the grower determines that the entire harvest is not in a homogenous form (intact-plant, flowers, ground materials, etc.), it must be determined to take additional samples or other course of action or take the pre-harvest results.
    ii. For intact-plant post-harvest samples:
        1. Ensure that the entire harvest is accounted for and in the same form (*i.e.*, intact-plants).
        2. Clip the top 12 inches of hemp plant, primary stem, including female floral material.
        3. Take cuttings from at least five (5) non-adjacent hemp plants within the harvest's storage/drying area.
        4. Place the complete sample in a paper bag.
        5. Seal the paper bag by folding over top once and stapling to keep closed.
        6. Complete sampling procedures in parts (d) and (e) above.
    iii. For ground plant or ground floral material post-harvest samples:
        1. Ensure that the entire harvest is accounted for and in the same form *(i.e.*, all harvested material whether whole plant or floral material only must be ground with no intact plants or whole flowers remaining from that harvest).
        2. Sample material from bag or container.
        3. Sample from a minimum of four (4) locations within the containers from a given harvest.
        4. Place the complete sample in a plastic sample container.
        5. Seal the plastic sample container.
        6. Complete sampling procedures in parts (d) and (e) above.
    iv. For post-harvest samples in other forms (*e.g.*, trimmed floral material, or floral material and stems, etc.):
        1. Ensure that the entire harvest is accounted for and in the same form (*i.e.*, all harvested material must be uniform).
        2. Randomly collect at least one cup of material by volume.
        3. Place the complete sample in a paper bag or plastic container and seal the container, as appropriate.
        4. Complete sampling procedures in parts (d) and (e) above.
    v. A separate sample must be taken for each plot designated for post-harvest sampling.
    vi. Samples shall be labeled and prepared for transport to the lab.
    vii. Label the sample container with a sample ID.

2.3.6 Handling Procedures of Post-Harvest Samples

a. The entire sample will be sent to the testing lab for analysis.
b. Hemp crops generated from certified seed will incur pre-harvest testing of at least five

percent (5%) of growing plots per variety, per seed source.

c. Hemp crops from planting materials other than certified seed will incur pre-harvest testing of at least fifty percent (50%) of growing plots per variety, per seed source.
d. 100% of post-harvest samples will be tested.

2.4 CONTAMINANT TESTING AND HEMP CANNABINOID QUANTIFICATION:

2.4.1 Potency / Strength Cannabinoid Quantification

To ensure that methods measuring cannabinoids are fit for purpose, laboratories should consider utilizing analytical methods which have achieved AOAC International First Action Status or Final Action (when available) Status to the attached SMPR 2017.002 approved by *Cannabis* stakeholders with AOAC[4],or use such methods for internal verification or validation purposes, except for the following revisions:

a. List only four compounds: THC, THCA, CBD, and CBDA as the main analytes of interest, with the other 10 listed in the SMPR optional.

b. List all target plant parts of hemp (flower, leaf, stalk, seed) and oils/extracts.

Cannabinoid potency/strength methods must be able to determine the concentration of target cannabinoids to effectively distinguish *Cannabis* as either legal hemp or marijuana. Specifically, methods must be accurate and precise at concentrations that bracket 0.3% THC.

2.4.2 Purity & Contaminants

Hemp products intended for human consumption or topical use may be subject to FDA and state or tribal government regulations regarding harmful substances and contaminants.

Guidance for contaminants (heavy metals, microorganisms, pesticides and residual solvents) has been published in the American Herbal Pharmacopoeia (AHP) *Cannabis* monograph and the American Herbal Products Association (AHPA) Guidance Policies.

Limits for the following contaminants are listed in the following references:

Heavy metals: AHP *Cannabis* Monograph/AHPA Guidance Document*

Microbiology: AHP *Cannabis* Monograph**/AHPA Guidance Document

Mycotoxins: AHPA Guidance Document

Pesticides: AHP *Cannabis* Monograph/FDA PAM/AHPA Guidance Document

Solvents: AHP *Cannabis* Monograph/USP <467>/AHPA Guidance Document

Note: * AHPA guidance does not include the stricter limits for lead consumption required in the state of California under Proposition 65

** Microbiology limits are based on products consumed orally.

[4] AOCA International (Association of Analytical Communities); SMPR is standard method performance requirements.

AOAC SMPR® 2017.002

Standard Method Performance Requirements (SMPRs) for Quantitation of Cannabinoids in Dried Plant Materials

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 Tables 1 and 2) in dried plant materials.

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

Dried plant materials.—Dried whole or milled flower plant material from *Cannabis sativa* and its hybrids.

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 Tables 3 and 4.

Table 1. Required cannabinoids

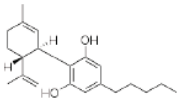
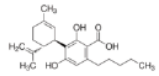
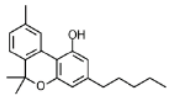
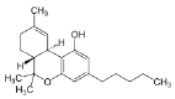
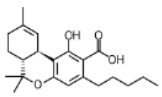
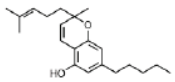
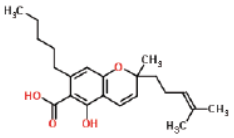
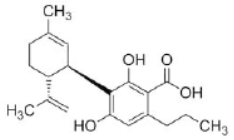
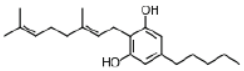
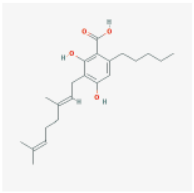
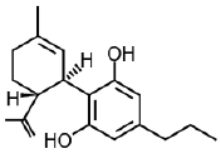
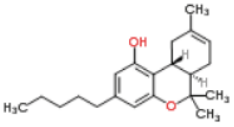
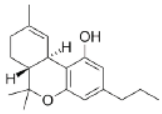
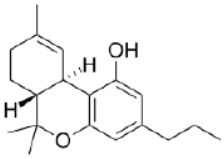
Common name	Abbreviation	IUPAC name	CAS No.	Molecular structure	Reference material
Cannabidiol	CBD	2-[(1*R*,6*R*)-6-Isopropenyl-3-methylcyclohex-2-en-1-yl]-5-pentylbenzene-1,3-diol	13956-29-1		Restek Cerilliant Sigma-Aldrich API Standards Echo Pharm Lipomed AG
Cannabidiolic acid	CBDA	2,4-Dihydroxy-3-[(1*R*,6*R*)-3-methyl-6-prop-1-en-2-ylcyclohex-2-en-1-yl]-6-pentylbenzoic 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

Table 2. Additional, desirable cannabinoids

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		No reference material
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
Tetrahydrocannabivarin acid	THCVA		28172-17-0		No reference material

2.5 STORAGE AND DISTRIBUTION

Storage of hemp shall be under appropriate conditions of temperature, humidity, and light so that the identity, purity, strength, and composition of the components and hemp are not affected.

Storage of hemp and hemp products shall be properly labeled at all times to prevent contaminations and unintended comingling.

Storage of hemp and hemp products shall be properly labeled to indicate a hold or available for release.

Storage of material in-process shall be identified under conditions that prevent mix-ups, contamination, and deterioration.

Storage of material in-process shall be held under appropriate conditions of temperature, humidity, and light.

Storage of packaging and labels shall be under conditions adequate to prevent the packaging and labels from being adversely affected.

Distribution of hemp products shall be under conditions that will protect the products against contamination and deterioration.