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CHAPTER 333
OREGON HEALTH AUTHORITY
PUBLIC HEALTH DIVISION

FILING CAPTION: Oregon Environmental Laboratory Accreditation Program (ORELAP) cannabis and psilocybin testing laboratory accreditation standards

EFFECTIVE DATE: 07/23/2024

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RULES:
333-064-0035, 333-064-0100, 333-064-0110, 333-064-0120, 333-064-0140

AMEND: 333-064-0035

NOTICE FILED DATE: 05/30/2024

RULE SUMMARY: Amend OAR 333-064-0035
This rule is being amended to add a requirement to obtain and maintain licensure from the Oregon Liquor and Cannabis Commission (OLCC) for laboratories accredited to perform compliance sampling and testing of cannabis. Similarly, a requirement to obtain and maintain licensure from Oregon Psilocybin Services for laboratories accredited to perform compliance sampling and testing of psilocybin products is being added to the rule. Failure to comply with these new requirements will result in a laboratory's loss of accreditation by ORELAP.

CHANGES TO RULE:

333-064-0035
Approval Requirements ¶

(1) This rule and the TNI Standards describe the procedure for obtaining and maintaining accreditation.¶
(2) Oregon Environmental Laboratory Accreditation Program (ORELAP) accreditation can be granted, denied, suspended, or revoked in total or in part as described in the TNI Standards.¶
(a) Reasons to deny an initial application shall include, but are not limited to:¶
(A) Failure to submit a completed application;¶
(B) Failure to pay fees;¶
(C) Failure of laboratory staff to meet the personnel qualifications of education, training, and experience as required by the TNI Standards;¶
(D) Failure to successfully analyze and report proficiency testing (PT) samples as required by the TNI Standards and these rules;¶
(E) Failure to respond to an assessment report from an on-site assessment with a corrective action report within 30 calendar days, or failure to respond to deficiencies identified in the first corrective action report review within 30 calendar days, as required by the TNI Standards;¶

(F) Failure to implement the corrective actions detailed in the corrective action report within the agreed upon time frame;¶
(G) Failure to implement a quality system as defined in the TNI Standards;¶
(H) Failure to pass required on-site assessments;¶
(I) Misrepresentation of any fact pertinent to receiving or maintaining accreditation; or¶
(J) Denial of entry during the laboratory's normal business hours for an on-site assessment.¶
(b) Reasons for suspension or revocation in total or in part shall include but are not limited to:¶
(A) If the accreditation body finds, during the on-site assessment, that the public interest, safety or welfare imperatively requires emergency action;¶
(B) Failure to complete proficiency testing studies as required by the TNI Standards and these rules;¶
(C) Failure to notify the accreditation body of any changes in key accreditation criteria defined in the TNI Standards within 35 calendar days of the effective change, including:¶
(i) General features of the laboratory, including corporate entity, name, addresses, legal status, technical directors and quality managers, and technical resources;¶
(ii) General information concerning the laboratory such as its activities, its relationship in a larger corporate entity if any, and addresses of all its physical location(s) to be covered by the scope of accreditation;¶
(D) Failure to maintain a quality system as required by the TNI Standards;¶
(E) Failure of the laboratory to employ staff that meets qualifications for education, training, and experience as required by the TNI Standards;¶
(F) Misrepresentation of any fact pertinent to receiving or maintaining accreditation;¶
(G) Denial of entry to an accreditation body's assessment team during the laboratory's normal business hours for the purpose of conducting an on-site assessment;¶
(H) Failure to pass an on-site assessment conducted by an accreditation body;¶
(I) Failure to respond to an assessment report from an on-site assessment with a corrective action report within 30 calendar days, or failure to respond to deficiencies identified in the first corrective action report review within 30 calendar days, as required by the TNI Standards;¶
(J) Failure to implement the corrective actions detailed in the corrective action report within the agreed upon time frame;¶
(K) Failure to pay fees;¶
(L) Failure to maintain licensure by the Oregon Liquor and Cannabis Commission (Commission) under ORS 475C.548 if the laboratory is accredited under ORS 475C.560 for sampling and laboratory testing of marijuana items and industrial hemp-derived vapor items required under ORS 475C.544; or¶
(~~K~~M) Failure to ~~pay fees.~~maintain licensure by Oregon Psilocybin Services under ORS 475A.594 if the laboratory is accredited under ORS 475A.606 for sampling and laboratory testing of psilocybin products required under ORS 475A.590. ¶
(3) In no case shall a laboratory be accredited that does not comply with the TNI Standards as specified in this rule.¶
(4) The elements for accreditation shall include but are not restricted to:¶
(a) Application for accreditation:¶
(A) ORELAP will make online, electronic applications available to all laboratories requesting an application.¶
(B) The laboratory must request ORELAP accreditation by completing and submitting to ORELAP an acceptable application that includes all elements as required by the TNI Standards. For primary accreditation this includes a completed application with all required documents. For secondary accreditation this includes a completed application with all of the required documents plus proof of accreditation from a primary accrediting body.¶
(b) The laboratory's participation in a biennial on-site assessment(s) as required by the TNI Standards. Environmental testing laboratories seeking initial, primary ORELAP accreditation shall not be granted accreditation prior to an acceptable on-site assessment;¶
(c) The laboratory's participation in ~~proficiency testing (~~PT~~)~~ and the obtaining of acceptable PT results according to the TNI Standards and these rules;¶
(d) A quality manual (QM) that includes all elements as set forth in the TNI Standards;¶
(e) Laboratory staff members that meet the TNI Standards for training and experience for their responsibilities within the laboratory;¶
(f) Creation and retention of all records pertaining to samples and analyses, including chain of custody documents, log books, work sheets, raw data, calculations, quality assurance data, and reports according to TNI Standards; and¶
(g) The laboratory's full payment of all appropriate fees as described in OAR 333-064-0060.¶
(5) Laboratories accredited under ORS 475C.560 that fail to obtain licensure with the Commission under ORS 475C.548 in accordance with subsection (5)(a) below, become unlicensed pursuant to subsection (5)(b) below, or otherwise become unlicensed under ORS 475C.548, shall have their accreditation denied or revoked in

accordance with ORS chapter 183.¶
(a) Laboratories that obtain initial accreditation under ORS 475C.560 must submit a complete application for licensure under ORS 475C.548 with the Commission within 30 days of accreditation by ORELAP and must obtain licensure within six months of accreditation. The Oregon Health Authority (Authority) may in its discretion extend the time to obtain initial licensure under ORS 475C.548 by six months upon request by an accredited laboratory due to processing delays with the Commission that are outside the control of the laboratory.¶
(b) The Authority shall deny accreditation renewal or revoke accreditation of a laboratory that surrenders its license, has its license revoked, is denied renewal of its license, or is otherwise no longer licensed under ORS 475C.548. ¶
(6) Laboratories accredited under ORS 475A.606 that fail to become licensed by the Oregon Psilocybin Services program under ORS 475A.594 in accordance with subsection (6)(a) below, become unlicensed pursuant to subsection (6)(b) below, or otherwise become unlicensed under ORS 475A.594, shall have their accreditation denied or revoked in accordance with ORS chapter 183.¶
(a) Laboratories that obtain initial accreditation under ORS 475A.606 must submit a complete application for licensure under ORS 475A.594 with Oregon Psilocybin Services within 30 days of accreditation by ORELAP and must obtain licensure within six months of accreditation. The Authority may in its discretion extend the time to obtain initial licensure under ORS 475A.594 by six months upon request by an accredited laboratory due to processing delays with Oregon Psilocybin Services that are outside the control of the laboratory.¶
(b) The Authority shall deny accreditation renewal or revoke accreditation of a laboratory that surrenders its license, has its license revoked, is denied renewal of its license, or is otherwise no longer licensed under ORS 475A.594.
Statutory/Other Authority: ORS 448.150, 448.131, 448.280, 438.605, 438.610, 438.615, 438.620, 475C.544, 475C.560, 475A.590, 475A.606
Statutes/Other Implemented: ORS 448.280, 438.605, 438.610, 438.615, 438.620, 475C.544, 475C.560, 475A.590, 475A.606

AMEND: 333-064-0100

NOTICE FILED DATE: 05/30/2024

RULE SUMMARY: Amend OAR 333-064-0100

This rule is being amended to include requirements previously shared with the regulated community via OLCC Guidance Bulletin CE2023-01 on how to homogenize pre-rolled usable marijuana and finished inhalable cannabinoid products that include a filter or tip. Another amended section describes required use by cannabis testing laboratories of internal amplification controls in microbiological Polymerase Chain Reaction (PCR) methods. This requirement was already in rule for psilocybin testing laboratories. Clarification has also been added that permission to deviate from the TNI Standard must be granted in writing by the Oregon Health Authority.

The rule is also being amended to specify a minimum sample material retention time for cannabis testing laboratories. Other small housekeeping updates to this rule include removing references to effective date of previous rule changes, removing reference to revision number 4.0 of the ORELAP sampling protocol for usable marijuana due to its sunset on July 1, 2022, and removal of revision 4.0 from the ORELAP-SOP-001 attachment to the rule, and typographical corrections.

CHANGES TO RULE:

333-064-0100
Cannabis Sampling Procedures and Testing ¶

(1) For purposes of this rule the definitions in OAR 333-007-0310 apply unless the context indicates otherwise.¶
(2) Sampling.¶
(a) A laboratory must have and follow marijuana item and industrial hemp-derived vapor items sampling policies and procedures, accredited by the Oregon Environmental Laboratory Accreditation Program (ORELAP), that:¶
(A) Ensure sampling will result in a sample that is representative of the batch being sampled.¶
(B) Require sampling and laboratory personnel to document and collect any information necessary for compliance with these rules, OAR chapter 333, division 7, and any applicable TNI ~~s~~Standards.¶
(C) Require chain of custody procedures consistent with TNI EL Standard V1M2 5.7 and 5.8.¶
(D) Are appropriate to the matrix being sampled.¶
(E) Are consistent with OAR 333-007-0360 and 333-007-0370 and the following ORELAP sampling protocols approved by the accrediting body, incorporated by reference:¶
(i) Usable Marijuana: ~~until July 1, 2022 ORELAP-SOP-001 Rev 4.0 and on or after July 1, 2022~~ ORELAP-SOP-001 Rev 4.1; and¶
(ii) Concentrates, Extracts, Products, and Industrial Hemp-derived Vapor Items: ORELAP-SOP-002 Rev 4.3.¶
(F) Ensure that only the finished cannabinoid concentrate, extract, product, or industrial hemp-derived vapor item is sampled if testing on the finished cannabinoid concentrate, extract, product, or industrial hemp-derived vapor item is required under OAR 333-007-0330 and OAR 333-007-0340.¶
(G) Contain training and education requirements for sampling personnel.¶
(b) Sampling policies and procedures must be accredited by ORELAP prior to any marijuana or industrial hemp-derived vapor item samples being taken.¶
(c) Laboratory personnel that perform sampling must:¶
(A) Comply with the laboratory's accredited sampling policies and procedures.¶
(B) After taking samples:¶
(i) Document the samples in accordance with subsection (2)(e) of this rule; and¶
(ii) If sampling for a licensee or a registrant required to comply with Cannabis Tracking System (CTS) tracking under ORS 475C.871, record the sampling and transfer information in the ~~Commission's~~Oregon Liquor and Cannabis Commission's (Commission's) seed to sale system, as required by the ~~Authority~~Oregon Health Authority (Authority) and the Commission; and¶
(C) Take care while sampling to avoid contamination of the non-sampled material. Sample containers must be free of analytes of interest and appropriate for the analyses requested.¶
(D) Take sample increments that are representative of the batch being sampled.¶
(d) A sufficient sample size must be taken for analysis of all requested tests and the quality control performed by the testing laboratory for these tests.¶
(e) A laboratory must comply with any recording requirements for samples and sample increments in the

accredited policies and procedures and at a minimum:¶
(A) Record the location of each sample and sample increment taken.¶
(B) Assign a field identification number for each sample, sample increment, duplicate sample, and replicate sample that have an unequivocal link to the laboratory analysis identification.¶
(C) Assign a unique identification number for the test batch in accordance with OAR 333-007-0370 and TNI ~~EL s~~Standard requirements.¶
(D) Have a documented system for uniquely identifying the samples to be tested to ensure there can be no confusion regarding the identity of such samples at any time. This system must include identification for all samples, sample increments, preservations, sample containers, tests, and subsequent extracts or digestates.¶
(E) Place the laboratory identification code as a durable mark on each sample container.¶
(F) Enter a unique identification number into the laboratory records. This number must be the link that associates the sample with related laboratory activities such as sample preparation. In cases where the sample collector and analyst are the same individual, or the laboratory pre-assigns numbers to sample containers, the unique identification number may be the same as the field identification code.¶
(f) Combining sample increments.¶
(A) Sample increments collected from the same batch of usable marijuana shall be combined into a single sample by a laboratory prior to testing.¶
(B) Prior to testing, a laboratory shall combine sample increments from a batch of a cannabinoid concentrate, extract, finished inhalable cannabinoid product, or industrial hemp-derived vapor item;¶
(i) Increments from a primary sample are combined into a single sample.¶
(ii) Increments from a duplicate sample are combined into a single sample.¶
(iii) Increments from each replicate sample are combined with each other but material from separate replicate samples are not combined.¶
(C) Prior to any testing or subsampling except as described in subparagraph (i) of this paragraph, the entire combined sample except as described in subparagraph (iii) of this paragraph must undergo the laboratory's homogenization process.¶
(i) If the homogenization process would invalidate the analysis for a required test, the laboratory must utilize a subsampling procedure to withdraw a portion of the sample prior to homogenization for the required test. Testing that would be invalidated by the homogenization process includes but is not limited to, cryogenic sterilization of the sample prior to microbiological analysis.¶
(ii) Potency analysis shall not be performed on material subsampled prior to homogenization steps.¶
(iii) Prior to homogenization, the filter or tip shall be removed from pre-rolled usable marijuana and finished inhalable cannabinoid products that include a filter or tip. Filters and tips are considered non-consumed packaging and are not part of the sample for the purpose of homogenization or testing. All other components of the pre-roll must be included in the laboratory's homogenization process. ¶
(D) Sample increments and samples collected from different batches may not be combined, except as permitted by OAR 333-007-0360 ~~and on or after July 1, 2022~~ using the process described in ORELAP-SOP-001 Rev. 4.1.¶
(E) Portions of homogenized samples that are combined for testing as permitted by OAR 333-007-0360(1)(c) shall use the laboratory's formal subsampling method to produce a proportionally representative sample from each batch involved.¶
(g) Sample replicate analysis for concentrates, extracts, finished inhalable cannabinoid products, and industrial hemp-derived vapor items.¶
(A) When replicate samples are required per OAR 333-007-0360 Exhibit B Table 7, each sample shall be analyzed individually for residual solvents as described in OAR 333-007-0410 and adult use cannabinoids and CBD as described in OAR 333-007-0430.¶
(B) Two samples shall be randomly selected according to the laboratory's policy and procedures and analyzed individually for the remaining required analyses as described in OAR 333-007-0330, 333-007-0341, and 333-007-0342.¶
(C) Adult use cannabinoid and CBD results for the batch or process lot are reported as the calculated average result from all sample replicates.¶
(3) Compliance testing validity.¶
(a) When testing a sample for the required microbiological compliance tests as described in OAR 333-007-0390, a laboratory must comply with additional method validation as follows:¶
(A) Run a negative control in accordance with TNI Standard requirements to demonstrate the procedure is free of microbiological contaminants that prevent accurate testing.¶
(B) Run a positive control in accordance with TNI Standard requirements to demonstrate acceptable performance of the procedure. Acceptable performance of the positive control means accurate identification and quantitation of all ~~regulated analytes~~organisms required in OAR 333-007-0390.¶
(C) Demonstrate acceptable performance as described in the manufacturer's instructions of an internal

amplification control (IAC) in each sample analyzed by a polymerase chain reaction PCR method. In the case of a positive result for a required organism in a sample with an unacceptable performance of the IAC, follow the manufacturer's instructions on interpretation of acceptability.¶
(b) When testing a sample for the required chemistry compliance tests as described in OAR 333-007-0400 to 333-007-0415 and 333-007-0425 to 333-007-0430, a laboratory must comply with additional method validation as follows:¶
(A) Run a method blank in accordance with TNI Standard requirements to demonstrate the procedure is free of contaminants at or above the limit of quantitation.¶
(BA) Run a laboratory control standard (LCS) in accordance with TNI Standard requirements to demonstrate acceptable performance of the procedure. Acceptable performance of the LCS means percent recovery for all regulated analytes are within the limits specified in Exhibit C, Table 1.¶
(CB) Analyze duplicate samples with a precision limit of 10 percent relative percent difference (RPD), if duplicates are required as specified in OAR 333-007-0360 and analyze all replicate samples with a precision limit of 10 percent relative standard deviation (RSD), if replicates are required as specified in OAR 333-007-0360.¶
(4) Calculating total delta-9 THC and total CBD.¶
(a) Total delta-9 THC must be calculated as follows, where M is the mass or mass fraction of delta-9 THC or delta-9 THCA:¶
M total delta-9 THC = M delta-9 THC + 0.877 * M delta-9 THCA.¶
(b) Total CBD must be calculated as follows, where M is the mass or mass fraction of CBD and CBDA:¶
M total CBD = M CBD + 0.877 * M CBDA.¶
(c) Each test report must include the results for delta-8 THC, total delta-9 THC, and total CBD.¶
(5) Report delta-8 THC, total delta-9 THC, and total CBD for useable marijuana as Ddry Wweight. A laboratory must analyze the sample as received and report delta-8 THC, total delta-9 THC and total CBD content by dry weight calculated as follows:¶
P delta-8 THC(dry) = P delta-8 THC(wet) / [1-(P moisture/100)]¶
P total delta-9 THC(dry) = P total delta-9 THC(wet) / [1-(P moisture/100)]¶
P total CBD(dry) = P total CBD(wet) / [1-(P moisture/100)]¶
(6) Calculating RPD and RSD.¶
(a) A laboratory must use the following calculation for determining RPD:¶
Relative Percent Difference¶
%RPD=|(sample-duplicate)|/((sample+duplicate)/2) * 100¶
(b) A laboratory must use the following calculation for determining RSD:¶
Standard Deviation¶
S= (((xi-x)^2)/(n-1))¶
Relative Standard Deviation¶
%RSD= (S/x)* 100¶
(c) For purposes of this section:¶
(A) S = standard deviation.¶
(B) n = total number of values.¶
(C) xi = each individual value used to calculate mean.¶
(D) x = mean of n values.¶
(d) For calculating both RPD and RSD if any results are less than the Limit of Quantitation (LOQ) the absolute value of the LOQ is used in the equation.¶
(e) The laboratory shall not substitute the LOQ for individual components of a totaled result, such as total delta-9 THC or total Hexanes, in the calculation of the totaled result for the purpose of calculating RPD or RSD.¶
(7) Tentative Identification of Unknown Compounds.¶
(a) If a laboratory is using a gas chromatography mass spectrometry instrument for analysis when testing cannabinoid concentrates, extracts, or industrial hemp-derived vapor items for solvents the laboratory shall have an established procedure for achieving tentative identification of unknown compounds in the sample. A tentatively identified compound (TIC) means an unknown chromatographic peak that is neither included in the list of analytes the laboratory is testing for nor expected to be naturally found in cannabis concentrates, extracts, or industrial hemp-derived vapor items such as terpenes.¶
(b) Tentative identification is achieved by searching NIST 2014 or an equivalent database (>250,000 compounds). Match scores for background subtracted or deconvoluted spectra should exceed 90 percent compared to the database library spectrum.¶
(c) Upon written request from the overseeing agency of the licensee or registrant, a laboratory shall report to the licensee or registrant and the Authority or the Commission all tentatively identified compounds (TICS) that meet the identification criteria in subsection (7)(b) of this rule.¶
(A) TIC quantitation is estimated by comparing analyte area to the closest internal standard area and assuming a

response factor (RF) =1.¶
(B) If a laboratory does not use internal standards, TICs shall be reported as "detected" and apparent relative concentration shall be judged based on peak area.¶
(8) A laboratory must provide:¶
(a) Any pesticide test result to the Department of Agriculture (Department) upon that agency's request.¶
(b) A sample or a portion of a sample to the Department ~~of Agriculture~~ upon that agency's request, document the chain of custody from the laboratory to the Department, and document that the sample or portion of the sample was provided to the Department in the Commission's seed to sale tracking system.¶
(9) A laboratory performing tests for a licensee or a registrant required to use CTS under ORS 475C.871 must enter any information required by the Commission or the Authority in CTS.¶
(10) A laboratory performing tests for a registrant must comply with the documentation requirements in OAR 333-007-0370 and must maintain the documentation required in these rules for at least three years and provide that information to the Authority upon request.¶
(11) The Authority may, in its discretion, permit a laboratory to deviate from the TNI Standard~~s~~ in order to comply with OAR 333-007-0300 to 333-007-0~~5~~600 and these rules based on the state's needs. Permission to deviate from the TNI Standard must be in writing from the Authority.¶
(12) A laboratory must be able to demonstrate that its limit of quantitation (LOQ) for all matrices in compliance testing is:¶
(a) Less than or equal to one-half of any action level established in OAR 333-007-0400, 333-007-0410, 333-007-0415, and 333-007-0425 Exhibit A, Tables 3, 4, 8, and 9 except for as outlined in 333-064-0110(6); and¶
(b) For total delta-9 THC concentration less than or equal to 0.15 percent; and¶
(c) For delta-8 THC concentration less than or equal to 0.15 percent.¶
(13) Non-compliance testing. A laboratory that conducts a quality control or research and development test for a registrant or licensee may use methods not approved by the Authority, but the laboratory may not identify those test results as accredited results.¶
(14) Sample material retention. A laboratory shall retain all of the unused sample material for a minimum of 30 calendar days after reporting results into the CTS.
Statutory/Other Authority: ORS 438.605, 438.610, 438.615 ~~& 438.620~~, 475C.544, 475C.560, 438.620
Statutes/Other Implemented: ORS 438.605, 438.610, 438.615 ~~& 438.620~~, 475C.544, 475C.560, 438.620

RULE ATTACHMENTS MAY NOT SHOW CHANGES. PLEASE CONTACT AGENCY REGARDING CHANGES.

ORELAP

Oregon
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Protocol for Collecting Samples of Usable Marijuana

ORELAP-SOP-001 Rev. 4.1

ORELAP Executive Board and Program Approval Signatures:

Lori Pillsbury, DEQ LEAD Division Administrator — Date: 03/10/2022

John Fontana Ph.D., Oregon State Public Health Laboratory Director — Date: 03/09/2022

Kathleen Wickman, ODA Laboratory Manager — Date: March 8, 2022

Travis Bartholomew, ORELAP Program Manager — Date: 3/08/2022

Sara Krepps, ORELAP QA Officer — Date: 03/10/2022

I. Table of Contents

Acknowledgements

Version 1.0 of this document was authored by the Cannabis Sub-Committee with input from Technical Experts and approved by the ORELAP Executive Board. See Revision 2.0 for original committee authorship. Revision 3.0 was authored and reviewed by NEFAP/GLP sampling experts. Revision 4.0 was authored by Steven Jetter and reviewed by Travis Bartholomew and the ORELAP Executive Board.

I. Introduction and Scope

Obtaining a representative sample from a larger batch is one of the key elements of accurate laboratory analysis. Laboratories collect representative samples by consistently using standard sampling methods and equipment, preventing contamination of the sample, and maintaining the sample identity within the batch. The laboratory must consistently use documented standard sampling practices, tools, and methods. These practices, tools, and methods must be appropriate for the matrix. If proper protocols are in place and adhered to for sample collection, the laboratory analysis of the sample should reflect the composition of the batch as a whole at the time the sampling occurred, within recognized tolerances.

This protocol is for use by ORELAP-accredited laboratories performing cannabis sampling as defined in OAR 333-064-0025. For the purposes of this protocol, cannabis sampling also includes activities related to obtaining a representative sample of post-harvest cured hemp material but excludes activities related to pre-harvest sampling of hemp material. The protocol focuses on standard and correct sampling practices that should be reflected in a laboratory's own sampling policies and procedures.

II. Records and Documentation

ORELAP-accredited laboratories shall maintain standard operating procedures (SOP) that accurately reflect current sampling activities.

1. The laboratory's SOP shall be readily accessible to all pertinent personnel.
2. The laboratory's SOP shall clearly indicate the effective date of the document, the revision number, and the signature of the approving authority.
3. The laboratory's SOP should use this protocol as minimum requirements and must include additional detail specific to laboratory procedures. In cases where the published method (this protocol) has been modified or where the referenced method (this protocol) is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described in the laboratory's SOP. Any changes to the laboratory's protocol, including use of a selected option, shall be documented and included on the laboratory's sampling form.
4. All documents shall be controlled and retained in accordance with the TNI Environmental Laboratory standard as defined in 333-064-0025.

ORELAP-accredited laboratories shall maintain sampling plans.

1. The laboratory's sampling plans shall be made available at their location of use.
2. The laboratory's sampling plans shall be based on appropriate statistical methods and shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch.
3. Any deviation from or addition to the laboratory's sampling plan must be documented in detail and shall be included in the final report. The standardized or generic sampling plans can be included in the SOP however specialized client requests or products may require additional information.
4. The laboratory's sampling plans shall document the date and time of sampling.

III. Client Contracts; Client Sampling and Testing Requests

The laboratory must have a sampling contract with a client that includes at least the following:

1. A test order containing the information required by OAR 333-007-0315
2. A site-specific sampling plan or process specific sampling plan that uses statistical design for each project to provide representative sampling.

IV. Planning

Prior to beginning the sampling procedure, the sampler shall survey the site to identify the conditions under which the Usable Marijuana is being kept, as this will determine the sampling plan. In cases where Usable Marijuana will be sold or transferred to a processor or processing site, analysis may occur prior to the drying and curing steps. All sampling must be performed by personnel employed by an ORELAP accredited laboratory and must be in accordance with OAR 333-007-0360 and OAR 333-064-0100.

The testing requirements for Usable Marijuana are in OAR 333-007-0320. The requirements for sampling and minimum sample size are in OAR 333-007-0360 and Appendix 2. Per Authority or Commission request or client request, additional analyses may be required and must be considered in the planning process.

To ensure representativeness, the sampling plan must be designed such that each flower bud, leaf, or other portion in the batch has an equal chance of being selected. **The sample size must be sufficient to complete all analyses required but shall in no case be less than 0.5% of the weight of the batch. The maximum batch size is 50.0 lbs or 22.68kg**.

V. Sampling Design and Plans

1. Sampling plans shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch. Standardized Sampling Plans can be included in the SOP however specialized client requests or products may require additional information. Any deviation from or addition to the sampling plan must be documented in detail and shall be included in the final report.
2. Sampling plans shall be designed to meet specified sample quality criteria. This includes using a sampling plan that meets a 95% confidence level for representative sampling and limits the fundamental sampling error. The most common way to achieve this is by increasing the number of sample increments from the minimum required to compensate for normal batch heterogeneity.
3. Sampling plans must ensure that adequate sample mass is collected for all analyses requested by the producer. This must include adequate sample mass for re-testing in the event a sample fails a criterion as well as adequate sample mass for any quality control samples required by the laboratory, such as duplicates or matrix spikes.
4. A sampling plan must include at a minimum:
    a. Shape, size, and number of container(s) holding the batch from which sample increments will be collected;
    b. Number of sample increments to be collected;
    c. Minimum weight or mass of each sample increment;
    d. Location of where sample increments will be taken within each container holding the

batch. See Appendix 2 for information on random selection of locations.

5. The laboratory must have details in its SOP or a sampling plan, from appropriate industry reference where possible, on how it will achieve random sampling in an unclear decision unit.

VI. Sampling Equipment and Supplies

1. A laboratory should, at a minimum, have the following equipment and supplies for sampling:
    a. Sampling equipment such as spoons, spatulas, transfer pipettes, or other matrix specific tools
    b. Tongs
    c. Corers
    d. Teri-wipes or equivalent
    e. Field balance (capable of 0.01 g measurements)
    f. Calibrated verification weights appropriate to verify accuracy of field balance
    g. Cleaning supplies – solvent, bleach, 70% Ethanol
    h. Gloves (powder-free, nitrile, sterile)
    i. Mylar bags (for final sample transport and storage) or amber glass jars (for final sample transport and storage)

2. Cleaning of Field Sampling Equipment
    a. Field sampling equipment shall be certified clean prior to use by the laboratory.
    b. Cleaning techniques will vary depending upon the desired analysis.
    c. In general, sampling equipment must be sterile for microbiology samples and clean for chemistry samples.
    d. The laboratory shall perform cleanliness checks on each batch of sampling equipment prior to taking that equipment into the field.
    e. Results from cleaning procedure tests must be below the reporting limit of the target analyte(s) for the associated analyses.
    f. If cleanliness checks fail, the sampling equipment must be re-cleaned, sterilized and tested.

3. Field balance calibration verification
    a. The laboratory sampling technician shall verify the calibration of the field balance at the sampling location.
    b. When multiple sampling events occur on the same day, the balance calibration shall be verified at each sampling location.
    c. Balance calibration verifications shall be documented.

VII. Procedures for Sampling Usable Marijuana

1. Locate the batch to be sampled. The sampler **must** have access to entire batch.
2. Check for any signs of non-uniformity within the batch and document the same.
    a. Some obvious indicators may be different types or sizes of containers, variations in marks and labels, or mixed batch numbers.
    b. During sampling, the sampler shall look for differences in the usable marijuana being sampled such as color, shape, size, and treatment.

c. By definition, the batch must be uniform for all factors that appear on the label; hence, variations in the product may indicate non-uniformity in the batch and that any sample drawn may not be representative for testing.

d. The sampler shall note these anomalies in the sample collection report.

e. Some batches may include more than one Metrc package associated with different strains or different types of material from the same strain where allowed in OAR 333-007-0360 (1) (c).

i. In the situation described in (e) above, sampling occurs by each Metrc package number composed of material that appears to be substantially similar in appearance and quality. Examples of materials that are not substantially similar include but are not limited to flower buds versus leafy stem material or "A buds" versus "B buds".

ii. Combining multiple Metrc packages shall occur in the laboratory AFTER sample homogenization steps are completed as required by OAR 333-064-0100 (2) (f).

3. Review the container label information for harvest lot number, producer, and other pertinent information. Each harvest lot must be separated into batches of 50.0 lbs. or less and must be assigned a unique batch number by the grower. Do not sample if a unique batch number is not available.
    a. The batch shall be presented for sampling in containers holding no greater than 15.0lbs.
4. Determine the number of containers in the batch and the batch size. Visually verify the batch size for each container and confirm batch weight with client. Do not sample if the batch size is unavailable or exceeds 50.0 lbs.
5. Determine the number of containers from which sample increments must be collected (Appendix 2).
6. Select the appropriate sampling tool to ensure that it reaches all portions of the container.
7. Sampling tool and other instruments like field balance must be clean prior to use to prevent cross-contamination of sample increments. Sampling tools which appear to be dirty or otherwise compromised shall not be used.
    a. To prevent contamination, sampling tools may be cleaned and sealed at the laboratory prior to use or may be cleaned in the field between batches using an appropriate solvent and decontaminant to prevent cross contamination of batches during sampling.
8. Results from cleaning procedure tests must be below the reporting limit of the target analyte(s) for the associated analyses.
9. Decontamination waste must be collected and properly disposed of if not used for analysis.
    a. Samplers must take extreme care if sampling from multiple sites in one day to ensure contaminants, pathogens, or organisms are not transferred between facilities. The sampler may clean sampling equipment in the field between samplings at a single facility. However, the sampler shall bring enough sets of sampling equipment to use a new set at each facility visited.
    b. All field equipment shall be returned to the laboratory following sampling and cleaned according to the laboratory's procedures or discarded.
    c. Where aseptic technique is required, samplers shall observe best practices to prevent microbiological contamination of samples. For an example of aseptic technique, see the FDA Aseptic Sample guidelines (Investigations Operations Manual Subchapter 4.3.6).
10. Visually inspect each test sample increment to assess uniformity. If non-uniformity is identified, record observation in the sampling report.

11. When collecting sample increments, approximately equal amounts of product are to be taken with each probing and from each container. Care must be taken by the sampler to not damage the portion of the product which is not being collected. Laboratory should refrain from sampling a batch from containers that because of their shape make it impossible to collect sample increments from all locations within the container. This includes subsurface or internal layers.
12. Weigh each sample increment, document weight on sampling report form, along with location sample increment was taken.
13. Combine all sample increments to form the composite sample.
14. Ensure sufficient sample increments are taken to meet sample size requirements for all analytical method(s) being performed.
15. Seal and label the composite sample with the following minimum requirements:
    a. Laboratory license number
    b. Unique identifier for sampling event
    c. Sampling date and name of sampler
    d. Producer's license or registration number
    e. Harvest lot and batch numbers
    f. Label "PRODUCT NOT TESTED" in bold capital letters in minimum 12-point font.
16. Apply a custody seal to the sample container in a manner which prevents the product from being tampered with or transferred prior to testing. This seal may contain the laboratory sample identification number.
17. Complete the sampling report while at the sampling location as well as an appropriate chain of custody form as outlined in the standards of accreditation.
18. Forward the sample and sampling report to the laboratory or other designated location using packaging appropriate for secure and timely transport.
19. Record the sampling event in the OLCC seed to sale system under the licensee number for recreational marijuana or record in the laboratory's records the registrant number for tracking medical marijuana.

VIII. Sampling Records/Field Data

1. At the time samples are collected the sampler must complete a sampling report form for each batch sampled. Sample report forms must include at a minimum the following information:
    a. Name and address of producer including licensee or registrant number;
    b. Product type.
    c. Total weight of batch.
    d. Unique laboratory batch ID#, Metrc batch ID #, and/or OHA batch ID#.
    e. Total number of containers sampled.
    f. Number of sample increments taken from each container.
    g. Number of sample containers collected.
    h. Weight and location of each sample increment.
    i. Total weight sampled.
    j. Sampling plan ID and revision date.
    k. Sampling Procedure ID and revision date.
    l. Description of equipment and tools used.
    m. Address where sampled.
    n. Date sampled.
    o. ORELAP Laboratory Identification number.

   p. Lab License Number.
   q. Sampler's identification and/or signature.
   r. Name of responsible party for the batch and transport information.
   s. Receiving laboratory and types of tests required or requested.
2. A chain of custody form must be used unless the laboratory is sampling for a client that is required to use Metrc. A chain of custody form must include at least the following information:
   a. Sampler's name
   b. Sample Identification (Lab ID number) if assigned before arrival at laboratory
   c. Sampling Date/Time
   d. Weight and location of increment samples
   e. Final weight of composite sample
   f. Custody transfer signatures
   g. Custody Transfer Dates/Times
3. If any of the above information requested on the sampling report form is unavailable, indicate "N/A" in the appropriate space with an explanation as to why the information is not available.
4. All sampling report forms must be signed by the sampler.

IX. Transportation and Handling of Samples

1. Samples must be transported to the laboratory performing the analysis by the most expedient, secure, and legal means to ensure that the sample continues to be representative of the harvest lot sampled and the chain of custody form continues to document sample integrity. Transportation must be done in compliance with OAR 845-025-5060. Note: The existing regulation does not permit shipping in any form such as USPS or FedEx.
2. Containers for sample transport must be designed to protect the sample from moisture and temperature extremes and to prevent damage, contamination, spillage, or commingling of the sample during transport. The required container for sampling is a glass, amber jar with a PTFE-lined lid or a Mylar bag. A tamper-proof seal is required and must be marked with the sampler's name, date, and sample number.
3. The laboratory must have detailed procedures on maintaining custody and sample integrity during transport. These procedures should take into consideration controlling temperature and other environmental factors.
4. Submit the composite sample to the laboratory in its entirety. In a situation where the composite sample must be split for analysis by two different laboratories, for example when pesticide analysis is subcontracted to another laboratory, the composite sample shall be homogenized by the primary laboratory using the laboratory's approved sample homogenization process prior to subsampling. This shall be reflected on the chain of custody.
5. Composite samples must always be identified by labeling or marking the sample container to associate them with the batch from which they originated and with the sampling report.

X. Quality Assurance and Quality Control

X.1 Sampler Qualifications

1. Basic qualifications for samplers of usable marijuana are:

a. Physically able to perform the duties of a sampler;
b. No conflict of interest;
c. Employed by an ORELAP accredited laboratory
d. Pass initial and ongoing demonstrations of capability as defined by the laboratory (see below);
e. Licensed under state law to transport the required quantity of usable marijuana items

2. Education and training for samplers:
    a. Initial training: training shall include principles, procedures, and policies of sampling; Initial training must be performed by an Instructor that has demonstrated competency in performing the sampling methods referenced or equivalent. After personnel goes through initial training, they are qualified to train others in their organization.
    b. Field or on-the-job training: 8-hours of training on various sampling techniques;
    c. Continuing education: periodic refresher training shall be done annually.

X.2 Demonstration of Capability

Prior to acceptance and institution of any accredited method, a satisfactory initial demonstration of capability (IDOC) is required. The laboratory shall have a documented procedure for performing the IDOC. The IDOC will be repeated: 1) every time there is a change in personnel or method, and, 2) when the method has not been performed by the laboratory or sampler within a 12-month period.

This procedure shall employ one of the following approaches to demonstrating capability:

1. Comparison of replicate samples within a defined Relative Standard Deviation (%RSD)[1].
2. Comparison of a sample collected to that of one collected by personnel with an existing IDOC within a defined RPD.

Thereafter, ongoing continuing demonstration of capability (CDOC) is required annually. The laboratory shall have a documented procedure for performing the CDOC. The laboratory shall retain documentation verifying CDOC for each sampler and make this documentation available to ORELAP upon request.

X.3 Field QC Samples

1. Duplicates
    a. Duplicates are recommended for any Usable Marijuana sampling event, but not required. The Duplicate must be collected using the same procedure and contain the same number of sample increments as the Primary Sample. The lab must have documentation of the client request for a Duplicate with any client specified Quality objectives and precision limits must meet the client's need.

2. Equipment Blanks
    a. Equipment rinse blank samples provide a QC check on the potential for cross contamination by measuring the effectiveness of the decontamination procedures on the sampling equipment. An equipment blank is required to validate equipment cleaning procedures for all required analyses. It is recommended but not required that an

[1] Standard Methods 20th Edition; 1020 B Quality Control, 11. QC Calculations, a. Initial Calibration.

equipment blank is collected upon each sampling event to demonstrate the equipment was not introduced to contamination after cleaning.

b. The equipment rinse blank samples consist of analyte-free matrix, as applicable, rinsed across sample collection and processing equipment. If the analytes of interest are detected in the equipment rinse blank samples, the detected concentrations will be compared to the associated sample results to evaluate the potential for contamination.

c. The Equipment Blank must pass the required analysis at <LOQ for cleaning validation.

d. If the Equipment Blank is collected at the sampling event, the lab must have detail in the sampling plan or procedures as to how to evaluate it and what actions to take if the evaluation demonstrates unacceptable results.

X.4 Field Audits

1. The laboratory shall adopt an ongoing system for performing audits of field activities. Field audits must be conducted periodically and in accordance with a predetermined schedule and procedure. The goal of the field audit is to verify that the sampling operation continues to comply with the requirements of the regulations and is being performed according to the laboratory's sampling SOP. Audits are to be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. The field audit shall address all elements of the sampling activities and shall be documented.
2. When field audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the field sampling activities, the associated laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that test results may have been affected. Laboratory management shall have a policy that specifies the time frame for notifying clients of events that cast doubt on the validity of the results. Follow up audit activities shall verify and document the implementation and effectiveness of any corrective actions taken as a result of the field audit.
3. Required components of the Field Audit program:
    a. Review sampling and performance records from the preceding year for deficiencies in the application of sampling protocol;
    b. Observe the sampler conducting sampling procedures;
    c. Record any deficiencies and initiate corrective action.

XI. References

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Oregon Administrative Rules, *Marijuana Labeling, Concentration limits, and Testing,* Chapter 333, Division 7.

Oregon Administrative Rules, *General Requirements Applicable to all Marijuana Licensees,* Chapter 845, Division 25.

Standard Methods 20th Edition (1998); 1020 Quality Assurance

Appendix 1 Definitions

**** If there are any inconsistencies between the definitions below and the definitions in OAR 333, Divisions 7 and 64, the definitions in the rules take precedence.**

Authority means Oregon Health Authority

Batch means a quantity, not to exceed 50.0 pounds or 22.68 kilograms, of marijuana or usable marijuana from a harvest lot.

Chain of Custody Form means a form completed by laboratory personnel that documents the collection, transport, and receipt of samples by the laboratory. (Sample tracking document)

Commission means the Oregon Liquor Control Commission.

Composite sample means a sample containing all sample increments taken from a batch.

Container means a sealable, hard- or soft-bodied receptacle in which a marijuana item is placed during sampling, transport, and storage; or a physical division into which a marijuana batch is placed for random and representative sampling.

Decision Unit (DU) means the material from which the primary sample(s) is collected and to which the inference(s) is made.

Duplicate Sample means sample increments taken in an identical manner to sample increments taken for the primary sample and representative of the same marijuana item being sampled that is prepared and analyzed separately from the primary sample.

Equipment Blank means a sample of analyte-free media, collected after decontamination and prior to sampling, which has been used to rinse the sampling equipment after cleaning to validate the cleaning procedure or between sampling batches to demonstrate lack of contamination.

Fundamental Sampling Error (FSE) means a measure of the compositional heterogeneity of the batch, which is controlled through the collection of sufficient sample mass (mass is inversely proportional to error).

Harvest Lot means a specifically identified quantity of marijuana that is cultivated utilizing the same growing practices, harvested within a 7 calendar day period at the same location, and cured under uniform conditions.

Heterogeneity means the state or quality of being heterogeneous.

Heterogeneous means non-uniform or consisting of dissimilar parts or components.

Homogeneous means of a uniform composition and with similar properties throughout a batch of useable marijuana; means a cannabinoid product, concentrate, or extract has uniform composition and properties throughout each process lot.

Label means a tag or other device attached to or written, stamped, or printed on any container or accompanying any batch in bulk stating all required batch information.

Laboratory means a laboratory that is accredited under ORS 438.605 to 438.620 to sample or conduct tests on marijuana items and licensed by the Oregon Liquor Control Commission under ORS475B.560.

Marijuana means the plant Cannabis family Cannabaceae, any part of the plant Cannabis family Cannabaceae and the seeds of the plant Cannabis family Cannabaceae. This does not include industrial hemp, as defined in ORS 571.300.

Marijuana item means marijuana, usable marijuana, a cannabinoid product or a cannabinoid concentrate or extract.

Metrc means the state-administered cannabis tracking system (CTS).

ORELAP means the Oregon Environmental Laboratory Accreditation Program administered by the Authority pursuant to ORS 438.605 to 438.620.

Primary Sample means a composite sample composed of sample increments and tested for the required analysis methods.

Producer means a person licensed by the Commission under ORS 475B.070 or a grower registered by the Authority under ORS 475B.810.

Registrant means a grower, marijuana processing site, or a medical marijuana dispensary registered with the Authority under ORS475B.810, 475B.840, or ORS 475B.858.

Relative Percent Difference means comparing two quantities while taking into account the size of what is being compared. If the final result (i.e. Total THC) is <LOQ in either sample, the absolute value of the LOQ is used in the equation.

$$\%RPD = \frac{|(sample - duplicate)|}{(sample + duplicate)/2}\ x\ 100$$

Relative Standard Deviation means the standard deviation expressed as a percentage of the mean recovery, i.e., the coefficient of variation multiplied by 100. If the final result (i.e. Total THC) is <LOQ in either sample , the absolute value of the LOQ is used in the equation.

Standard Deviation

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

Relative Standard Deviation

$$\%RSD = \frac{S}{\bar{x}}\ x\ 100$$

S = standard deviation.
n = total number of values.
x_i = each individual value used to calculate mean.
$\bar{x}$ = mean of n values.

Representative Sample means a sample obtained according to an incremental sampling procedure designed to ensure that the different parts of a batch or lot or the different properties of a batch or lot are proportionally represented.

Sample means an amount of a marijuana item collected by laboratory personnel from a registrant or

licensee and provided to a laboratory for testing.

Sample Increment means an amount of a marijuana item collected by laboratory personnel from a registrant or licensee that is combined into a sample for purposes of testing.

Sample Quality Criteria (SQC) means a series of statements that clarify a sampling program's technical and quality needs to support defensible decisions, including statement of the question to be answered, definition of the decision unit, and the desired confidence in the inference.

Sealed means secured in such a way as to prove authenticity or integrity of the sample.

Sterilization means the removal of all microorganisms and other pathogens from a marijuana item by treating it with approved chemicals or subjecting it to high heat.

TNI Standard: TNI Environmental Laboratory Standard as defined in 333-064-0025.

Usable Marijuana means the dried leaves and flowers of marijuana. Usable Marijuana does not include the seeds, stalks and roots of marijuana or waste material that is a by-product of producing or processing marijuana.

Appendix 2 Sampling Requirements

I. Random Sampling

As specified in the sampling plan, select random sample increments from different locations within a container or set of containers. Laboratories must develop procedures describing how to:

1. Assign location numbers within containers and among a set of containers;
2. Use a random number generator to determine which locations to sample; and
3. Document where each sample increment was sampled from and the volume collected from each increment.

Assign divisions based on the type of container in the site-specific sampling plan. For container types that are greater than four (4) inches deep, divisions must also include a layer or layers beneath the accessible portion of the batch. Use a random number generator with the higher number equal to the number of divisions for the container. When there are multiple containers use existing or arbitrary order of containers to assign numbers to the total of "divisions multiplied by total number of containers" (divisions x # containers = total number of random increments) and record in the sampling report.

The laboratory must have details in its SOP or Sampling Plan, from appropriate industry reference where possible, on how it will achieve random sampling in an unclear decision unit.

II. Sample size

Per OAR 333-007-0360, the sample size must be sufficient to complete all analyses required but shall in no case be less than 0.5% of the weight of the batch. The batch shall be presented for sampling in containers holding no greater than 15.0lbs. Per OAR 333-007-0350, the maximum batch size is 50.0 lbs.

The required sample size for a given batch size based on OAR 333-007-0360 varies depending upon the size of the batch. Example batch sizes and the corresponding sample mass are presented in Table . The laboratory shall calculate the actual minimum required sample size based on the actual batch weight. Taking more sample than the minimum required mass or more increments than the required number of increments is encouraged and will improve representativeness of the sample in relation to the batch.

Table 1 – Examples of required sample size based on size of batch.

Batch size		**Required sample size**	
Pounds	Kilograms	**Pounds (lbs)**	**Grams (g)**
1.0	0.45	0.005	2.3
2.0	0.90	0.010	4.5
3.0	1.36	0.015	6.8
5.0	2.27	0.025	11.4
10.0	4.54	0.050	22.7

Batch size		Required sample size	
Pounds	Kilograms	**Pounds (lbs)**	**Grams (g)**
15.0	6.80	0.075	34.0
20.0	9.07	0.100	45.4
30.0	13.61	0.150	68.1
40.0	18.14	0.200	90.7
50.0	22.68	0.250	113.4

III. Sampling a batch

1. When collecting a primary sample from a batch, a minimum of seven (7) sample increments shall be collected. Collect the sample increments according to the sampling plan or the procedure described in the laboratory's SOP. The procedure used shall ensure that any part or portion of the batch has equal odds of being selected in order to provide a representative sample.
2. As the batch increases in size, it is necessary to collect additional sample increments to make up the primary sample (Table 2).

Table 2 – Minimum number of sample increments for the primary sample based on batch size.

Size of batch (lbs)	≤ 4.0	≤ 8.0	≤ 12.0	≤ 16.0	≤ 20.0
No. of increments	7	8	9	10	12

Size of batch (lbs)	≤ 24.0	≤ 28.0	≤ 32.0	≤36.0	≤40.0
No. of increments	14	16	18	20	24

Size of batch (lbs)	≤44.0	≤48.0	≤50.0
No. of increments	28	32	36

Table 3 – Revision history of this SOP.

Revision	Date	Summary of changes made, and initials of editor
4.0	7/20/2020	Major updates and re-formatting, with input from Scott Hoatson and Department of Justice. Updated: OSPHL address; executive board and ORELAP staff names as needed; definitions in order to match OARs and ORS and arranged in alphabetical order. Added: this table (Revision history); subsection VI.3; additional information about subsampling for subcontracted analyses; mention of assigning layers for sampling deep containers; required calibration verification of field balances. Combined: information in section IX with information from former section X (Forwarding samples to the Primary and/or Retesting Laboratory) and deleted

		former section X and combined former section X.5 with section X.4. Minor updates for consistency and typo fixes. Includes fixing reference to minimum number of sample increments in Table 2. STJ 7/20/2020
4.1		Minor formatting updates, removed historical authorship captured in previous versions. Added to Section I that the protocol also applies to post-harvest cured hemp. Clarification in section IV that the samples must be representative and that increased sampled material improves representativeness. Addition to section VII on how to sample for pooled sampling, explanation on what batch material homogeneity means with descriptions of dissimilar materials, and requirement that batch be presented for sampling in containers holding no more than 15.0 lbs. Removed reference to "field" when discussing duplicate samples in section X.3. Updated definitions of "batch" and "field duplicate" and "harvest lot". Updated Table 1 and Table 2 to reflect increased batch size. STJ 11/17/2021

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Protocol for Collecting Samples of Cannabinoid Concentrates, Extracts, Products, and Industrial Hemp-derived Vapor Items

ORELAP-SOP-002 Rev 4.3

ORELAP Executive Board and Program Approval Signatures:

Lori Pillsbury, DEQ LEAD Division Administrator — Date: 03/10/2022

John Fontana Ph.D., Oregon State Public Health Laboratory Director — Date: 03/08/2022

Kathleen Wickman, ODA Laboratory Manager — Date: March 8, 2022

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Acknowledgements

Version 1.0 of this document was authored by Cannabis Sub-Committee with input from Technical Experts and approved by the ORELAP Executive Board. See Revision 2.0 for original committee authorship. Revision 3.0 was authored and reviewed by NEFAP/GLP sampling experts. Revision 4.0 was authored by Steven Jetter and reviewed by Travis Bartholomew and the ORELAP Executive Board.

I. Introduction and Scope

Obtaining a representative sample from a larger batch is one of the key elements of accurate laboratory analysis. Laboratories collect representative samples by consistently using standard sampling methods and equipment, preventing contamination of the sample, and maintaining the sample identity within the batch. The laboratory must consistently use documented standard sampling practices, tools, and methods. These practices, tools, and methods must be appropriate for the matrix. If proper protocols are in place and adhered to for sample collection, the laboratory analysis of the sample should reflect the composition of the batch as a whole at the time the sampling occurred, within recognized tolerances.

Controlling manufacturing error is the responsibility of the processor of the cannabinoid concentrate, extract or product. Sampling error must be controlled by the laboratory in order to obtain a representative sample of the defined batch. This is accomplished by maintaining the sample identity within the defined batch, prevention of contamination of the sample, and consistent use of standard sampling methods and equipment.

This protocol is for use by ORELAP-accredited laboratories performing finished cannabinoid concentrate or extract, finished cannabinoid product, finished inhalable cannabinoid product, or industrial hemp-derived vapor item sampling as defined in OAR 333-064-0025. It focuses on standard and correct sampling practices that should be reflected in a laboratory's own sampling policies and procedures

II. Records and Documentation

1. ORELAP-accredited laboratories shall maintain standard operating procedures (SOP) that accurately reflect current sampling activities.
    a. The laboratory's SOP shall be readily accessible to all pertinent personnel.
    b. The laboratory's SOP shall clearly indicate the effective date of the document, the revision number, and the signature of the approving authority.
    c. The laboratory's SOP should use this protocol as minimum requirements and must include additional detail specific to laboratory procedures. In cases where the published method (this protocol) has been modified or where the referenced method (this protocol) is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described in the laboratory's SOP. Any changes to the laboratory's protocol, including use of a selected option, shall be documented and included on the laboratory's sampling form.
    d. All documents shall be controlled and retained in accordance with the TNI Environmental Laboratory standard as defined in 333-064-0025.

2. ORELAP-accredited laboratories shall maintain sampling plans.

a. The laboratory's sampling plans shall be made available at their location of use.
b. The laboratory's sampling plans shall be based on appropriate statistical methods and shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch.
c. Any deviation from or addition to the laboratory's sampling plan must be documented in detail and shall be included in the final report. The standardized or generic sampling plans can be included in the SOP however specialized client requests or products may require additional information.
d. The laboratory's sampling plans shall document the date and time of sampling.

III. Client Contracts; Client Sampling and Testing Requests

The laboratory must have a sampling contract with a client that includes at least the following:

1. A test order containing the information required by OAR 333-007-0315
2. A site-specific sampling plan or process specific sampling plan that uses statistical design for each project to provide representative sampling.

IV. Planning

Prior to beginning the sampling procedure, the sampler shall survey the site to identify the conditions under which the cannabinoid concentrate, extract, or product is being kept, as this will determine the sampling plan. All sampling must be performed by personnel employed by an ORELAP accredited laboratory and must be in accordance with OAR 333-007-0360 and OAR 333-064-0100.

The testing requirements for cannabinoid concentrates and extracts are in OAR 333-007-0330; those for cannabinoid products are in OAR 333-007-0340; those for industrial hemp-derived vapor items are in OAR 333-007-0342. The requirements for sampling and sample size are in OAR 333-007-0360 and Appendix 2 of this protocol. Per Authority or Commission request or client request, additional analyses may be required and must be considered in the planning process.

To ensure representativeness, the sampling plan must be designed such that any part or individual unit of sale in the batch or process lot has an equal chance of being selected. **The sample size must be sufficient to complete all analyses required, including necessary re-analyses and laboratory QC samples**.

V. Sampling Design and Plans

1. Sampling plans shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch or process

lot. Standardized Sampling Plans can be included in the SOP however specialized client requests or products may require additional information. Any deviation from or addition to the sampling plan must be documented in detail and shall be included in the final report.

2. Sampling plans shall be designed to meet specified sample quality criteria. This includes using a sampling plan that meets a 95% confidence level for representative sampling and limits the fundamental sampling error. The most common way to achieve this is by increasing the number of sample increments from the minimum required to compensate for normal batch heterogeneity.
3. Sampling plans must ensure that adequate sample mass is collected for all analyses requested by the producer. This must include adequate sample mass for re-testing in the event a sample fails a criterion as well as adequate sample mass for any quality control samples required by the laboratory, such as duplicates or matrix spikes.
4. A sampling plan must include at a minimum:
    a. Shape, size, and number of container(s) holding the batch or process lot from which sample increments will be collected;
    b. Number of sample increments to be collected;
    c. Total mass of sample needed to perform testing and approximate mass needed for each increment to ensure adequate mass;
    d. Location of where sample increments will be taken within each container holding the batch. See Appendix 2 for information on random selection of locations.
5. The laboratory must have details in its SOP or a sampling plan, from appropriate industry reference where possible, on how it will achieve random sampling in an unclear decision unit.

VI. Sampling Equipment and Supplies

1. A laboratory should, at a minimum, have the following equipment and supplies for sampling:
    a. Sampling equipment such as spoons, spatulas, transfer pipettes, or other matrix specific tools
    b. Tongs
    c. Corers
    d. Teri-wipes or equivalent
    e. Field balance (capable of 0.01 g measurements)
    f. Calibrated verification weights appropriate to verify accuracy of field balance
    g. Cleaning supplies – solvent, bleach, 70% Ethanol
    h. Gloves (powder-free, nitrile, sterile)
    i. Mylar bags (for final sample transport and storage) or amber or colorless glass jars (for final sample transport and storage)

2. Cleaning of Field Sampling Equipment

a. Field sampling equipment shall be certified clean prior to use by the laboratory.
b. Cleaning techniques will vary depending upon the desired analysis.
c. In general, sampling equipment must be sterile for microbiology samples and clean for chemistry samples.
d. The laboratory shall perform cleanliness checks on each batch of sampling equipment prior to taking that equipment into the field.
e. Results from cleaning procedure tests must be below the reporting limit of the target analyte(s) for the associated analyses.
f. If cleanliness checks fail, the sampling equipment must be re-cleaned, sterilized and tested.

3. Field balance calibration verification
    a. The laboratory sampling technician shall verify the calibration of the field balance at the sampling location.
    b. When multiple sampling events occur on the same day, the balance calibration shall be verified at each sampling location.
    c. Balance calibration verifications shall be documented.

VII. Procedures for Sampling Concentrates, Extracts, Products, Finished Inhalable Cannabinoid Products, and Industrial Hemp-derived Vapor Items.

1. Locate the cannabinoid concentrate, extract, product, finished inhalable cannabinoid product, or industrial hemp-derived vapor item batch to be sampled. The sampler **must** have access to the entire batch or process lot.
2. Check for any signs of non-uniformity within the batch or process lot and document the observations.
    a. Some obvious indicators may be different types or sizes of containers, variations in marks and labels, or mixed batch numbers
    b. During sampling, the sampler shall look for differences in the marijuana items or industrial hemp-derived vapor items being sampled such as color, visible layers, size of items, or texture.
    c. By definition, the batch must be uniform for all factors that appear on the label; hence, variations in the product may indicate non-uniformity in the batch or process lot and that any sample drawn may not be representative for testing.
    d. The sampler shall note these anomalies in the sample collection report.
3. Review the container label information for batch and process lot number and other pertinent information. Do not sample if a unique batch and process lot numbers are not available.

4. Determine if the sample matrix is a liquid, semi- solid, solid, or freshly-baked edible item either in bulk form or in packaged units. Determine and record the total batch weight or volume and the number of containers or units of sale comprising the batch. If the product is already in final packaging, determine and record the total number of final package units. Do not sample if there are deviations from the manifest or questions about the statistical certainty of the sampling plan.
5. Establish which tests will be performed. Ensure sufficient sample increments are taken to meet sample size requirements determined in the sampling plan and record the number of increments collected. The minimum sample amount is determined by the analytical method(s) being performed but for cannabinoid concentrates, extracts, finished inhalable cannabinoid products, or industrial hemp-derived vapor items can be no less than number of increments in OAR 333-007-0360, Exhibit B, Table 7 (see Appendix 2.) For cannabinoid products, a minimum of one unit of sale each is required for the primary and duplicate. If the product is sold in packages with variable units of sale, the smallest unit of sale is the minimum amount required for sampling.
6. Ensure that appropriate equipment and containers are used for the tests being performed. For residual solvent analysis, use glass containers that can be properly sealed to prevent the loss of solvent gas and minimize the headspace remaining in the sample container. If colorless glass containers are used, the container must also be enclosed in a mylar bag to protect the sample from light.
7. Select the appropriate sampling tool to ensure that it reaches all portions of the batch.
8. Collection instruments must be cleaned appropriately prior to use to prevent cross-contamination of samples. Sampling tools which appear to be dirty or otherwise compromised shall not be used.
    a. To prevent contamination, sampling tools may be cleaned and sealed at the laboratory prior to use or may be cleaned in the field between batches using an appropriate solvent and decontaminant to prevent cross contamination of batches during sampling.
9. Results from cleaning procedures must be below the reporting limit of the target analyte(s) for the associated analyses.
10. Decontamination waste must be collected and properly disposed of if not used for analysis.
    a. Samplers must take extreme care if sampling from multiple sites in one day to ensure contaminants, pathogens, or organisms are not transferred between facilities. The sampler may clean sampling equipment in the field between samplings at a single facility. However, the sampler shall bring enough sets of sampling equipment to use a new set at each facility visited.
    b. All field equipment shall be returned to the laboratory following sampling and cleaned according to the laboratory's procedures.

c. Where aseptic technique is required, samplers shall observe best practices to prevent microbiological contamination of samples. For an example of aseptic technique, see the FDA (2015) Aseptic Sample Guidelines (Investigations Operations Manual Subchapter 4.3.6).

11. When collecting sample increments, approximately equal amounts are to be taken with each probing and from each container. Care must be taken by the sampler to not damage the portion of the material which is not being collected. See sections below for more detail on sampling liquid, semi-solid, or solid sample matrices.
12. Once taken, seal and label the sample increments, composite sample, primary sample, duplicate sample, or replicate sample as applicable with the following minimum requirements:
    a. Laboratory license number
    b. Unique identifier for sampling event
    c. Sampling date and name of sampler
    d. Processor's license or registration number
    e. Process lot and batch numbers
    f. Label "PRODUCT NOT TESTED" in bold capital letters in minimum 12-point font
13. Apply a custody seal to the sample container in a manner that prevents the marijuana item or industrial hemp-derived vapor item from being tampered with prior to testing. This seal may contain the laboratory sample identification number.
14. Complete the sampling report while at the sampling location as well as an appropriate chain of custody form as outlined in the standards of accreditation.
15. Forward the sample and sampling report to the laboratory or other designated location using packaging appropriate for secure and timely transport.
16. Record the sampling event in the OLCC seed to sale system under the licensee number or under the registrant number, as applicable.
17. Apply the following steps when taking **Solid** and **Semi-Solid** samples:
    a. Establish the total batch weight or volume. If the batch is in final product packaging, determine how many units of sale there are and the total batch mass.
    b. Each sample increment should be taken from a randomly chosen position in the batch, as far as practically possible. A sample increment should be taken from each container if possible. If more containers exist than sample increments required, sample from as many as possible to obtain a representative sample. If permitted by OHA's rules, sample increments may be combined into a composite sample, or a primary sample, duplicate sample, or replicate sample as applicable.

c. The samples shall consist of sufficient material to perform the required laboratory methods. The mass of the sample increments can be increased or decreased as long as they are equivalent to each other.

d. The minimum number of sample increments is in OAR 333-007-0360, Exhibit B and included in Appendix 2, but more sample increments may be collected if needed for laboratory analysis or at client request based on the statistical design in the site-specific sampling plan. If not using the minimum requirements in rule the laboratory shall use its statistical design training, procedures, and calculators to determine the increments needed for a confidence interval that meets the client request.

e. Consideration must be taken for specific concentrate, extract, product, finished inhalable cannabinoid product, or industrial hemp-derived vapor item types that may be difficult to sample or weigh due to the physical nature of the item. When a sample type, such as kief, moonrocks, or infused pre-rolled joints, requires deviation from laboratory protocols, it is the responsibility of the sampler to document the actions taken.

f. Store each sample increment or combine all sample increments if allowed, as specified in the site-specific sampling plan, in a glass container with PTFE-lined screw cap to form the sample for testing. If residual solvent testing is required, ensure minimal headspace remains in sample container and lid is secure. If the sample increments are combined into a primary sample, complete the same procedure with a second set of equivalent sample increments to form the duplicate sample. Repeat the same procedure with equivalent sample increments to form the replicate sample(s), as specified in Appendix 2.

18. Apply the following steps when taking **Liquid** samples:

a. If the sample increments are to be taken from a bulk container, ensure proper homogenization of the product prior to taking the sample by mixing the container thoroughly and employing any process for homogenization that the processor would use to disperse the concentrate, extract, product, finished inhalable cannabinoid product, or industrial hemp-derived vapor item into packaging. Use an appropriate sample device for sampling bulk liquid in a container. Collect the appropriate number of sample increments based on the site-specific sampling plan for the client.

b. Store each sample increment or combine all sample increments if allowed, as specified in the site-specific sampling plan, in a glass container with PTFE-lined screw cap to form the sample for testing. If residual solvent testing is required, ensure minimal headspace remains in sample container and lid is secure. If the sample increments are combined into a primary sample, complete the same procedure with a second set of equivalent sample increments to form the duplicate sample. Repeat the same procedure with equivalent sample increments to form the replicate sample(s), as specified in Appendix 2.

19. Apply the following steps when sampling fresh-baked edible products:
    a. The batch or process lot must be presented in its final portioned form where the only remaining step to create a finished cannabinoid product is the baking step.
    b. Select the required number of unbaked units of sale (with the minimum being one unit of sale) to provide sufficient material for all required testing.
    c. Repeat the process outlined in b) above to select units of sale for the duplicate.
    d. **While remaining onsite** and in custody of the selected samples, request that the samples are baked.
    e. Combine the units of sale as applicable to form the primary and duplicate sample. Store each sample in a glass container with PTFE-lined screw cap or a mylar bag as appropriate.

VIII. Sampling Records/Field Data

1. At the time samples are collected the sampler must complete a sampling report form for each batch or process lot sampled. Sample report forms must include at a minimum the following information:
    a. Name and address of producer including licensee or registrant number
    b. Item type.
    c. Total weight of batch or total number of units of sale of batch.
    d. Unique laboratory batch ID#, Metrc batch ID #, and/or OHA batch ID#.
    e. Total number of containers sampled.
    f. Number of sample increments taken from each container.
    g. Number of sample increments combined into a primary, duplicate, and replicate sample, if applicable
    h. Number of sample containers collected.
    i. Weight and location of each sample increment.
    j. Total weight sampled.
    k. Sampling plan ID and revision date.
    l. Sampling Procedure ID and revision date.
    m. Description of equipment and tools used.
    n. Address where sampled.
    o. Date sampled.
    p. ORELAP Laboratory Identification number.
    q. Lab License Number.
    r. Sampler's identification and/or signature.
    s. Name of responsible party for the batch and transport information.
    t. Receiving laboratory and types of tests required or requested.
2. A chain of custody form must be used unless the laboratory is sampling for a client that is required to use Metrc. A chain of custody form must include at least the

following information:

a. Sampler's name
b. Sample Identification (Lab ID number) if assigned before arrival at laboratory
c. Sampling Date/Time
d. Weight and location of increment samples
e. Final weight of composite sample
f. Custody transfer signatures
g. Custody Transfer Dates/Times

3. If any of the above information requested on the sampling report form is unavailable, indicate "N/A" in the appropriate space with an explanation as to why the information is not available.
4. All sampling report forms must be signed by the sampler.

IX. Transportation and Handling of Samples

1. Transport the sample increments or composite sample to the laboratory performing the analysis by the most expedient, secure, and legal means to ensure that the sample continues to be representative of the process lot sampled and the chain of custody form continues to document sample integrity. Transportation must be done in compliance with OAR 845-025-5060. Note: Current law does not permit shipping in any form such as USPS or FedEx.
2. Containers for sample transport must be designed to protect the sample from moisture and temperature extremes and to prevent damage, contamination, spillage, or commingling of the sample during transport. The required container for sampling is an amber or colorless glass jar with a PTFE-lined lid or a Mylar bag and should be appropriate for the sample matrix and the tests required. If a colorless glass jar is used, the container must also be placed in a mylar bag to protect the sample from light exposure. A tamper-proof seal is required and must be marked with the sampler's name, date, and sample number.
3. The laboratory must have detailed procedures on maintaining custody and sample integrity during transport. These procedures should take into consideration controlling temperature and other environmental factors.
4. Submit the sample increments or composite samples to the laboratory in their entirety. In a situation where the composite sample must be split for analysis by two different laboratories, for example when residual solvent analysis is subcontracted to another laboratory, the composite sample(s) shall be homogenized by the laboratory's approved sample homogenization process prior to subsampling. Care must be taken to maintain sample integrity during this process and to prevent the loss of volatile components. This shall be

reflected on the chain of custody.

5. Composite samples must always be identified by labeling or marking the sample container to associate them with the batch from which they originated and with the sampling report.

X. Quality Assurance and Quality Control

The sampler must be prepared to collect adequate sample mass for all analyses requested by the producer. This must include adequate sample mass for re-testing in the event a sample fails a criterion as well as adequate sample mass for any quality control samples required by the laboratory, such as duplicates or matrix spikes.

1. Sampler qualifications
    a. Basic qualifications for samplers of marijuana items and industrial hemp-derived vapor items are:
        i. Physically able to perform the duties of a sampler;
        ii. No conflict of interest;
        iii. Employed by an ORELAP accredited laboratory;
        iv. Pass initial and ongoing demonstrations of capability as defined by the laboratory (see below);
        v. Licensed under state law to transport the required quantity of marijuana items or industrial hemp-derived vapor items.
    b. Required education and training for samplers:
        i. Initial training: training shall include principles, procedures, and policies of sampling; Initial Training must be performed by an Instructor that has demonstrated competency in performing the sampling methods referenced or equivalent. After personnel goes through initial training, they are qualified to train others in their organization.
        ii. Field or on-the-job training: 8-hours of training on various sampling techniques.
        iii. Continuing education: periodic refresher training shall be done annually.

2. Demonstration of Capability
    a. Prior to acceptance and institution of any accredited method, a satisfactory initial demonstration of capability (IDOC) is required. The laboratory shall have a documented procedure for performing the IDOC. The IDOC will be repeated: 1) every time there is a change in personnel or method; and 2)

when the method has not been performed by the laboratory within a 12-month period.

b. This procedure shall employ one of the following approaches to demonstrating capability:

   i. Comparison of replicate samples within defined Relative Standard Deviation (%RSD) acceptance criteria.

   ii. Comparison of a sample collected to that of one collected by personnel with an existing IDOC within defined Relative Percent Difference (%RPD) acceptance criteria.

c. Thereafter, ongoing continuing demonstration of capability (CDOC) is required annually. The laboratory shall have a documented procedure for performing the CDOC. The laboratory shall retain documentation verifying CDOC for each sampler and make this documentation available to ORELAP upon request.

3. Field QC Samples

   a. Duplicates

      i. A Duplicate Sample is required for any sampling event that takes place according to this protocol.. The duplicate and replicate samples must be collected using the same procedure as the primary sample. Comparison of primary and duplicate results must be evaluated against %RPD requirements as specified in the applicable OAR sections. Comparison of primary, duplicate, and replicate results must be evaluated against %RSD requirements as specified in the applicable OAR sections.

   b. Equipment Blanks

      i. Equipment rinse blank samples provide a QC check on the potential for cross contamination by measuring the effectiveness of the decontamination procedures on the sampling equipment. An equipment blank is required to validate equipment cleaning procedures for all required analyses. It is recommended but not required that an equipment blank is collected upon each sampling event to demonstrate the equipment was not introduced to contamination after cleaning.

      ii. The equipment rinse blank samples consist of analyte-free matrix, as applicable, rinsed across sample collection and processing equipment. If the analytes of interest are detected in the equipment rinse blank

samples, the detected concentrations will be compared to the associated sample results to evaluate the potential for contamination.

iii. The equipment blank must pass the required analysis at <LOQ for cleaning validation.

iv. If the equipment blank is collected at the sampling event, the lab must have detail in the sampling plan or procedures as to how to evaluate it and what actions to take if the evaluation demonstrates unacceptable results.

c. Transport Blank
   i. A transport blank is **required** as part of a sampling plan that includes collection for residual solvent analysis.
   ii. A single transport blank must be collected and analyzed per trip regardless of amount of sampling events and each event's samples must be linked to the acceptability of its result.
   iii. The transport blank must pass solvent analysis at <LOQ for the sampling event to be considered valid.

4. Field Audits

   a. The laboratory shall adopt an ongoing system for performing audits of field activities. Field audits must be conducted periodically and in accordance with a predetermined schedule and procedure. The goal of the field audit is to verify that the sampling operation continues to comply with the requirements of the regulations and is being performed according to the laboratory's sampling SOP. Audits are to be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. The field audit shall address all elements of the sampling activities and shall be documented.

   b. When field audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the field sampling activities, the associated laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that test results may have been affected. Laboratory management shall have a policy that specifies the time frame for notifying clients of events that cast doubt on the validity of the results. Follow up audit activities shall verify and document the implementation and effectiveness of any corrective actions taken as a result of the field audit.

   c. Required components of the Field Audit program:
      i. Review sampling and performance records from the preceding year for deficiencies in the application of sampling protocol.
      ii. Observe the sampler conducting sampling procedures.
      iii. Record any deficiencies and initiate corrective action.

XI. References

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FDA, *Guidelines for Food Spice Labeling.* Code of Federal Regulations Title 21, Volume 2. http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=101.22)

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Sampling and Sample Handling Working Group FDA, AAFCO, AFDO, APHL and Industry, October 2015. *Good Samples: Guidance on Obtaining Defensible Samples*.

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Oregon Administrative Rules, *Marijuana Labeling, Concentration limits, and Testing,* Chapter 333, Division 7.

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Technical and Regulatory Guidance, Incremental Sampling Methodology, February 2012, Prepared by The Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team

Appendix 1 – Definitions

**If there are any inconsistencies between the definitions below and the definitions in OAR 333, Divisions 7 or 64, the definitions in the rules take precedence.

Authority means Oregon Health Authority

Batch means a quantity of cannabinoid concentrate, extract, product, finished inhalable cannabinoid product, or industrial hemp-derived vapor item from a process lot.

Chain of Custody Form means a form completed by laboratory personnel that documents the collection, transport, and receipt of samples by the laboratory. (Sample tracking document)

Commission means the Oregon Liquor and Cannabis Commission.

Composite Sample means a sample containing all sample increments taken from a batch.

Container means a sealable, hard- or soft-bodied receptacle in which a marijuana item is placed during sampling, transport, and storage; or a physical division of an extract or concentrate process lot for random sampling.

Decision Unit (DU) or Sampling Unit means the material from which the primary sample(s) is collected and to which the inference(s) is made.

Duplicate Sample means sample increments taken in an identical manner to sample increments taken for the primary sample and representative of the same marijuana item being sampled that is prepared and analyzed separately from the primary sample.

Equipment Blank means a sample of analyte-free media, collected after decontamination and prior to sampling, which has been used to rinse the sampling equipment after cleaning to validate cleaning procedure or between sampling batches to demonstrate lack of contamination.

Fundamental Sampling Error (FSE) means a measure of the compositional heterogeneity, which is controlled through the collection of sufficient sample mass (mass is inversely proportional to error).

Heterogeneity means the state or quality of being heterogeneous.

Heterogeneous means non-uniform or consisting of dissimilar parts or components.

Homogeneous means a cannabinoid product, concentrate, or extract has uniform composition and properties throughout each process lot.

Industrial hemp-derived vapor item has the meaning given that term in OAR 333-007-0310.

Kief means the resinous trichomes of marijuana that accumulate or fall off when marijuana flowers are sifted through a mesh screen or sieve.

Label means a tag or other device attached to or written, stamped, or printed on any container or accompanying any batch in bulk stating all required batch information.

Laboratory means a laboratory that is accredited under ORS 438.605 to 438.620 to

sample or conduct tests on marijuana items and licensed by the Oregon Liquor and Cannabis Commission under ORS475B.560.

Marijuana has the meaning given that term in OAR 333-007-0310.

Marijuana Item has the meaning given that term in OAR 333-007-0310.

Metrc means the state-administered cannabis tracking system (CTS).

ORELAP means the Oregon Environmental Laboratory Accreditation Program administered by the Authority pursuant to ORS 438.605 to 438.620.

Primary Sample means a composite sample composed of sample increments and tested for the required analysis methods.

Process Lot has the meaning given that term in OAR 333-007-0310.

Producer has the meaning given that term in OAR 845-025-1015.

Registrant has the meaning given that term in OAR 333-007-0310.

Relative Percent Difference means the comparison of two quantities while taking into account the size of what is being compared. If the final result (i.e., Total THC) is <LOQ in either sample, the absolute value of the LOQ is used in the equation.

$$\%RPD = \frac{|(sample - duplicate)|}{(sample + duplicate)/2}\ x\ 100$$

Relative Standard Deviation means the standard deviation expressed as a percentage of the mean recovery, i.e., the coefficient of variation multiplied by 100. If the final result (i.e., Total THC) is <LOQ in either sample, the absolute value of the LOQ is used in the equation.

Standard Deviation

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

Relative Standard Deviation

$$\%RSD = \frac{S}{\bar{x}}\ x\ 100$$

S = standard deviation.

n = total number of values.

x_i = each individual value used to calculate mean.

$\bar{x}$ = mean of n values.

Replicate sample is a sample in addition to the primary and duplicate samples that consists of the same number of increments and taken in the same manner as the primary and duplicate samples.

Representative Sample means a sample obtained according to an incremental sampling procedure designed to ensure that the different parts of a batch or lot or the different properties of a batch or lot are proportionally represented.

Sample means an amount of marijuana item or industrial hemp-derived vapor item collected by laboratory personnel from a registrant or licensee and provided to a laboratory for testing.

Sample Increment means an amount of a marijuana item or industrial hemp-derived vapor item collected by laboratory personnel from a registrant or licensee that may be combined into a sample for purposes of testing.

Sample Quality Criteria (SQC) means a series of statements that clarify a sampling program's technical and quality needs to support defensible decisions, including statement of the question to be answered, definition of the decision unit, and the desired confidence in the inference.

Sealed means secured in such a way as to provide authenticity or integrity of the sample.

Sterilization means the removal of all microorganisms and other pathogens from a marijuana item or industrial hemp-derived vapor item by treating it with approved chemicals or subjecting it to high heat.

TNI Standard means the TNI Environmental Laboratory Standard as defined in OAR 333-064-0025.

Transport Blank means a sample of analyte-free media which has been carried to the field and returned to the lab and is used to demonstrate that the process did not add volatile contamination in solvent analysis.

Usable marijuana has the meaning given that term in OAR 333-007-0310.

Appendix 2 – Sampling Requirements

Random sampling

1. As specified in the sampling plan, sample increments should be randomly selected from different locations within a container or set of containers. Laboratories must develop procedures describing how to:
   a. Assign location numbers within containers and among a set of containers;
   b. Use a random number generator to determine which location to sample; and
   c. Document where each sample increment was sampled and the volume or mass collected from each increment.
2. Assign divisions based on the type of container in the site-specific sampling plan. For container types that are greater than four (4) inches deep, divisions must also include a layer or layers beneath the upper portion of the container. Use a random number generator with the higher number equal to the number of divisions for the container. When there are multiple containers, use existing or arbitrary order of containers to assign numbers to the total of "divisions multiplied by total number of containers" (divisions x # containers = total number of random sample increments) and record in the sampling report.
3. The laboratory must have details in their SOP or Sampling Plan, from appropriate industry reference where possible, on how they will achieve random sampling in unclear decision unit.

Sample size and increments

1. Per OAR 333-007-0360, the sample size must be sufficient to complete all analyses required.
2. The required sample increments for a given batch or process lot of cannabinoid concentrate, extract, finished inhalable cannabinoid product, or industrial hemp-derived vapor item varies depending upon the size of the batch. Taking more sample increments than required is encouraged and will improve representativeness of the sample in relation to the batch. (See Table 1)
3. Sample increments are combined into a primary sample. An equivalent number of increments sampled using the same procedure are combined into the duplicate sample. An equivalent number of increments sampled using the same procedure are combined into the replicate sample. The combined samples are put in separate containers and are prepared and analyzed separately.

Table 1 – Sample increment and replicate requirements based on size of concentrate, extract, finished inhalable cannabinoid product, or industrial hemp-derived vapor item batch. (From 333-007-0360, Exhibit

B, Table 7)

Batch Weight		Sample Increments Required		Number of Replicates
Pounds	Kilograms	Primary	Duplicate	
0-3.31	0-1.50	1	1	
3.32-6.61	1.51-3.00	3	3	
6.62-13.23	3.01-6.00	5	5	
13.24-26.46	6.01-12.00	7	7	
26.47-55.12	12.01-25.00	7	7	1
55.13-110.23	25.01-50.00	7	7	2
110.24-220.46	50.01-100.00	7	7	3

For batches exceeding 100.00kg: apply the following formula to determine number of replicate samples: X=(batch weight in kg/50)*1.5 where X is the number of replicates, rounded to the nearest whole number.

4. Finished cannabinoid products require a primary and duplicate sample. A minimum of one unit of sale each shall be selected for the primary and duplicate sample. The primary and duplicate samples are placed in separate containers and are prepared and analyzed separately.

Table 2 – Revision history of this SOP.

Revision	Date	Summary of changes made, and initials of editor
4.0	07/22/2020	Major updates and re-formatting based on input from Scott Hoatson (former ORELAP QA Officer) and Department of Justice. Updated: OSPHL address; executive board and ORELAP staff names/titles as needed; definitions in order to match OARs and ORS. Added: Tables 1, 2, 3, and 4 (this table); information regarding required calibration verification of field balances; mention of assigning divisions to layers in deep containers; section II; section III; condensed general document requirements in new section II, and specific sampling forms under section VIII; condensed Planning section, now section IV; reference to FDA aseptic sampling document; definition of Metrc. Combined: sampling design and plans and representative sampling sections; forwarding samples section with transportation section Minor updates and typo fixes for consistency with Useable Marijuana sampling SOP. STJ 07/22/2020
4.1	10/19/2020	Minor updates to include definition of kief and inclusion of consideration of tricky/unusual sample matrices in section 17 e. STJ 10/19/2020
4.2	9/27/2021	Minor formatting updates, removed historical authorship captured in previous versions, added reference to industrial hemp-derived vapor items. Updated definitions in Appendix 1 to align with those in applicable administrative rules. Updated Tables 5 and 7 to show more accurate mass ranges. STJ 9/27/2021
4.3	11/18/2021	Added information about finished inhalable cannabinoid products. Removed reference to control studies. Updated container requirements

		that amber glass is not required so long as the clear glass containers are stored in mylar bags. Added instruction on sampling fresh-baked edible products. Added information about sampling of finished cannabinoid products. Added definition of 'replicate sample' and removed 'field' from reference to duplicate samples for consistency with OARs. Updated table on sample increments (formerly Table 7) to be copy of new sampling format from 333-007-0360 Exhibit B. STJ12/15/2021

OAR 333-064-0100, Exhibit C

Table 1 – Accuracy requirements for laboratory control samples (LCS).

Test	Analyte	LCS Limits (%R)
Pesticides in accordance with OAR 333-007-0400	Abamectin	50 - 150
	Acephate	60 - 120
	Acequinocyl	40 - 160
	Acetamiprid	60 - 120
	Aldicarb	60 - 120
	Azoxystrobin	60 - 120
	Bifenazate	60 - 120
	Bifenthrin	50 - 150
	Boscalid	60 - 120
	Carbaryl	60 - 120
	Carbofuran	60 - 120
	Chlorantraniliprole	60 - 120
	Chlorfenapyr	60 - 120
	Chlorpyrifos	60 - 120
	Clofentezine	60 - 120
	Cyfluthrin	50 - 150
	Cypermethrin	50 - 150
	Daminozide	60 - 120
	DDVP (Dichlorvos)	60 - 120
	Diazinon	60 - 120
	Dimethoate	60 - 120
	Ethoprophos	60 - 120
	Etofenprox	50 - 150
	Etoxazole	60 - 120
	Fenoxycarb	60 - 120
	Fenpyroximate	60 - 120
	Fipronil	60 - 120
	Flonicamid	60 - 120
	Fludioxonil	50 - 150
	Hexythiazox	60 - 120
	Imazalil	60 - 120
	Imidacloprid	60 - 120
	Kresoxim-methyl	60 - 120
	Malathion	60 - 120
	Metalaxyl	60 - 120
	Methiocarb	60 - 120
	Methomyl	60 - 120

Test	Analyte	LCS Limits (%R)
Pesticides in accordance with OAR 333-007-0400 continued	Methyl parathion	50 - 150
	MGK-264	50 - 150
	Myclobutanil	60 - 120
	Naled	50 - 150
	Oxamyl	60 - 120
	Paclobutrazol	60 - 120
	Permethrins	50 - 150
	Phosmet	50 - 150
	Piperonyl butoxide	60 - 120
	Prallethrin	60 - 120
	Propiconazole	60 - 120
	Propoxur	60 - 120
	Pyrethrins	60 - 120
	Pyridaben	50 - 150
	Spinosad	50 - 150
	Spiromesifen	60 - 120
	Spirotetramat	60 - 120
	Spiroxamine	60 - 120
	Tebuconazole	60 - 120
	Thiacloprid	60 - 120
	Thiamethoxam	60 - 120
	Trifloxystrobin	60 - 120
Residual Solvents in accordance with OAR 333-007-0410	1,4-Dioxane	60 - 120
	2-Butanol	60 - 120
	2-Ethoxyethanol	60 - 120
	2-Propanol (IPA)	60 - 120
	Acetone	60 - 120
	Acetonitrile	60 - 120
	Benzene	60 - 120
	Butanes	60 - 120
	Cumene (aka isopropylbenzene)	60 - 120
	Cyclohexane	60 - 120
	Dichloromethane	60 - 120
	Ethyl acetate	60 - 120
	Ethyl benzene	60 - 120
	Ethyl ether	60 - 120
	Ethylene glycol	60 - 120
	Ethylene Oxide	60 - 120

Test	Analyte	LCS Limits (%R)
Residual Solvents in accordance with OAR 333-007-0410 continued	Heptane	60 - 120
	Hexanes	60 - 120
	Isopropyl acetate	60 - 120
	Methanol	60 - 120
	Pentanes	60 - 120
	Propane	60 - 120
	Tetrahydrofuran	60 - 120
	Toluene	60 - 120
	Xylenes	60 - 120
Adult use cannabinoids and CBD in accordance with OAR 333-007-0430	delta-9 THC	90 - 110
	delta-9 THCA	90 - 110
	delta-8 THC	90 - 110
	CBD	90 - 110
	CBDA	90 - 110
Heavy Metals in accordance with OAR 333-007-0415	Arsenic	80-115
	Cadmium	80-115
	Lead	80-115
	Mercury	80-115
Mycotoxins in accordance with OAR 333-007-0425	Aflatoxin B1	60-120
	Aflatoxin B2	60-120
	Aflatoxin G1	60-120
	Aflatoxin G2	60-120
	Ochratoxin A	60-120

AMEND: 333-064-0110

NOTICE FILED DATE: 05/30/2024

RULE SUMMARY: Amend OAR 333-064-0110

This rule is being amended to clarify some of the test report components required by the TNI Standard that are not required to be included on reports by cannabis testing laboratories. This also required the restructuring of the numbering in this rule. Batch quality control samples are clarified to include negative control and positive control in microbiology testing.

CHANGES TO RULE:

333-064-0110
Reporting Cannabis Test Results ¶

(1) For purposes of this rule the definitions in OAR 333-007-0310 apply unless the context indicates otherwise.¶
(2) A test report must clearly~~i~~: ¶
(a) Identify for the licensee or registrant:¶
(~~a~~A) Whether a sample has exceeded an action limit for an analyte in OAR 333-007-0400, 333-007-0410, 333-007-0415, and 333-007-0425 Exhibit A, Tables 3, 4, 8, or 9, or has otherwise failed a test as described in OAR 333-007-0300 to 333-007-0500.¶
(~~b~~B) A "detected" pesticide result as required in section (6) of this rule.¶
(~~c~~C) The batch unique identification number required under OAR 333-007-0350 and the test batch number associated with the samples tested, as required by OAR 333-064-0100.¶
(~~d~~D) Identification of the test as a compliance test or a quality control or research and development test. If the test is not for compliance, the report shall indicate clearly on the first page the testing was for quality control or research and development.¶
(~~e~~E) If applicable, a statement that the test was done on a sample from a remediated marijuana item or industrial hemp-derived vapor item.¶
(b) Include all information required in Volume 1 Module 2 section 5.10 of the 2016 TNI Standard with the following exceptions:¶
(A) The address of the customer; and¶
(B) The location of sampling, including any diagrams, sketches, or photographs.¶
(3) Within 24 hours of completion of the laboratory's data review and approval procedures a laboratory must report all failed tests for testing required under OAR 333-007-0300 to 333-007-0500 except for failed water activity, whether or not the laboratory is reanalyzing the sample under OAR 333-007-0450:¶
(a) Into the Cannabis Tracking System (CTS) if performing testing for a licensee or a registrant who is subject to CTS tracking under OAR chapter 333, division 8; and¶
(b) To the ~~Authority~~Oregon Health Authority (Authority) electronically at www.healthoregon.org/ommp if performing testing for a registrant, along with a copy of the test order information required in OAR 333-007-0315, regardless of whether the laboratory is also reporting into CTS on behalf of a registrant that is subject to CTS tracking under OAR chapter 333, division 8.¶
(c) If the laboratory discovers that an error has occurred after reporting, an amended report shall be generated and communicated to the licensee or registrant, the ~~Commission~~Oregon Liquor and Cannabis Commission (Commission) for licensees, and the Authority for registrants. The laboratory shall ensure that results entered into the CTS are accurate and updated if necessary to reflect the amended report. The laboratory shall ensure that the amended report, communication, and updates to CTS as described in this rule are completed within 48 hours of learning of the error.¶
(4) The laboratory must report all test results required under OAR 333-007-0300 to 333-007-0500 that have not been reported under section (3) of this rule into the Commission's seed to sale tracking system if performing testing for a licensee or a registrant who is subject to CTS tracking under OAR chapter 333, division 8.¶
(5) A laboratory must determine and include on each test report its limit of quantitation (LOQ) and action level for each analyte listed in OAR 333-007-0400 Table 3, 333-007-0410 Table 4, 333-007-0415 Table 8, and 333-007-0425 Table 9.¶
(6) When reporting pesticide testing results the laboratory must include in the report any target compound that falls below the LOQ that has a signal to noise ratio of greater than 5:1 and meets identification criteria with a result of "detected." This additional reporting is not required if the laboratory's LOQ is less than or equal to one half of the action level in OAR 333-007-0400 Table 3.¶
(7) A laboratory must include in a test report the results of all associated batch quality control samples, with the

date of analysis of the quality control samples and the acceptance limits used to determine acceptability.¶
(a) Batch quality control samples are the method blank or negative control and laboratory control sample or positive control.¶
(b) The report must clearly show the association to the client samples in the report by listing the batch identification numbers.¶
(8) A laboratory that is reporting failed test results to the Commission or the Authority in accordance with section (3) of this rule must report the failed test at the same time or before reporting to the licensee or registrant.¶
(9) If requested by the Authority, a laboratory must report sampling and testing information to the Authority, in a manner prescribed by the Authority.¶
(10) If a laboratory's calculated adult use cannabinoid or CBD result exceeds 100 percent and the difference between the result and 100 percent is within the laboratory's calculated analytical uncertainty, the laboratory may report the result as 100 percent with a qualifying statement on the certificate of analysis or the laboratory may report the calculated result with or without a qualifying statement. If the difference between the result and 100 percent is outside the calculated analytical uncertainty, the calculated result shall be reported without correction.¶
(a) The qualifying statement on the certificate of analysis shall clearly state the calculated value and the laboratory's analytical uncertainty.¶
(b) For the purposes of calculating relative percent difference (RPD) or relative standard deviation (RSD), a laboratory shall use the calculated result and not the adjusted result described in this rule.¶
(11) A primary accredited laboratory may subcontract with accredited laboratories to perform required compliance testing. The primary accredited laboratory shall issue the final report.¶
(a) Accredited, subcontracted laboratories shall validate the results of any sample analysis and report that analysis to their client laboratory within 24 hours of completing the analytical run if the analysis results in a failed compliance test.¶
(b) The accredited laboratory that issues the final test report shall validate and report the results of any failed sample analysis as described in section (3) of this rule.
Statutory/Other Authority: ORS 475C.544, ORS 475C.560
Statutes/Other Implemented: ORS 475C.544, ORS 475C.560

AMEND: 333-064-0120

NOTICE FILED DATE: 05/30/2024

RULE SUMMARY: Amend OAR 333-064-0120

This rule is being amended to bring proficiency testing requirements from the 2016 TNI Standard that already apply to cannabis testing laboratories into the rule text. While the addition on reporting requirements and evaluation for acceptability by the proficiency testing (PT) provider and ORELAP doesn't represent a change in requirements, ORELAP is adding it to the rules to help laboratories better comply with the existing requirements. The proficiency testing reporting limit is also added to rule to support the additional language from the TNI Standard. Mycotoxin analysis is added to existing requirements for PT samples to be made in a usable marijuana matrix alongside pesticides and potency. This is because mycotoxins are similarly affected by the usable marijuana matrix as pesticides, so the in-matrix requirement is an important test on the laboratory's performance.

CHANGES TO RULE:

333-064-0120
Proficiency Testing for Laboratories Accredited for Cannabis Testing
The purpose of a proficiency testing (PT) study is to provide a means for the Oregon Environmental Laboratory Accreditation Program (ORELAP) to evaluate a laboratory's performance, under specified conditions related to a given set of criteria in a specific area of accreditation, through analysis of PT samples provided by an external source.¶
(1) A laboratory accredited to test marijuana items and industrial hemp-derived vapor items must at all times have two successful PT studies out of the most recent three attempts for each field of accreditation for which the laboratory holds accreditation.¶
(a) The closing dates of ~~a~~consecutive PT stud~~y~~ies for a particular field of accreditation can be no more than seven months apart.¶
(b) The opening date of a PT study for a particular field of accreditation must be at least seven calendar days after the closing date of the previous PT study for the same field of accreditation.¶
(2) For purposes of this rule ~~a~~:¶
(a) A PT study is a scheduled PT study or a supplemental PT study.¶
(b) The proficiency testing reporting limit (PTRL) is equal to required maximum laboratory limits of quantitation (LOQs) as established in OAR 333-064-0100(12).¶
(3) When a laboratory submits its PT study results to the PT ~~vendo~~provider, the laboratory must:¶
(a) Ensure that the information provided to the ~~vendo~~provider reflects accurate information about the laboratory that corresponds to the information in the laboratory's accreditation or application for accreditation, including but not limited to:¶
(A) The laboratory's name and address;¶
(B) The laboratory's ORELAP ID number; and¶
(C) The method and analyte codes.¶
(b) Instruct the PT ~~vendor to send the PT results directly to ORELAP~~provider to send the PT results directly to ORELAP.¶
(c) Report each quantitative field of accreditation as follows:¶
(A) The obtained numeric analytical result when the result is greater than or equal to the PTRL. ¶
(B) If the PTRL is less than the laboratory's LOQ as allowed in OAR 333-064-0110(6), the laboratory shall report the analytical result without qualifying the result.¶
(C) One of the following when the obtained numeric analytical result is less than the PTRL:¶
(i) <PTRL; or¶
(ii) The obtained analytical result if greater than or equal to the laboratory's LOQ; or¶
(iii) <LOQ if the obtained analytical result is less than the laboratory's LOQ.¶
(D) For non-detect results, <PTRL.¶
(d) Report each qualitative field of accreditation according to the PT provider's instructions. Presence or absence determinations in the PT shall be made following the laboratory's normal procedure.¶
(4) Any of the following will be considered to be an unsuccessful PT study and if a study was done, may not be counted toward the laboratory's PT history of the most recent three attempts:¶
(a) If a PT study for a particular field of accreditation is not performed within seven months, the laboratory will be charged with a failed study for each analyte.¶
(b) A PT study for a particular field of accreditation that has an opening date less than seven days from the closing

date of the previous PT study for that same field of accreditation.¶
(c) Information on study results received from the ~~vendo~~provider does not match any of the items in OAR 333-064-0120(3).¶
(5) For pesticide, mycotoxin analysis on or after August 1, 2024, and potency analyses in usable marijuana, a laboratory must use PT samples made with a usable marijuana matrix. If a usable marijuana matrix is unavailable, then a PT sample made with usable hemp matrix may be used with written permission from ORELAP. If a PT sample made with a usable hemp matrix is used for accreditation of potency analysis, then the PT ~~vendo~~provider must prepare the sample in usable hemp material itself and may not provide a separate spiking solution with the sample.¶
(6) In accordance with ORS chapter 183, the Oregon Health Authority may:¶
(a) Suspend the affected field of accreditation if a laboratory fails to comply with this rule.¶
(b) Revoke the affected field of accreditation if a laboratory fails three consecutive PT studies or fails to participate in a PT study as is required by these rules.¶
(7) For purposes of this rule a successful PT study means the testing results have been ~~evaluated~~reported according to these rules and evaluated by the PT provider and ORELAP as acceptable.
Statutory/Other Authority: ORS 475~~B~~C.55~~5~~44
Statutes/Other Implemented: ORS 475~~B~~C.55~~5~~44

AMEND: 333-064-0140

NOTICE FILED DATE: 05/30/2024

RULE SUMMARY: Amend OAR 333-064-0140

A small change was made to the structuring of sections in the rule concerning combining sample increments. While the wording of the rule did not change, the restructuring changes how the rule can be read. This aligns the reading of the rule with the original intent. The existing section on internal amplification control was edited to improve readability, remove extraneous wording, and add instruction on what to do in the case of an unacceptable internal amplification control but an otherwise acceptable test. These changes also harmonize the paragraph with the similar requirement in OAR 333-064-0100. The rule is also amended to specify that accredited laboratories may not receive psilocybin products from unlicensed sources, harmonizing with OAR 333-333-7010 and ORS chapter 475A. The rule also includes specific situations where a laboratory may receive psilocybin containing materials for the purposes of establishing instrument calibrations or validating laboratory processes necessary for required testing. The rule also includes entities the psilocybin containing materials can be received from.

CHANGES TO RULE:

333-064-0140
Psilocybin Products Sampling Procedures and Testing
(1) For purposes of this rule the definitions in OAR 333-333-1010 apply unless the context indicates otherwise.¶
(2) Sampling.¶
(a) A laboratory must have and follow psilocybin products sampling policies and procedures, accredited by the Oregon Environmental Laboratory Accreditation Program (ORELAP), that:¶
(A) Ensure sampling will result in a sample that is representative of the batch being sampled.¶
(B) Require sampling and laboratory personnel to document and collect any information necessary for compliance with these rules, OAR chapter 333, division 333, and any applicable TNI Standards.¶
(C) Require chain of custody procedures consistent with the TNI Standards.¶
(D) Are appropriate to the matrix being sampled.¶
(E) Are consistent with OAR 333-333-7100 and the ORELAP sampling protocol, ORELAP-SOP-004 Rev 1.0, approved by the accrediting body and incorporated by reference.¶
(F) Ensure that only the finished psilocybin product is sampled if testing the finished product is required under OAR 333-333-7100.¶
(G) Contain training and education requirements for sampling personnel.¶
(b) Sampling policies and procedures must be accredited by ORELAP prior to any psilocybin product samples being taken.¶
(c) Laboratory personnel that perform sampling must:¶
(A) Comply with the laboratory's accredited sampling policies and procedures.¶
(B) After taking samples:¶
(i) Document the samples in accordance with subsection (2)(e) of this rule; and¶
(ii) If sampling for a manufacturer required to comply with P~~TS~~silocybin Tracking System (PTS) tracking under ORS 475A.400, record the sampling and transfer information in the tracking system, as required by the ~~Authority~~Oregon Health Authority (Authority).¶
(C) Take care while sampling to avoid contamination of the non-sampled material. Sample containers must be free of analytes of interest and appropriate for the analyses requested.¶
(D) Take sample increments that are representative of the batch being sampled.¶
(d) A sufficient sample size must be taken for analysis of all requested tests and the quality control performed by the testing laboratory for these tests.¶
(e) A laboratory must comply with any recording requirements for samples and sample increments in the accredited policies and procedures and at a minimum:¶
(A) Record the location of each sample and sample increment taken.¶
(B) Assign a field identification number for each sample and duplicate sample that have an unequivocal link to the laboratory analysis identification.¶
(C) Assign a unique identification number for the test batch in accordance with OAR 333-333-7110 and TNI Standard requirements.¶
(D) Have a documented system for uniquely identifying the samples to be tested to ensure there can be no confusion regarding the identity of such samples at any time. This system must include identification for all

samples, sample increments, preservations, sample containers, tests, and subsequent extracts or digestates.¶
(E) Place the laboratory identification code as a durable mark on each sample container.¶
(F) Enter a unique identification number into the laboratory records. This number must be the link that associates the sample with related laboratory activities such as sample preparation. In cases where the sample collector and analyst are the same individual, or the laboratory pre-assigns numbers to sample containers, the unique identification number may be the same as the field identification code.¶
(f) Combining sample increments.¶
(A) Sample increments collected from the same batch of whole fungi shall be combined into a composite sample and homogenized prior to testing.¶
(B) Sample increments collected from the same batch of homogenized fungi, psilocybin extract, or edibles shall be combined into a composite sample.¶
(i) Increments from a primary sample must be combined into a single composite sample.¶
(ii) Increments from a duplicate sample must be combined into a composite sample separate from the primary sample composite sample.¶
(~~iii~~C) Prior to any testing or subsampling, each composite sample must undergo the laboratory's homogenization process.¶
(~~iv~~D) If the homogenization process would invalidate the analysis for a required test, the laboratory must utilize a subsampling procedure to withdraw a portion of the sample prior to homogenization for the required test. Testing that would be invalidated by the homogenization process includes but is not limited to, cryogenic sterilization of the sample prior to microbiological analysis.¶
(3) Compliance testing validity.¶
(a) When testing a sample for the required chemistry compliance tests as described in OAR 333-333-7040 and 333-333-7050, a laboratory shall comply with additional method validation as follows:¶
(A) Run a method blank in accordance with TNI Standard requirements to demonstrate the procedure is free of contaminants at or above the limit of quantitation.¶
(B) Run a laboratory control sample (LCS) in accordance with TNI Standard requirements to demonstrate acceptable performance of the procedure. Acceptable performance of the LCS means percent recovery for all regulated analytes are within the limits specified in Exhibit D, Table 1.¶
(C) Calculate a measure of precision within each analytical batch. This may be done by analyzing an analytical duplicate sample or a laboratory control sample duplicate (LCSD) and calculating the ~~percent~~ relative percent difference (RPD). An analytical duplicate sample is prepared from a second aliquot of material from the same sample to determine variability of measurements within the laboratory.¶
(b) When performing a speciation test as described in OAR-333-333-7030, a laboratory shall use any DNA-based approach that has been validated to show acceptable inclusivity, exclusivity, and probability of detection (POD). A laboratory shall comply with additional method validation as follows:¶
(A) The laboratory must perform initial method validation to include inclusivity and exclusivity testing using whole tissue or cultured organisms. This is to show the laboratory has proficiency with the DNA extraction, replication, and detection processes and can demonstrate the ability to differentiate between the target organism and other organisms that may be found in samples. Nothing in these rules prohibits testing laboratories from possessing Psilocybe cubensis for purposes of method validation and testing.¶
(B) Run a negative control to show laboratory reagents and equipment are not causing false positives.¶
(C) Run a positive control with each batch to show acceptable performance of the method. Acceptable performance means detection of target DNA sequences in the positive control that has been spiked with Psilocybe cubensis genetic material.¶
(D) ~~Show~~Demonstrate acceptable performance as described in the manufacturer's instructions of an internal amplification control (IAC) in each sample ~~to indicate DNA extraction, replication, and detection occurred.~~ analyzed by a polymerase chain reaction (PCR) method. In the case of a positive result for Psilocybe cubensis in a sample with an unacceptable performance of the IAC, follow the manufacturer's instructions on interpretation of acceptability.¶
(4) Compliance testing results should not be adjusted for percent moisture, or on any other basis except to account for extraction and dilution during laboratory preparation.¶
(5) Calculating RPD and relative standard deviation (RSD.)¶
(a) A laboratory must use the following calculation for determining RPD:¶
Relative Percent Difference¶
%RPD=|(sample-duplicate)|/((sample+duplicate)/2) * 100¶
(b) A laboratory must use the following calculation for determining RSD:¶
Standard Deviation¶
S= (((xi-x)^2)/(n-1))¶
Relative Standard Deviation¶

%RSD= (S/x)* 100¶
(c) For purposes of this section:¶
(A) S = standard deviation.¶
(B) n = total number of values.¶
(C) xi = each individual value used to calculate mean.¶
(D) x = mean of n values.¶
(d) For calculating both RPD and RSD if any results are less than the Limit of Quantitation (LOQ) the absolute value of the LOQ is used in the equation.¶
(6) A laboratory must provide any pesticide test result to the Authority upon its request.¶
(7) A laboratory performing tests for a manufacturer required to use PTS under ORS 475A.400 must enter any information required by the Authority in PTS.¶
(8) A laboratory performing tests for a manufacturer must comply with the documentation requirements in OAR 333-333-7100 and must maintain the documentation required in these rules for at least three years and provide that information to the Authority upon request.¶
(9) The Authority may, in its discretion, permit a laboratory to deviate from TNI Standards in order to comply with OAR 333-333-7020 to 333-333-7150 and these rules based on the state's needs. Permission to deviate from TNI Standards must be in writing from the Authority.¶
(10) A laboratory must be able to demonstrate that its limit of quantitation (LOQ) for compliance testing is less than or equal to one-half of any action level established in OAR 333-333-7050.¶
(11) Non-compliance testing. A laboratory that conducts a quality control or research and development test for a manufacturer may use methods not approved by the Authority, but the laboratory may not identify those test results as accredited results.¶
(a) Accredited laboratories may not receive psilocybin product from a source that is not licensed under ORS 475A.290.¶
(b) Notwithstanding subsection (11)(a), it is not a violation of these rules for an accredited laboratory to receive materials containing psilocybin for purposes of establishing instrument calibrations or validating laboratory processes necessary for testing required under ORS 475A.590 from the following entities: ¶
(A) Providers of certified reference materials; ¶
(B) Approved or accredited providers of proficiency tests; ¶
(C) The Oregon State Reference Laboratory or any agent acting in an official capacity for the Authority or the Oregon Department of Agriculture; or¶
(D) Individuals providing material that is not tracked in PTS for the exclusive purpose of laboratory method development.
Statutory/Other Authority: ORS 438.605, 438.610, 438.615, 438.620, 475A.590, 475A.606
Statutes/Other Implemented: ORS 438.605, 438.610, 438.615, 438.620, 475A.590, 475A.606

ORELAP

Oregon

Environmental Laboratory Accreditation Program

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Protocol for Collecting Samples of Psilocybin Products

ORELAP-SOP-004 Rev 1.0

ORELAP Executive Board and Program Approval Signatures:

Lori Pillsbury, DEQ LEAD Division Administrator — Date: 12/14/2022

Patrick Luedtke, MD, MPH, Oregon State Public Health Laboratory Director — Date: 12/19/2022

Kathleen Wickman, ODA Laboratory Manager — Date: 12/19/2022

Travis Bartholomew, ORELAP Manager — Date: 12/12/2022

Sara Krepps, ORELAP QA Officer — Date: 12/12/2022

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Acknowledgements

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I. Introduction and Scope

Obtaining a representative sample from a decision unit is one of the key elements of accurate laboratory analysis. Laboratories collect representative samples by consistently using standard sampling methods and equipment, preventing contamination of the sample, and maintaining the sample identity within the batch. The laboratory must consistently use documented standard sampling practices, tools, and methods. These practices, tools, and methods must be appropriate for the matrix. If proper protocols are in place and adhered to for sample collection, the laboratory analysis of the sample should reflect the composition of the batch as a whole at the time the sampling occurred, within recognized tolerances.

Controlling cultivation or manufacturing error is the responsibility of the manufacturer of the psilocybin product. Sampling error must be controlled by the laboratory in order to obtain a representative sample of the defined batch. This is accomplished by maintaining the sample identity within the defined batch, prevention of contamination of the sample, and consistent use of standard sampling methods and equipment. Sampling bias must also be controlled by the laboratory to ensure the sample remains representative of the decision unit. Randomized increment locations, as determined by the laboratory's sampling plan, prevent intentional or unintentional sampling bias.

This protocol is for use by ORELAP-accredited laboratories performing psilocybin product sampling as defined in OAR 333-064-0140. It focuses on standard and correct sampling practices that should be reflected in a laboratory's own sampling policies and procedures

II. Records and Documentation

1. ORELAP-accredited laboratories shall maintain standard operating procedures (SOPs) that accurately reflect current sampling activities.
    a. The laboratory's SOP shall be readily accessible to all pertinent personnel.
    b. The laboratory's SOP shall clearly indicate the effective date of the document, the revision number, and the signature of the approving authority.
    c. The laboratory's SOP should use this protocol as minimum requirements and must include additional detail specific to laboratory procedures. In cases where the published method (this protocol) has been modified or where the referenced method (this protocol) is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described in the laboratory's SOP. Any changes to the laboratory's protocol, including use of a selected option, shall be documented and included on the laboratory's sampling form.
    d. All documents shall be controlled and retained in accordance with the TNI Standard as defined in 333-064-0025.

2. ORELAP-accredited laboratories shall maintain sampling plans.

a. The laboratory's sampling plans shall be made available at their location of use.
b. The laboratory's sampling plans shall be based on appropriate statistical methods and shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch.
c. Any deviation from or addition to the laboratory's sampling plan must be documented in detail and shall be included in the final report. The standardized or generic sampling plans can be included in the SOP however specialized client requests or products may require additional information.
d. The laboratory's sampling plans shall document the date and time of sampling.

III. Client Sampling and Testing Requests

The laboratory must have a sampling contract with a client that includes at least the following:

1. A test order containing the information required by OAR 333-333-7020

IV. Planning

Prior to beginning the sampling procedure, the laboratory shall gather information about the type(s) of psilocybin product being sampled, the conditions under which the psilocybin product is being kept, and batch size. This information may be included in the sampling contract or test order. All sampling must be performed by personnel employed by an ORELAP accredited laboratory and must be in accordance with OAR 333-333-7100 and OAR 333-064-0140.

The testing requirements for psilocybin products are in OAR 333-333-7010 to 333-333-7080. The requirements for batch sampling and sample size are in OAR 333-333-7090 to 333-333-7110 and Section VII and Appendix 2 of this protocol. Per Authority request or client request, additional analyses may be required and must be considered in the planning process.

To ensure representativeness, the sampling plan shall be designed such that any part or individual unit in the batch has an equal chance of being selected. The laboratory shall develop procedures, and implement them in the sampling plan, which achieve randomized incremental sampling. Procedures shall include how to:

1. Assign location numbers within containers and among a set of containers holding batch material.
2. Use a random number generator to determine which location to take increments from.
3. Document where each sample increment was taken from batch container(s) and the mass collected for each increment.

Samplers must take extreme care if planning to sample from multiple sites in one day to

ensure contaminants, pathogens, or organisms are not transferred between facilities. Samplers must follow any client requirements on personal protective equipment, sterilization, or sanitization when sampling at the client's facility. If the test order or sampling request includes speciation testing, sampler must ensure equipment used is free of interfering genetic material. The sampler must clean reusable sampling equipment between samplings at a single facility. However, the sampler shall bring enough sets of sampling equipment to use a new set at each facility visited.

V. Sampling Design and Plans

1. Sampling plans shall address factors to be controlled to ensure the subsequent laboratory test results accurately reflect the composition of the batch at the time of sampling. Standardized sampling plans can be included in the laboratory's SOP however specialized client requests or products may require additional information.
2. A site-specific sampling plan that uses statistical design for each project to provide representative sampling must be generated prior to beginning the sampling procedure. The plan shall guide samplers on how to assign divisions based on the type of container holding the batch material. Container types greater than four inches deep shall have divisions assigned to the layer or layers beneath the upper portion of the container. A random number generator programmed to provide assignments based on the total number of divisions in the containers will be employed to indicate which locations increments are pulled from. When there are multiple containers, use existing or arbitrary order of containers to assign numbers to the total of "divisions multiplied by total number of containers."
3. Sampling plans shall be designed to meet specified sample quality criteria. This includes using a sampling plan that meets a 95% confidence level for representative sampling and limits the fundamental sampling error. The most common way to reduce error is by increasing the number of sample increments from the minimum required to compensate for normal batch heterogeneity. Any deviation from or addition to the sampling plan must be documented in detail and shall be maintained in the laboratory's sample records.
4. **Sampling plans must ensure that adequate sample mass is collected for all analyses requested by the manufacturer**. This must include adequate sample mass for re-testing in the event a sample fails a criterion as well as adequate sample mass for any quality control samples required by the laboratory, such as duplicates or matrix spikes. Sampling plans must also indicate the minimum number of sample increments required in Table 1 and Table 2 in Appendix 2 of this protocol.
5. A sampling plan must include at a minimum:
    a. Shape, size, and number of container(s) holding the batch from which sample increments will be collected;
    b. Number of sample increments to be collected;
    c. Total mass of sample needed to perform testing and approximate mass needed for each increment to ensure adequate mass;
    d. Location of where sample increments will be taken within each container

holding the batch.

6. The laboratory must have details in its SOP or a sampling plan, from appropriate industry reference where possible, on how it will achieve random sampling in an unclear decision unit.

VI. Sampling Equipment and Supplies

1. A laboratory should, at a minimum, have the following equipment and supplies for sampling:
    a. Sampling equipment such as spoons, spatulas, transfer pipettes, or other matrix specific tools
    b. Tongs
    c. Corers
    d. Teri-wipes or equivalent
    e. Calibrated field balance (capable of 0.01 g measurements)
    f. Calibrated verification weights appropriate to verify accuracy of field balance
    g. Cleaning supplies – ex: solvent, bleach, 70% Ethanol
    h. Gloves (powder-free, nitrile, sterile)
    i. Mylar bags or amber or colorless glass jars that have been verified to be clean or sterile as needed (for final sample transport and storage)
    j. Desiccant packets or similar to provide moisture control if necessary

2. Cleaning of reusable field sampling equipment:
    a. Reusable field sampling equipment shall be certified clean prior to use by the laboratory.
    b. Cleaning techniques for reusable equipment will vary depending upon the desired analysis.
    c. In general, sampling equipment must be sterile for microbiology samples and clean for chemistry samples.
    d. The laboratory shall perform cleanliness checks on each batch of reusable sampling equipment prior to taking that equipment into the field.
    e. Results from tests following the cleaning procedures must be below the reporting limit of the target analyte(s) for the associated analyses.
    f. If cleanliness checks fail, the sampling equipment must be re-cleaned, sterilized if required, and tested.

3. Field balance calibration verification
    a. The laboratory sampling technician shall verify the calibration of the field balance at the sampling location.
    b. When multiple sampling events occur on the same day, the balance calibration shall be verified at each sampling location.
    c. Balance calibration verifications shall be documented.

VII. Procedure for Sampling Psilocybin Products.

1. Locate the batch of psilocybin product to be sampled. The sampler **must** have access to the entire batch.
2. Check for any signs of non-uniformity within the batch and document the observations.
    a. Some obvious indicators may be different types or sizes of containers, variations in marks and labels, or mixed batch numbers
    b. During sampling, the sampler shall look for physical differences in the psilocybin product being sampled such as color, visible layers, age of whole dried fungi, relative size of items, or texture.
    c. By definition, the batch must be uniform for all factors that appear on the label; hence, variations in the product may indicate non-uniformity in the batch and any sample drawn may not be representative for testing.
    d. The sampler shall note these anomalies in the sample collection report.
3. Review the container label information for batch number and other pertinent information. Do not sample if unique batch numbers are not available.
4. Determine the sample matrix. Psilocybin products fall into two groups for purposes of sampling and testing:
    a. Whole fungi
    b. Homogenized fungi, psilocybin extracts, or edibles.
5. Record the total batch weight and the number of containers comprising the batch. If the product is already in final packaging, record the total number of final package units.
6. Establish which tests will be performed.
7. Ensure that appropriate equipment and containers are available for the tests being performed. For residual solvent analysis, use glass containers that can be properly sealed to prevent the loss of solvent gas and minimize the headspace remaining in the sample container. If colorless glass containers are used, the container must also be enclosed in a mylar bag to protect the sample from light. For whole dried fungi, ensure sample containers contain desiccant packets to maintain product dryness during transport and storage.
8. Select the appropriate sampling tool to ensure that it reaches all portions of the batch.
    a. Sampling tools must be unused or cleaned appropriately prior to use if reusable to prevent cross-contamination of samples. Sampling tools which appear to be dirty or otherwise compromised shall not be used.
    b. To prevent contamination, sampling tools may be cleaned and sealed at the laboratory prior to use or may be cleaned in the field between batches using an appropriate solvent and decontaminant to prevent cross contamination of batches during sampling.
    c. Decontamination waste shall be collected and properly disposed of if not used for analysis.
    d. Where aseptic technique is required, samplers shall observe best practices to prevent microbiological contamination of samples. For an example of aseptic technique, see the FDA (2015) Aseptic Sample Guidelines (Investigations Operations Manual Subchapter 4.3.6).

9. Collect at minimum the required number of sample increments according to Table 1 and Table 2 in Appendix 2 of this protocol and the laboratory sampling plan. Approximately equal amounts of material are to be taken with each probing and from each container. Care must be taken by the sampler to not damage the portion of the material which is not being collected. See sections below for more detail on sampling whole fungi, liquid, semi-solid, or solid sample matrices.
10. Record the number of increments collected, the mass of each increment, and the location within containers the increment was taken.
11. Once taken, seal and label the sample increments, composite sample, primary sample, or duplicate sample as applicable with the following minimum requirements:
    a. Harvest or process lot unique identification number
    b. Name of the laboratory
    c. Laboratory's unique sample identifier
    d. Sampling date and name of sampler
    e. The phrase "PRODUCT NOT TESTED" in bold capital letters no smaller than 12-point font
12. Apply a custody seal to the sample container in a manner that prevents the psilocybin product sample from being tampered with prior to testing.
13. Complete the sampling report while at the sampling location as well as an appropriate chain of custody form as outlined in the TNI Standard.
14. Record the sampling event and material transfer in the psilocybin tracking system (PTS) under the manufacturer's number.

15. **Apply the following guidelines when taking whole fungi samples:**
    a. Determine the total batch weight. Per OAR 333-333-7090, harvest lots must be separated into batches weighing no larger than 1.0 kg. Do not proceed with sampling if batch weight exceeds 1.0 kg.
    b. Determine the required minimum number of increments based on Table 1 in Appendix 2 of this protocol and the laboratory's site-specific sampling plan. Additional sample increments may be collected if needed for laboratory analysis or at client request based on the statistical design in the sampling plan.
    c. Determine the minimum mass of each sample increment such that the total mass of all increments is equal to or greater than 2.0% by weight of the batch.
    d. Carefully pull sufficient mass for each increment as determined above. The combined mass of sample increments shall consist of sufficient material to perform the required laboratory methods. Increments should be approximately equivalent to each other.
    e. Each sample increment shall be taken from a randomly chosen position in the batch, as practically possible. A sample increment shall be taken from each container if possible. If more containers exist than sample increments required, sample from as many as possible to obtain a representative sample.
    f. Psilocybin analyte and psilocin analyte concentrations may vary widely between fruiting bodies in a batch. A high degree of variability may be found between young mushrooms (sometimes referred to as aborts) and older

mushrooms. It is critically important the sampler follow the randomized plan for taking sample increments while also ensuring the sampled material is representative of the batch.

g. Combine each sample increment into the composite sample and store in a mylar bag to protect sample material from light and moisture. Secure the bag or bags with tamper-proof seals.

16. **Apply the following guidelines when taking liquid samples:**
    a. If the sample increments are to be taken from a bulk container, ensure proper homogenization of the product prior to taking the sample by mixing the container thoroughly and employing any process for homogenization that the manufacturer would use to disperse the liquid material into packaging.
    b. Determine the total batch weight and the required number of increments based on Table 2 in Appendix 2 of this protocol and the site-specific sampling plan for the client.
    c. Select an appropriate sampling device for pulling bulk liquid from a container.
    d. Collect the appropriate number of sample increments.
    e. Combine all sample increments in the selected container type to form the primary sample for testing. If residual solvent testing is required, ensure minimal headspace remains in sample container and lid is secure.
    f. Complete the same procedure with a second set of equivalent sample increments to form the duplicate sample.
17. **Apply the following guidelines when taking solid or semi-solid samples:**
    a. Determine the total batch weight. If the batch is in final product packaging, determine how many final package units there are and the total batch mass.
    b. Determine the required number of increments based on Table 2 in Appendix 2 of this protocol and the site-specific sampling plan for the client.
    c. Select an appropriate sampling device based on the batch matrix.
    d. Collect the appropriate number of sample increments.
    e. Combine all sample increments in the selected container type to form the primary sample for testing. If residual solvent testing is required, ensure minimal headspace remains in sample container and lid is secure.
    f. Complete the same procedure with a second set of equivalent sample increments to form the duplicate sample.

VIII. Sampling Records/Field Data

1. At the time samples are collected the sampler shall complete a sampling report form for each batch sampled. Sample report forms shall include at a minimum the following information:
    a. Name and address of manufacturer including license number
    b. Psilocybin product type.
    c. Total weight of batch.
    d. Sample identification number (ID) which can be linked through documentation to the manufacturer's unique batch ID.
    e. Total number of containers sampled.

   f. Number of sample increments taken from each container.
   g. Number of sample increments combined into a primary and duplicate sample, if applicable
   h. Number of sample containers collected.
   i. Weight and location of each sample increment.
   j. Total weight sampled.
   k. Sampling plan document control ID and revision date.
   l. Sampling procedure document control ID and revision date.
   m. Description of equipment and tools used.
   n. Address where sampled.
   o. Date sampled.
   p. ORELAP laboratory identification number.
   q. Lab license number.
   r. Sampler's identification and/or signature.
   s. Name of responsible party for the batch and transport information.
   t. Receiving laboratory and types of tests required or requested.
2. A chain of custody form shall be used. The tracking manifest in the psilocybin tracking system (PTS) may function as the chain of custody so long as it includes at least the following information:
   a. Sampler's name
   b. Sampling location
   c. Unique sample ID
   d. Sampling date/time
   e. Sample mass
   f. Custody transfer signatures
   g. Custody transfer dates/times
3. If any of the above information requested on the sampling report form or chain of custody is unavailable, indicate "N/A" in the appropriate space with an explanation as to why the information is not available.
4. All sampling report forms must be signed by the sampler.

IX. Transportation and Handling of Samples

1. Transport the composite sample to the laboratory performing the analysis by the most expedient, secure, and legal means to ensure that the sample continues to be representative of the batch sampled and the chain of custody form continues to document sample integrity. Note: Current law does not permit shipping in any form such as USPS or FedEx.
2. Containers for sample transport must be designed to protect the sample from moisture and temperature extremes and to prevent damage, contamination, spillage, or commingling of the sample during transport. The required container for sampling is a mylar bag or amber or colorless glass jar with a PTFE-lined lid and should be appropriate for the sample matrix and the tests required. If a colorless glass jar is used, the container must also be placed in a mylar bag to protect the sample from light exposure. A tamper-proof seal is required on each sample container.
3. The laboratory must have detailed procedures on maintaining custody and sample

integrity during transport. These procedures should take into consideration controlling temperature and other environmental factors.

4. Submit the composite sample to the laboratory in its entirety. In a situation where the sample must be split for analysis by two different laboratories, for example when solvent analysis is subcontracted to another laboratory, the composite sample(s) shall be homogenized by the laboratory's approved sample homogenization process prior to subsampling. Care must be taken to maintain sample integrity during this process. This shall be reflected on the chain of custody.
5. Samples must always be identified by labeling or marking the sample container to associate them with the batch from which they originated and with the sampling report.

X. Quality Assurance and Quality Control

The sampler must be prepared to collect adequate sample mass for all analyses requested by the manufacturer. This must include adequate sample mass for re-testing in the event a sample fails a criterion as well as adequate sample mass for any internal quality control samples required by the laboratory, such as laboratory duplicates or matrix spikes.

1. Sampler qualifications
    a. Basic qualifications for samplers of psilocybin products are:
        i. Physically able to perform the duties of a sampler;
        ii. No conflict of interest;
        iii. Employed by an ORELAP accredited laboratory;
        iv. Pass initial and ongoing demonstrations of capability as defined by the laboratory (see below);
        v. Permitted as a licensed representative under Oregon Psilocybin Services rules to transport the required quantity of psilocybin products.
    b. Required education and training for samplers:
        i. Initial training: training shall include principles, procedures, and policies of sampling. The training shall be performed by an instructor that has demonstrated competency in performing the sampling methods referenced or equivalent. After personnel go through initial training, they are qualified to train others in their organization.
        ii. Initial field or on-the-job training: 8-hours of training on various sampling techniques.
        iii. Continuing education: periodic refresher training shall be done annually.

2. Demonstration of Capability

    a. Prior to acceptance and institution of any accredited method, a satisfactory initial demonstration of capability (IDOC) is required. The laboratory shall have a documented procedure for performing the sampling IDOC. The IDOC will be repeated: 1) every time there is a change in personnel or method; and 2) when the method has not been performed by the laboratory within a 12-month period.

    b. This procedure shall employ one of the following approaches to demonstrating capability:

        i. Comparison of replicate samples within defined Relative Standard Deviation (%RSD) acceptance criteria.

        ii. Comparison of a sample collected to that of one collected by personnel with an existing IDOC within defined Relative Percent Difference (%RPD) acceptance criteria.

    c. Thereafter, ongoing continuing demonstration of capability (CDOC) is required annually. The laboratory shall have a documented procedure for performing the CDOC. The laboratory shall retain documentation verifying CDOC for each sampler and make this documentation available to ORELAP upon request.

3. Field QC samples

    a. Duplicates

        i. A duplicate sample is required when sampling a batch of homogenized fungi, extract, or edible psilocybin product. The sample duplicate must be collected using the same procedure as the primary sample. Comparison of primary and duplicate potency results must be evaluated against %RPD or RSD requirements as specified in OAR 333-333-7040.

    b. Equipment blanks

        i. Equipment rinse blank samples provide a QC check on the potential for cross contamination by measuring the effectiveness of the decontamination procedures on the sampling equipment. An equipment blank is required to validate equipment cleaning procedures that occur in the field during sampling. It is recommended but not required that an equipment blank is collected upon each sampling event using new or previously certified equipment to demonstrate the equipment was not a source of contamination.

ii. The equipment blank consists of an aliquot of the cleaning solution as applicable, rinsed across sample collection equipment after cleaning has taken place. If the analytes of interest are detected in the equipment rinsate, the detected concentrations will be compared to the associated sample results to evaluate the potential for contamination.
iii. The equipment blank must pass the required analysis at <LOQ for cleaning validation.
iv. If the equipment blank is collected at the sampling event, the lab must have detail in the sampling plan or procedures as to how to evaluate it and what actions to take if the evaluation demonstrates unacceptable results.

c. Transport blank
   i. A transport blank is **required** as part of a sampling plan that includes collection for solvent analysis.
   ii. A single transport blank must be collected and analyzed per trip regardless of the amount of sampling events during the trip and each event's samples must be linked to the acceptability of its result.
   iii. The transport blank must pass solvent analysis at <LOQ for the sampling event to be considered valid.

4. Field audits

   a. The laboratory shall adopt an ongoing system for performing audits of field activities. Field audits must be conducted periodically and in accordance with a predetermined schedule and procedure. The goal of the field audit is to verify that the sampling operation continues to comply with the requirements of the regulations and is being performed according to the laboratory's sampling SOP. Audits are to be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. The field audit shall address all elements of the sampling activities and shall be documented.

   b. When field audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the field sampling activities, the associated laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that test results may have been affected. Laboratory management shall have a policy that specifies the time frame for notifying clients of events that cast doubt on the validity of the results. Follow up audit activities shall verify and document the implementation and effectiveness of any corrective actions taken as a result of the field audit.

   c. Required components of the field audit program:
      i. Review sampling and performance records from the preceding year for deficiencies in the application of sampling protocol.

ii. Observe the sampler conducting sampling procedures.
iii. Record any deficiencies and initiate corrective action.

XI. References

NDA (2006). *Standard operating procedure on sampling and analysis of agricultural products of plant origin to determine agrochemical residue levels and risk management as part of the export inspection and certification in terms of agricultural products standards act.*

FDA (2015). *Salmonella sampling plan*. Investigations Operations Manual 2015. ASTA.

Clean, Safe Spices. Guidance from the American Spice Trade Association.

FDA, *Guidelines for Food Spice Labeling.* Code of Federal Regulations Title 21, Volume 2. http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=101.2 2)

FDA. The Food Defect Action Levels: *Levels of natural or unavoidable defects in foods that present no health hazards for humans.* Code of Federal Regulations Title 21, Part 110.

FDA (2015). Subchapter 4.3.6: Aseptic Sample. *In:* Investigations Operations Manual Chapter 4: Sampling. 106 pp.

Sampling and Sample Handling Working Group FDA, AAFCO, AFDO, APHL and Industry, October 2015. *Good Samples: Guidance on Obtaining Defensible Samples*.

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http://www.nelac-institute.org/content/CSDP/standards.php

Oregon Administrative Rules, *Psilocybin* Chapter 333, Division 333.

Oregon Administrative Rules, *Accreditation of Laboratories,* Chapter 333, Division 64.

ORELAP-SOP-001 Revision 4.1 *Protocol for Collecting Samples of Usable Marijuana.*

ORELAP-SOP-002 Revision 4.3 *Protocol for Collecting Samples of Cannabinoid Concentrates, Extracts, Products, and Industrial Hemp-derived Vapor Items.*

Standard Methods 20th Edition (1998); 1020 Quality Assurance

Technical and Regulatory Guidance, Incremental Sampling Methodology, February 2012, Prepared by The Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team

Appendix 1 – Definitions

**If there are any inconsistencies between the definitions below and the definitions in OAR 333, Divisions 333 or 64, the definitions in the rules take precedence.

Authority means Oregon Health Authority

Batch means a quantity of psilocybin product from a harvest lot or a process lot.

Chain of Custody Form means a form completed by laboratory personnel that documents the collection, transport, and receipt of samples by the laboratory. (Sample tracking document)

Composite Sample means a sample composed of all sample increments taken from a batch.

Container means a sealable, hard- or soft-bodied receptacle in which a psilocybin product is placed during sampling, transport, and storage.

Decision Unit or Sampling Unit means the material from which the primary sample(s) is collected and to which the inference(s) is made.

Duplicate Sample means sample increments taken in an identical manner to sample increments taken for the primary sample and representative of the same psilocybin product being sampled that is prepared and analyzed separately from the primary sample.

Edible psilocybin product means psilocybin extract or homogenized fungi that has been incorporated into a food item or potable beverage.

Equipment Blank means a sample of analyte-free media, collected after decontamination and prior to sampling, which has been used to rinse the sampling equipment after cleaning to validate cleaning procedure or between sampling batches to demonstrate lack of contamination.

Extract means a product made by separating psilocybin from fungi by using a solvent.

Fundamental Sampling Error (FSE) means a measure of the compositional heterogeneity, which is controlled through the collection of sufficient sample mass (mass is inversely proportional to error).

Heterogeneity means the state or quality of being heterogeneous.

Heterogeneous means non-uniform or consisting of dissimilar parts or components.

Homogeneous means a psilocybin product has uniform composition and properties throughout each batch or process lot.

Homogenized fungi means dried fruiting bodies or mycelium that have been mixed by powdering or other techniques which uniformly distribute psilocybin throughout the product.

Label means a tag or other device attached to or written, stamped, or printed on any container or accompanying any batch in bulk stating all required batch information.

Laboratory means a laboratory that is accredited under ORS 475A.606 to sample or

conduct tests on psilocybin products and licensed by the Authority under ORS475A.594.

ORELAP means the Oregon Environmental Laboratory Accreditation Program administered by the Authority pursuant to ORS 438.605 to 438.620.

Primary Sample means a composite sample composed of sample increments and tested for the required analysis methods.

Process Lot has the meaning given that term in OAR 333-333-1010.

Psilocin analyte means 4-hydroxy-N,N-dimethyltryptamine, Chemical Abstracts Service Number 520-53-6.

Psilocybin analyte means 4-phosphoryloxy-N,N-dimethyltryptamine, Chemical Abstracts Service Number 520-52-5.

Psilocybin product has the meaning given that term in OAR 333-333-1010.

Relative Percent Difference means the comparison of two quantities while taking into account the size of what is being compared. If the final result (i.e., psilocin analyte) is <LOQ in either sample, the absolute value of the LOQ is used in the equation.

$$\%RPD = \frac{|(sample - duplicate)|}{(sample + duplicate)/2}\ x\ 100$$

Relative Standard Deviation means the standard deviation expressed as a percentage of the mean recovery, i.e., the coefficient of variation multiplied by 100. If the final result (i.e., psilocin analyte) is <LOQ in either sample, the absolute value of the LOQ is used in the equation.

Standard Deviation

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

Relative Standard Deviation

$$\%RSD = \frac{S}{\bar{x}}\ x\ 100$$

S = standard deviation.

n = total number of values.

x_i = each individual value used to calculate mean.

$\bar{x}$ = mean of n values.

Representative Sample means a sample obtained according to an incremental sampling procedure designed to ensure that the different parts of a batch or lot or the different properties of a batch or lot are proportionally represented.

Sample means an amount of psilocybin product collected by laboratory personnel from a manufacturer for the purpose of laboratory testing.

Sample Increment means an amount of a psilocybin product collected by laboratory personnel from a manufacturer that may be combined into a sample for purposes of testing.

Sample Quality Criteria (SQC) means a series of statements that clarify a sampling program's technical and quality needs to support defensible decisions, including statement of the question to be answered, definition of the decision unit, and the desired confidence in the inference.

Sealed means secured in such a way as to provide authenticity or integrity of the sample.

Sterile means the removal of all living microorganisms and other pathogens from a psilocybin product by treating it with approved chemicals or subjecting it to high heat.

TNI Standard means the TNI Environmental Laboratory Standard as defined in OAR 333-064-0025.

Transport Blank means a sample of analyte-free media which has been carried into the field and returned to the laboratory and is used to demonstrate transportation of samples did not add volatile contamination in solvent analysis.

Whole fungi means dried fruiting bodies or mycelium of *Psilocybe cubensis*, or portions thereof, that have not been homogenized.

Appendix 2 – Sample size and increments

Per OAR 333-333-7100, the sample size must be sufficient to complete all analyses required.

The required sample increments for a given batch of psilocybin products varies depending upon the product type and the size of the batch. Taking more sample increments than required is encouraged and will improve representativeness of the sample in relation to the batch.

Whole fungi:

1. The number of required sample increments for a batch are based on the size of the batch. See Table 1, below.
2. Each increment shall be taken from the batch according to the random and representative sampling approach described in this protocol and in the laboratory's sampling plan.
3. Record the mass and location within the batch for each increment.
4. The total mass of all increments must be equal to or greater than 2.0% of the batch mass.
5. Each increment is placed into a mylar bag to form the composite sample. After homogenization at the laboratory, the composite sample is prepared and analyzed for required tests.

Table 1 – Sample increment requirements based on size of dried whole fungi batch.

Batch Weight		Sample Increments Required
Ounces	Grams	
0-3.52	0-100	7
3.53-10.58	100.1-300	8
10.59-21.16	300.1-600	9
21.17-35.27	600.1-1000	10

Homogenized fungi, psilocybin extracts, or edibles:

1. The number of required increments for a batch are based on the size of the batch. See Table 2, below.
2. The mass of each increment is not specified, but the combined mass of all increments must be sufficient to complete all required analyses, laboratory QC, and re-analyses.
3. The specified number of increments are taken from the batch following the random and representative sampling approach described in this protocol and in the laboratory's sampling plan.
4. The mass and location within the batch for each increment is recorded and each increment is placed into the selected sample container. This is the primary sample.
5. An equivalent number of increments sampled using the same random and

representative procedure are combined into the duplicate sample.

6. The primary and duplicate samples are put in separate containers and are prepared and analyzed separately.

Table 2 – Sample increment requirements based on size of psilocybin extract, edible, or homogenized fungi batch.

Batch Weight		Sample Increments Required	
Pounds	Kilograms	Primary	Duplicate
0-3.31	0-1.50	3	3
3.32-6.61	1.51-3.00	4	4
6.62-13.23	3.01-6.00	5	5
13.24 +	6.01 +	6	6

Appendix 3 – Document history

Table 3 – Revision history of this protocol

Revision	Date	Summary of changes made, and initials of editor
1.0	12/9/2022	Initial draft. STJ 07/22/2022. Incorporated changes suggested during RAC convened on 9/12/2022. STJ 10/3/2022. Incorporated editorial change raised during ORELAP executive team review. TJB/STJ 12/12/2022.

OAR 333-064-0140, Exhibit D

Table 1 – Accuracy requirements for laboratory control samples (LCS).

Test	Analyte	LCS Limits (%R)
Potency in accordance with OAR 333-333-7040	Psilocybin analyte	70 - 130
	Psilocin analyte	70 - 130
Solvent testing in accordance with OAR 333-333-7050	Methanol	60 - 120
	Acetic acid	60 - 120