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CONTACT: Melissa Jackson

503-746-7454

publichealth.rules@odhsoha.oregon.gov

7202 NE Evergreen Parkway

Hillsboro, OR 97124

Filed By:

Public Health Division

Rules Coordinator

AMEND: 333-064-0100

REPEAL: Temporary 333-064-0100 from PH 18-2025

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RULE SUMMARY: Amend OAR 333-064-0100: Reattaching three documents referenced in OAR 333-064-0100 that were inadvertently left off of a statutory minor correction (SMC) that was filed on April 21, 2025, to correct the outlining of the rule text. The three documents are: Usable Marijuana: ORELAP-SOP-001 Rev 4.1; Concentrates, Extracts, Products, and Industrial Hemp-derived Vapor Items: ORELAP-SOP-002 Rev 4.3; and Exhibit C, Table 1: Accuracy requirements for laboratory control samples (LCS). No changes are being made to the rule text or to the attached documents.

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 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: 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 Oregon Liquor and Cannabis Commission's (Commission's) seed to sale system, as required by the 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 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 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 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.¶¶

(B) 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.¶¶

(C) 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:¶¶

$$M \text{ total delta-9 THC} = M \text{ delta-9 THC} + 0.877 * M \text{ delta-9 THCA.}¶¶$$

(b) Total CBD must be calculated as follows, where M is the mass or mass fraction of CBD and CBDA:¶¶

$$M \text{ total CBD} = M \text{ CBD} + 0.877 * M \text{ CBDA.}¶¶$$

(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 dry weight. 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:¶¶

$$P \text{ delta-8 THC(dry)} = P \text{ delta-8 THC(wet)} / [1 - (P \text{ moisture}/100)]¶¶$$

$$P \text{ total delta-9 THC(dry)} = P \text{ total delta-9 THC(wet)} / [1 - (P \text{ moisture}/100)]¶¶$$

$$P \text{ total CBD(dry)} = P \text{ total CBD(wet)} / [1 - (P \text{ moisture}/100)]¶¶$$

(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¶¶

$$S = (((x_i - x)^2) / (n - 1))¶¶$$

Relative Standard Deviation¶¶

$$\%RSD = (S / x) * 100¶¶$$

(c) For purposes of this section:¶¶

(A) S = standard deviation.¶¶

(B) n = total number of values.¶¶

(C) x_i = 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 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 in order to comply with OAR 333-007-0300 to 333-007-0600 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, 475C.544, 475C.560, 438.620
- Statutes/Other Implemented: ORS 438.605, 438.610, 438.615, 475C.544, 475C.560, 438.620

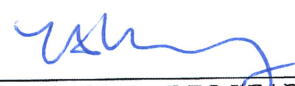
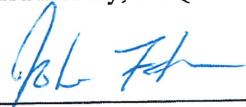
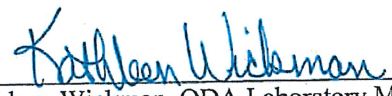
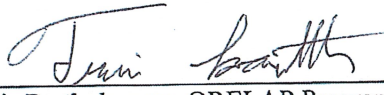
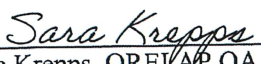
RULE ATTACHMENTS MAY NOT SHOW CHANGES. PLEASE CONTACT AGENCY REGARDING CHANGES.

	<h1 style="margin: 0;">Oregon</h1> <h2 style="margin: 0;">Environmental Laboratory Accreditation Program</h2>	
<p>Department of Agriculture, Laboratory Services Division Department of Environmental Quality, Laboratory and Environmental Assessment Division Oregon Health Authority, Public Health Laboratory</p>	<p>Oregon State Public Health Laboratory 7202 NE Evergreen Pkwy, Suite 100 Hillsboro, OR 97124 (503) 693-4122 FAX (503) 693-5602 TTY (503) 731-4031</p>	

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	03/10/2022 _____ Date
 _____ John Fontana Ph.D., Oregon State Public Health Laboratory Director	03/09/2022 _____ Date
 _____ Kathleen Wickman, ODA Laboratory Manager	March 8, 2022 _____ Date
 _____ Travis Bartholomew, ORELAP Program Manager	3/08/2022 _____ Date
 _____ Sara Krepps, ORELAP QA Officer	03/10/2022 _____ Date

Protocol for Collecting Samples of Usable Marijuana

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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.

Protocol for Collecting Samples of Usable Marijuana

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.

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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

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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.

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- 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.

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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.

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- 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:

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- 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.

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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

Association of American Seed Control Officials. 2006. *AASCP Handbook on Seed Sampling*, Rev. Ed. 2006 (W.R. Guerke, Ed.). 41 pp.

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*

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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*.

TNI Environmental Laboratory Standard, Volume 1 *Management and Technical Requirements for Laboratories Performing Environmental Analysis*. TNI EL Standard as defined in 333-064-0025.

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

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

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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 ORS 475B.560.

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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 ORS 475B.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.

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

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

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

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

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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.

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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

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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

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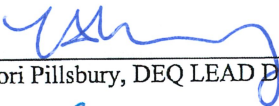
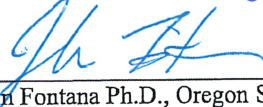
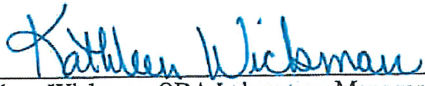

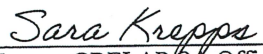
		former section X and combined former section X.5 with section X.4. Minor updates for consistency and typo fixes. Includes fixing reference to minimum number of sample increments in Table 2. STJ 7/20/2020
4.1		Minor formatting updates, removed historical authorship captured in previous versions. Added 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

	<h1 style="margin: 0;">Oregon</h1> <h2 style="margin: 0;">Environmental Laboratory Accreditation Program</h2>	
<p>Department of Agriculture, Laboratory Services Division Department of Environmental Quality, Laboratory and Environmental Assessment Division Oregon Health Authority, Public Health Laboratory</p>	<p>Oregon State Public Health Laboratory 7202 NE Evergreen Parkway, Suite 100 Hillsboro, OR 97124 (503) 693-4122 FAX (503) 693-5602 TTY (503) 731-4031</p>	

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	<u>03/10/2022</u> _____ Date
 _____ John Fontana Ph.D., Oregon State Public Health Laboratory Director	<u>03/08/2022</u> _____ Date
 _____ Kathleen Wickman, ODA Laboratory Manager	<u>March 8, 2022</u> _____ Date
 _____ Travis Bartholomew, ORELAP Program Manager	<u>3/08/2022</u> _____ Date
 _____ Sara Krepps, ORELAP QA Officer	<u>03/10/2022</u> _____ Date

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

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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.

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

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.

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- 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

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- 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

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- 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.

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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.

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- 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.

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- 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.

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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

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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

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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)

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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

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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.

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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=101.22>)

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*.

National Environmental Field Activities Program (NEFAP); TNI EL Standard (2009), Volume 1 *Management and Technical Requirements for Laboratories Performing Environmental Analysis*.

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

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

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

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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

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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.

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

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

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

S = standard deviation.

n = total number of values.

x_i = each individual value used to calculate mean.

\bar{x} = mean of n values.

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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.

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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

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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: $X = (\text{batch weight in kg} / 50) * 1.5$ 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

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		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
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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

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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

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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

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