ORELAP

# Oregon
## Environmental Laboratory Accreditation Program

Department of Agriculture, Laboratory Services Division
Department of Environmental Quality, Laboratory and Environmental Assessment Division
Oregon Health Authority, Public Health Laboratory

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

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

*ORELAP-SOP-002 Rev 4.3*

**ORELAP Executive Board and Program Approval Signatures:**

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

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

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

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

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

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## Acknowledgements

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

## I. Introduction and Scope

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

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

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

## II. Records and Documentation

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

2. ORELAP-accredited laboratories shall maintain sampling plans.

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

## III. Client Contracts; Client Sampling and Testing Requests

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

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

## IV. Planning

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

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

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

## V. Sampling Design and Plans

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

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

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

## VI. Sampling Equipment and Supplies

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

2. Cleaning of Field Sampling Equipment

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

## VIII. Sampling Records/Field Data

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

following information:

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

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

## IX. Transportation and Handling of Samples

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

reflected on the chain of custody.

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

## X. Quality Assurance and Quality Control

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

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

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

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

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

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

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

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

3. Field QC Samples

   a. Duplicates

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

   b. Equipment Blanks

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

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

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

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

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

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

4. Field Audits

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

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

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

## XI. References

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## Appendix 1 – Definitions

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

**Authority** means Oregon Health Authority

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

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

**Commission** means the Oregon Liquor and Cannabis Commission.

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

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

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

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

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

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

**Heterogeneity** means the state or quality of being heterogeneous.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Standard Deviation

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

Relative Standard Deviation

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

S = standard deviation.

n = total number of values.

$x_i$ = each individual value used to calculate mean.

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

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

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

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

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

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

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

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

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

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

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

## Appendix 2 – Sampling Requirements

### Random sampling

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

### Sample size and increments

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

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

**B, Table 7)**

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

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

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

## Table 2 – Revision history of this SOP.

| Revision | Date | Summary of changes made, and initials of editor |
|---|---|---|
| 4.0 | 07/22/2020 | Major updates and re-formatting based on input from Scott Hoatson (former ORELAP QA Officer) and Department of Justice.<br>Updated: OSPHL address; executive board and ORELAP staff names/titles as needed; definitions in order to match OARs and ORS.<br>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.<br>Combined: sampling design and plans and representative sampling sections; forwarding samples section with transportation section<br>Minor updates and typo fixes for consistency with Useable Marijuana sampling SOP.<br>STJ 07/22/2020 |
| 4.1 | 10/19/2020 | Minor updates to include definition of kief and inclusion of consideration of tricky/unusual sample matrices in section 17 e. STJ 10/19/2020 |
| 4.2 | 9/27/2021 | Minor formatting updates, removed historical authorship captured in previous versions, added reference to industrial hemp-derived vapor items. Updated definitions in Appendix 1 to align with those in applicable administrative rules. Updated Tables 5 and 7 to show more accurate mass ranges. STJ 9/27/2021 |
| 4.3 | 11/18/2021 | Added information about finished inhalable cannabinoid products. Removed reference to control studies. Updated container requirements |

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