ORELAP

Oregon

Environmental Laboratory Accreditation Program

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Protocol for Collecting Samples of Psilocybin Products

ORELAP-SOP-004 Rev 1.0

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I. Introduction and Scope

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

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

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

II. Records and Documentation

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

2. ORELAP-accredited laboratories shall maintain sampling plans.

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

III. Client Sampling and Testing Requests

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

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

IV. Planning

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

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

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

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

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

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

V. Sampling Design and Plans

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

holding the batch.

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

VI. Sampling Equipment and Supplies

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

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

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

VII. Procedure for Sampling Psilocybin Products.

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

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

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

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

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

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

VIII. Sampling Records/Field Data

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

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

IX. Transportation and Handling of Samples

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

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

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

X. Quality Assurance and Quality Control

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

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

2. Demonstration of Capability

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

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

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

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

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

3. Field QC samples

    a. Duplicates

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

    b. Equipment blanks

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

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

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

4. Field audits

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

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

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

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

XI. References

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Appendix 1 – Definitions

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

Authority means Oregon Health Authority

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

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

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

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

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

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

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

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

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

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

Heterogeneity means the state or quality of being heterogeneous.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Standard Deviation

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

Relative Standard Deviation

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

S = standard deviation.

n = total number of values.

x_i = each individual value used to calculate mean.

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

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

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

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

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

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

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

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

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

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

Appendix 2 – Sample size and increments

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

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

Whole fungi:

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

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

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

Homogenized fungi, psilocybin extracts, or edibles:

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

representative procedure are combined into the duplicate sample.

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

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

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

Appendix 3 – Document history

Table 3 – Revision history of this protocol

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