THE HIGHGRADE TESTING LAB

Standard Operating Procedure 301

Collecting Samples of Usable Marijuana

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Approved by: Date

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1. Introduction

1.1 Overview

All medical marijuana products in Oklahoma must pass OMMA testing guidelines before going into the market. This SOP outlines how to collect samples from growers, processors, and dispensaries in order to get an accurate sample that is representative of the entire batch of product.

1.2 Scope

This procedure is designed to standardize the collection of medical marijuana of samples by Highgrade analysts or other staff.

1.3 Referenced Documents

Osterbaur, N., S. Krepps, J. Sackett, C. Holladay, E. Wendt, D. Wells, S. Price, and J. Kristof. "Protocol for Collecting Samples of Usable Marijuana." Ed. S. Swantek, M. Moore, L. Garcia, C. Redman, and G. Ward. *Oregon Environmental Laboratory Accreditation Program* Rev. 2.0 ORELAP-SOP.001 (2016). *Oregon Health Authority*. Oregon Public Health Division, 21 June 2016. Web. July. 2016. <https://public.health.oregon.gov/LaboratoryServices/EnvironmentalLaboratoryAccreditation/Documents/sop-001.pdf>.

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

Protocol for Collecting Samples of Usable Marijuana ORELAP SOP-001 Revision 2.0

Protocol for Collecting Samples of Cannabis Concentrates and Extracts: ORELAP SOP-002 Revision 2.0

Protocol for Collecting Samples of Cannabinoid Products: ORELAP SOP-003 Revision 2.0

Standard Methods 20th Edition; 1020 B Quality Control, 11. QC Calculations, f. Duplicate Sample.

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

Standard Methods 20th Edition; 9020 B, 8. & 9.

EPA School Guidance; Appendix E, Field Duplicates.

1.4 Definitions

Batch

A quantity of cannabinoid concentrate or extract or cannabinoid product from a process lot.

CBD

Cannabindiol

Chain of Custody

The chronological documentation showing the collection, custody, control, transfer, analysis, and disposition of a sample

Composite sample
A sample containing all primary samples taken from a batch.

Container
A sealed, hard or soft bodied receptacle in which a marijuana item is placed or a physical division of an extract or concentrate process lot for random sampling.

Deep Container
A Three-Dimensional receptacle containing marijuana items - length × width × height (i.e. bulk liquid in a bucket, packaged edibles or bulk flower in a Tupperware bin)

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

Equal Portions
+/- 20%.

Equipment blank
A sample of analyte-free media, collected after decontamination 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.

Field Duplicate Sample
Sample taken in an identical manner to primary sample, representative of the same marijuana items, that will be prepared and analyzed separate from the primary sample.

Flat Container
A Two-Dimensional surface containing marijuana items – length × width only (i.e. solids on a sheet, a single layer of flowers on a tray, or packaged edibles on a table).

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

Harvest Lot
A specifically identified quantity of marijuana that is cultivated utilizing the same growing practices and harvested within a 72-hour period at the same location and cured under uniform conditions.

Heterogeneity
The state or quality of being heterogeneous.

Heterogeneous
Non-uniform or consisting of dissimilar parts or components.

Homogeneous

Uniform in composition within recognized tolerances.

Label

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.

Marijuana item

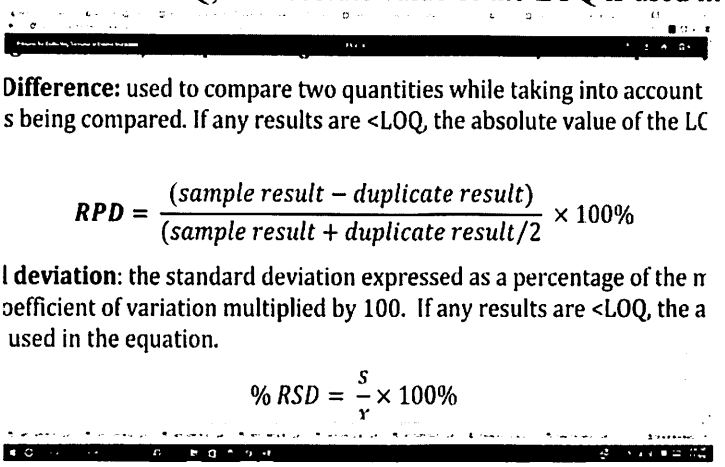
Marijuana, Usable Marijuana, a cannabinoid product or a cannabinoid

Primary Sample

A sample composed of sample increments and tested for the required and primary analysis methods.

Relative Percent Difference

Comparing two quantities while taking into account the "sizes" of the things being compared. If any results are < LOQ, the absolute value of the LOQ is used in the equation.



Difference: used to compare two quantities while taking into account
s being compared. If any results are <LOQ, the absolute value of the LC

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

l **deviation**: the standard deviation expressed as a percentage of the n
oefficient of variation multiplied by 100. If any results are <LOQ, the a
used in the equation.

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

Relative Standard Deviation

The standard deviation expressed as a percentage of the mean recovery, ie; the coefficient of variation multiplied by 100. If any results are < LOQ, the absolute value of the LOQ is used in the equation.

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

$$s = \sqrt{\sum_{i=0}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

where:
s = standard deviation,
n = total number of values,
x_i = each individual value used to calculate mean, and
$\bar{x}$ = mean of n values

Representative Sample
A sample obtained according to a 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
An amount of marijuana item collected by sampling personnel from a registrant or licensee and provided to a laboratory for testing.

Sample Increment
An amount of a marijuana item collected by laboratory personnel from a registrant or licensee that may be combined into a sample for purposes of testing, or in the case of a control study, is tested individually.

Sample Quality Criteria
A series of statements that clarify program technical and quality needs to support defensible decisions, including statements of the questions to be answered, definition of the decision unit, and the desired confidence in the inference.

Sealed
Secured to provide authenticity or integrity.

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

Usable Marijuana
The dried and cured 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.

2. Quality Control

2.1 Equipment

2.1.1 Instrumentation

a) Field Balance
b) Thermometer

2.1.2 Supplies

a) Calibrated verification weights
b) Ice packs
c) Gloves
d) Transfer syringes
e) Tamper-proof tape
f) Mylar bags or other storage containers
g) Cooler
h) 70% isopropyl alcohol
i) 70% Ethanol
j) Kim wipes

2.2 Control Samples

2.2.1 Field Duplicates

Field duplicates are recommended for any usable marijuana sampling event. Duplicates must use the same procedure and contain the same number of sample increments as the primary sample. The field duplicate may serve as the reserve testing sample as required per OMMA.

2.3 Quality Assurance Measures

Sampling plans must meet a 95% confidence level for representative sampling and limit fundamental sampling errors. The most common way to achieve this is by increasing the number of sample increments to compensate for normal batch heterogeneity. It is recommended that a minimum of 7 increments be taken for the sample to be considered representative.

The sampler must be prepared to collect adequate sample masses for all analyses requested by the grower or processor. This mass must adequately include sample masses for any quality-controlled samplers required by the laboratory

To prevent contamination, filed sampling tools must be certified cleaned at the laboratory prior to use. They may also be cleaned in the field between batches using the appropriate solvent and decontaminant to prevent cross-contamination. Any disposable sampling items used may be properly discarded.

3. PROCEDURE

A. Locate the batch to be sampled
B. Ensure the minimum required information is given for the sample, such as batch number, lot number, sample name.
C. The maximum batch size is 10 pounds. One full compliance test covers 10 pounds of product. A minimum sample of 5 grams is required for flower tests and 2 grams are required for extract tests. A duplicate sample to be held in reserve is also required per OMMA guidelines. This means a total sample size of 10 grams for flower tests and 4 grams for extract tests.
D. Determine the minimum number of sample increments needed for sampling. At least 7 increments are recommended
E. Determine the number of quadrants for sampling based off of the size and shape of the container. This number must be greater than or equal to the number of increments determined in D. See Appendix A for model of quadrants.
F. Use a random number generator or dice to randomly number the quadrants.
G. Sample randomly from the quadrants in a random order.
H. Visually inspect samples for uniformity. Note any irregularities.
I. Combine all increments into the sample container, record the final weight of the sample, and label with the appropriate Confident Cannabis assigned number and "FOR TESTING ONLY." Confident Cannabis ID numbers are only generated for samples when the order is placed online before transporting to the lab. If the order has not been placed online, then labeling the sample with the strain name and batch number is sufficient.
J. Fill out 2 copies of a chain of custody form. One copy goes to the client, the other copy goes to the lab.

3.1 SAMPLE TRANSPORTATION

A. Transport the sample to Highgrade Labs following OMMA license transportation regulations. Note: Shipping samples to Highgrade Labs is not permitted under any circumstance.
B. Protect the samples from moisture and temperature extremes. When outdoor temperatures are greater than 15°C, the use of an insulated cooler and ice packs is necessary.

3.2 Preventing Cross-contamination between Samples

A. If multiple batches are being sampled, sample each batch individually. Use a separate container for each batch, labeled with the confident cannabis ID number or the strain name and batch number.
B. In order to prevent cross-contamination, gloves should be switched out in between each sample.
C. In order to prevent cross-contamination, any tools or scales used during sampling should be thoroughly cleaned with either an isopropanol or ethanol solution in between batches. The alcohol solution should be at least 70% alcohol to maintain sterility.

3.3 Samples Other than Flower or Concentrate

A. Highgrade Labs has the ability to test a variety of medical-marijuana products and medical-marijuana derived products. This includes:
    - Bath bombs and other detergent based products (minimum sample size of 1 bath bomb, or at least 5 grams, plus a reserve sample)
    - Topicals/salves (minimum sample size of 1 jar, or at least 5 grams, plus a reserve sample)
    - Edibles of any nature (minimum sample size of 1 package, or at least 5 grams, plus a reserve sample)
    - pre-rolls and enhanced pre-rolls (minimum sample size of 5 grams, plus a reserve sample)
B. Because most of these products have been homogenized during the production of the product, increments and quadrants are not necessary for sampling these products. For example, 1 bath bomb should already be representative of the entire batch of bath bombs made from one extract.

Appendix A: Visual Sampling Quadrant Model

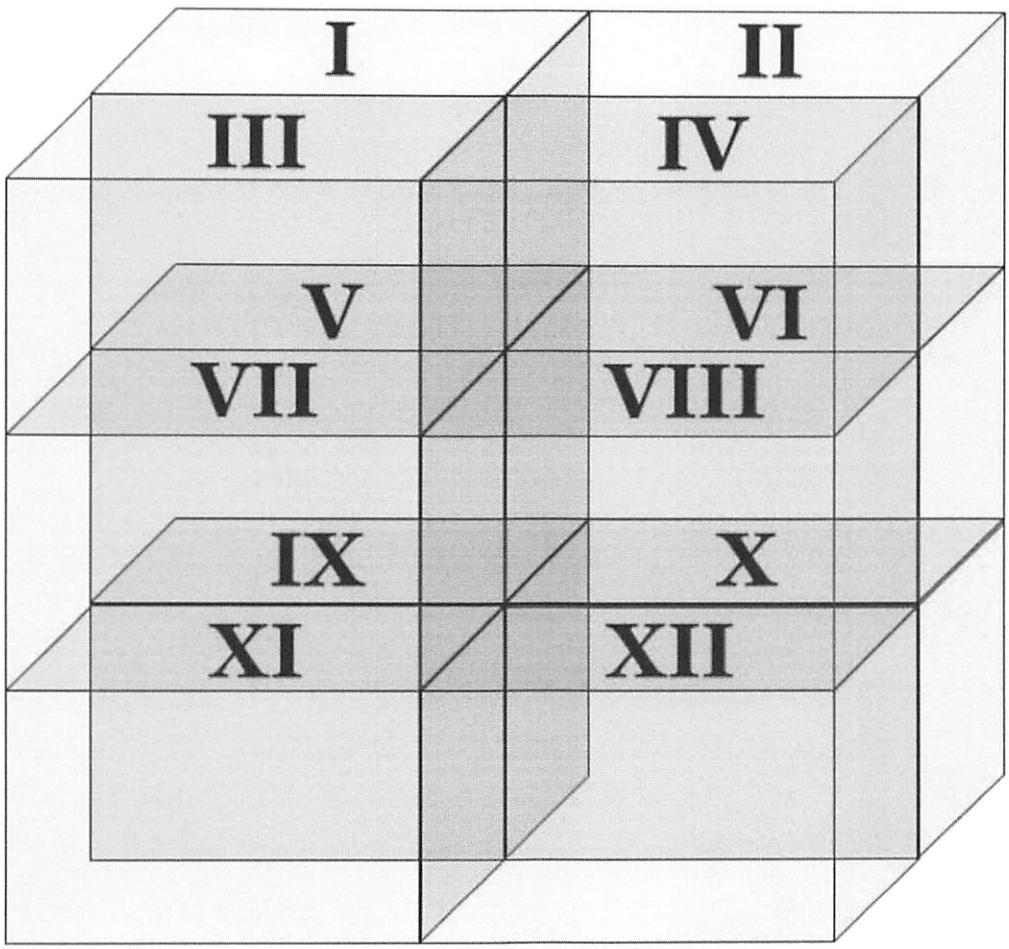


Figure 1: Visual sampling quadrant model

Standard Revision History

Version	Action	Date (MM/DD/YY)
1.0	Initial Version	09/03/19
1.1	Changed format to match other SOP's, removed Oregon specific laws and simplified language. Abigail Crutchmer, Lab Director	01/02/20
2.0	Updated SOP to reflect OMMA guidelines published in emergency rules on 10/15/20. Abigail Burkhart, Lab Director	11/11/20
2.1	Removed Producer and Registrant definitions. Added quadrant model to appendix A. Abigail Burkhart, Lab Director	02/12/21