



SAMPLING AND TESTING TECHNICAL GUIDANCE FOR MARIJUANA PRODUCTS

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This information is intended for use by licensed safety compliance facilities and licensed marihuana safety compliance facilities, collectively defined as laboratories, regulated by the Marijuana Regulatory Agency (MRA).

This version of the technical guide combines and replaces all prior iterations published under the purview of both MMFLA and MRTMA.

This information does not constitute legal advice and is subject to change. Licensees are encouraged to seek legal counsel to ensure their operations comply with the Medical Marihuana Facilities Licensing Act and associated Administrative Rules.

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INTRODUCTION

The Marijuana Regulatory Agency (MRA) is committed to evidence-based decision-making when implementing technical guidance for licensed laboratories. As research into marijuana use, safety, and testing advances, this guide may be revised and updated to reflect these changes where appropriate.

The sampling and analysis described in this guide shall be conducted by a laboratory licensed by the MRA and accredited to the International Organization for Standardization (ISO), ISO/IEC 17025:2017 by an International Laboratory Accreditation Cooperation (ILAC) recognized accreditation body or by an entity approved by the agency by 1 year after the date the license is issued Rule 5(1)(a) in the Sampling and Testing Rule Set R 420.305(1)(a).

Accreditation to ISO/IEC 17025 standards demonstrates successful implementation of a quality management system which includes but is not limited to: organizational structure, quality procedures, internal audits, and corrective actions. In addition to the quality management system, technical matters such as the equipment calibration, traceability of controls, relevance of the method, and the validation data will be reviewed by the auditor. Verification of the performance of all methods employed by the laboratory is required. Marijuana safety and potency should be analyzed using testing methodology validated by an independent third party and approved by the MRA

Analytical testing of marijuana for safety and potency is increasingly recognized as a critical and necessary component of the industry for several reasons ^[12]:

Laboratory testing minimizes the risk of exposure to pesticides, microbes, heavy metals, molds, and residual solvents by providing consumers information about the products they are purchasing and helps to prevent consumption by sensitive populations.

- Quantification of cannabinoid profiles and potency is available to the consumer to inform decision making and aids in determining appropriate dosing for individual use.
- Laboratory testing provides transparency and promotes a sense of reassurance to consumers as well as ensuring product quality for the tested marijuana.

QUALITY ASSURANCE

According to Rule 7(3)(f) in the Marihuana Licensees Rule Set R 420.107(3)(f).:

All laboratories must retain and employ at least one laboratory manager with a relevant advanced degree in a medical or laboratory science. This person must fill the role of a laboratory manager who is responsible for duties including the following:

- Ensure tests are conducted in accordance with Rule 5 in the Marijuana Sampling and Testing Rule Set R 420.305.
- Ensure test results are accurate and valid
- Oversee day-to-day operations
- Validate reporting requirements in the statewide monitoring system (Metrc)
- Verify ongoing conformity with ISO 17025 standards
- Any other duties required and published by the agency Rule 7(3)(f) in the Marihuana Licensees Rule Set R 420.107(3)(f).

All results entered in the statewide monitoring system (Metrc) must be valid and defensible. The laboratory should ensure that all test results are legally defensible by keeping thorough and accurate records as well as detailed chain of custody (COC) documents. The quality assurance (QA) plan and/or standard operating procedures (SOPs) should describe the policies and procedures used by the facility for record integrity, retention, and storage. The laboratory must have a procedure for monitoring the validity of results. The resulting data must be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to review the results. This monitoring should occur on an ongoing basis and should be reviewed by the laboratory manager/quality assurance manager and shall include, but not be limited to:

- Use of reference materials or quality control (QC) materials
- Functional check(s) of measuring and testing equipment
- Use of check or working standards with control charts, where applicable
- Intermediate checks on measuring equipment
- Review of reported results
- Intra-laboratory comparisons (proficiency testing)

Laboratories analyzing marijuana samples must adhere to all required QC procedures specified in the reference method(s) to ensure that routinely generated analytical data is scientifically valid and defensible and is of known and acceptable precision and accuracy. To accomplish these goals, each laboratory must prepare a written

description of its QA activities, included as part of a Quality Assurance Manual and as part of all relevant SOPs Rule 5(1)(c) in the Sampling and Testing Rule set R 420.305(1)(c). It is the responsibility of the laboratory manager/quality assurance manager to keep these documents up to date. A controlled version of the Quality Assurance Manual and all SOPs should be made readily accessible to all laboratory employees. Additionally, all laboratory personnel should be familiar with the contents of the Quality Assurance Manual as well as all SOPs for which they are responsible.

QUALITY ASSURANCE MANUAL

At a minimum, the following items should be addressed in the Quality Assurance Manual:

Laboratory organization and responsibility

- A chart or table showing the laboratory organization and lines of responsibility, including QA managers
- List of key individuals who are responsible for ensuring the production of valid measurements and the routine assessment of measurement systems for precision and accuracy (e.g., who is responsible for internal audits and reviews of the implementation of the plan and its requirements)
- Clearly defined job descriptions of all personnel
- Descriptive training program aimed at keeping personnel updated on regulations and methodology
- Records documenting Demonstration of Capability (DOC) for all laboratory personnel.

Standard Operating Procedures (SOPs)

- All SOPs must be formatted to include the date(s) of revision(s) in accordance with Rule 5(1)(b) in the Sampling and Testing Rule set R 420.305(1)(b).
- All SOPs should be routinely updated to reflect all phases of current laboratory activities.
- Current copies of all SOPs must be made available and be easily accessible to all staff.
- SOPs are reviewed not less than annually and should be revised as changes are made.
- SOPs should include signature pages.

Note: All SOPs related to the analysis of samples must be approved by the MRA. Any changes to a previously approved SOP must be sent to MRA for review and approval prior to use. The MRA may request copies of any SOP if it is deemed necessary.

Field sampling procedures

- Describe all processes used to collect samples, including sanitation protocol.
- Ensure that all forms are legible and written in indelible ink and/or electronic data are retained and adequately stored.
- Ensure that sampling protocol is clearly documented in an SOP and available to field samplers.
- Store both unprocessed and processed samples at the proper temperature, isolated from laboratory contaminants.
- Maintain integrity throughout the life of all samples (e.g., track samples from receipt through analysis to disposal by utilizing the identification number from the Metrc).
- Create and retain adequate Chain of Custody (COC) for samples.

Instrument calibration procedures (may reference SOP)

- Specify type of calibration used for each method and frequency of use
- Describe calibration standards' source, age, storage, and labeling
- Perform data comparability checks

Analytical procedures (may reference SOP)

- Cite complete method
- Accurately describe and maintain quality control procedures required by the method(s).

Data reduction, validation, reporting, and verification (may reference SOP)

- Describe data reduction process: method of conversion of raw data to reported unit of measure
- Describe data validation process
- Describe reporting procedures, include processes and format
- Describe data verification process
- Describe procedure for data corrections

Quality control (QC) checks and frequency of use (may reference SOP)***Parameters for chemistry should include or reference***

- Instrument performance check standards
- Frequency and acceptability of method detection limit (MDL) calculations
- Frequency and acceptability of demonstration of low-level capability, calibration, internal and surrogate standards, laboratory reagent blank, field reagent blank, field, and laboratory matrix replicates
- QC and proficiency testing samples
- Laboratory fortified blank and laboratory fortified sample matrix replicates
- Initial demonstration of method capability
- Use of control charts
- Qualitative identification/confirmation of contaminants

Parameters for microbiology should include or reference

- Positive and negative culture controls
- Proficiency testing and quality control samples

Preventive maintenance procedures and schedules

- Describe location of instrument manuals and schedules and documentation of all routine equipment maintenance
- Describe availability of instrument spare parts in the laboratory
- List any maintenance contracts in place

Corrective action contingencies

- Describe response to obtaining unacceptable results from analysis of proficiency testing samples and from internal QC checks
- Name person(s) responsible for corrective actions
- Describe how corrective actions are documented

Record keeping procedures

- Describe procedures and documentation of those procedures
- List length of storage, media type (electronic or hard copy)
- Describe security policy of electronic databases

QUALITY CONTROL REQUIREMENTS

When methods do not include quality control parameters, the agency has adopted the following requirements:^[35]

DAILY REQUIREMENTS

Analytical Batch

- Must be clearly defined (e.g., every 10 samples or every 20 samples).

Laboratory Reagent Blank

- Checks for background contamination, and should be the first sample on the sample run; a blank should also be run before and after a calibration check (CC) and initial calibration verification (ICV) /continuing calibration verification (CCV) and at the end of the run.

ICV/CCV

- ICV - Analyze a known concentration as a sample after calibration and before samples to verify; should also be done after every 10 samples.
- CCV - Analyze a known concentration at the end of all samples daily.

Laboratory Fortified Blank (LFB)

- Analyze a known standard.

Laboratory Fortified Matrix (LFM)/Laboratory Fortified Matrix Duplicate (LFMD)

- Analyze a sample with a known amount of standard added (spike).
- Add a known amount of standard to a sample and expect that amount to be added to your sample concentration and repeat process for LFMD.
- Calculate Relative Percent Difference (RPD) between spiked sample and spiked duplicate, target value should be close to the first value and have a small RPD (less than 20%).
- Spike volume should be less than 1% of the volume. Example: spike with 1 mL of 1000 mg/L into 100 mL sample will equal a 10 mg/L increase in concentration.

Duplicate

- Analyze the same sample twice - should be a set procedure (e.g., every 7th sample will be repeated in duplicate).

ANNUAL REQUIREMENTS

Demonstration of Capability (DOC)

- Each analyst must have a DOC which includes documentation they can accurately run each test
- Documentation that an analyst has read and understands all appropriate SOPs and methods
- Backup analysts should do this once a year or any time there is a reason to question competence
- Competency assessments should be completed not less than annually including having staff run a previously reported sample from sample prep through result reporting to assure all staff are following the written SOPs.

Method Detection Limit (MDL)

- Run at least 7 samples at low levels following procedure outlined below, i.e. Daily Requirements.

Control Charts

- Create and maintain control charts if you have 20-30 data points within 90 days.

Corrective Action

- Corrective actions must be included in the SOPs for each method and should include what to do if QC tests fail or are out of range
For example, if standards fail, re-calibrate and run test again.

QC Acceptance

- Include in the SOP for each method the acceptance ranges for standards, duplicates, spikes, etc. and verify that they match the method requirements

QC ACCEPTANCE CRITERIA

*unless otherwise specified in reference method

- LRB < MDL
- LFB \pm 15%
- ICV/CCV \pm 10%
- LFM/LFMD \pm 20%
- RPD < 20%
- Reporting limit = MDL Calculations
- % Recovery for LFB = $\frac{\text{LFB Result} \times 100\%}{\text{Expected Concentration}}$
- RPD – relative percent differences for duplicates and LFM/LFMD

$$\text{RPD} = (|\text{Num1} - \text{Num2}| / ((\text{Num1} + \text{Num2}) / 2)) \times 100$$

Where,

Num1= Original Number

Num2= Second Number

- % Recovery for LFM – when using less than or equal to 1% spike volume compared to sample volume

$$\% \text{ Recovery} = \frac{\text{LFM Result} - \text{Sample Result} \times 100\%}{\text{Actual Concentration of spike}}$$

DEFINITIONS

A variety of definitions relating to detection limits and quantitation limits are used in literature and by government agencies, however, universally accepted procedures for calculating these limits do not exist. The definitions below attempt to clarify the meaning of these terms as recognized by the MRA. ^[13]

Limit of Detection (LOD) or detection limit: The lowest concentration level that can be determined to be statistically different from a blank (99% confidence). The LOD is typically determined to be in the region where the signal to noise ratio is greater than 5. Limits of detection are matrix, method, and analyte specific. The LOD is approximately equal to the MDL for those tests which the MDL can be calculated.

Method Detection Limit (MDL): To calculate MDLs, please follow this [procedure](#). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL procedure is designed to be a straightforward technique for estimation of the detection limit for a broad variety of physical and chemical methods. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps used by the laboratory be included in the determination of the MDL.

Limit of Quantitation (LOQ): The minimum concentration or mass of an analyte in a given matrix that can be reported as a quantitative result. For all analyses aside from Target Analytes, **the LOQ must be at least 1/2 of the published action limit.** The analyst should determine LOQs, when any change is made which could affect the LOQs, or more frequently if required by the method. In addition, the analyst must demonstrate low-level capability on an ongoing basis through an LOQ determination or repeated low-level analyses.

Low Level Quantitation: The laboratory's minimum reporting limits (MRL) should be reported to the client along with the data. The reporting limit must be below the agency's published action limits. Laboratories should run an LFB at their MRL every analysis day and should not report contaminants at levels less than the level at which they routinely analyze their lowest standard.

Initial Demonstration of Capability (IDOC): Before beginning the analysis of samples, an (IDOC) must be performed. The IDOC includes a demonstration of the ability to achieve a low background, the precision and accuracy required by the method, and determination of the limit of detection (LOD) (see below). An IDOC should be performed for each instrument. It is also recommended that an IDOC be performed by each analyst. In addition, it is recommended that the IDOC also address the variability introduced if more than one sample preparation analyst is used. Precision, accuracy, and LOD should be comparable for each technician.

Demonstration of Capability (DOC): Each analyst should be assigned a file in which records are maintained that demonstrate the capability of the individual to perform analyses for which they are responsible on frequent or infrequent basis. At minimum, the analyst should calibrate and analyze four standards to demonstrate their ability to run each test. Additionally, the file should contain documentation (signed form) that the analyst has read and understands all assigned SOPs and methods. Backup analysts should do this once a year. The primary analyst should recalculate DOCs when a change in the method, analyst, or instrument is made which could affect precision, accuracy, or sensitivity. Minor changes in methodology should prompt a check to verify that the precision, accuracy, and sensitivity have been maintained.

Laboratory Reagent Blank (LRB): Depending on the method, the LRB may be appropriate for this. An LRB should be carried through the full analytical procedure with every sample batch. In general, results from LRBs should not exceed the laboratory's Minimum Reporting Limit (MRL), the lowest concentration of standard used for quantitation.

Calibration requirements: Calibration must occur not less than monthly. At the beginning of each day samples are to be analyzed, a calibration curve composed of four or more points including all target analytes should be generated according to the approved SOP. Where the determinative time is extensive and the instrument is very stable, the calibration curve should be initially developed; thereafter, each day that samples are to be analyzed, this curve should be verified by analysis of a Calibration Check (CC) following the requirements listed below. The check must be +/- 10% of the known value.

Calibration Check (CC): This verification should be done at both the beginning and end of the analyses, including at least one standard for each of the target analytes at the expected concentration range. It is recommended that a calibration standard of one component of a multicomponent analyte also be analyzed each day or work shift. All checks must be within 10% of the known value or the instrument is to be recalibrated as specified in the calibration requirements.

Linear Calibration Range (LCR): The region of a calibration curve within which a plot of the concentration versus the response of a particular analyte remains linear, and the correlation coefficient of the line is approximately 1 (0.995 for most analytes). The plot may be normal-normal, log-normal, or log-log where allowed by the analytical method. At the upper and lower bounds of this region (upper and lower limits of quantitation), the response of the analyte's signal versus concentration deviates from the line.

Laboratory Fortified Sample Matrix: Laboratory fortified sample matrix requirements in the methods must be met. If there are no laboratory fortified sample matrix requirements in the method, the following guidelines are to be used. The laboratory should add a known quantity of analytes to a percentage (to be described in the approved SOP) of the routine samples to determine sample matrix interference. The fortified concentration should not be less than the concentration of the sample selected for fortification unless specified by the method. If the sample concentration is unknown or less than

detectable, the analyst should choose an appropriate concentration (e.g., a percentage of the published action limit or mid-point in the calibration range). Over time, samples from all routine sample sources should be fortified. The procedure should be described in the SOP. If any of these checks are not within the criteria specified in the method or control limits specified in this document, and the laboratory performance is in control, the result for that sample should be flagged to inform the data user that the results are suspect due to matrix effects.

STANDARD OPERATING PROCEDURE (SOP) REQUIREMENTS

All testing, QA, and QC procedures must be documented in an SOP and must include enough detail for someone who is not familiar with the test to perform it. Additionally, all SOPs should include:

- Step by step instructions detailing how to perform the analyses
- Step by step instructions describing how data is generated and how values are reported
- QC Acceptance Criteria and definition of a “Batch” (e.g., every 10 or 20 samples) as well as the minimum frequency of QC checks.

VALIDATION REQUIREMENTS

In accordance with Rule 5(2) in the Sampling and Testing Rule Set R 420.305(2).

The laboratory must present a validation report styled after peer-reviewed scientific publications, that includes enough information that an experienced analyst could repeat the validation study. The validation report must accompany the raw data and include, at minimum, the following:

- Introduction and brief summary
- Materials and preparation methods
- Method parameters
- Chromatograms/spectra, calculations and results
- Method acceptance and limit performance data
- Acceptable results of a graded external proficiency test that includes all analytes require by the agency
- Conclusion, discussion and references

Literature references must be peer-reviewed if not published by a government agency. Application notes are also acceptable.

Analytical chemistry methods:

A laboratory validation is required to show that the method is fit for purpose for the intended matrix and that any modifications to the original method do not negatively impact performance. All test methods must be based on compendia or published methods. AOAC Appendix K must be followed and, at a minimum, validations must verify accuracy, precision, analytical sensitivity, analytical selectivity, limit of detection, limit of quantitation, reportable range, and the identification of interfering substances. All validations must be submitted to the agency for approval with an acceptable and graded external proficiency test where all required analytes are shown to have passed. Validation protocols should include marijuana matrices (e.g., flower, infused products and concentrates). If the initial validation study was not performed on marijuana matrices, an interference study should be performed for each and should include each matrix type analyzed by the laboratory Rule 5(2) in the Sampling and Testing Rule Set R 420.305(2).

Microbiological methods:

A laboratory validation is required to show that the method is fit for purpose for the intended matrix and that any modifications to the original method do not negatively impact performance. All test methods must be based on compendia or published methods. AOAC Appendix J must be followed and, at a minimum, validations must address accuracy precision, repeatability, reproducibility, robustness, inclusivity/exclusivity, limit of detection, limit of quantitation, reportable range and the identification of interfering substances. All validations must be submitted to the agency for approval with an acceptable and graded external proficiency test where all required analytes are shown to have passed. Validation protocols should perform inoculation of marijuana matrices with live organisms where feasible in order to ensure that both extraction and detection for the assay are tested. To further test the accuracy of the assay, probability of detection (POD) analyses, inclusivity, exclusivity, lot-to-lot stability, and robustness studies must be included in the validation studies. Methods adopted from a matrix specific standard method, inclusivity/exclusivity do not require a comprehensive reassessment, provided that the referenced media, primers, probes, antibodies, critical chemistries, etc., were not modified Rule 5(2) in the Sampling and Testing Rule Set R 420.305(2).

Proficiency Testing

Rule 5(14) in the Sampling and Testing Rule Set R420.305(14) requires the agency to establish a proficiency testing program and designate laboratory participation. A laboratory shall analyze proficiency test (PT) samples using the same procedures with the same number of replicate analyses, standards, testing analysts and equipment as used for marijuana product testing. If the PT deviates from normal sample preparation this must be documented in the SOP. Analysis of PT's must occur on a rotating schedule and should include all analysts who perform the testing. Proficiency tests should be thoroughly documented and completed by a single analyst.

The following guidelines for proficiency testing must be followed by all laboratories on an annual basis:

- Complete one set of acceptable, externally graded PT for all analyses performed. The PT samples must be procured from an accredited third-party vendor.
- Any ISO 17043 accredited PT vendor may be used.
- For parameters where there is no commercially available PT, laboratories may opt to do one of the following:
 - Request that an ISO 17043 accredited PT vendor create a test that meets the laboratory's needs.
- The agency will require unscheduled random testing of matrix matched samples to evaluate the overall performance of the laboratories not less than annually.
- The agency will require randomized audits of previously analyzed samples on a quarterly basis or in response to an active investigation.

Note: Copies of all proficiency testing results, both acceptable (pass) and unacceptable (fail) must be sent to the agency for review direct from the PT vendor via email to **MRA-SCF@michigan.gov**.

REQUIRED TESTS

All compliance testing must conform to ISO/IEC 17025 standards for quality control and quality assurance, including the validation of matrices not listed here.

Potency

Rule 5(16) in the Sampling and Testing Rule Set R 420.305(16) prohibits a laboratory from desiccating samples prior to performing potency analysis.

For the analysis of potency, a standard method for the quantitative analysis of cannabinoids has not yet been published., however the following references may be helpful:

De Backer, B., Debrus, B., Lebrun, P., Theunis, L., Dubois, N., Decock, L., & Charlier, C. (2009). Innovative development and validation of an HPLC/DAD method for the qualitative and quantitative determination of major cannabinoids in cannabis plant material. *Journal of Chromatography B*, 877(32), 4115-4124.

Patel, B., Wene, D., & Fan, Z. T. (2017). Qualitative and quantitative measurement of cannabinoids in cannabis using modified HPLC/DAD method. *Journal of pharmaceutical and biomedical analysis*, 146, 15-23.

All testing must meet the standard method performance requirements listed below. For matrices not listed, the method performance requirements should be as close to the published Standard Method Performance Requirements (SMPRs) as possible, using standard analytical methods.

- Concentrates: AOAC SMPR 2017.001
- Dried Plant Materials: AOAC SMPR 2017.002
- Edible Chocolates: AOAC SMPR 2017.019

Residual Solvent Testing

All testing must meet the SMPRs for the adopted reference method.

- United States Pharmacopeia (USP), 2008. “<467> Residual Solvents.”

Chemical Residue Testing

All testing must meet the SMPRs listed below.

- AOAC Official Method 2007.01 Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate- Gas Chromatography/Mass Spectrometry and Liquid Chromatography/Tandem Mass Spectrometry.
- Pesticides in Cannabis Flower: AOAC SMPR 2018.011

Heavy Metals

All testing must meet the standard method performance requirements for the adopted reference methods.

Applicable references may include, but are not limited to:

- United States Pharmacopeia (USP), 233
- Using EPA 3052 (microwave digestion) for the sample prep.

Water Activity

- ASTM D8196-18

Microbial Assays (Qualitative)

Methods applicable to pathogenic Salmonella spp., Shiga toxic producing Escherichia coli (STEC), and Aspergillus spp.

All microbiological methods employed must include applicable controls. Qualitative pathogen methods should confirm presumptive results as either positive or negative by inclusion of a confirmation step. The confirmation step should be performed by utilizing a method different from the one used to obtain the first result for any STEC, or Salmonella presumptive positive results initially performed utilizing qPCR.

Microbial Assays (Quantitative)

Methods applicable to total coliforms and total yeast and mold:

- Association of Analytical Communities (AOAC) 2019. “Yeast and Mold Counts in Foods, 997.02.”
- Association of Analytical Communities (AOAC) 2019. “Total Coliform 991.14”
- USP 61, 62, 1223
- Quantitative PCR assays.

Foreign Matter/Filth Analysis

- AOAC Official Method 970.74 Foreign Matter in Drugs (Leafy, Crude).

For further information regarding the use of a microscope, please refer to the ORA Laboratory Manual, FDA Office of Regulatory Affairs. In Section 4, Microanalytical & Filth Analysis, please see 4.2.2. Microscopic Examination and Microscope Accessories. Within that section, please refer to Section B (Discussion, Part 1 C re: stereomicroscopes) as well as Section B (Discussion, Part 2 Fundamental Microscopic Techniques and Procedures, part a).

Vitamin E Acetate

Rule 1(dd) in the Sampling and Testing Rule Set R 420.301(dd) defines a target analyte as a non-marihuana active ingredient designated for analysis. Vitamin E Acetate has been identified as a target analyte by the MRA.

- Brabcová, I., Kovářová, L., Šatinský, D., Havlíková, L., & Solich, P. (2013). A fast HPLC method for determination of vitamin E acetate in dietary supplements using monolithic column. *Food analytical methods*, 6(2), 380-385

FIELD SAMPLINGS

According to Rule 4(2)(b) in the Sampling and Testing Rule Set R 420.304(2)(b) Except otherwise required by the agency, the laboratory shall collect a sample size that is sufficient to complete all required analyses and not less than 0.5% of the weight of the harvest batch.

- Prior to September 1, 2020, the maximum harvest batch size is 15 pounds.
- From September 1, 2020 through December 31, 2020, the maximum harvest batch size is 20 pounds.
- From January 1, 2021 through March 31, 2021, the maximum harvest batch size is 25 pounds.
- After March 31, 2021, the maximum harvest batch is 50 pounds.

At least 50% of the sample taken must be homogenized for testing.

The agency may publish sample sizes for other marijuana products being tested. The laboratory must develop a statistically valid sampling method to collect a representative sample from each batch of marijuana product. The laboratory must have access to the entire batch for the purposes of sampling.

Standardized sampling procedures are an integral component to all laboratory testing. The objective of a well-defined sampling procedure is to ensure proper collection, clear labeling, careful sample transportation, and proper storage of samples to ensure sample integrity. The best way to track as they make their way from the originating facility and throughout the analysis process is to create and maintain a well-organized and detailed Chain of Custody

According to Rule 5(1)(b) in the Sampling and Testing Rule Set R 420.305(1)(b) a laboratory shall maintain internal SOPs. Accurate and consistent results should be the primary goal of all laboratories. To support this goal, it is critical that all samples are obtained in a way that ensures the integrity of the analytical process. Sampling events should be performed consistently by all field staff using the accepted methodology. All field staff should be thoroughly trained in sampling procedures and confident in their ability to accurately perform all aspects of the sampling process.

For additional information regarding sample collection procedures, please refer to:

- Osterbauer, N., Krepps, S., Sackett, J., Holladay, C., Wendt, E., Wells, D., & Kristof, J. *Protocol for Collecting Samples of Usable Marijuana*. ORELAP-SOP-001 Rev. 3.0. 2016 December.
- Sexton, M., & Ziskind, J. (2013). Sampling cannabis for analytical purposes. *BOTEC Analysis Corp*, 26.
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FIELD SAMPLING STAFF

Sampler Qualifications

Model qualifications for samplers are:

- Capable of performing all required duties
- Free from undue burden that would cause a conflict of interest or bias in sampling
- Capable of passing initial and ongoing demonstrations of capability

Prior to testing samples, a satisfactory initial demonstration of capability (IDOC) or competency assessment must be documented and approved by the laboratory manager for all samplers. The IDOC should include principles, procedures, and policies for sampling. The IDOC should be repeated:

1. Every time there is a change in personnel or SOP.
2. When the sampler has not performed a sampling within a 12-month period.

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

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

Thereafter, ongoing/continuing demonstration of capability (CDOC) as per the quality control requirements referenced in the SOP should be done at least annually. The laboratory should have a documented procedure for performing the CDOC, should retain documentation verifying CDOC for each sampler and must make this documentation available upon request.

SAMPLING IN THE FIELD

Field Duplicates

Field duplicates are recommended for any marijuana sampling event. Field duplicates must be sampled in such a way as to replicate the primary sampling event and all requirements should be clearly outlined in the field sampling SOP. If the laboratory opts to take an amount greater than 0.5% for the purposes of collecting a field duplicate, Rule 4(2)(a)(i) in the Sampling and Testing Rule Set R 420.304(2)(a)(i) permits them to do so.

Field Audits

The laboratory must adopt an ongoing system for performing audits of field activities which the laboratory manager will oversee. Field audits should be conducted periodically and in accordance with a predetermined schedule and procedure. The goal of the field audit is to verify that the sampling events comply with the requirements of the regulations and demonstrate compliance with the designated sampling SOP. If the findings of a field audit cast doubt on the effectiveness or validity of the field sampling events, corrective action should be taken, and preventative action must be enacted. If the audit shows that improper sampling may have led to incorrect test results, the laboratory should notify customers and identify any test results that may have been affected.

Auditing Checks

Using audit checklists:

- Review sampling and performance records from the preceding year for deficiencies in the application of sampling protocol
- Observe the sampler conducting sampling procedures
- Have the auditor and sampler collect samples from the same harvest lot for evaluation and comparison of results
- Record any deficiencies and initiate corrective action

Field Quality Control

Field sampling equipment should be certified clean/sterile prior to use by the laboratory. In general, sampling equipment should be sterile for microbiology samples and clean for chemistry samples. The laboratory should perform cleanliness checks on each batch of sampling equipment prior to use in the field. Results from cleaning procedure tests must be below the reporting limit of the target analyte(s) for the associated analyses. If cleanliness checks fail, the sampling equipment must be re-cleaned/sterilized, and retested.

Representative sampling should meet a 95% confidence level and limit sampling error. Increasing the number of sample increments to compensate for normal batch heterogeneity is the simplest means to achieve a representative sample.

Sample size

Rule 12(7) in the Marijuana Operations Rule Set R 420.212(7) requires a laboratory to establish an adequate chain of custody (COC) as well as instructions for sample and storage requirements. Additionally, laboratories are required to develop a statistically valid sampling method to collect a representative sample from each batch. The laboratory shall collect a sample size that is sufficient to complete all required analyses

and is not less than 0.5% of the weight of the harvest batch. At least 50% of the sample taken must be homogenized for testing as outlined in Rule 4(2)(b) in the Marijuana Sampling and Testing Rule Set R 420.304 (2)(b). The MRA requires all laboratories to develop and submit an SOP that meets all aspects of the administrative rules and shall ensure that the procedures outlined are statistically valid.

Additionally, the agency may publish sample sizes for other marijuana products being tested. The laboratory must develop a statistically valid sampling method to collect a representative sample from each batch of marijuana product. The laboratory must have access to the entire batch for the purposes of sampling.

Sampling a Batch of Marijuana

1. Physically locate the batch to be sampled as well as the source package and tag information from Metrc. **Each batch mass should be compared to the Metrc system to confirm access to the entirety of the batch.**
2. Review the container label information for batch number, producer, and other pertinent information. Each harvest batch should be separated and must be assigned a source package tag in the Metrc system. **Do not sample if the product is not in the Metrc system or if the batch weight or details do not match.**
3. Determine the number of containers in the batch and the batch size. Verify the batch size for each container. **Do not sample if the batch size is unavailable, or the batch weight does not match the information in the Metrc.**
4. Determine the total number of containers from which sample increments must be collected using a random number generator.
5. Select the appropriate sampling tool to ensure that it reaches all portions of the container.
 - a. *Please note: All samples must be collected in compliance with Rule 2(2) in the Marijuana Employees Rule Set 420.602(2)(h) current good manufacturing practice in manufacturing, packing, or holding human food, 21 CFR part 110, as specified in these rules.*
6. To prevent contamination, sampling tools should be cleaned and prior to use and may be cleaned in the field between batches using an appropriate solvent and decontaminant to prevent cross-contamination of batches during sampling. Where aseptic technique is required, please refer to the FDA Aseptic Sample guidelines ([Investigations Operations Manual Subchapter 4.3.6](#)) for information. Sampling tools which appear to be contaminated or appear to be otherwise compromised should not be used.
7. Visually inspect each test sample increment to assess uniformity.
8. If non-uniformity is identified, record observation on the COC. Marijuana flowers often have variably sized flowers. When drawing sample increments, approximately equal amounts of product are to be taken with each probing and from each container. Care must be taken by the sampler to not damage or contaminate the portion of the product which is not being collected.
9. Combine all sample increments to form the composite sample.

10. Ensure enough sample increments are taken to meet sample size requirements for all analytical methods being performed.
11. Seal and label the composite sample with the following minimum requirements:
 - Sample package tag assigned in the Metrc system
 - Sampling date and name of sampler
 - Producer's license.
12. Complete the COC form while onsite at the facility.
13. The sample, COC, and manifest from Metrc must be transported to the SCF using packaging appropriate for secure and timely transport.

Sampling Procedure for Concentrates

The procedure is designed to ensure that each sampling event shall produce samples that are representative of the production batch specified.

1. The laboratory's sampling shall ensure that samples are collected randomly from the entire production batch. The COC shall document the total number of storage containers for a production batch and clearly identify containers selected for sampling.
- 2. Sample weights have yet to be determined, however, all licensees will be notified when the determination is made.**
3. The samples shall be collected from product in the state which it is intended to be consumed, but prior to final form.
4. The sampler shall wear sterile nitrile, latex, or equivalent gloves during sample collection. The gloves shall be changed between each production batch to minimize potential cross contamination.
5. The sample collection vials shall be labeled at the time of sampling.
6. All vials shall be immediately stored between 2° -21° C.

Shatter/Wax/Slab Concentrates

Shatter, wax, or other concentrate slab may have varying degrees of thickness resulting in varying concentrations of cannabinoids, potential residual solvent(s), or pesticides. Thinner portions of the concentrate slab have greater exposed surface area, thus allowing for a higher rate of diffusion of residual solvents. Samples should be obtained from each region of thickness to provide a representative sampling of the overall product. Samples should be obtained from each region of thickness to provide a representative sampling of the overall product. To ensure representative sampling, the following is recommended:

1. Visually identify regions of varying thicknesses of the product.
2. Using forceps or a spatula collect the correct number of subsamples needed from each region of the overall production batch to meet the minimum number of samples described above.
3. The sample shall be collected and weighed to ensure the correct sample amount has been collected.

4. The sample vials shall be weighed and tared to ensure that the correct amount of sample is collected. Record the weight of each aliquot.

Distillate and Viscous Liquids

Viscous liquid concentrates of a single batch should be stored in a single container. The final size of the container will not be dictated by the MRA, however, it is important to note that a laboratory should consider viscous liquids in a single container to be homogeneous and any viscous liquids contained in multiple containers should be considered as the same number of separate batches. If the viscous liquids are separated into containers as they are intended for sale, the laboratory should sample using the same sampling protocol they would employ for the statistically random selection of other marijuana products.

Viscous Liquids in Small Container (1 mL or less)

1. If the oil is a viscosity that can be homogenized by hand through simple inversion, the oil shall be inverted a minimum of three (3) times to ensure that the oil is homogenous.
 - a. Each inversion must be complete, i.e. the oil must flow to the cap of the vial and back to the base three times.
2. Alternatively, if the oil is too viscous to invert, the laboratory should confirm with the facility representative that the sample is homogenous.
3. If the oil is stored and sampled in a container (syringe, dart, vial, etc.), an empty container shall be weighed, and the weight recorded to confirm that the weight of all samples collected are accurate.
4. Record the weight and/or volume of each aliquot on the sampling document / COC

Viscous Liquids in Medium to Large Volume Containers

1. Using a 0.5 mL or 1.0 mL pipette or syringe remove the sample amount for each sample to be collected.
2. The aliquots shall be taken at different depths of the oil to ensure that the oil is sampled representatively.
3. The top third of the container, middle third of the container and the bottom third of the container shall be sampled.
4. Each collection vial shall be weighed and tared prior to aliquoting the sample.
5. Record the weight and/or volume of each aliquot.

Marijuana-Infused Products (Edibles)

Marijuana infused products must be in the form that is intended for consumption prior to sampling. **Sample increments have yet to be determined, however, all licensees will be notified when the determination is made.**

1. The samples shall be collected randomly from a batch of product in final form and will follow the statistically valid sampling method employed by the laboratory.
2. The sampler shall wear sterile/sanitized nitrile, latex, or rubber gloves during sample collection.
3. The gloves shall be changed between each production batch to minimize potential cross contamination.
4. The sample product containers shall be labeled at the time of sampling.
5. All vials shall be immediately stored at a temperature appropriate to completely maintain the integrity of the sample and not to exceed 21° C prior to shipping.
6. In order to collect random samples, the laboratory may choose to employ the following method:
 - This can be accomplished by dividing the production process into thirds and selecting representative and random samples from each 1/3 of the production batch.
 - Take the total number of samples produced, divide by one-third and select randomly from each third section of the batch.
 - Each sample within each 1/3 of the batch must be selected randomly. For example, a production batch of 1000 sample units, 6 samples are required; therefore, 2 samples shall be taken from different locations within the beginning third, 2 from different locations within the middle third, and 2 from different locations within the end third.

Chain of Custody Documentation

Rule 12(7) in the Operations Rule Set 420.212(7) requires SCFs to have an adequate chain of custody (COC). The following information is what the agency has deemed adequate and must be included at a minimum:

- Marijuana facility where the samples were collected (name, address, and license number)
- Date and time
- Indication whether the samples are for retesting due to an initial test failure
- If samples are for retesting, ensure that the product is not prohibited from retesting
- Product type (e.g., buds, concentrate, infused product)
- Whether products are in final package, i.e., individually wrapped as intended for sale, with or without labeling

- If the product is labeled with a concentration, make note of the intended concentration.
- Total mass of the source package and unique Metrc package tag
- Total container number, # of sample increments, # of containers sampled
- # of sample containers collected
- Unique Metrc package tag for the sample package including the total mass sampled
- Laboratory license number
- Sampler's signature
- Signature from the originating marijuana facility representative(s) present during the sample collection process. The signatures are attesting to the accuracy of the sampling information
- Creation of a transfer manifest in the Metrc system
- Sampling Procedure ID and revision date

Please ensure that all information is legible. Do not scribble or write over errors. Any errors should have one line through them with the initials of the person correcting the information, the date and time.

ADDITIONAL TESTING GUIDELINES FOR FIELD SAMPLERS

Testing marijuana product after failed initial safety testing

Rule 6(1) in the Sampling and Testing Rule Set 420.306(1) A laboratory may test marijuana product that has failed initial safety testing, except as indicated under subrule (3) of this rule.

(2) A failed marijuana product must pass 2 separate tests with new samples consecutively to be eligible to proceed to sale or transfer.

(3) The agency may publish a remediation protocol including, but not limited to, the sale or transfer of marijuana product after a failed safety test as provided in these rules.

(4) The marijuana business that provided the sample is responsible for all costs involved in a retest.

Research and Development (R&D) Testing

Rule 7(1) in the Sampling and Testing Rule set 420.307(1) As used in this rule, "research and development testing" means optional testing performed **before** final compliance testing.

(2) Except for R 420.304(2)(b), when performing research and development testing, the laboratory must comply with these rules.

(3) Punitive action shall not be taken against a marijuana business for conducting research and development testing.

(4) The agency may publish guidance for research and development testing that must be followed by all marijuana businesses.

(5) All research and development testing must be entered into the Metrc.

The licensee will select R&D testing at the time of the sampling. After the testing is completed, the appropriate laboratory staff will select the appropriate tests from the list below, aside from mycotoxins:

R&D Testing	Select R&D Testing Batch Result #1 "R&D Chemical Residue" enter the value found during analysis and then select pass or fail as the status. Result #2 "R&D Foreign Matter" enter the value found during analysis and then select pass or fail as the status. Result #3 "R&D Metals" enter the value found during analysis and then select pass or fail as the status. Result #4 "R&D Microbials" enter the value found during analysis and then select pass or fail as the status. Result #5 "R&D Moisture Content" enter the value found during analysis and then select pass or fail as the status. Result #6 "R&D Mycotoxins" enter the value found during analysis and then select pass or fail as the status. Result #7 "R&D Potency" enter the value found during analysis and then select pass or fail as the status. Result #8 "R&D Residual Solvents" enter the value found during analysis and then select pass or fail as the status. Result #9 "R&D Water Activity" enter the value found during analysis and then select pass or fail as the status.
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If the sample passes, please enter pass. If the sample fails, please enter the failure(s) into the notes section. You do not need to enter individual results for each test. The sample status will remain "testing in progress" until compliance testing is performed, at which point the status will update accordingly.

REQUIRED SAFETY TESTS AND ACTION LIMITS

The MRA has established action limits for all required analytes based on the most current literature. The laboratory shall report the results of the testing by indicating "pass" or "fail" in the Metrc system and on the Certificate of Analysis (CofA). The results in the Metrc system and the CofA should be identical aside from the reported results of testing not required by the agency (i.e. terpenes). For the purposes of chemical residue testing and target analyte testing, the agency shall publish a list of quantification levels. All results will be reported in parts per million (PPM) and to 3 decimal places unless otherwise specified.

POTENCY

The analytes required for potency analysis include Tetrahydrocannabinol (THC), Tetrahydrocannabinol acid (THC-A), Cannabidiol (CBD), and Cannabidiol acid (CBD-A). Laboratories can analyze additional cannabinoids CBN, CBG, CBC, THC-V, CBD-V, CBG-A with approval from the agency, and should be included in the scope of accreditation when obtained.

Concentrates and Flower

Total THC and CBD values should be calculated and reported as follows:

$$\text{Total THC\%} = (\text{THCa} * 0.877) + \text{d9-THC}$$

$$\text{Total CBD\%} = (\text{CBDa} * 0.877) + \text{CBD}$$

Marijuana Infused Products

For marijuana-infused products, the Delta-9-THC concentration can be reported in mg/serving. If the item contains more than one serving, the mg/container should also be included.

Rule 4 in the Marihuana-Infused Products and Edible Marihuana Product Rule Set R 420.404 (4) A marihuana sales location shall not sell or transfer marihuana-infused products that exceed the maximum THC concentrations established by the agency by more than 10%. For the purposes of maximum THC for marihuana-infused products, the agency shall publish a list of maximum THC concentrations and serving size limits.

If the reported delta-9 THC exceeds the maximum allowed, the product is considered failing.

Table 1. Maximum THC Concentrations for Marihuana-Infused Products

The Maximum Delta-9-THC Concentrations for Medical Marijuana-Infused Products		
	Per Dose	Per Container
Gummies, baked goods, etc.	50 mg	200 mg
Capsules, tinctures, etc.	100 mg	2000 mg
Suppositories and tampons	100 mg	2000 mg
Transdermal Patches	100 mg	2000 mg
Topical products including lubricants, spa products, lotions, balms or rubs	N/A	N/A
Products not listed	10 mg	100 mg

The Maximum Delta-9-THC Concentrations for Adult-Use Marijuana-Infused Products		
	Per Serving	Per Container
Gummies, baked goods, etc.	10 mg	100 mg
Capsules, tinctures, etc.	10 mg	200 mg
Topical products including lubricants, spa products, lotions, balms or rubs	N/A	N/A
Products not listed	10 mg	100 mg

TERPENE TESTING

Rule 5(18) in the Sampling and Testing Rule Set 420.305(18) A laboratory may perform terpene analysis on a marijuana product by a method approved by the agency. There are no established safety standards for this analysis. The laboratory shall analyze a sample of marijuana or marijuana-infused product to determine whether the terpenoid profile of the sample conforms to the labeled content of terpenoid(s).

CHEMICAL RESIDUE

Rule 5(11) in the Sampling and Testing Rule Set 420.305(11) For the purposes of chemical residue testing and target analyte testing, the agency shall publish a list of quantification levels. Any result that exceeds the action limit is a failed sample. The LOQ's required for chemical residue testing are provided in the table below and are set at ½ the action limit. The list is evaluated and an ongoing basis and updated based on the best available scientific and industry data, or if the federal government adds marijuana to 40 CFR part 180, subpart C, or the federal insecticide, fungicide, and rodenticide act, 7 USC 136 to 136y.

Table 2. List of Banned Chemical Ingredients: Action Limit and LOQ ^[11]

Analytes	CAS Number	Action Limit (ppm)	LOQ (ppm)
Abamectin	71751-41-2	0.5	0.25
Acephate	30560-19-1	0.4	0.2
Acequinocyl	57960-19-7	2	1
Acetamiprid	135410-20-7	0.2	0.1
Aldicarb	116-06-3	0.4	0.2
Azoxystrobin	131860-33-8	0.2	0.1
Bifenazate	149877-41-8	0.2	0.1
Bifenthrin	82657-04-3	0.2	0.1
Boscalid	188425-85-6	0.4	0.2

Carbaryl	63-25-2	0.2	0.1
Carbofuran	1563-66-2	0.2	0.1
Chlorantraniliprole	500008-45-7	0.2	0.1
Chlorfenapyr	122453-73-0	1	0.5
Chlorpyrifos	2921-88-2	0.2	0.1
Clofentezine	74115-24-5	0.2	0.1
Cyfluthrin	68359-37-5	1	0.5
Cypermethrin	52315-07-8	1	0.5
Daminozide	1596-84-5	1	0.5
DDVP (Dichlorvos)	62-73-7	1	0.5
Diazinon	333-41-5	0.2	0.1
Dimethoate	60-51-5	0.2	0.1
Ethoprophos	13194-48-4	0.2	0.1
Etofenprox	80844-07-1	0.4	0.2
Etoxazole	153233-91-1	0.2	0.1
Fenoxycarb	72490-01-8	0.2	0.1
Fenpyroximate	134098-61-6	0.4	0.2
Fipronil	120068-37-3	0.4	0.2
Fonicamid	158062-67-0	1	0.5
Fludioxonil	131341-86-1	0.4	0.2
Hexythiazox	78587-05-0	1	0.5
Imazalil	35554-44-0	0.2	0.1
Imidacloprid	138261-41-3	0.4	0.2
Kresoxim-methyl	143390-89-0	0.4	0.2
Malathion	121-75-5	0.2	0.1
Metalaxyl	57837-19-1	0.2	0.1
Methiocarb	2032-65-7	0.2	0.1
Methomyl	16752-77-5	0.4	0.2
Methyl parathion	298-00-0	0.2	0.1
MGK-264	113-48-4	0.2	0.1
Myclobutanil	88671-89-0	0.2	0.1
Naled	300-76-5	0.5	0.25
Oxamyl	23135-22-0	1	0.5
Paclobutrazol	76738-62-0	0.4	0.2
Permethrins*	52645-53-1	0.2	0.1
Prallethrin	23031-36-9	0.2	0.1
Phosmet	732-11-6	0.2	0.1
Propiconazole	60207-90-1	0.4	0.2
Propoxur	114-26-1	0.2	0.1
Pyridaben	96489-71-3	0.2	0.1
Pyrethrins+	8003-34-7	1	0.5
Spinosad	168316-95-8	0.2	0.1
Spiromesifen	283594-90-1	0.2	0.1
Spirotetramat	203313-25-1	0.2	0.1

Spiroxamine	118134-30-8	0.4	0.2
Tebuconazole	80443-41-0	0.4	0.2
Thiacloprid	111988-49-9	0.2	0.1
Thiamethoxam	153719-23-4	0.2	0.1
Trifloxystrobin	141517-21-7	0.2	0.1

* Permethrins should be measured as cumulative residue of cis- and trans-permethrin isomers (CAS numbers 54774-45-7 and 51877-74-8).

+ Pyrethrins should be measured as the cumulative residues of pyrethrin 1, cinerin 1 and jasmolin 1 (CAS numbers 121-21-1, 25402-06-6, and 4466-14-2 respectively)

RESIDUAL SOLVENTS

Table 3 lists the residual solvents required with their associated action limits. Action limits are based on the “International Conference for Harmonisation (ICH) Guideline Q3C (R5) on Impurities: Guidelines for residual solvents” and information provided by states with current marijuana programs. Please note that the LOQ for residual solvents must be ½ the action limit. Any detection that exceeds the published action limit is considered a fail.

Table 3. Action Limits for Residual Solvents

Solvent	CAS Number	Action Limit for Inhaled Products (ppm)	Action Limit for Non-inhaled Products (ppm)
1,2-Dichloroethane	107-06-2	2	5
Acetone	67-64-1	750	5000
Acetonitrile	75-05-8	60	410
Benzene	71-43-2	1	2
Butanes all isomers	106-97-8	800	5000
Chloroform	67-66-3	2	60
Ethanol	64-17-5	1000	5000
Ethyl acetate	141-78-6	400	5000
Ethyl ether	60-29-7	500	5000
Ethylene oxide	75-21-8	5	50
Heptane	142-82-5	500	5000
Hexanes all isomers	110-54-3	50	290
Isopropyl alcohol	67-63-0	500	5000
Methanol	67-56-1	250	3000
Methylene chloride	75-09-2	125	600
Pentanes all isomers	109-66-0	750	5000
Propane	74-98-6	2100	5000
Trichloroethylene	79-01-6	25	80
Toluene	108-88-3	150	890

Total xylenes (ortho-, meta-, para-)	1330-20-7	150	2170
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HEAVY METALS

Table 4 lists the heavy metals required with their associated action limits based on a 5 gram/day consumption of marijuana. These values were derived from USP 232-Elemental Impurities-Limits with additional information derived from the most recent consumption data available in the United States. ^{[18][21]} Please note that the LOQ for heavy metals must be ½ the action limit. Any detection that exceeds the published action limit is considered a fail.

Table 4. Heavy Metal Action Limits

Heavy metal	Action Limit for Inhaled Marijuana flower (ppm)	Action Limit for Inhaled Marijuana concentrates (ppm)	Action Limit for other Marijuana products (ppm)
Lead	1.0	0.5	0.5
Inorganic Arsenic	0.4	0.2	1.5
Mercury	0.2	0.1	3.0
Cadmium	0.4	0.2	0.5
Total Chromium	1.2	0.6	2.0
Nickel*	1.0	0.5	N/A
Copper*	N/A	3.0 ⁺	N/A

*Nickel and Copper requirements not effective until July 1, 2020.

+ Copper is required for vaping products only

FOREIGN MATTER ANALYSIS

Pests and other foreign matter including fungi, metal or plastic fragments, and both organic and non-organic debris are found in food as well as tobacco products and may also be detected in marijuana. The FDA considers debris of this kind in food to pose a negligible health hazard but recognizes that quality and user experience is compromised. Because of this, the FDA has methods for monitoring this type of contamination (FDA 2013b), which can be consulted to compare standards for different food commodities.

The action limit for crude marijuana is not more than 5.0% of stems and not more than 2.0% of other foreign matter. All failures should be documented photographically. It is recommended that the laboratory clearly delineate calculation guidelines and ranges for total surface area contamination. Foreign matter analysis should be performed prior to all other testing, aside from microbials. The material remaining after Foreign Matter

Analysis is acceptable for all chemical testing but should not be used for microbial testing. The amount of marijuana or marijuana product used for testing should be no less than 30% of the total gram weight or total sample lot obtained for compliance testing.

In the case of marijuana flower, the allotted 30% should come from separate, intact buds.

1. The buds should be separated into no less than 10 increments. The results from which can be averaged together as total foreign matter contamination.
2. Dissection of nodes should be done whenever physically possible.
3. If dissection of distinct nodes is deemed unnecessary, due to the small and compact nature of the buds (“popcorn” buds), the buds then should be examined in their entirety and additionally cut in half to observe the inside portion.
4. In the case of marijuana trim, kief, concentrate or infused product, the calculation to determine 30% of the sampling batch should be included in the SOP.
5. Filth analysis should be performed at a low-power magnification.
6. Quantitation of filth should be done as a total surface area calculation. The laboratory-derived calculation should be included in the SOP.
7. If a sample fails for foreign matter, the laboratory should include a note in Metrc listing all contaminants identified.

Analysis for Organic Matter

The 2.0% action limit is defined by approximate surface area (SA) and a detailed example of how this value is estimated can be found below:

1 node of an average sized marijuana bud has a total surface area of approximately 1.0 inch. It is reasonable to assume that there are approximately 5 nodes / 1-gram bud and an average surface area of 5.25 inches / 1-gram bud resulting in a total SA of 52.5 inches for 10, 1-gram buds of flower material.

It is recommended that the laboratory determine average surface area for common pests and create an easy-to-use scale for identifying passing and failing samples. Total surface area for contamination by mold could be done by estimating total surface coverage by mycelia or as otherwise determined by the SOP.

Analysis for Inorganic Matter

For these purposes, inorganic matter includes, but is not limited to, any material that would not normally be found on a living organism (plant) and includes materials such as glass, metal shavings, or synthetic fibers. In this case, the presence of any inorganic

matter on any marijuana plant, concentrate, or infused product would result in an automatic failure for foreign matter. The observed matter should be documented photographically, and a note of the results should be included in Metrc.

WATER ACTIVITY (a_w)

Water activity (a_w) is the partial vapor pressure of water divided by the standard state partial vapor pressure of water. With pure, distilled water having a water activity of exactly one. For testing purposes, water activity is a measure of the available water that can be utilized for microbiological growth. Values range from 0 to 1 with microbial growth unlikely below a_w 0.6. Most marijuana is dried and cured to a final water activity level of a_w 0.3-0.6. Most pathogens cannot grow below a_w 0.9.^[38] Based on the available research the following guidelines have been developed.

- A marijuana sample shall be deemed to have passed water activity testing if the water activity does not exceed 0.65 a_w .
- An **edible** marijuana-infused product shall be deemed to have passed water activity testing if the water activity does not exceed 0.85 a_w .
- Non-edible marijuana-infused products are **not** subject to water activity testing.
- Any detection that exceeds the published action limit is considered a fail.

MICROBIALS

The criteria for acceptability are in Table 5 (below). Any detection that exceeds the published action limit is considered a fail.

Table 5. Microbial Screening Action Limits (CFU/g)

	Bud, shake/trim from Harvest Batch (CFU/g)	Marijuana-Infused Product (CFU/g)	Marijuana Extract non-solvent & non-CO2* (CFU/g)
Medical Total Yeast & Mold Count	10,000	10,000	1,000
Adult-Use Total Yeast & Mold Count	100,000	10,000	1,000
Total Coliform	1000	100	100
Shiga toxin-producing <i>E. coli</i> (STEC)	If STEC is detected, the sample fails	If STEC is detected, the sample fails	If STEC is detected, the sample fails
Pathogenic <i>Salmonella spp.</i>	If Pathogenic <i>Salmonella spp.</i> are detected, the sample fails	If Pathogenic <i>Salmonella spp.</i> are detected, the sample fails	If Pathogenic <i>Salmonella spp.</i> are detected, the sample fails

<i>Aspergillus flavus, fumigatus, niger & terreus</i>	If <i>Aspergillus spp.</i> are detected, the sample fails	If <i>Aspergillus spp.</i> are detected, the sample fails	If <i>Aspergillus spp.</i> are detected, the sample fails
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TARGET ANALYTES

Rule 1(1)(dd) of the Marijuana Sampling and Testing Rule Set (R 420.301(1)(dd)) defines “Target Analyte” in the following way:

“Target Analyte” means a non-marijuana inactive ingredient designated for analysis.

Vitamin E Acetate has been identified as a target analyte by the MRA. All vape cartridges must be tested for Vitamin E Acetate. Any detection that exceeds the published LOQ is considered a fail.

Table 6. Target Analytes for Marijuana Products Intended for Inhalation

Target Analyte	(CAS) Number	LOQ (ppm)
Vitamin E Acetate	58-95-7	100

HOMOGENEITY

Homogeneity testing or the process of homogeneous sampling, is when all items in a sample are chosen at random to be representative of product batch so they have similar or identical traits. To perform homogeneity testing each dose or serving shall be treated as a separate individual sample and a total of 10 doses or servings shall be sampled at random. The weight and concentration of Delta-9 THC must be recorded and the variability of weight and concentration of Delta-9 THC among servings in a single package may not exceed +/- 15%.

This can be determined by first calculating the Standard Deviation (SD) among samples and subsequently calculating the Relative Standard Deviation (RSD) for both weight and concentration of Delta-9 THC.

The SD is calculated using the following formula:

$$SD = \sqrt{\frac{(sample1 - mean)^2 + (sample2 - mean)^2, \dots, (sample10 - mean)^2}{total\ number\ of\ samples - 1}}$$

The RSD is calculated using the following formula:

$$RSD = \frac{SD}{mean} \times 100$$

The MRA has determined that the laboratories should complete potency testing based on the information provided by the processors at the time of the sampling event. If a product is sampled as an individual serving/dosage, the facility will report total THC by serving/dose. If the product is sampled in final package, total THC content for the package will be reported.

It is not incumbent on the laboratory to determine if the product will meet the package labeling requirements. The laboratory testing results provided on the package will report the calculated THC as the product was submitted for testing and is not required to replicate the processor designated package label for THC content. The processor designated package label will be considered the target THC.

Table 8. Marijuana Testing Requirements

	Bud, shake/trim from Harvest Batch	Marijuana Extract non-solvent & non-CO₂^a	Marijuana Concentrate Co2 & Solvent-based	Marijuana Vape Product	Marijuana-Infused Product
Vitamin E Acetate				√	
Homogeneity					√
Potency Analysis	√	√	√	√	√
Foreign Matter Inspection	√	√			√
Microbial Screen	√	√			√
Water Activity	√				√ ^b
Heavy Metal Screen	√	√	√	√	√ (If Extract or concentrate not previously tested)
Residual Solvents			√	√	√ (If Extract or concentrate not previously tested)
Chemical Residue Analysis	√	√	√	√	√ (If Extract or concentrate not previously tested)

^a Extraction using ice water, rosin press or dry ice, this also includes kief

^b Not required for non-edible marijuana product

APPENDIX A-DEFINITIONS

Accuracy – A combination of the bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value. (Standard Methods, 18th edition). For the purposes of laboratory certification, accuracy means the closeness of a measured value to its generally accepted value or its value based upon an accepted reference standard.

Action Limit – The MRA defines an action limit as the maximum permissible level of contaminant in marijuana product allowable by the agency. These standards are toxicologically derived to protect human health. A marijuana product with a value that exceeds the published action limit is considered to be a failed sample. A marijuana sample that is at or below the action limit is considered to be a passing sample.

Analytical Sensitivity – The assay's ability to detect very low concentrations of a given substance. Analytical sensitivity is often referred to as the limit of detection (LOD). LOD is the actual concentration of an analyte in a sample that can be consistently detected \geq 95% of the time.

Analytical Selectivity – The degree to which the method can quantify the analyte of interest in the presence of other analytes, matrices, or other potentially interfering materials. This is usually achieved by isolation of the analyte through selective solvent extraction, chromatographic or other phase separations, or by application of analyte-specific techniques such as biochemical reactions (enzymes, antibodies) or instrumentation mass spectrometry (MS).

Batch – All marijuana product of the same variety that has been processed together and exposed to substantially similar conditions throughout processing.

Bias – Provides a measure of systematic, or determinative error in an analytical method. Bias is determined by assessing the percent recovery of spiked samples. Historically, the term accuracy has been used interchangeably with bias, although many sources make a distinction between the two. (Standard Methods, 18th edition).

Calibration – Modern instrumental methods depend upon the comparison of a signal from the unknown concentration of an analyte to that from a known concentration of the same or similar analyte. The simplest calibration procedure requires preparation of a series of standard solutions from the reference material, by dilution of a stock solution, covering a reasonable range of signal response from the instrument. Four or more points, approximately equally spaced over the concentration range of interest, performed in duplicate but measured at random (to avoid confusing nonlinearity with drift) is a suitable calibration pattern. Fit the calibration line and plot the residuals as a function of concentration. An acceptable fit produces a random pattern of residuals with a zero mean.

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

CFU/g – Colony forming units per gram.

Final Form- means the form a marijuana product is in when it is available for sale by a marijuana sales location. For marijuana products intended for inhalation, the marijuana concentrate in the e-cigarette or vaping device.

Final Package- means the form a marijuana product is in when it is available for sale by a marijuana sales location.

Harvest batch – A designated quantity of harvested marijuana, all of which is identical in strain and has been grown and harvested together and exposed to substantially similar conditions throughout cultivation.

Instrument Detection Limit (IDL) – The concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument. The IDL should always be below the method detection limit, and is not used for compliance data reporting, but may be used for statistical data analysis and comparing the attributes of different instruments.

Interfering Substances – One that at the given concentration causes a systematic error in the analytical result.

Linear Calibration Range (LCR) or Range of Linearity – The region of a calibration curve within which a plot of the concentration of an analyte versus the response of that particular analyte remains linear and the correlation coefficient of the line is approximately 1 (0.995 for most analytes). The plot may be normal-normal, log-normal, or log-log where allowed by the analytical method. At the upper and lower bounds of this region (upper and lower limits of quantitation), the response of the analyte's signal versus concentration deviates from the line.

Limit of Detection (LOD) or detection limit – The lowest concentration level that can be determined to be statistically different from a blank (99% confidence). The LOD is typically determined to be in the region where the signal to noise ratio is greater than 5. Limits of detection are matrix, method, and analyte specific. The LOD is approximately equal to the MDL for those tests which the MDL can be calculated.

Limit of Quantitation or lower limit of quantitation (LOQ) – Limit of quantitation is defined in rule as the minimum concentration or mass of an analyte in a given matrix that can be reported as a quantitative result.

The LOQ is mathematically defined as equal to 10 times the standard deviation of the results for a series of replicates used to determine a justifiable limit of detection. *Limits of quantitation are matrix, method, and analyte specific.*

Marijuana-Infused Product – Any product containing marijuana that is intended for human consumption in a manner other than smoke inhalation.

Marijuana Product – Marijuana or a marijuana-infused product, or both, as those terms are defined in the act unless otherwise provided for in these rules.

Marijuana Concentrate – A product derived from marijuana that is kief, hashish, bubble hash, oil, wax, or other product, or that includes cannabinoids extracted from the plant by any means.

Marihuana product intended for inhalation- Means any marihuana concentrate that is intended to be inhaled using an e-cigarette or vaping device.

Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL procedure is designed to be a straightforward technique for estimation of the detection limit for a broad variety of physical and chemical methods. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps used by the laboratory be included in the determination of the method detection limit. To calculate MDLs please follow this [procedure](#).

Milligrams (mg) – A unit of mass equal to one thousandth (10^{-3}) of a gram.

Precision – A measure of the random error associated with a series of repeated measurements of the same parameter within a sample. Precision describes the closeness with which multiple analyses of a given sample agree with each other and is sometimes referred to as reproducibility. Precision is determined by the absolute standard deviation, relative standard deviation, variance, coefficient of variation, relative percent difference, or the absolute range of a series of measurements.

Production batch – A manufacturing event of batch of material containing any amount of Medical or Adult-use Marijuana Concentrate of the same category and produced using the same extraction methods, standard operating procedures and an identical group of Harvest Batch(es) of Medical or Adult-use Marijuana; or (b) any amount of Medical or Adult-use Marijuana Product of the same exact type, produced using the same ingredients, standard operating procedures and the same Production Batch(es) of Medical or Adult-use Marijuana Concentrate.

Reportable Range – The upper limit of the reportable range will be set at the concentration of the highest standard tested which exhibited acceptable results for linearity, accuracy and precision. the lower limit of the reportable range will be set at the lowest standard tested which exhibits acceptable results

Reporting Limit – Arbitrary number below which data is not reported. The reporting limit may or may not be statistically determined or may be an estimate that is based upon the experience and judgement of the analyst. Analytical results below the reporting limit are expressed as "less than" the reporting limit. Reporting limits are not acceptable substitutes for detection limits.

Representative Sample – A sample obtained according to a sampling procedure designed to ensure that the different parts of a batch or the different properties of a batch are proportionally represented.

Sample Matrix, or Matrix – Defines the general physical-chemical composition of a sample other than the analyte of interest. General classes of matrices commonly referred to in the marijuana industry include flower, concentrate, marijuana-infused product, and all associated subtypes.

Sample Standard Deviation, or Standard Deviation (s) – A measure of the degree of agreement, or precision, among replicate analyses of a sample.

Sample – An amount of marijuana collected by laboratory personnel from a licensee and provided to a laboratory for testing.

Solvent – A substance that can dissolve another substance, or in which another substance is dissolved, forming a solution. Examples of solvents include water, acetone, turpentine and ethanol.

Statewide Monitoring System – Marijuana Enforcement Tracking Reporting & Compliance (Metrc).

Statistical Outlier, or Outlier – An observation or data point that appears to deviate markedly from other members of the population in which it occurs. The presence of outliers must be verified using an approved statistical method, at the 1% significance level.

Target Analyte – Non-marijuana inactive ingredient designated for analysis.

Test batch – A group of samples that are derived from a single Production Batch. The combined subset of Samples is collectively submitted for to a licensed testing facility for testing purposes.

Validation – The process of demonstrating or confirming the performance characteristics of a method of analysis. The validation of a method of analysis results in the specification of various aspects of reliability and applicability. Changes to the validation will require revalidation.

Water Activity – The partial vapor pressure of water in a substance divided by the standard state partial vapor pressure of water.

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