

Sample Collection

Alaska has enacted only basic sampling requirements, and sampling is performed by the producers (self sampling). Harvest batch packages are limited to ten pounds, and sampling requirements specify samples be composed of “representative” 1 gram increments. Samples are to be between 4 and 16 increments, for 1 lbs to 16 lbs harvest batch packages.

Sample Homogenization

Samples are processed at room temperature first by course grinding, then homogenized by mixing and quartering. One gram test aliquots are accurately weighed for extraction.

Sample Preparation

A surrogate solution of ibuprofen in methanol is added to each sample. Extraction solvent is 9:1 methanol:chloroform (v/v). Extractions are agitated, sonicated by water bath, and centrifuged. The final extraction is filtered through PTFE membrane. For potency testing, the extraction is diluted in methanol prior to instrumental analysis.

Sample Analysis:**Potency:**

Dilutions are injected into an Agilent 1100 HPLC system including a G1322A degasser, a G1311A QuatPump, a G1313A autosampler, a G1316A thermostated column compartment, a Zorbax Eclipse Plus C18 4.6x100mm analytical column with 3.5 micron medium, and either a G1314A variable wavelength detector or a G1315B Diode Array Detector reading at 230 nm. Data acquisition and analysis are via OpenLAB CDS EZChrom software. Mobile phase is a gradient of acetonitrile and 50 mM ammonium formate, pH 3.75.

Terpenes:

Undiluted extraction is introduced by headspace sampling into an Agilent 6890N GC system with a ResTek Sky 1mm split inlet liner, a ResTek Rxi-624Sil MS 30 m x 0.25 mm id x 1.4 µm film column, and a flame ionization detector (FID), coupled with a 7697A headspace sampler. Data acquisition and analysis are via OpenLAB CDS EZChrom software. Mobile phase is helium.

Sample Collection

Upon sampling, the entire batch of cannabis flower (not to exceed 15lbs per batch) must be presented for sampling. The sample increments are selected at random and increments taken must be greater than or equal to 0.5% of the batch's total mass.

Sample Homogenization

The state of Oregon defines usable cannabis as “dried leaves and flowers of marijuana [and]... does not include the seeds, stalks, and roots of marijuana; or waste material that is a by-product of processing marijuana.” Based on this definition, there is a “de-stemming” procedure performed in which the buds are broken down by hand and the stems are removed. From here, the samples are then cryo-ground in order to produce a fine powder material that is homogenous.

Sample Preparation

Once the samples have been homogenized, they are weighed out for extraction and placed into conical tubes where the extraction solvent (MeOH fortified with Ibuprofen as an internal standard) is introduced. The sample and solvent tubes are then agitated at high speed for 5 minutes. Once fully extracted, the sample solution is then filtered using PTFE filters and further diluted 50x and transferred to 2mL autosampler vials.

Sample Analysis:**Potency:**

Once the sample vials are prepared, they are put onto our instruments which are Agilent 1260 Infinity II HPLC instruments using UV-Vis diode array detectors. The method is a gradient using Water and Acetonitrile as mobile phases. Each analytical sequence is run along with a solvent blank, a laboratory control standard (characterized cannabis products for each matrix type), a matrix duplicate, a matrix spike sample, and is bracketed by continuing calibration verification standards.

Terpenes:

If a sample is to be tested for both cannabinoid potency and terpenes, two vials of the same extract are prepared. One for the HPLC for potency and one for the FID to assess terpene recovery. For terpene analysis, we use a liquid injection into a split inlet onto a GC column designed for cannabinoid pesticide analysis. This allows for a full representation of the sample's aromatic and volatile compounds on the column and in the analysis.

Sample Collection

Samples are collected from licensed cannabis production facilities in Washington state. Each sample represents a production lot of cured, dried, and usually trimmed cannabis flower. The WSLCB defines a production lot as “the flowers from one or more marijuana plants of the same strain. A single lot of flowers cannot weigh more than five pounds.”

Sample Homogenization

Cannabis flower samples are homogenized by way of a frozen-ball-milling technique using a blast freezer set to -25°C and a Spex Geno Grinder 2010 or similar.

Sample Preparation

Subsamples are deducted with a metal spatula and weighed on an analytical balance to the nearest 0.1 mg into a 15 ml conical tube. Cannabinoids are extracted with 10 ml HPLC grade methanol spiked with internal standard propyl paraben. Samples are filtered before instrumental analysis at 0.2 micron.

Sample Analysis:**Cannabinoids and Terpenes:**

Shimadzu GC-FID+MS (dual detection)

Shimadzu HS-20 Headspace

Shimadzu GC-2030 GCMS-QP2020 NX

Injection Mode: Split

Carrier Gas: He

Column: Restek Rxi-624Sil MS

H2 Flow: 40.0 mL/min

Air Flow: 400.0 mL/min

Shimadzu Nexera-i UPLC system

Model LC-2040C 3D with a PDA detector

Mobile Phase A: H₂O, 0.18% Formic Acid, 4 mM Ammonium Formate

Mobile Phase B: Acetonitrile

Flow Rate: 1.8 mL/min gradient to 3 mL/min

Column: Restek ARC-18, 2.7µm, 150 x 4.6 mm

Wavelength: 190-400 nm

Sample Collection

Flower samples are collected from Medical Marijuana Treatment Centers (MMTCs) throughout the state of Florida. The amount of sample collected is dependent on the total batch size of the product. The laboratory collects 15g of sample, or 0.35% of the total retail batch, whichever is greater.

Sample Homogenization

Upon arrival to the laboratory, the flower material is broken into small pieces and the entire collected sample is mixed. The small pieces are transferred into 50ml conical tubes with steel grinding balls and placed into a freezer prior to being fully ground using a SPEX 2010 Geno Grinder. If the total sample is spread across multiple conical tubes, all tubes of the same sample are thoroughly mixed to ensure full homogenization.

Sample Preparation

After homogenization, samples are weighed (0.2g) into a 20ml vial using an analytical balance and 10ml of 9:1 (Methanol:Chloroform) are added to the samples. Once this step is complete, the samples are agitated prior to being transferred to an ultrasonic water bath for 5 minutes. After sonication, samples are agitated once more, prior to allowing the particulate to separate. An aliquot of the extracted material is taken from the top layer of the vial and transferred to a 2ml autosampler vial. An additional dilution of 100X is performed and both the undiluted and diluted extract are run on the HPLC.

For terpene preparation, samples are weighed (0.1g) into a 15ml conical tube using an analytical balance and 10ml of Methylene Chloride are added to the samples. Once this step is complete, the samples are agitated prior to being transferred to an ultrasonic water bath for 5 minutes. After sonication, samples are agitated once more, prior to being centrifuged at 5000rpm for 5 minutes. A 1ml aliquot of the extracted material is taken from the top layer of the tube and transferred to a 2ml autosampler vial. Internal standard (2-fluorobiphenyl) is added to the autosampler vial, and the extract is run on the GC-MS.

Sample Analysis:

Potency:

After the samples have been prepared, they are analyzed using a Shimadzu LC-40 equipped with a UV-Vis detector on a binary gradient. The samples are analyzed in an analytical batch that contains a method blank, laboratory control sample, laboratory control sample duplicate, matrix spike, and matrix spike duplicate to ensure the preparation was done correctly.

Additionally, continuing calibration verifications are analyzed every 10 injections to ensure the instrument is operating in the proper specifications.

Terpenes:

After the samples have been prepared, they are analyzed using liquid injection on an Agilent 8890-GC coupled with a 5977B-MS. The samples are analyzed in an analytical batch that contains a method blank, laboratory control sample, and a sample duplicate to ensure the preparation was done correctly. Additionally, continuing calibration verifications are analyzed every 10 injections to ensure the instrument is operating in the proper specifications.

Sample Collection

Samples of cannabis flower that are tested for State of Michigan compliance are collected from licensed cultivation facilities by PSI Labs samplers. These samples are randomly selected in increments from the bulk batch with a total sample weight corresponding to 0.5% of the total batch weight. Samples are also dropped off over the counter at PSI Labs, Ann Arbor, MI by medical caregivers.

Sample Homogenization

Approximately half of each sample is ground and homogenized to a finer size using glass mortar and pestles or a food processor.

Sample Preparation

For potency analysis, around 100mg of ground flower is weighed into a 15mL centrifuge tube and extracted for about 15 min in an ultrasonic bath with 12.5 mL of methanol delivered from a calibrated solvent dispenser. These sample extractions are diluted by a factor of 10× with methanol prior to injection into the HPLC for potency analysis. For terpene analysis, 80-100mg of ground flower is weighed into a microcentrifuge tube and extracted with 1mL of methanol by vortexing for about 1 min. A 20µl aliquot of the terpene sample extractions are placed into a headspace vial and sealed with spike containing isotopically labeled p-xylene as an internal standard.

Sample Analysis:**Potency:**

Cannabinoid analyses of flower extracts are performed using an Agilent 1200 series HPLC equipped with a UV-vis diode array detector. Cannabinoids are separated on a C-18 column using a gradient method with water and acetonitrile (both at 0.1% formic acid) as the mobile phases.

Terpenes:

Terpenes are analyzed by headspace analysis using an Agilent 8890 GC equipped with a 5977 MS and a 7697 HS autosampler. After heating the sample extractions in the headspace autosampler, the terpenes are separated using a G43 phase column with He carrier gas and quantified using the MS detector.

For both methods, blanks and calibration verification checks are performed prior to analysis of a batch of samples. Throughout the analytical session continuing calibration verification

checks are also analyzed between every 20 sample injections to ensure acceptable instrument performance over the batch.

Sample Preparation & Analysis:

Potency:

Samples were measured for cannabinoid content according to the method described in “Appendix 2. HPTLC Chromatographic Profile” of the document, “JNP Appendices.pdf.”

Terpenes:

Samples were measured for terpene content according to the methods details found in the document, “SC-Labs-ISO-17025_2017-Accreditation-Certificate-2022-01-25.pdf,” including measurement uncertainties.