Cole-Parmer®
sampleprep

Cannabis and Hemp
Sample Preparation Protocols

APPARATUS: Geno/Grinder®, MiniG®, GenoLyte®, Freezer/Mill®

APPLICATION: Sample Preparation

The following procedures are provided as a basic guideline for cannabis and hemp sample preparation and are in no way required or absolute validated methodologies. Spex makes no claims in regard to instrument parameters and environmental conditions related to the specific physical and chemical properties of individual samples. Spex SamplePrep does not perform analytical testing and results are based on customer research publications or limited in-house studies. These suggested procedures are a starting point for method development and would require appropriate method validation and verification to be included as part of standard protocols, publications, procedures, good laboratory procedures, or SOPs.

Geno/Grinder® and MiniG®

Cannabis and Hemp (Flower, Seeds and Leaves)

Grinding method for small samples up to 2 grams:

1. Place two 11 mm stainless steel balls into a 50 mL centrifuge tube.
2. Weigh up to 2 g of the sample and place in the tube.
3. Place tube in the homogenizer and shake at 1500 rpm for 1 to 2 minutes.
4. Add half of the desired volume extraction solvent (methanol or acetonitrile, etc.) to the tube.
5. Swirl contents of the tube until steel balls are free of any residual material.
6. Remove balls with magnetic pickup tool. Rinse balls with solvent into the sample to reduce sample loss.
7. Remove grinding balls for cleaning and reuse.
8. Add solvent to reach desired volume.
9. Reseal the centrifuge tube and shake at 1000 rpm for 30 seconds to ensure homogeneity and complete extraction.
10. For samples with an exceptionally high moisture content or ‘sticky’ characteristics:
    a. Freeze for one hour or more prior to grinding, or place sample in liquid nitrogen/dry ice for several minutes to reduce sample temperature.

Grinding method for mid-size samples from 2 grams to 5 grams:

1. Chill sample before homogenization.
2. Place one 11 mm and two 9.5 mm stainless steel balls into the bottom of a 50 mL centrifuge tube before weighing the cannabis sample.
3. Weigh 2 g and up to 5 g of material and place in the tube.
4. Shake at 1500 rpm for 1 to 2 minutes.

Before and After Samples

Grinding method for large samples from 5 grams to 30 grams::

1. Using either one 12 oz or two 5 oz PET jars (PN 2248, 2258) in conjunction with our large format foam holder (PN 2259), add sample to jar with up to fifteen 11 mm stainless steel grinding balls.
2. Grind sample at 1500 rpm for 1 to 5 minutes.
3. The number of balls will depend on the volume of the sample:
   a. Samples between 5 g and 30 g: 3-6 balls.
   b. Samples between 40 g and 80 g: 10-15 balls, and a run time of 3-5 minutes.
4. For samples with an exceptionally high moisture content or ‘sticky’ characteristics:
   a. Freeze for one hour or more prior to grinding, or place sample in liquid nitrogen/dry ice for several minutes to reduce sample temperature.
5. The grinding balls must be cleaned prior to extraction to prevent sample loss. Rinse the beads in methanol or acetonitrile, etc., or use liquid nitrogen to freeze the adhered sample and scrape it from the grinding balls into the bulk sample.

Examples

10 g sample of cannabis buds/flower ground for one minute at 1500 rpm using five 11 mm stainless steel balls.

30 g sample of cannabis trimmings ground for two minutes at 1500 rpm using eight 11 mm stainless steel balls.

1200 GenoLyte®

Cannabis and Hemp (Flower, Seeds and Leaves)

Grinding method for small samples up to 2 grams:

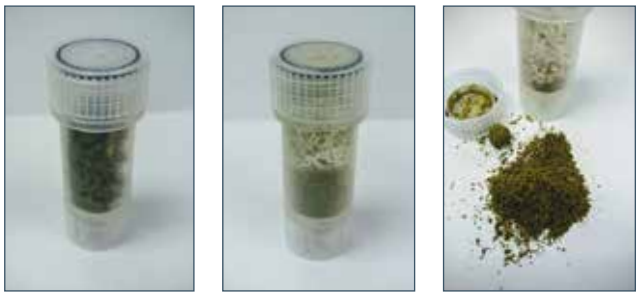
1. Place one to two, 1/4-inch (6.35 mm) stainless steel balls into 7 mL centrifuge tubes (PN 2142-PE).
2. Weigh sample and place in the tubes (typical sample 0.5 g).
3. Place tubes into the GenoLyte and shake at 3000 rpm for 15-30 seconds.
4. Add the desired volume of extraction solvent (methanol, acetonitrile, etc.) to the tube.
5. Shake for an additional 10 seconds at 750 rpm or until steel balls are free of any residual material.
6. Remove balls with magnetic pickup tool and rinse with solvent to reduce sample loss.
7. Remove grinding balls for cleaning and reuse. Clean with detergent and warm water or autoclave.
8. Add solvent (if needed) to reach desired volume.
9. Reseal the centrifuge tube and shake at 750 rpm for 30 seconds to ensure homogeneity and complete extraction.

Alternate grinding method for small samples up to 2 grams:

1. Place one to two, 1/4-inch (6.35 mm) stainless steel balls into 7 mL centrifuge tubes (PN 2142-PE).
2. Weigh sample and place in the tubes (typical sample 0.5 g).
3. Add extraction solvent to tubes, seal and grind at 3000 rpm for 15-30 seconds.
4. For samples with an exceptionally high moisture content or ‘sticky’ characteristics:
    a. Freeze sample for one hour or more prior to grinding, or place sample in liquid nitrogen/dry ice for several minutes to reduce sample temperature.

GenoLyte configured for 2 x 7 mL tubes loaded with 0.5 g of cannabis (hemp).

Examples



0.5 g of cannabis (hemp) before and after being ground in the GenoLyte configured for 7 mL tubes with one 1/4-inch stainless steel bead. Sample was ground for 30 seconds at the 3000 rpm setting.

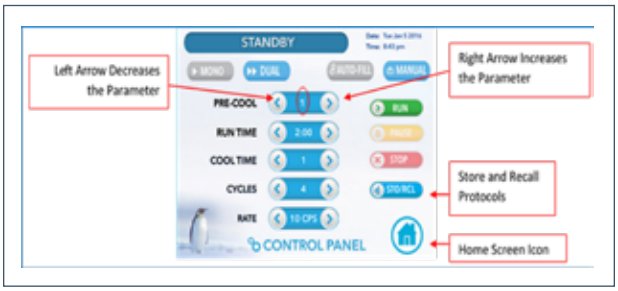
0.5 g of cannabis (hemp) before and after being ground in the GenoLyte configured for 7 mL tubes with one 1/4-inch stainless steel bead and 3 mL of EtOH. Sample was ground for 30 seconds at the 3000 rpm setting.

6875D/6875 Freezer/Mill®

Edibles

Grinding method for edibles, concentrates, and infused consumables:

1. The Freezer/Mill has a magnetically driven impactor which is inserted with sample into the large (#6801) grinding vials.
   a. Ensure that the impactor has free movement after the sample is placed into the vial and the endcap is replaced.
   b. If the impactor becomes lodged or obstructed from movement, the sample will not be ground.
2. Insert vial into the grinding chamber (please refer to the instruction manual for loading and operation of the instrument).
3. Depending on the sample viscosity, consistency and moisture content, it may be necessary to pre-chill the sample in liquid nitrogen before inserting the impactor.
4. Set the pre-cool time to approximately 5 minutes (up to 10 minutes depending on the sample) and the run time between 1-2 minutes.
5. Set the rates/cycles per second to 12.
6. For most samples in this category, a single cycle will be necessary, so you may set the cycle to 1.
   a. The instrument will start grinding as soon as the pre-cool period has elapsed and the impactor movement will be audible. The run can be paused or canceled at any time to inspect the sample for the desired particle size.
7. Once the run is complete, remove the vial from the instrument and immediately transfer the ground sample to a container for testing or storage.
   a. If the sample is going to be stored, it is advisable that it be kept as cool as possible to prevent agglomeration.
   b. For later testing, most samples will retain their powder consistency if they are transported to and stored at -80 °C immediately after grinding.



Touchscreen user interface to program desired method parameters.

8. The cleaning procedure is crucial for longevity of the vials.
   a. Vials have center cylinders made of polycarbonate and are incompatible with organic solvents (alcohols, cyclohexanes, etc.).
   b. Polycarbonate is not able to be autoclaved or exposed to high temperatures.
   c. Clean tubes with mild laboratory grade detergents and lukewarm water or a 5% bleach solution.
   d. These tubes can be decontaminated with RNAse/DNAse solution, UV and gaseous decontaminants (ethylene oxide).
   e. The end plugs and impactor, which are made from stainless steel, do not require these special cleaning considerations.

Before and After Samples

Before and after results using the Freezer/Mill for several representative matrices commonly encountered with regard to infused/edible products.

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