CBD Isolation by Crystallization

# I. Purpose

The following procedures describe the isolation of chemically pure CBD by crystallization using pentane.

# II. Objective

The objective of these procedures is to prioritize the safety of personnel while producing a consistent, quality controlled final product.

# III. Responsibility

This SOP is the responsibility of the following personnel:

* Director of Extraction
* Extraction Technician

# V. Definition of Terms

**The following terms are used in this document:**

**Personal Protective Equipment (PPE) -** Specialized clothing or equipment worn by employees for protection against health and safety hazards.

**Standard Operating Procedures (SOP)** - established or prescribed methods to be followed routinely for the performance of designated operations or in designated situations —called also standing operating procedure.

**CBD Isolate** – Purified CBD in crystalline form.

**Mother Liquor** – The liquid mixture remaining after crystal filtration.

**Pentane** – A non-polar solvent with a flash temperature of 57F/14C boiling temperature of 97F/36C

**Distilled Oil** – In this application Distilled Oil will mean winterized, decarboxylated and distilled oleoresin of the cannabis plant containing 70% or more CBD by weight.

## Materials Needed:

* Stainless Steel Mixing Vessel
* Stainless Steel overhead or hand mixer/homogenizer
* Mortar & Pestle or stainless steel grinder (manual, pneumatic, or appropriately rated electric motor)
* Stainless Steel mesh screen basket (as large as opening of stainless steel vessel)
* Dry Ice/Alcohol Slurry & Storage Bucket (capable of accommodating stainless steel mixing vessel)
* Chemically Pure Pentane
* Distilled Oil containing 70% or more CBD by weight

## Personal Protective Equipment:

* 1. Gloves
  2. Lab coat
  3. Safety glasses
  4. Shoe covers
  5. Hairnet or lab hat
  6. Beard cover

Note: All PPE must be worn for the duration of all procedures.

Note: Any employee who will be conducting work in the Laboratory must be trained on “SOP - Chemical Hygiene Plan”, before any process can be started. Also training must be completed on “SOP – Pentane Use” before the solvent can be used in any process.

# VI. Procedure

## Preparing Distilled Oil

* 1. Fill a clean stainless steel vessel no more than half way with Distilled Oil
  2. Warm the Distilled Oil in the vessel to 90F/32C, stirring constantly with overhead mixer or homogenizer on lowest setting.

1. **Mixing Pentane** 
   1. Slowly introduce room temperature pentane while constantly stirring the distilled oil on the lowest mixer/homogenizer setting.
      1. Do not allow the mixture to exceed 90F/32C
      2. Do not mix pentane outside of an appropriately ventilated environment.
      3. Do not mix pentane without wearing all required PPE.
   2. Continue to mix until the distilled oil is uniformly distributed within the pentane.
2. **Chilling the Solution**
   1. Using an appropriately rated, solvent safe freezer or dry ice slurry, slowly bring the solution down to -31F/-35C
      1. It is important to decrease solution temperature slowly to achieve the cleanest possible initial crystal formation.
   2. Allow solution to remain at -31F/-35C until substantial formation of crystals is visible at the bottom of the stainless steel vessel.
3. **Removing Mother Liquor**
   1. Pour the chilled solution out of the stainless steel mixing vessel into another stainless steel mixing vessel through a fine stainless steel mesh screen.
   2. Recover the pentane from the mother liquor using an appropriate solvent recovery system and set aside the remaining oil to be refined later.
4. **Rinsing the Crystals** 
   1. Chill pentane to -31F/-35C in appropriately rated freezer or in dry ice slurry.
   2. Remove CBD Crystals from the bottom of the stainless steel mixing vessel and place in a stainless steel mesh screen basket resting securely over the mother liquor solution.
   3. Pour the chilled pentane over the crystals, agitating/stirring as little as necessary to rinse all crystals until they are completely colorless.
      1. Use as little pentane as necessary to remove impurities.
      2. Use a wash bottle with a nozzle to maximize efficiency.
      3. Ensure the pentane stays chilled to -31F/-35C during the entire rinsing process.
5. **Pulverize the Crystals** 
   1. Chill the CBD Crystals in an appropriate freezer or dry ice slurry to -31F/-35C
   2. Remove from freezer/slurry and filter any residual liquid pentane through a stainless steel mesh screen.
   3. Return crystals to stainless steel vessel and pulverize into a uniform powder using a mortar and pestle, or a manual, pneumatic or appropriately rated electric grinder.
   4. Evenly distribute the CBD powder over a large flat surface area (in stainless steel trays lined with parchment or shallow pyrex dishes) at a thickness of approximately 1inch/2.5cm
   5. Place the CBD powder in a vacuum oven at ambient/room temperature and pull vacuum in one or two hour intervals until the residual pentane is removed.
      1. Break vacuum only to stir and redistribute the powder as needed.

# Change Log

| Date | Revision | Modified By | Description of Changes |
| --- | --- | --- | --- |
| [Date] | 1.0 | [Department F/-Head] | Initial release. |
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**Document Release**

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| Name: | Signature: | Date: |