Standard Operating Procedure
Cannabinoid Conversion
CBD Isolate – Δ8 Crude
Reactor Method
Full Overview

Conducted by:
Grim Leadingham
WKU CONSULTING
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Scope:
Outlining the procedures necessary to convert 10-20Kg's of high purity CBD Isolate into Δ8THC Crude Oil via a 100L Reactor via p-Toluenesulfonic acid reagent (TsOH/PTSA).

Processing Needs (Equipment):

1. Reaction Apparatus (100L Reactor)
2. Reactor heat circulating unit
3. Reactor Condenser chilling unit
4. 5 Gallon Containers (Food Grade)
5. 1 Gallon Containers (Food Grade)

Processing Needs (Solvents & Consumables):

1. nHeptane
2. Distilled Water
3. Sodium BiCarbonate (NaHCO3)
4. Salt (NaCl)
5. p-Toluenesulfonic acid (TsOH/PTSA)

Personal Protective Equipment (PPE):

1. Lab Coat (Long sleeve)
2. Eye Protection
3. Respirator (Chemical Rated, 3M)
4. Black Nitrile Gloves (5-7 mil)
5. Pants (No shorts)
6. Closed-Toe, Slip Resistant Shoes

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3.2. Wash #2, Sodium BiCarbonate Wash (NaHCO3). (x2)
3.3. Wash #3, Salt/NaCl Wash (NaCl) (x2)
3.4. Wash #4, Distilled Water Wash. (x2-4)
3.5. Δ8 Crude:Heptane Collection
4. Further Refinement Needs

Procedure:

1. **Reactor & Reaction Preparations**
   a. Addition of CBD Isolate into the reactor, melting down the isolate into a liquified state, adding equal parts of Heptane into the reactor. 10-20Kg Scaled Method via 100L Reactor.

1.1. Reactor Preparation. Turn ON the reactor's jacketed circulating heater to 80c.
1.1.1. Turn ON the reactor's reflux condenser circulating chiller to -10c (or as cold as your circulator will go).
1.2. ***CBD Isolate Addition.*** Weigh 10,000g's/10Kg's of CBD Isolate in glass beakers or stainless-steel pot.
1.2.1. Can melt down the CBD Isolate pre-addition on mag stir hot plates or in a vacuum chamber if desired. Then add liquified CBD into reactor. Follow continued steps.
1.3. Add the CBD Isolate into the glass jacketed reactor.
1.4. Heat the CBD isolate until in a semi-liquified state.
1.4.1. Once the CBD isolate begins to melt and liquify, turn ON the reactor's stirrer to 100rpm's.
1.4.2. If the stirrer is not moving or is struggling to move, turn OFF the stirrer and allow the CBD isolate to heat longer until in a more liquified state. Once in a more liquified state, attempt to stir the solution again.
1.5. Solvent Addition. Add equal parts (1:1)(CBD:Heptane) of room temperature/clean nHeptane by volume of CBD input. ***Ex.*** 10Kg's of CBD input = (10,000mL's/10L's/2.64 gallons) of heptane added into the reactor containing the liquified CBD.
1.6. Allow the CBD and nHeptane to fully homogenize with the stirrer set at 100-125rpm's.
1.7. Lower the temperature of the reactor's circulating heater to 50c!
1.7.1. Allow the CBD/Heptane solution and the circulating heater to fully stabilize at 50c before continuing to the next step (important!).

2. **Reaction**
   a. Outlining the procedures needed to successfully convert CBD into Δ8 Crude via p-Toluenesulfonic acid reagent (PTSA/TsOH).

2.1. ***Reagent Prep.*** Weigh the p-Toluenesulfonic acid reagent at a 5% ratio by volume of the input CBD Isolate. ***Ex.*** 5% PTSA reagent for 10,000g's/10Kg's of CBD isolate = 500 grams of PTSA reagent needed.
2.1.1. Be sure to wear all PPE during the weighing and addition of the reagent.
2.2. ***Reagent Addition.*** Once the CBD:Heptane solution temperature within the reactor has fully stabilized to 50c and is fully homogenized, slowly add the PTSA into the reactor.
2.2.1. The addition of PTSA will cause an exothermic reaction and will increase the temperature of the solution by 20-30c.

2.3. Reaction. Once the temperature increase has stabilized, increase the reactor's jacketed circulating heater to 90-102c (This temperature will vary depending on ambient temp, amount of reagent:solution, stirrer rpm's, etc. The boiling point of heptane is 98.4c).

2.3.1. Increase the temperature until a minor reflux has occurred on the reactor's condensing unit. Look for minor sweating on the condenser and on the sides of the reactor. A vigorous reflux is NOT needed nor intended for this method.

2.4. Once a steady and slow reflux is achieved, allow the solution to reflux, and convert for a duration of 4 hours, undisturbed.

2.5. After the 4-hour conversion period is concluded, turn OFF the reactor's circulating heater and allow the solution to cool to room temperature (25c).

3. Liquid-Liquid-Extraction (LLE)(Washes)

a. Utilizing LLE methods within the reactor via distilled water, sodium bicarbonate (NaHCO3), and salt (NaCl). Removing undesirables and neutralizing the pH of the Δ8 Crude. Solutions will be homogenized in containers then added to the reactor containing the Δ8 Crude:Heptane solution. This process will occur at room temperature, with the heat circulator ON and set temp equal to room temperature (25c) to eliminate any major temperature swings during LLE.

3.1. Wash #1, Distilled Water Wash. (x2) Add equal parts (1:1) of distilled water by the total volume of the solution within the reactor. 10L's of CBD + 10L's of Heptane = 20L's of total solution. Add 20L's/5.28 gallons (per wash) of distilled water into the reactor containing the Δ8 crude:Heptane solution.

3.1.1. Turn ON the stirrer to 100rpm's for 5 minutes.

3.1.2. Turn OFF the stirrer and allow the solution to separate for 30 minutes.

3.1.3. After the 30-minute separation time, discard the aqueous/water layer at the bottom of the reactor.

3.2. Wash #2, Sodium BiCarbonate Wash (NaHCO3). (x2). Add 50g's of Sodium BiCarbonate to each gallon of Distilled water needed for the wash. Thoroughly shake/homogenize each gallon before adding into the reactor. 50g's per gallon of distilled water. 50g's x 5.28 gallons = 264g's of total NaHCO3 added to distilled water. Add equal parts (1:1) of NaHCO3/Distilled water solution into the reactor.

3.2.1. Turn ON the stirrer to 100rpm's for 5 minutes.

3.2.2. Turn OFF the stirrer and allow the solution to separate for 15 minutes.

3.2.3. After the 15-minute separation time, discard the aqueous/water/NaHCO3 layer at the bottom of the reactor.

3.3. Wash #3, Salt/NaCl Wash (NaCl) (x2). Add 50g's of salt to each gallon of the distilled water needed for the wash. Thoroughly shake/homogenize each gallon before adding into the reactor. Same ratio and weighing procedures as step 3.2. (NaHCO3 Wash).

3.3.1. Turn ON the stirrer to 100rpm's for 5 minutes.

3.3.2. Turn OFF the stirrer and allow the solution to separate for 15 minutes.

3.3.3. After the 15-minute separation time, discard the aqueous/water/NaCl layer at the bottom of the reactor.

3.4. Wash #4, Distilled Water Wash. (x2-4) Add equal parts (1:1) of distilled water by volume of the entire CBD:Heptane solution, into the reactor. Same volume as the previous washes.

3.4.1. Turn ON the stirrer to 100rpm's for 5 minutes.

3.4.2. Turn OFF the stirrer and allow the solution to separate for 30 minutes.

3.4.3. After the 30-minute separation time, discard the aqueous/water layer at the bottom of the reactor.

3.4.3.1. Collect a small amount of the washed Δ8 Crude:Heptane solution from the reactor into a small 100mL beaker.

3.4.3.2. Test the pH of the solution with basic pH test strips. Target pH is 6.5 pH.

3.4.3.3. If the solution is far off from the targeted pH, repeat steps 3.4 - 3.4.3.1. one or two more times to help neutralize the pH.

3.4.3.4. Wash Tips. Purchase all the distilled water from the same supplier to try and ensure that the distilled water is relatively the same pH. Test the pH of the distilled water before the addition of Sodium BiCarbonate or Salt. Since both NaHCO3 and NaCl will increase the pH of the solution, if the Δ8 Crude:Heptane solution remains a highly basic solution (10-13 pH), you can do 50 grams of NaHCO3 per gallon and 25 grams of NaCl per gallon, and 3-5 clean distilled water washes to assist in neutralizing the solution. The more neutral the final solution is… the clearer of a final product you will achieve upon distillation.

3.5. Δ8 Crude:Heptane Collection. Remove and collect all of the Δ8 Crude:Heptane solution from the reactor into food-grade 5 gallon containers for further processing.

3.5.1. If the solution is not going to be directly processed for solvent recovery and distillation, place the air-tight containers in a chemical storage cabinet at room temperature.

4. Further Refinement Needs.

a. The Δ8 Crude:Heptane solution needs further processing in order to create a high potency Δ8 Distillate. Solution needs to be stripped of any residual moisture and solvents via rotary evaporator, and distillation of the crude oil via Short Path or Thin Film Distillation to create a final product. See Roto and Distillation SOP's for these processing procedures.

CONGRATULATIONS, YOU'VE SUCCESSFULLY CREATED HIGH-PURITY Δ8 Crude Oil!

See Flow Chart on next page…

CBD - D8 Crude Conversion (Reactor) & Procedural Flow Chart