THC TO THCA SOP:

Notes: Please read all corresponding information and instructions first! This SOP has virtually the same efficacy using either cbd or thc as a feedstock material for carboxylation.

Equipment needed:

A. Jacketed Glass reactor with auxiliary chiller and vacuum pump
B. Stirred Autoclave high pressure reactor, jacketed with auxiliary chiller and vacuum pump
C. Rotary evaporator with auxiliary chiller and vacuum pump.
D. Filtration apparatus
E. Vacuum oven

Diagram A:

Diagram B:

Diagram C:

Diagram D:

Diagram E:

Reagents needed:

1. Pentane: CAS 109-66-0
2. Acetonitrile: CAS 75-05-08
3. l,8-diazabicyclo[5.4.0]undec-7-ene (DBU): CAS 6674-22-2
4. Pressurized CO2
5. Hydrochloric acid (HCL) CAS: 7647-01-0
6. Magnesium sulfate CAS: 7487-88-9
7. Glacial Acetic Acid CAS: 64-19-7

DBU-THC complex:

1. Inspect your pressure reactor for integrity before application. Reactor should adequately handle, seal, stir and pressurize without leaks before proceeding with the reaction.
2. 400 grams of thc (as pure as possible) is placed inside of a 2.5L borosilicate glass beaker.
3. 1 Liter of acetonitrile is added to the beaker.
4. Homogenize the the cannabinoid/solvent mixture until the cannabinoids have completely dissolved in the solvent.
5. Place your beaker inside of your pressure reactor.
6. Make sure your reactor is ready to be sealed before adding the DBU!
7. Pour 570ml of DBU into the beaker and seal the reactor immediately.
8. Turn on your stirring mechanism for a slight mixture speed. If no sight glass in present in the reaction apparatus then aim for approximately 75 rpms.
9. Using your high pressure C02 cylinder pressurize the reactor to 30 bar.
10. Set aside the reaction apparatus for the next 24 hours.

Metal complex:

1. After 24 hours turn off stirring mechanism and slowly vent your apparatus of carbon dioxide to atmospheric conditions. Preferably under a fume hood using safety precautions well known in the art.
2. Open your reaction apparatus and remove the solution contents directly into your liquid addition funnel equipped on your glass reactor.
3. Transfer the liquid addition funnel to the glass reactionary apparatus.
4. Discharge the solution into the glass reactor.
5. Turn on your chilling pump to 15C and allow the reactor to reach temperature
6. Open the white circular port at the top of the reactor and add 450g of anhydrous magnesium sulfate
7. Turn on the stir motor to approximately 100 rpm
8. Add 600ml of pentane to the reactor whilst stirring. A white precipitate will begin to form.
9. Stir for 15 minutes
10. Add 600ml of distilled water slowly. If temperature inside the reactor exceeds 23C discontinue water addition and allow the reaction to stabilize before continuing.
11. Stir for 15 minutes
12. Discontinue the stirring mechanism and allow the solution to form its separation. There is no time limit here but adequate layers can be observed in the glass apparatus.
13. Decant the yellow organic layer at the top of the solution.

Filtration/Drying:

1. Prepare your filtration apparatus with 2.5 micron filter paper.

2. Discharge your solution over the filter paper to recover your solids.
3. Take ice cold distilled water and rinse slightly, gently rotating the solids with a glass rod or suitable tool to enable an appropriate amount of water to rinse as much surface area of the material as possible.
4. Discharge liquid material into, if necessary, a drying vessel where solvent can be passed through an appropriate desiccant and then recovered in the rotary evaporator. OR because of the relative loss in cost comparison to the final product and taking into consideration time and manpower the solution can be discarded.
5. Recover your precipitate and spread evenly over parchment paper.
6. Place inside of your vacuum oven.
7. Place the oven under full vacuum and dry at 30C for 24 hours. Optionally since the rinsing agent was water this precipitate can be dried at room temperature with adequate air flow for 24 hours if vacuum oven is not available.
8. Place your dried precipitate over a fresh filter paper in a clean filtration apparatus.
9. Mix up a washing solution 44ml of chilled pentane with 1% glacial acetic acid.
10. Lightly rinse your precipitate. Rinsing solution should be chilled to prevent dissolving any precipitate.
11. Repeat step 7 in vacuum oven only. Or suitable solvent safe warming station.
12. Rotovap your rinsed solution.

Acidification:

1. Place your precipitate in your glass reactor and add 1:1 pentane:precipitate
2. Stir your reactor for 15 minutes
3. Slowly add 10% hcl in water solution by weight of cbda used to the reaction.
4. Stir for 30 minutes.
5. Add two liters of distilled water to the reaction solution.
6. Stir for 20 minutes.
7. Discontinue stirring mechanism and allow layers to separate.
8. Discharge aqueous solution.
9. Take organic phase to the rotovap and rotovap under maximum vacuum until approximately 80% of the pentane used is recovered.
10. Place the remaining solution in glass pyrex dishes evenly in your vacuum oven.
11. Place vacuum oven under complete vacuum and leave the pump running. Solution should not be poured into pyrex so full as to have overspill.
12. Heat your oven to 45C
13. Vacuum dry your solution until a purified crystalline structure, solvent free, is observed through the viewing window.

Recover your purified THCA.

THCA can be decarboxylated by warming at 120C for 1.2 hours.

Please follow all appropriate safety guidelines and precautions, use all proper PPE and have an adequate understanding/documentation of all chemicals used, their SDS sheets and proper handling and disposal procedures according your local, federal and state regulations.