Conversion of Delta 9 THC to Delta 10 THC

Materials:

Reagent grade Sulphur was purchased from Sigma Aldrich
YETHAN Copper scouring pads were purchased from Amazon
25 L short path distillation unit with packable distillation head was purchased from Lab Society
>50% Delta 9 crude oil was obtained from conversion of 99.5% CBD ISOLATE
IF NECESSARY INPUT MATERIAL OF RAW CBD ISOLATE CAN BE SUBSTITUTED WITH 1ST PASS DELTA 9 DISTILLATE. RESULTS SLIGHTLY IMPROVED USING THIS FEED STOCK

Method:

1. SPD was thoroughly cleaned, lubricated and placed under vacuum to check for any leaks.
2. SPD was brought back to atmospheric conditions and boiling flask temperature probe was replaced with loading funnel.
3. Sealed beakers with tare weight were filled with D9 oil and weighed
4. Sealed beakers containing Delta 9 crude oil were warmed delicately to 45 C to improve product distribution and inhibit degradation.
5. PTFE magnetic stir bar was centered in SPD boiling flask, and set to 300 RPM
6. 15 L of crude oil was added to SPD boiling flask
7. Copper scouring pads were separated into finer strand lengths and packed to a medium degree inside of distillation head
8. Leibig condenser heating/chilling unit was set to 50 C and circulated
9. Cold Trap chilling unit was set to -15 C
10. 15% by weight Reagent Grade Sulphur was added to SPD boiling flask, slowly allowing for proper homogeneity
11. Stir bar was increased to 400 RPM
12. Loading funnel was removed and replaced with temperature probe ensuring probe did not make contact with magnetic stir bar and was centered in the mixture.
13. Boiling flask temperature was set to 65 C
14. Boiling flask temperature was ramped up by 10 C every time parameter was reached until 100C was obtained.
15. Vacuum regulator was securely closed.
16. Vacuum pump was turned on.
17. Vacuum regulator was slowly opened gauging reaction until regulator was all the way open.
18. Timer was set for 60 minutes.
19. After timer conclusion boiling flask temperature was ramped up by 10C every time parameter was reached until 185 C was obtained.
20. Vapor temperature was closely monitored.
21. At approximately 145 C heads fraction began distribution into receiving flask

22. Heads fraction consistency was monitored until stability improved and thin strands of oil began to coil at the bottom of receiving flask.
23. Vacuum was isolated via vacuum isolation valve at boiling flask.
24. Receiving flask was brought back to atmospheric condition and replaced with fresh receiving flask
25. Vacuum Isolation valve was slowly opened and reaction was allowed to continue.
26. Vapor temperature was approximately 160 C
27. Oil was collected in receiving flask until conclusive evidence of drastic consistency change inside of liebig condenser was observed.
28. Vacuum was isolated at boiling flask via vacuum isolation valve and the receiving flask was brought back to atmospheric conditions and collected.
29. Receiving flask was turned upside down over graduated beaker inside of warming oven at 45 C
30. Receiving flask was replaced with a fresh flask and vacuum isolation valve was slowly opened.
31. Reaction was allowed to continue.
32. To improve process flow liebig condenser heating/chilling unit was ramped up to 60 C
33. Stir bar was increased to 500 rpm
34. Tails fraction was collected inside of receiving flask until flow at condenser had slowed to almost non existent.
35. Stir bar was stopped
36. Boiling flask temp was stopped
37. Vacuum was stopped
38. SPD was brought back to atmospheric conditions and tails fraction was collected and labeled.
39. SPD was worked up to room temperature and cleaned according to laboratory procedure.
40. Main body fraction was removed from warming chamber and redissolved in a 2:1 ethanol ratio. Ethanol:Distillate
41. Copper scouring pads were placed inside of Ethanol:distillate mixture to aid in absorption of residual sulphur.
42. Mixture was monitored and pads were replaced every 2 hours until no discoloration was observed in scouring pad.
43. Mixture was rotovapped at 70C heating bath and -10 C condensing coil
44. Ethanol was recovered from mixture until drips had slowed to almost non existent on condensing coil and oil began to climb the walls of rotary orb.
45. Mixture was removed from rotary evaporator and lightly decarboxylated at 120C for 30 minutes using magnetic hot plate and stir bar.
46. Sample was sent for testing.
47. SOP was repeated until 15L of converted oil was obtained from main body
48. Freshly converted Delta 10/CBN/Delta8 oil was then loaded into SPD boiling flask and distilled according to normal procedures
49. Sample of main body/heads/tails fractions were sent for testing.

50. Delta 10 oil was placed in a vacuum beaker, blanketed with nitrogen and stored in a dark cool location for shelf life testing purposes.
51. After 3 weeks crystal formulation was noticed.
52. After 6 weeks extreme crystallization was noted.
53. Introduction of 5% organic terpenes and propylene glycol was noted to make a suitable vaporizable solution.
54. After vapor cartridges were stored for 12 weeks leakage and slight crystallization was noted but further research needed to provide conclusive evidence that crystallization or cartridge failure or a combination of both were contributing factors.

Troubleshooting:

IT WAS NOTED OVER THE PROCESS OF MULTIPLE BATCHES ACROSS DIFFERENT EQUIPMENT SETUPS THAT RESULTS VARIED.
CONTRIBUTING VARIABLES ARE AS FOLLOWS.

1. IF: Delta 10 concentrations are not over 40% THEN: feedstock could be of poor purity, Sulphur was not monitored upon addition and stir bar was suffocated, vacuum leak.
2. IF: CBN concentrations are too high. THEN: Sulphur can be decreased by 5%. Reaction exposed to heat for too long, decrease timer by 15 minutes until results are satisfactory. Boiling Mantle coil malfunction. Temperature probe not centered in boiling flask
3. IF: Vacuum will not hold. THEN: check all fittings/connectors for integrity and proper amount of grease. Leak is present.
4. IF: Oil has a strange color in receiving flask. THEN: repack distillation head properly.
5. IF: oil is not moving. THEN: ensure boiling flask is heating properly, if unsure use vapor temperature as reference point and lightly disregard mantle temp. Increase stir bar by 100 RPM. Decarboxylate properly.
6. IF: when cleaning boiling flask you notice burned cake like substance in bottom of flask. THEN: make sure sulphur was added and mixed properly and stir bar was functioning.

Safety:

Proper PPE and Safe Laboratory Practices should be adhered to at all times. Adequate ventilation should be provided. Techs should wear goggles, lab coat, face shield, gloves and non-slip shoes at all times. When the addition of sulphur is taking place an organic respirator can be worn for additional safety precaution.