NEW LEVEL
PROCESS SOLUTIONS

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1. Purpose

1.1. To outline the startup procedure, standard operation, troubleshooting, and shut down procedure of the KDL-6,10 and 30 Short path distillation units.

2. Scope/Objective

2.1. To provide operators with a reference manual for the safe operation of the distillation units used by [Client], Inc. personnel.

2.2. The general process used for raw CBD/THC distillate production begins with decarboxylation, followed by winterization, filtration, solvent recovery, and distillation. The overall scheme may be changed due to ethanol extraction preceding distillation (in house extraction), product inconsistency, due to planned deviations, or at manager's discretion.

2.3. Any unintended changes to the process require a deviation and/or a CAPA report to be included in the batch record.

2.4. If in house extraction is being performed, thorough decarboxylation is performed prior to distillation.

3. Related Documents

3.1. Following SOPs: Julabo Heater Chillers, Mokon HTF heater chiller, Leybold Rotary Vane vacuum pumps, Leybold Roots Blower vacuum pump, Leybold Turbo i/ix vacuum pumps, Edwards Diffusion pumps, Ohaus Ranger 3000 scale, AI Stabletemp ovens, Binder explosion proof C1D1 ovens.

4. Responsibilities & Accountabilities

4.1. Distillation technicians are responsible for the proper usage of the information in this SOP. Any Changes to the process require a deviation and/or a CAPA report to be included in the batch record.

5. Safety Equipment/PPE:

5.1. Standard PPE includes – hair net and beard net (if applicable), lab shoes, gloves, lab coat or overalls, and safety glasses

5.2. Additional PPE for specific process – face shield, thermal safety gloves (hot).

6. Start Up Procedure

6.1. The KDL-6, KDL-10, and KDL-30 Short path distillation units require high temperatures and deep vacuums, and these systems take time to reach to desired values - keep this in mind during process set up. Always turn the chiller on as soon as possible to start the vacuum. **Never start the rotary vane vacuum pump without the chiller ≤ - 1°C.**

6.2. Put crude product load pots in the oven at a reasonable temperature to liquify. Do not rush this process, higher temperatures will oxidize the product and reduce yield! Generally, 80 - 100 ° C for S processes and 85 ° C and lower for P processes.

6.3. Refer to **Table 1** and **Table 2** below to set the temperatures on each heat recirculatory baths for the desired process. **Note:** During start up, it is best to set the residue and evaporator temperatures to 100 °C so that the operator allows the rollers to warm up to avoid chipping of rollers upon startup. It is best to wait for the baths to reach temperature as prolonged exposure to heat in one spot can begin to warp the roller blades. After 100 °C has been reached, wipers may be turned on an normal temperatures may be set.

6.4. After setting the temperatures on the heating units on the KDL unit of the desired process, obtain clean product pots (vacuum jars if using the KDL-6) or final product bottles, record weights, and place jars on top of the lab cart or jack stand under each of the residue and distillate outlets. If final product is being distilled, a scale can be placed on top of one of the laboratory jacks, then balanced, calibration checked and tared for determination of product weights.

Table 1: Process Parameters for standard distillation of THC

System	**S1**	**P1**	**P2**
Feed	95 °C		
Evaporator	175-180 °C	155 - 160 °C*	145 - 150 °C
Residue	140 °C		160 °C
Cold Trap	-50 °C or as cold as possible		
Condenser	45 °C	95 °C	95 °C
Pressure	$8.0e^{-1}$ – $9.0e^{-1}$ Torr	$1.0e^{-2}$ – $1.0\ e^{-1}$ Torr	$1.0e^{-2}$ – $1.0e^{-4}$ Torr

*may be set higher based on color and vacuum behavior

Table 2: Process Parameters for standard distillation of CBD

System	S1	P1	P2
Feed	95 °C		
Evaporator	170-180 °C	160 °C	145 - 150 °C
Residue	140 °C		160 °C
Cold Trap	-50 °C or as cold as possible		
Condenser	45 °C	95 °C	95 °C
Pressure	$8.0e^{-1}$ – $9.0e^{-1}$ Torr	$1.0e^{-2}$ – $1.0\ e^{-1}$ Torr	$1.0e^{-2}$ – $1.0e^{-4}$ Torr

6.5. Ensure that the cold trap light molecular weight waste bleed valve outlet is closed with the red K-clamp securing the flask.
6.6. Ensure the feed is plugged with the rubber tipped wand, the vacuum control knob is fully open, and that temperatures are at or very near to the running parameters. Open the air ballast prior to powering the rotary vane vacuum pump to extend oil life while pulling down, operator may choose to leave it on for the duration of startup if oil is cold, but it should be turned off prior to beginning processing.
6.7. With all vacuum gates open for desired process (turbo vs. standard path), turn the power on for the primary vacuum, the rotary vane pump. If using the KDL-6, it is important to ensure the vacuum flasks are making an even and flush contact with the distillation unit outlets as they will suck on to the unit for the duration of the run and a misalignment could cause time loss due to heating and cooling of diffusion pump/turbo.
6.8. After turning the primary vacuum pump on, the pressure should begin to drop from the display pressure of 750 Torr or ~7.68 e^2 Torr. As the pressure drops, the operator should slowly but steadily close the black Leybold vacuum adjustment valve until it is all the way closed. If the process uses a turbo pump or diffusion pump, close the white collar to ensure that no air gets into the system even if the black knob is touched. **Do not over tighten the valve or its collar.**
6.9. Allow the primary vacuum pump to drop to its lowest pressure. Record this pressure on the vacuum pump use log. Close the air ballast.
6.10. Depending on the process, bleed the pressure to the appropriate value for the selected process using the Leybold vacuum bleed knob or keep the unit sealed. This is done by adjusting the black knob counterclockwise until the display reads the appropriate value.
6.11. If using the KDL-30, at 10.0 to 1.00 Torr the roots blower system may be turned on to assist with vacuum stability.
6.12. If all seals are properly made, the system will continue to drop to $2.00e^{-1}$ Torr or lower without bleeding the vacuum via the bleed knob.

6.13. For diffusion pumps, the system pressure must be below 4.9 e-1 Torr and for turbo pumps, the pressure should be below 3.9 e-1 Torr to be properly operated. To activate the diffusion pump – switch the toggle switch to the bottom position "fan + heater". To activate the turbo pump, either turn the ignition key switch or the turnkey switch on the electronic cabinet interface labelled "turbo pump." **Note**: some pump suppliers say it is allowable to start pumps up as soon as 10 Torr. We have seen that if the pressures mentioned in 5.13 are not met at the beginning of the run, the vacuum will not reach its ultimate low during the processing portion of the run which makes it hard to determine if the product is causing the pressure issue or if it is the system.

7. Distillation

7.1. Put a pair of thermal gloves on.

7.2. Obtain the liquified starting material from the oven. Record the starting weight using the Ohaus 3000 bulk balance.

7.3. Filter load through one of the fine wire filter screens to capture any debris that could damage the feed on the unit. This can be done from steel pot to steel pot or from steel pot to the distillation unit feed. Ensure product is hot enough to pass through filter screen, this process should be done at or near 85 °C or greater.

7.4. Carefully and slowly, pour the process load stock pot into the distillation unit feed tank.

7.5. Record the weight of the pour and return the pot or jar to the oven where it was queued for processing. **Record the weight of all subsequent additions on the batch record.**

7.6. The feed should only be loaded while in use to protect the product and its impurities from degradation.

7.7. After loading the unit, unplug the inlet (KD-6,10) or open the feed valve (KD-30).

7.8. Begin the feed drive pump by turning the ignition key on the interface of the unit (all units).

7.9. Set the feed rate to a process appropriate feed rate.

KD-6: Depending on the generation of KD-6, the feed may be set to 110 rpm for a strip process and 80 – 110 for a polish process, or using a gearbox 30 rpm for strip and 25 rpm for polish. The KD-6 is capable of stripping approximately 2 liters of material per hour, and polishing between 0.7 L and 1.1 L per hour.

KD-10: For a strip, 10 – 12 Hz may be used depending on the amount of terpene waste. For a crude to polish, 8 – 9.5 Hz may be used, the product will come from the system at approximately 1.45 liters per hour. For a polished product going into a second polish, the speed may be determined by the vacuum level, at 10 Hz the unit will produce roughly 2 liters of final material per hour.

KD-30: For both strip and polish, run the system as slow as it will allow without giving a f13 or f12 error. Unit should strip up to 15 L per hour and distill between 6 and 9 liters an hour.

7.10. Record the process starting vacuum in the second vacuum field.

7.11. Adjust the vacuum as needed, stopping the feed when the vacuum falls out of range for the desired process and troubleshooting error.

7.12. When stripping material of lights and terpenes, the product is the residue and the waste is the distillate. It is normal to see a loss of about 10 - 20% during a lights strip on cannabis. Terpenes will be much lower in commercial hemp.

7.13. When distilling material, the product is the distillate and the waste is the residue. Distillation is not as efficient as stripping, so slower speeds and re-runs may be necessary to get a proper yield if the first pass is not performing well.

7.14. The first 10 minutes of all distillations are the most crucial points in the distillation. During this point operators must ensure product is flowing out of both streams and waste products are being formed according to the process and in correct ratios.

7.15. It is normal for the formation of waste to start late. If waste has not formed by 15 minutes, the operator should troubleshoot the instrument for potential clogs in the feed or in the discharge systems.

7.16. If waste and product are forming properly, the operator may now begin priming the residue and distillate pumps (KD-10,30). The pump that is forming more product should be primed first.

7.17. Obtain a heating gun, a small waste transfer jar, a clean product pot, and ask for help from a second operator. Both operators must don full face splash masks incase a clog is pushed through – extremely hot waste can be discharged in any direction in this case. One operator should stand next to the electronics interface and turn on/off the feed pump power switch and adjust the rate as needed. The other operator should place a finger on the outlet of the selected product outlet, use the heating gun to liquify the contents of the steel tube, put the heating gun down, and then slowly open the red handled valve while observing the pressure changes on the fingertip placed against the tube outlet. At first the operator should notice a pressure increase on the display, feel a bit of suction on the gloved fingertip, and see the product in the glass sight begin to jump. **Go slow enough to not allow the pressure change to fracture the glass sight. This can be dangerous if an implosion happens.** The pump may now run for the duration of the distillation so long as there remains a constant feed to “wet” the Witte pump. Never run the Witte pump dry for any extended period. To stop the feed, simply close the red handle and turn the feed pump to the off position.

7.18. Operators **must** pay attention to feed levels, especially while turbo fans are being ran. The feed must continually be replenished to ensure no atmosphere gets into the system. This can cause burning of in process product and damage to vacuum parts. During the final few minutes of any run, the operator should watch the distillate flow into the feed port and plug the feed following the last of the load. A spatula may be used to push the entire contents of the feed into the feed hole.

7.19. After 2-5 seconds, the feed may be turned off at the system interface.

7.20. If a diffusion or turbo pump was running for the process, it is wise to turn it off early so that there is plenty of time to cool unit down as these take 15 minutes or more to cool down. To turn a turbomolecular pump off, ensure the evaporator main body is clear of product, the condenser coil has completed clearing itself of distillate, and the system is ready to be shut down. Gate off the turbo pump completely. The vacuum gauge may begin to rise, this is normal. Turn the turbo off at the interface of the cabinet. The turbo will blink for 30 to 45 minutes, do not turn the rotary vane or roots vane pumps off during this time or damage can occur to the turbo fans. For diffusion pumps, simply turn the switch from “Fan+heater” to “fan”. Once the diffusion pump cools enough to touch again, it is okay to bleed the system and turn the rotary vane pump off.

8. Shut Down Procedure

8.1. Turn evaporator and residue baths to 100 ° C.

8.2. Allow system to cool while turbo/diffusion pump is cooling.

8.3. Once diffusion/turbo pump is cooled (determined by heat sensor on diffusion pump and green light to stop blinking on the turbo), bleed the vacuum slightly until it begins to climb upward toward 750 torr. After an **increase** in pressure is observed, turn the roots vane pump off (KD-30 only), and the rotary vane pump off.

8.4. Turn the knob so that the system bleeds slow but at a steady rate of about 5-10 torr per second. This should continue to 750 torr, or approximate room pressure.

8.5. Close the vacuum pathway gate on the system, if applicable.

8.6. After vacuums have been powered down, cold trap and condenser baths may be turned off at the system interface.

8.7. When 100 ° C has been reached on the heating baths, they may be turned off.

8.8. After Julabo heating baths/Mokon HTF Heating recirculators are turned off, the wiper blades may be turned off.

8.9. The unit is now completely shut down. The main knob should be turned off, and the system should shut off at the breaker panel.

8.10. Register the time on the unit, lot number and process done.

9. Emergency Protocol

9.1. Nearest emergency medical center is at 3700 E South St, Lakewood, CA 90805

9.2. In case of any emergency call 911 immediately. Notify supervisors if not already notified.

9.3. In case of any fire, call 911 immediately and request fire department assistance. Refer to Fire Safety SOP for fire safety instructions.

9.4. In case of large heating bath oil spill, use spill kit to contain spill. Spill kit should include spill bats which are long tube-shaped sponges that contain spills and stops the spread of the oil. Shovel cat litter or sand over the oil and scoop into temporary waste transport container such as a bucket. Take bucket to the solid waste storage container to dispose of oil contaminated sand or cat litter. Wipe floor clean of oil using spill rags. Report spill to supervisors.

10. Planned Maintenance

10.1. Wiper blades are to be changed out and balanced at least once every year.
10.2. Quarterly major clean out according to CTS cleaning SOP.
10.3. Yearly gasket rebuilds on entire short path.
10.4. Change Leybold rotary vane vacuum pump oil bi-weekly or as needed according to use.

11. Distribution

11.1. This SOP is to be distributed to manufacturing and quality assurance personnel.

12. Health, Safety and Environmental

12.1. Always wear a laboratory frock or coat, gloves, hair nets, shoe covers, safety goggles, ear plugs, face masks where applicable, face shields where applicable, and heat resistant gloves where applicable.
12.2. All Chemical waste and by-products must be segregated appropriately, and by-product waste must include date of production and lot number produced from.
12.3. If an emergency or injury occurs, call 911 to report immediately.
12.4. Visitors may not enter manufacturing C1D2 area without proper orientation or prior authorization.