**Section I** (version 5.1.19b)

**Starting a new BATCH**

Process Summary

When work begins on a new BATCH a folder should be started and labeled with the super sack number. This folder will then follow the material from that super sack as it moves through the stages of our process. All FORMS related to the processing of that super sack should be kept in this one folder with the FORM for the most recent action on top of the previous action. On top of all the FORMS in the folder is the Process TOTALS FORM. This FORM tells the technicians the amount of product created during the previous process.

**Procedure**:

1. Use the Process Schedule on the large white board near the door to identify the next super sack to be processed
2. Locate the super sack in building C
3. Using the BATCH Generation FORM record the super sack name, total weight, and make note of all physical characteristics
4. Assign a BATCH number by adding the date in reverse with no space or dash to the end of the super sack name using the format below:

BATCH#: ABC001**190204**

1. Start a new folder with the tab labeled with the super sack name **(no date)**
2. Add the BATCH Gen FORM you filled out at Step 3 above to the folder
3. Fill out the heading of the Process TOTALS FORM
4. Add the BATCH TOTAL to the first line of the Process TOTALS FORM
5. Place the Process TOTALS FORM in the BATCH folder
6. Move the folder to the Extraction table near the Falling Film Evaporator (FFE)

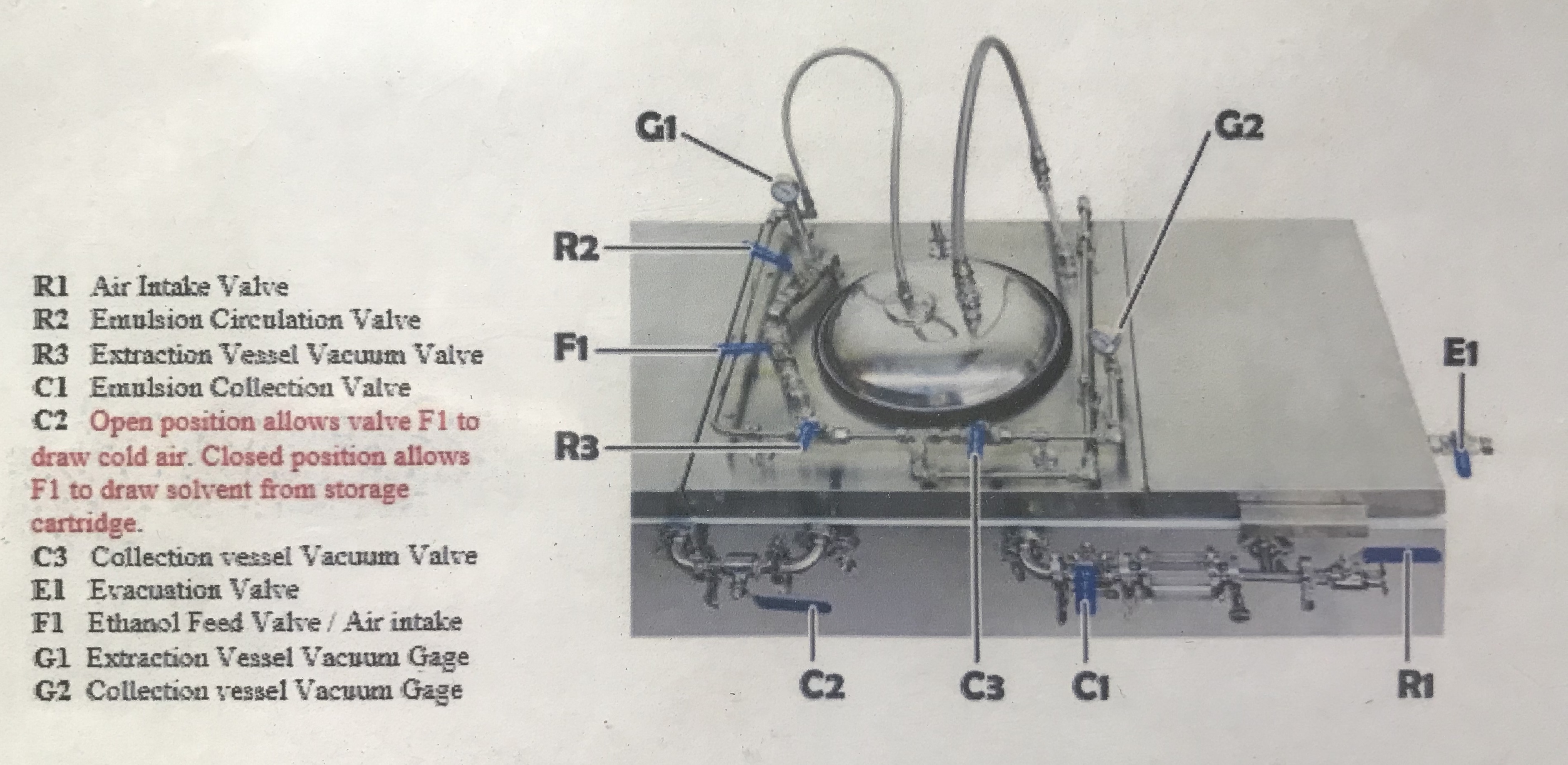
**Section II** (ver.5.1.19b)

**Extraction**

ProcessSummary

This process is the first step in separating our target compound(s) from the biomass. Here we use sub-zero ethanol to extract the crude oil from the dried biomass. By using ethanol between -60˚C and -40˚C we avoid extracting a large amount of the waxes present, which allows us to skip the time-consuming process of removing these waxes later via freeze precipitation, sometimes called “winterizing.”

**(Fig. 2.1)**



**Procedure**:

I. Flooding Procedure

1. Load 2.3Kg of biomass into the nylon extraction bag
2. Place the loaded bag into the extraction vessel and secure the nylon bag with the stainless steal pressure ring
3. Close the extraction vessel lid
4. Close all wet plumbing by closing valves F1, C1, C2, R1, R2, E1
5. Open valve R3 to engage the vacuum in the extraction vessel. De-compress vacuum gauge G1 to -10inHg
6. Open valve F1. **(This action will saturate the biomass with solvent (ethanol) transferred from the holding cartridge into the extraction vessel)**

**NOTE: The flow rate at -10inHg vacuum pressure is typically 1gal/5sec**

1. Open valve C2 to stop the flow of solvent **(ethanol)**

**NOTE: Valves C2 and F1 should remain open during the extract solution collection**

1. Close valve R3 to disengage the vacuum in the extraction vessel

II. Collection Procedure

**NOTE: Valves C2 and F1 MUST be open prior to starting this procedure**

1. Open valve C3. De-compress the vacuum gauge G2 down to -25inHg
2. Close valve C3
3. Open valve C1 to engage the transfer of extract solution from the extraction vessel into the collection vessel
4. Once the solution has been transferred, close valve C1
5. Prepare for the recirculation procedure by closing valve F1
6. Prepare for the recirculation procedure by opening valve R1

III. Recirculation Procedure

**NOTE: Valve F1 MUST be CLOSED prior to this procedure**

**and**

**NOTE: Valve R1 MUST be OPEN prior to this procedure**

1. Open valve R3 to de-compress vacuum gauge G1 to -10inHg
2. Open valve R2 to engage transfer of the extract solution into the extraction vessel
3. When transfer is complete, close valve R2
4. Allow gauge G2 to de-compress to 0inHg
5. Close valve R1
6. Open valve C3. De-compress the vacuum gauge G2 down to -25inHg
7. Close valve C3
8. Open valve C1 to engage the transfer of extract solution from the extraction vessel into the collection vessel
9. Once the solution has been transferred, close valve C1
10. Prepare for the recirculation procedure by closing valve F1
11. Prepare for the recirculation procedure by opening valve R1
12. Repeat this process at least five times to maximize yield efficiency

IV. Draining Procedure

1. De-compress vacuum gauge G2 to -25inHg
2. OPEN valve C1
3. Repeat these steps up to twelve times

V. Evacuation Procedure

1. OPEN valves C1, C2, and F1 in preparation for evacuation
2. De-compress the evacuation tank
3. OPEN valve E1 and allow the extract solution to evacuate into the collection vessel
4. Transfer the extract solution from the collection vessel to a storage vessel **(5gal bucket)**
5. Seal the storage vessel **(5gal bucket)** with a lid
6. Label the lid using the following format:

BATCH#: **ABC001190430**

DATE: **5/22/19**

TECH: **MJM**

1. Once the entire BATCH has been extracted, record the total number of storage vessels **(5gal buckets)** associated with the BATCH on the Process TOTALS FORM

VI. Removal of the Spent Biomass

1. Open the extraction vessel lid
2. Remove the stainless steel pressure ring holding the nylon extraction bag in place
3. Remove the nylon extraction bag containing the spent biomass and place it in the transfer vessel in preparation for centrifugal removal **(PANDA)** of the retained extract solution

VII. Centrifugal Removal (PANDA) of Retained Extract Solution

1. Load a single nylon extraction bag of previously extracted biomass into the PANDA **(If the PANDA struggles to spin, reduce the load by splitting the biomass between the two PANDAs)**

**NOTE: It is sometimes simpler to place a fresh nylon bag in the PANDA, and then transfer the biomass from the extraction bag into this fresh bag**

1. Fit the retaining gasket into place
2. Place a collection vessel **(5gal bucket)** at the effluent opening found near the bottom of the PANDA
3. Start the PANDA
4. Continue until the PANDA sounds the Complete Signal **(Usually 8-10 minutes)**
5. Remove the now filled collection vessel **(5gal bucket)** and seal with a lid in preparation for filtration
6. Label the lid using the following format:

BATCH#: **ABC001190223-P**

DATE: **05/12/19**

TIME: **17:12**

TECH: **MJM**

1. Remove the nylon extraction bag from the PANDA
2. Dump the spent biomass into the waste vessel **(garbage bag**) in preparation for moving it into the dumpster
3. Once all material from the BATCH has been processed through the PANDA, record the number of PANDA vessels **(5gal buckets)** associated with the BATCH on the Process TOTALS FORM

**Section III** (ver. 5.30.19)

**Filtration**

ProcessSummary

This process removes chlorophyll and other pigments and water contamination, as well as a small percentage of any ∆9-THC present. Here we pass the extraction solution through a Buchner-style vacuum filtration apparatus using a media pad composed of layers of Celite 545, hardwood activated carbon, and MagnaSol. The black-green extract solution passes through the media pad, which adsorbs the pigments and water, and a yellow-to-red extract solution is collected.

This Section assumes that ALL extract solutions and materials from the PANDA are to be filtered. For reasons concerning time-management and/or equipment limitations, sometimes only the PANDA material, or rarely none of the material is filtered. Confirm with your supervisor whether ALL material, just PANDA material, or no material is to be filtered.

**Procedure:**

I. Preparing the Filtration Media Pad

1. Carefully remove the spent media pad(s) from the stainless steel filtration funnel(s)
2. Clean the funnel(s) and rinse the receiving flask(s) of any media
3. Confirm the diaphragm vacuum pump is attached to the receiving flask and that ALL valves are CLOSED
4. Turn the vacuum pump ON
5. Confirm the funnel is under vacuum **(The funnel MUST be under vacuum during this entire process)**
6. Lay down a new trimmed filter paper
7. Wet the entire filter paper with ethanol
8. Using a stainless steel spoon or scooper, carefully begin adding approximately 2000g of Celite 545, starting from the center of the filter paper

**CAUTION: Celite 545 contains silicated diatoms, which are dangerous to breath. Make sure other techs and yourself use proper protection equipment**

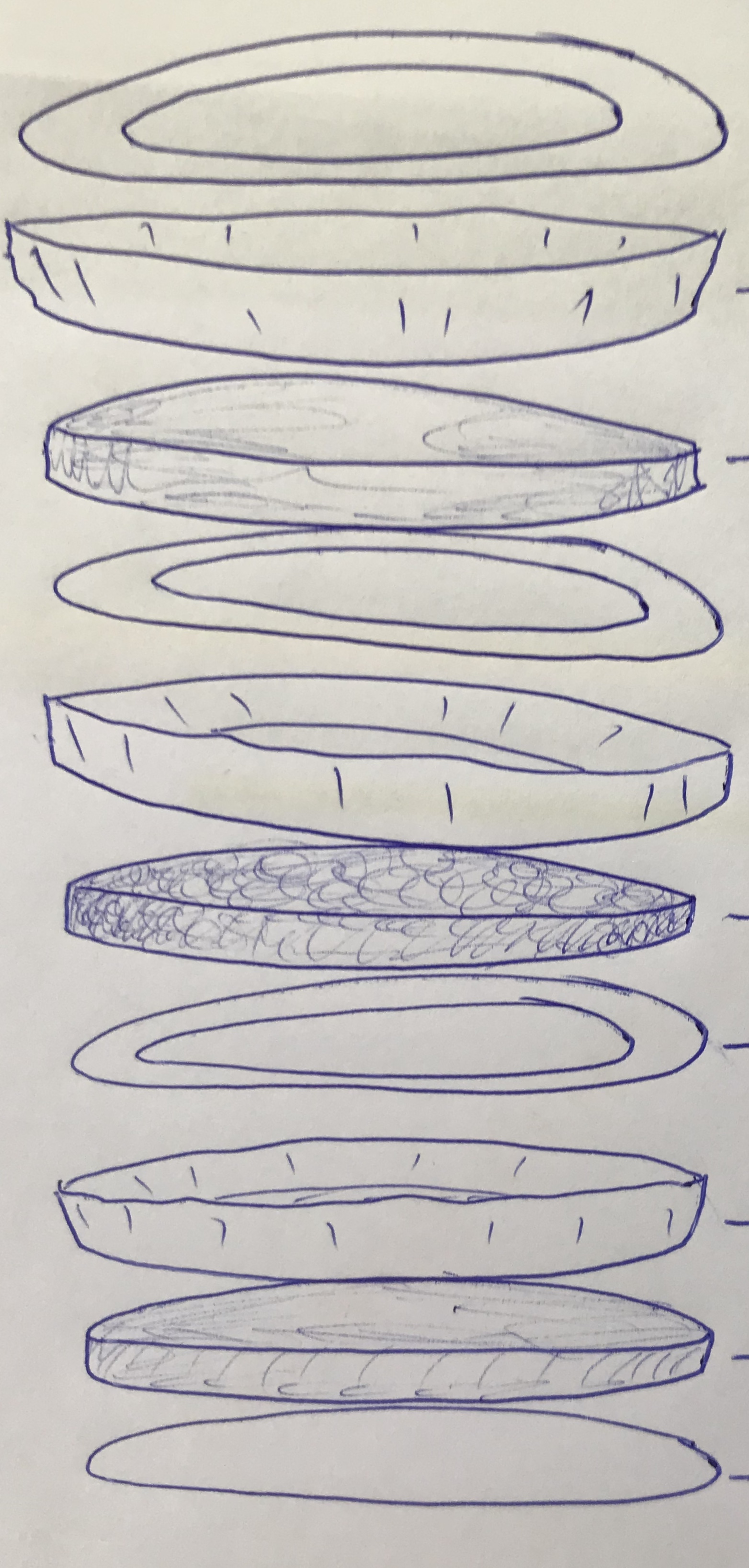
1. Use the bottom of a clean 100mL beaker to pack the Celite 545 tightly, creating a solid pad with no pitting or irregular areas
2. Gently pour clean ethanol onto the Celite 545 layer, being sure that you wet the entire pad thoroughly **(Be sure you do not allow the ethanol to dig a hole in the Celite layer, consider use a stainless steel spoon as a shield as you pour to disperse the ethanol stream)**
3. Lay down a new untrimmed filter paper and place the stainless steel retaining ring in place
4. Carefully begin adding approximately 2000g of hardwood activated carbon, starting from the center of the filter paper

**CAUTION: Activated Carbon is dangerous to breath. Be sure that other technicians and yourself use proper protection equipment**

**NOTE: A slurry of ethanol and hardwood activated carbon can be made in order to reduce possible exposure, (This can help reduce the risk of inhaling the carbon dust) this slurry can then be added to the filter paper above the Celite 545**

**NOTE: It is preferable to use a non-powdered, hardwood activated carbon, as extreme caution must be taken to avoid breathing in the black carbon dust**

**(Fig. 3.1 Exploded view of media filter pad)**

****

**SS retaining ring**

**Untrimmed filter paper**

**MagnaSol**

**SS retaining ring**

**Untrimmed filter paper**

**Hardwood activated carbon**

**SS retaining ring**

**Untrimmed filter paper**

**Celite 545**

**Trimmed filter paper**

1. Gently pour ethanol onto the activated carbon pad, being sure you wet the entire pad thoroughly **(Be sure you do not allow the ethanol to dig a hole in the activated carbon layer, consider use a stainless steel spoon as a shield to disperse the ethanol stream as you pour)**
2. Lay down another new filter paper and place the stainless steel retaining ring in place
3. Carefully begin adding approximately 2000g of MagnaSol, starting from the center of the filter paper

**CAUTION: MagnaSol is dangerous to breath. Be sure that other technicians and yourself use proper protection equipment**

1. Gently pour ethanol onto the MagnaSol pad, being sure you wet the entire pad thoroughly **(Be sure you do not allow the ethanol to dig a hole in the MagnaSol layer, consider use a stainless steel spoon as a shield to disperse the ethanol stream as you pour)**
2. Lay down another filter paper centered so that a wall of paper encircles the side of the funnel, and add the stainless steel retaining ring
3. Set up a feed bucket on top of the funnel
4. Pass approximately 5 gallons of clean ethanol over the newly prepared media pad **(Be sure the entire pad is saturated, leaving no dry spots, cracks, or pitting)**
5. Drain the receiving flask of ethanol and rinse out any media that may have passed through during the preparation of the media pad

II. Filtering the Ethanol Extract

1. Confirm the number of buckets using the Process TOTALS FORM located in the BATCH folder
2. Locate all the extract solution buckets associated with your BATCH# **(Use the Process TOTALS FORM to confirm the number of buckets associated with you BATCH)**

**NOTE: Do not forget about the material reclaimed from the Panda centrifuges**

1. Confirm your feed bucket(s) valve(s) are closed
2. Place the feed bucket(s) on the grating cover above the funnel
3. Fill the feed bucket with your first bucket of extract solution
4. OPEN the feed valve
5. Adjust the feed rate so that there is no danger of filling the funnel higher than the paper side walls **(Confirm this often, consider setting a timer)**

**NOTE: DO NOT ALLOW THE EXTRACT SOLUTION TO RISE ABOVE THE FILTER PAPER WALLS**

1. Continue to refill the feed bucket(s), being sure to monitor the level of extract solution so as not to rise above the filter paper walls
2. Once the receiving flask is ¾ full, stop the feed bucket valve
3. Allow the extract solution remaining in the funnel to drain into the receiving flask
4. Once all the extract solution has been pulled through the media pad and into the receiving flask, vent the receiving flask by opening the pump line valve
5. Now turn the vacuum pump OFF
6. Leave the pump line valve OPEN
7. Place a collection vessel **(3gal bucket)** below the drain valve of the funnel’s receiving flask
8. OPEN the receiving flask’s drain valve and empty the filtered extract solution into the collection vessel **(3gal bucket)**
9. Transfer the filtered extract solution into a storage vessel **(5gal bucket)**
10. Label the bucket with the BATCH# followed by “**-F**” for filtered (ABC001190321**-F**) to distinguish it from the unfiltered extract solution still awaiting filtration
11. Continue filtration of all buckets of extract solution for the entire BATCH

**Section IV** (ver. 5.1.19b)

**Drying**

ProcessSummary

This process separates the ethanol used during extraction from the crude extract via a Falling Film Evaporator (FFE). The extract solution is sprayed from a nozzle down through a series of hot-water heated cylinders under vacuum during which time the extract falls to the bottom of the collection pot area, and the vaporized ethanol is pulled up through a second cylinder and over into a condenser via vacuum pressure. At this point it falls out as a liquid, which is moved via vacuum action into the ethanol-recovery holding tank.

**Procedure:**

1. Transfer the buckets of filtered extract solution to the 55gal feed drum, being sure to record the volumes added on the Drying FORM
2. Confirm that the Chiller is ON and running at 29˚F-32˚F
3. Confirm that the Water heater is ON, set to 185˚F and that the current temperature reads at least 175˚F
4. Adjust the feed valve on the FFE so that a fine mist-spray is achieved and record this as the START TIME on the Drying FORM
5. Continue to monitor the feed, occasionally adjusting the feed valve to maintain a fine mist-spray throughout the entire process.
6. Continue adding extract solution to the feed barrel until all vessels from your BATCH have been added. **(Be sure to record the additional volumes of extraction solution being added to the feed barrel on the Drying FORM)**
7. Continue to monitor the feed, maintaining a fine mist-spray until the feed barrel is empty **(Be extra mindful of the feed barrel as you near the end of a run, as it will begin to introduce air into the feed line causing the nozzle to drip instead of spray, thereby reducing the effectiveness of the separation)**
8. Record the time the process is complete as the END TIME on the Drying FORM and list any problems or unusual circumstances in the COMMENTS section

**Section V** (ver. 5.28.19)

**De-gassing**

ProcessSummary

This procedure is designed to remove the majority of any ethanol left behind after the extract solution is dried using the FFE. Ideally, the FFE is run effectively enough that this step is not required, or alternately, requires the least amount of time to de-gas. Here we place the dried crude extract from the FFE into a De-gassing Pot on a hotplate with overhead stirring and boil the ethanol out. This de-gassing procedure will also decarboxylate crude, although not necessarily to 100% completion.

**Procedure**:

1. Record the TARE weight of your clean de-gassing pot(s) on the De-gassing FORM
2. Open the Bleed Valve on top of the second cylinder of the FFE
3. Open the Crude Drain Valve leading to the collection pot using the incoming pressure from the Bleed Valve to push the extract into the collection pot
4. Once all the extract has been flushed into the collection pot, close the Crude Drain Valve
5. Remove the collection pot from the FFE
6. Close the Bleed Valve on top of the second cylinder
7. Thoroughly wipe down the collection pot and remove any water/moisture

**NOTE: WATER CONTAMINTAION IS TO BE AVOIDED AT ALL TIMES**

1. Transfer the crude from the FFE collection pot into the de-gassing pot(s), being mindful that no water is introduced during the transfer
2. Record the Gross weight of the de-gassing pot(s) containing the crude on the De-gassing FORM
3. Calculate the NET weight by subtracting the TARE weight of the de-gassing pot(s) from the Gross weight of the filled de-gassing pot(s)
4. Label the de-gassing pot(s) using the following format:

BATCH# **ABC001190223**

DATE IN: **05/12/19**

TIME IN: **17:12**

TARE Wt.**: 1427g**

Gross Wt.**: 3934g**

Net Wt. IN: **2507g**

TECH: **MJM**

1. Copy the NET weight IN on the De-gassing FORM
2. Place the de-gassing pot(s) on the hotplate(s)
3. Adjust the overhead stirrer(s) into position
4. Confirm that the overhead stirrer(s) are turned down to the lowest setting
5. Turn the overhead stirrer(s) ON
6. Adjust the speed to about a quarter speed on the control dial, until strong mixing occurs, but not so fast it splatters
7. Adjust the hotplate temperature(s) so that the crude reaches 125˚C
8. Use the temp laser gun to read the crude temperature(s) **(Do not allow the crude to get hot enough to vaporize, use only enough heat to reach 125˚C. Usually this means turning the hotplate no higher than 250˚F-300˚F)**
9. Allow the crude to remain at 125˚C until no more boiling occurs

**NOTE: Be mindful that depending on the level of ethanol present as well as the consistency of the crude itself, a foaming action can sometimes occur. Set a timer for every 2-5min when setting up a new BATCH. Confirm that the crude is not going to boil over**

1. Once the crude has reached 125˚C and is no longer showing signs of the presence of ethanol **(bubbling)** remove the de-gassing pot(s) from the hotplate
2. Obtain the Gross weight of the de-gassing pot(s) using the appropriate balance
3. Subtract the TARE weight(s) from the Gross weight(s) to obtain the NET weight(s) OUT
4. Record the NET weight(s) OUT on the De-gassing FORM
5. Record the number of hours this BATCH spent de-gassing
6. Calculate the % lost to de-gassing using the formula below **(Fig 5.1)** and record the value on the De-gassing FORM:

**(Fig 5.1)**

**grams lost to de-gas/ NET weight IN x 100 = % Loss to De-gassing**

1. If the crude is added directly to either the WFMS de-gassing/holding pot or the SPD reaction flask, then check the appropriate box near the bottom of the De-gassing FORM
2. If the crude is jarred for later distillation, record the storage jar label IDs and weights in the appropriate place on the bottom of the De-gassing FORM

**Section VI** (ver. 5.28.19)

**Distillation**

ProcessSummary

This procedure uses one of two types of distillation to separate the target cannabinoid(s) from both the lighter and heavier compounds in our de-gassed crude extract.

One type of distillation uses a Short-Path, Round Bottom Still and is often referred to as SPD (Short Path Distillation).

Here the de-gassed crude extract is loaded into a large double or triple-neck, round bottom boiling flask and seated in a heat/stir mantle. The reaction flask is then fitted with a special distillation head, condenser, and collection flask(s). The system is then connected to a vacuum source and the heat gradually increased. Compounds with a lower boiling point are collected and removed first. Then, the collection flask is changed and the main target compounds (the “Mains”) are collected. The collection flask is changed again and “tails” collected. This is referred to as a First Pass SPD distillation. These “Mains” can then be sent to isolation as-is, or loaded into a clean Round-bottom boiling flask and returned to the mantle for a “Second Pass” distillation. This “second pass” distillation produces a product much more suitable for sale as a distillate.

A second type of distillation uses a Wiped Film Molecular Still (WFMS), which has many advantages over the SPD-type still described above.

Here the de-gassed crude extract is loaded into a heated de-gassing/holding pot, where it’s transferred to the vertical body of the still via a peristaltic dosing pump. This allows for total control of the feed rate, which is as simple as setting a value on the peristaltic pump’s controller. The crude is fed into the body of the still where PTFE blades wipe the material into a thin film as it progresses via gravity from the top of the still toward the bottom. As the crude moves down the vaporization surface the target molecules are vaporized and collected on the surface of the center condenser, sometimes referred to as a “cold finger.” The distillate continues to flow down the cold finger condenser and is directed into a collection flask. The un-distilled material, or Raffinate continues to flow down the outer edges and is directed into a separate collection flask. This process is performed first at a lower temperature under mild vacuum (80-700mTORR) to remove volatiles and terpenes. The Raffinate is then loaded back into the de-gassing/feed pot of a second WFMS, where extra vacuum is applied via a diffusion pump to bring the pressure to below 10mTORR and with the temperature raised slightly higher. By lowering the vacuum pressure to below 10mTORR a special distillation condition called molecular distillation is achieved. Here the mean free path is cleared of all interfering molecules and vaporization of the target molecules occurs in strictly straight lines from the vaporization surface to the condensing surface. This allows for cleaner separations at lower temperatures than is possible with traditional or SPD-type distillations, thereby producing a higher quality product with an increased potency and yield.

**SPD-type Distillation:**

**Procedure**:

*MISSING SECTION*

**WFMS-type Distillation:** (ver. 5.28.19)

**Procedure:**

I. Volatiles Removal/Terpenoid Stripping

1. Transfer your de-gassed crude extract from the de-gassing pot or storage vessel to the WFMS de-gassing/holding pot **(If the crude is not coming directly off the de-gassing hotplate you’ll need to warm it up enough to allow for an easy transfer. Use the de-gassing hotplate with an overhead stirrer if it’s still in the de-gassing pot(s). If it has been stored in a jar, you can remove the lid and use the microwave in short 30 second intervals until its fluid enough to allow an easy transfer)**
2. Turn on the WFMS and set the still body temperature to 137˚C
3. Turn on and set the internal cold finger chiller to 35˚C
4. Turn on and set the primary cold trap chiller to -50˚C
5. Confirm the secondary cold trap is on and has reached -90˚C

**CAUTION: Running without the cold trap chillers on and at the proper temperature exposes your pump to volatile vapors and other contaminates. DO NOT operate the still without ALL chillers on and at the proper temperature**

1. Confirm that ALL chillers and heating elements are at the proper temperature
2. Confirm that both cold traps have a clean collection flask attached **(Change if necessary)**
3. Engage the vacuum pump
4. Allow the pressure to drop to below 600mTORR before beginning distillation
5. Record the still body temperature setting and cold-trap chillers temperature settings at the top of the POPE 4.1 Distillation FORM
6. Record the DATE, TECH, BATCH number and BATCH weight at the top of the POPE 4.1 Distillation FORM
7. Set the peristaltic dosing pump to **1.25L/hour**
8. Turn on the blades motor
9. Set the blades motor to **80**
10. Confirm that the dosing pump is delivering crude by observing the inlet joint at the top right of the still body
11. Record the TIME and instrument readings on the POPE\_4.1 Distillation FORM
12. Continue the distillation, being sure to record the instrument readings at least twice per hour
13. Adjust the feed rate and blade speed using their respective controllers so as to maintain a good yield, loosing only a small amount of product to the distillate collection flask
14. Be sure to record instrument readings a minimum of twice per hour

II. Cannabinoid Distillation

1. Transfer your de-terped/volatile stripped crude extract from the previous procedure above, to the 6” WFMS de-gassing/holding pot **(You may need to warm it up enough to allow for an easy transfer. Place the flask, or if it has been stored in a jar, you can remove the lid and use the microwave in short 30 second intervals until its fluid enough to allow an easy transfer)**
2. Turn on the WFMS and set the still body temperature via the hot oil circulator to **165˚C**
3. Turn on the and set the internal cold finger chiller to **70˚C**
4. Confirm the cold trap is ON
5. Confirm that ALL chillers and heating elements are at the proper temperature
6. Confirm that both cold traps have a clean collection flask attached
7. Engage the main vacuum pump
8. Once the pressure has dropped below 400mTORR, engage the diffusion pump
9. Allow the diffusion pump to reduce the pressure to below 10mTORR
10. Record the still body temperature and cold-trap chiller temperature settings at the top of the POPE\_6.1 Distillation FORM
11. Record the DATE, TECH, and BATCH number and BATCH weight at the top of the POPE\_6.1 Distillation FORM
12. Set the peristaltic dosing pump to **1.25L/hour**
13. Turn on the blades motor
14. Set the blades motor to **80**
15. Confirm that the dosing pump is delivering crude by observing the inlet joint at the top right of the still body
16. Record the TIME and instrument readings on the POPE\_6.1 Distillation FORM
17. Continue the distillation, being sure to record the instrument readings at least twice per hour, or whenever a change is made or action taken
18. Adjust the feed rate and blade speed using their respective controllers so as to maintain a good yield, loosing only a small amount of product to the raffinate collection flask
19. Be sure to record instrument readings a minimum of twice per hour and any changes made or actions taken

**Section VII** (ver. 5.30.19)

**Isolation**

Process Summary

This procedure is a Winterization technique, which takes advantage of CBD’s poor solubility in heptane at temperatures below 0˚C. While soluble in heptane at room temperature and above, CBD is susceptible to precipitation at lower temperatures. By mixing the distillate into heptane at room temperature and then freezing, the CBD is liberated from the solution via precipitation and can then be filtered from the mother liquor. Washing with additional frozen heptane removes the residual mother liquor, leaving a fine, brilliant white powder.

The general rule governing this procedure is to calculate 60% of the weight of distillate in grams to obtain the amount of heptane required in mL for SPD distilled material, or 1:1 for POPE\_6.1 distilled material

**(Fig. 7.1)**

***1000g distillate x 0.6 = 600mL heptane (for SPD distilled material)***

***1000g distillate x 1 = 1000mL heptane (for POPE\_6.1 dist. material)***

Here the two are mixed together using an overhead stirrer until the distillate is fully dissolved in the heptane. The mixture is then frozen. The CBD becomes liberated from the mixture and precipitation occurs forming a suspension of CBD in heptane with a “butter-like” consistency. This mixture is then placed into a Hochstrom filtration apparatus and washed with frozen heptane, leaving a fine white powdered CBD behind.

**NOTE**: **In order to promote a more rapid precipitation, approximately 2-5 hours after the mixture is placed in the freezer the solution should be agitated with a hand blender and then returned to the freezer**

**NOTE: “Tails” and “heads” should be isolated separately from the “mains”**

**Procedure:**

I. Estimating distillate jars needed

1. Use approximately 2500g of distillate for this procedure
2. Locate all the jars for the next BATCH to be isolated
3. Using the appropriate balance take a measurement of the Gross weight of each jar
4. Record the Gross weight of each jar of distillate in the NOTES section of the Initial Isolation FORM **(Be sure to label all values)**
5. Subtract the engraved TARE weight from the Gross weight to obtain the NET weight of the distillate inside each jar **(Never assume the label weights are correct, always re-weigh)**
6. Use these NET weights to estimate how many jars will be needed to remove 2500g of distillate **(You’ll need more than 2500g NET to account for transfer loss)**

II. Preparing the isolation/mixing pot

1. Adjust the overhead mixer so that the mixing head is to one side of the isolation/mixing pot
2. Using a clean beaker or graduated cylinder, measure and add the required amount of Heptane (**heptane to distillate 0.6:1 for SPD distilled material, 1:1 for Pope\_6.1 distilled material**) to the stainless steal isolation/mixing pot **(The estimated graduations of a beaker and/or graduated cylinder are suitable here as the volumes do not need to be exact)**
3. Record the volume of heptane you added using the Initial Isolation FORM
4. Make sure the overhead mixer RPMs are turned all the way to the lowest setting
5. Turn the overhead mixer ON
6. Adjust the RPMs until a strong mixing begins

III. Mixing the distillate/heptane solution

1. Return to the distillate jars you selected above
2. Take the metal lids off the jars and use the microwave in 30sec intervals to warm the jars of distillate **(Be sure to take the jars out and mix by rotation in between each 30sec heating period)**
3. Slowly pour the estimated 2500g of warmed distillate into the isolation/mixing pot with the already mixing heptane
4. Using the appropriate balance record the Gross weight of the now emptied or partially emptied distillate jars
5. Subtract the Gross weight of these emptied jars from the Gross weight of the full jars before adding the distillate recorded above
6. This is the NET weight added to your Isolation Pot
7. Record the NET weight of distillate added on the Initial Isolation FORM
8. Continue mixing, being sure that ALL distillate has completely dissolved in the heptane
9. Stop the overhead mixer and confirm ALL distillate is fully dissolved **(You may need to use a spoon to dislodge any distillate stuck to the sides or bottom of the isolation pot)**
10. Once the solution is fully homogenized and no un-dissolved distillate is visible, turn the over head mixer OFF
11. Fill out a label with the format:

BATCH# **ABC001190223**

DATE IN: **05/12/19**

TIME IN: **17:12**

Dist. Wt.: **2507g**

TECH: **MJM**

1. Record the information from the label on the Initial Isolation FORM and place the FORM in order of DATE **(Most recent on top)** in the proper super sack folder
2. Attach the label to the lid of the isolation/mixing pot and move it to the chest freezer

IV. Preparing the Hochstrom Filtration Apparatus

1. Be sure the Hochstrom filter is clean **(If it is not clean, disassemble the unit and use Isopropanol to clean all parts, paying particular attention to the threads on both the base and upper wall piece)**
2. Place a single disc of 0.8µm pore size filter paper into the Hochstrom funnel
3. Center the beveled retaining ring on top of the filter paper and screw the upper walls of the funnel to the base **(Be sure the pieces are attached properly and tightened all the way)**
4. Confirm the collection vessel is empty **(If not, see *XI. Draining the Supernatant Liquid from the Hochstrom Apparatus*)**
5. Attach the tubing from the collection vessel to the Hochstrom funnel
6. Turn the diaphragm pump ON

V. Filtration and Washing the Raw Isolate

1. Remove the isolation/mixing pot from the freezer and record the TIME on the Initial Isolation FORM
2. Confirm that the mixture has homogenized **(It should appear as a light-yellow to orange butter-like solid)**
3. If the mixture is not solid use the hand blender to homogenize the Distillate/heptane mixture, you should see it cloud up
4. Record agitation times on the Initial Isolation FORM
5. Place the lid back on the isolation/mixing pot and return to the chest freezer for at least 60min or until solid
6. Once the mixture has solidified, transfer the contents of the isolation/mixing pot to the Hochstrom funnel, being sure to leave enough space for additional heptane
7. Confirm that the inlet valve on the collection vessel is in the OPEN position
8. Add additional frozen heptane to the Hochstrom funnel
9. Using a clean hand blender, thoroughly blend the contents in the Hochstrom funnel **(Be careful not to puncture the filter paper!)**
10. Allow the mother liquor to drain into the collection vessel **(Do NOT allow the mixture to dry out between washings or channeling may occur. Once the rinsing heptane is pulled below the surface add the next wash of heptane)**
11. Once the liquid level has lowered, begin washing the isolate mixture with small amounts of frozen heptane **(Use enough frozen heptane with each wash to cover all the isolate. Ideally, you want the frozen heptane to be about 2cm above the isolate mixture. Remember, many washes with a smaller volume of frozen heptane is better than fewer washes with a larger volume of frozen heptane)**
12. Continue washing the isolate mixture until you are left with pure white powder
13. Once the isolate appears completely rinsed of the mother liquor, use a clean hand blender to once again to mix the contents in the funnel **(Be careful not to puncture the filter paper!)**

**NOTE: If you accidentally puncture the filter paper, immediately turn the inlet valve to the CLOSED position and notify the Shift-Lead. Then, drain the vessel into a large beaker. Replace the filter paper on the Hochstrom funnel and return the contents of the beaker to the funnel and return to step 7 and continue**

1. Continue with successive washings until the isolate appears pure white and the heptane washes are quickly pulled beneath the surface of the isolate **(Allow the vacuum pump to partially dry the isolate before removing it from the Hochstrom funnel)**
2. Transfer the isolate from the funnel to a clean glass collection dish(s)
3. Create a label for the dish using the format:

BATCH# **ABC001190223**

DATE: **05/12/19**

TIME: **17:12**

TECH: **MJM**

1. Allow the Isolate to sit loosely covered with Aluminum foil for 4 hours
2. At the four-hour mark weigh the glass dish and its contents
3. Record the NET weight of the isolate on the Initial Isolation FORM
4. This material is now ready for re-crystallization

**Section VIII** *(ver. 5.30.19)*

***Re-crystallization***

Process Summary

This procedure is used as a purification process. By creating a supersaturated solution of CBD isolate in warm heptane, and allowing it to slowly cool to room temperature, we force the CBD isolate out of solution to form a lattice structure (crystal). If the initial molecules of CBD come together with no impurities incorporated into the lattice, then the CBD molecules will continue to build on that pure lattice and push the impurities in front of them. This leaves the purified crystals growing from the sides and bottom of the beaker, and the impurities left in solution. By repeating this process with clean heptane, the CBD crystals are able to grow in an environment, which becomes increasingly free of impurities, thereby producing a higher purity product with each repetition.

The general rule governing this procedure is tocalculate 120% of the weight of isolate in grams to obtain the amount of heptane required in mL.

**(Fig. 8.1)**

**1000g(CBD isolate) x 1.2 = 1200mL (heptane)**

The heptane is warmed to 50˚C and the isolate slowly added until it’s completely dissolved. This solution is then vacuum filtered through a Buchner funnel packed with Celite 545. The filtered solution is then gently reheated on a hotplate and mixed using a glass stir rod to re-dissolve any CBD that may have crystallized during filtration. The solution is then allowed to cool to room temperature until crystallization occurs.

**In order to promote a larger more pure crystal structure, the solution should be gently reheated using the hotplate once crystallization has covered the bottom and sides of the beaker. Do this the same day crystallization occurs so as not to re-dissolve a beaker that would be ready for collection. If a solution does not crystallize, a “seed” crystal can be added once the solution is cool enough so as not to melt the ”seed” once added.**

Once crystallization has stopped, the mother liquor is decanted into a jar, labeled, and placed into the walk-in freezer to precipitate the remaining CBD. The crystals are removed from the beaker, ground to a powder, placed into a Hochstrom vacuum filtration apparatus and washed with frozen heptane. The washed, powdered CBD isolate is then either sent back through re-crystallization for further purification, or spread on a glass dish and placed in the vacuum oven for 48hrs to purge the residual heptane.

**Procedure:**

I. Making a Supersaturated CBD Solution with Heptane

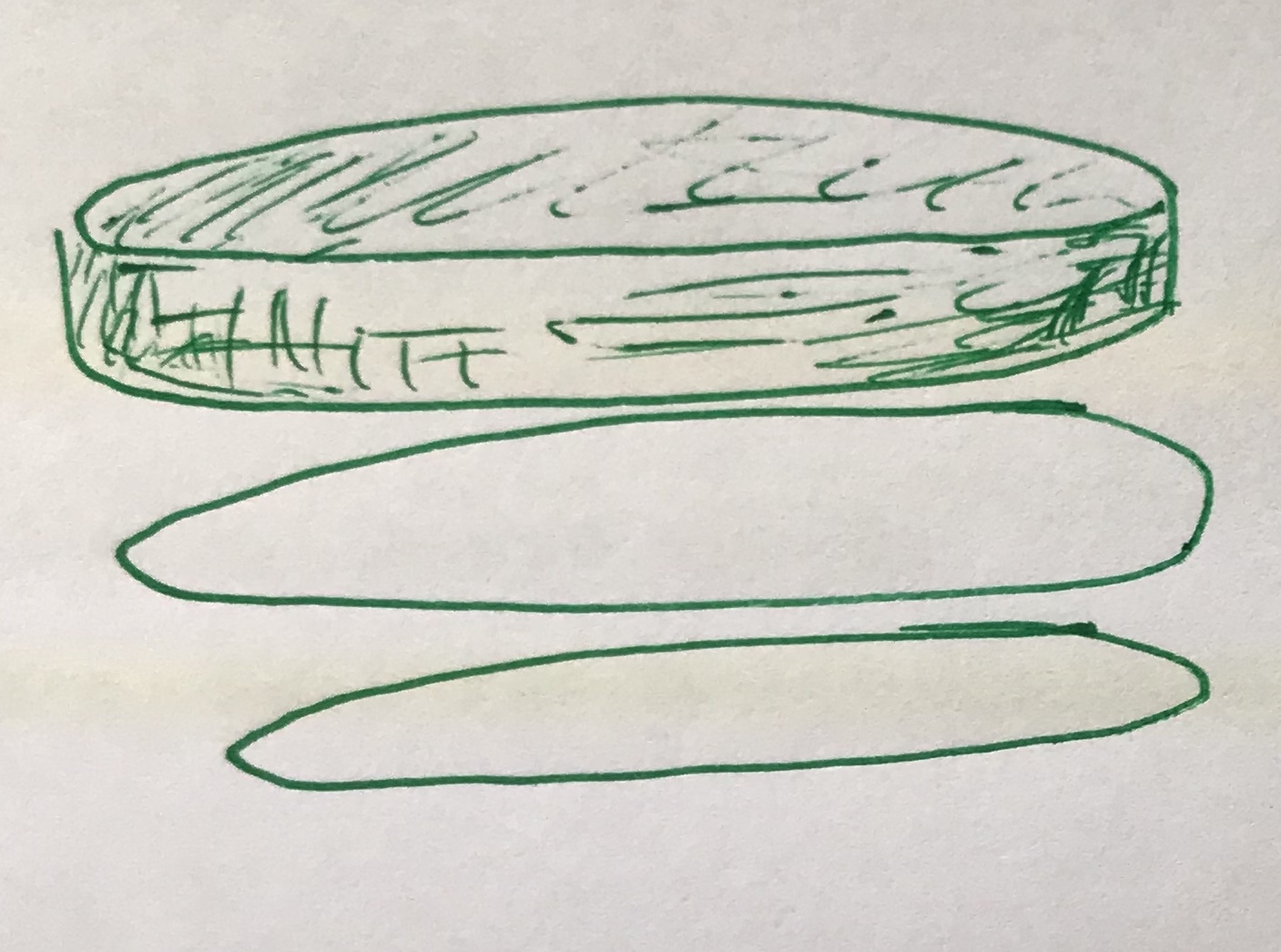
1. Weigh your isolate to be re-crystallized.
2. Use the formula above **(Fig 8.1)** to calculate the volume of heptane needed
3. Record these values on the Re-Crystallization FORM
4. Add the heptane to a clean 5L beaker, using the estimated graduations to measure the required amount from you calculation at step 2
5. Place the heptane filled beaker onto a MagStir hot plate **(Be sure its centered)**
6. Adjust the attached probe to hang in the heptane **(Be sure not to allow it to rest on the bottom of the beaker)**
7. Add a stir bar
8. Make sure the stir mechanism is in the OFF position
9. Turn the MagStir hotplate ON, and set it to 50˚C
10. Slowly increase the magnetic stir bar RPM until a strong mixing begins

**(Be careful not to turn the rotation too high, or have the beaker un-centered or the stir-bar will knock itself off balance and get stuck on one side, thereby stopping the mixing action)**

1. Place a piece of aluminum foil on the bench top along the front edge of the MagStir to catch any isolate that may spill during transfer from the dish to the beaker.
2. Once the heptane has reached 40˚C begin slowly adding the isolate **(Be careful to avoid knocking the stir bar off center. If this happens turn the stirring mechanism off and allow the stir bar to re-center its self. If that fails to re-center the stir bar, use a glass stir rod to push the stir bar back to center. Don’t forget to re-start the stirring mechanism and adjust the stir speed)**
3. Once all the isolate has been added, allow the MagStir to reach 50˚C **(Don’t forget to add any isolate that may have spilled on the aluminum foil)**
4. Confirm that all the isolate has fully dissolved

II. Preparing a Celite 545 Filter Pad

**(Fig. 8.2 Exploded view of the Re-crystallization filtration media pad)**

****

**Celite 545**

**1µm silca filter paper**

**qualitative filter paper**

1. Set-up a Buchner filtration apparatus
2. Connect the Buchner Flask to the diaphragm pump using the vacuum tubing
3. Turn the diaphragm vacuum pump ON
4. Confirm that the Buchner Funnel is under vacuum **(Check the line valve and confirm it is in the closed position)**
5. Place a qualitative filter paper in the center of the funnel
6. Wet the filter paper using clean heptane
7. Place a 1µm silica filter paper centered on top of the first filter paper
8. Wet the filter paper with clean heptane
9. Using a spoon, begin adding the Celite 545, starting from the center of the filter papers
10. Use the spoon to lay down a uniform 5-6cm layer of Celite 545, with no cracks or pitting
11. Use the bottom of a clean 100mL beaker to compress the Celite 545 as much as possible
12. Confirm the Celite 545 pad is uniform and shows no signs of irregularities or holes
13. Completely wet the Celite 545 by pouring heptane over the entire pad **(Be sure that the heptane soaks the entire pad or “channeling” may occur)**
14. Fix any channeling and re-wet the Celite 545 pad using clean heptane
15. Confirm the Celite 545 pad is uniform and shows no signs of irregularities or holes
16. This pad is now good to use for approximately one week, or until the Celite 545 is impaired by “junk”

III. Filtering the Supersaturated CBD/Heptane Solution

1. Turn the vacuum pump connected to your Buchner filtration apparatus ON
2. Take your supersaturated solution and slowly pour the contents over the Celite 545 filter pad
3. If water is present it will be at the bottom of your beaker, try to decant the heptane solution, leaving the water **(if present)** behind
4. If water is NOT present, then rinse the 5L beaker into the Celite 545 pad with clean heptane a minimum of two times to collect any residual solution
5. Allow the last of the solution to drain into the Erlenmeyer collection flask
6. Use clean heptane to rinse the Celite 545 pad a minimum of three times, allowing the heptane to fully drain between each rinse
7. Turn the vacuum pump OFF
8. Disconnect the Buchner funnel and place in a holding jar
9. Remove the rubber gasket from the filter flask
10. Slowly pour the filtered solution into a 3L beaker **(If you must split the solution into more than one beaker, be sure to divide it evenly)**
11. Rinse the filter flask into the 3L beaker(s) with clean heptane three times to collect any residual solution

IV. Crystallization & Labeling

1. Place the beaker(s) on a hotplate and gently reheat to re-dissolve any isolate that may have begun to crystallize during filtration
2. Use a glass stir rod to mix the solution until homogenous
3. Confirm the solution is fully homogenized
4. Turn the hot plate OFF
5. Cover the beaker(s) with Aluminum foil
6. Print two identical labels using the RECRYSTALLIZATION label on the Dymo label printer
7. Attach one label to the foil beaker cover
8. Attach the second label to the back of the Re-Crystallization FORM in the designated area(s)
9. Record the information from the label on the Re-Crystallization FORM

V. Decanting the Mother Liquor

1. Once crystallization has stopped **(Usually within 24 hours)** remove the beaker(s) from the hot plate
2. Step up a ring stand with a chain clamp
3. Slowly decant the solution into a collection jar
4. Label the collection jar with the BATCH number
5. Position the collection jar under the chain clamp
6. Securely attach the beaker to the chain clamp
7. The beaker should first be positioned so that any solution can easily drain from the bottom
8. The angle of the beaker is then increased so that any remaining solution can easily drain into the collection jar
9. Confirm that the beaker has stopped dripping
10. Carefully remove the beaker from the chain clamp
11. Re-seal the collection jar, being sure to invert the inner half of the lid so as not to degrade the rubber seal **(The inner half of the lid should be face up, with the white coating with the rubber seal facing up)**
12. Place the collection jar in a box and move it to the walk-in freezer for at least 24 hours to force the CBD remaining in solution to crystallize

VI. Removing the Crystals

1. Return to the now decanted beaker containing your crystals
2. If a darker “crust” has formed around the upper-most edge of the beaker, you’ll need to separate and remove this first
3. Place a piece of Aluminum foil on the bench-top
4. Invert the beaker so that as you scrape the material from the upper edge it falls out onto the foil, and not back into the beaker **(Remember we are trying to separate this dark “crust” from our purified crystals, DO NOT let this material fall back into the beaker)**
5. Using a clean plastic-handled, extendable utility knife, carefully scape the darker “crust” from around the upper edge of the beaker onto the foil
6. Confirm that all the darker “crust” has been removed
7. Transfer the separated “crust” you’ve removed from the collection foil to an Isolate “Spills” jar
8. Place a clean glass baking dish on the bench-top
9. Return to the recrystallization beaker
10. Hold the beaker above the glass dish
11. Using a clean plastic-handled, extendable utility knife carefully remove the crystals from the sides of the beaker, using the glass dish to collect the material as it’s freed **(Be careful not to score the sides of the beaker with the knife blade as you work. This weakens the glass and increases the possibility of the beaker cracking or breaking the next time its heated or frozen)**
12. Once the crystals have been removed from the sides of the beaker you can begin working to dislodge the layer that forms on the bottom **(This layer is often thick and firmly “stuck” to the beaker)**
13. Using a clean, plastic-handled, extendable utility knife carefully cut out a small “pie” wedge from the bottom “puck” **(Again, be careful not to score the bottom of the beaker with the knife blade as you work. This weakens the glass and increases the possibility of the beaker cracking or breaking the next time its heated or frozen)**
14. Now carefully begin removing the crystals from around the outer edge of the “puck” **(Once you have removed a sufficient portion of the outer edge, you should be able to spin the “puck” in the bottom of the beaker)**
15. Once the “puck” has been dislodged from the bottom of the beaker, carefully invert the beaker over the glass collection dish and allow the “puck” to slide out
16. Be sure to carefully scape any remaining isolate from the beaker into the glass collection dish

VII. Crushing & Milling the Crystals

1. Once the crystals have been removed from the beaker, use the back of a spoon to crush the large pieces into smaller pieces **(You may not have room in the glass collection dish to do this without spilling isolate onto the bench top. In this case use another clean glass baking dish to crush the larger pieces)**

**NOTE: Depending on how much residual heptane remains in the crystals, you may need to hand-crush the crystal masses and let the product air-dry in the glass dish before milling the pieces or clumping will occur in the blender, and a fine powder will not be achieved**

1. Once the isolate has been broken into smaller pieces and is no longer “wet” with residual heptane, its ready to be milled to a fine powder using a blender set to the “Flour Mill” function
2. Carefully transfer your crushed, re-crystallized isolate into the blender and clip the lid with the plastic pestle in place **(Be sure the lid is properly attached and clipped down)**
3. Turn the power to the blender ON
4. Set the function to “Flour Mill”
5. Press START
6. Using the plastic pestle assist the milling action by pulling the blending isolate from the outer upper edges and corners toward the center
7. Mill the isolate until its homogenous and no more “crackling” sounds can be heard
8. Press STOP
9. Turn the blender OFF
10. Remove the blender lid, and detach the pitcher from the motor base of the blender
11. Carefully transfer the milled isolate back to the same glass collection dish that it was crushed in
12. Confirm that all the isolate was ground to a fine powder and no large or small chunks remain **(If any chunks or un-milled pieces are found, return just those pieces back to the blender and repeat steps 3-13)**

VIII. Preparing the Hochstrom Filtration Apparatus

1. Be sure the Hochstrom filter is clean **(If it is not clean, disassemble the unit and use Isopropanol to clean all parts, paying particular attention to the threads on both the base and upper piece)**
2. Place a single disc of 0.8µm pore size filter paper into the Hochstrom funnel
3. Center the beveled retaining ring on top of the filter paper and screw the upper walls of the funnel to the base **(Be sure the pieces are attached properly and tightened all the way)**
4. Confirm the collection vessel is empty **(If not, drain the mother liquor from the collection vessel using the drain valve into an appropriate and labeled collection drum)**
5. Attach the tubing from the collection vessel
6. Turn the diaphragm pump ON

IX. Filtration and Washing of the Re-Crystallized Isolate

1. Transfer the powdered re-crystallized isolate from the glass dish to the Hochstrom funnel, being sure to leave enough space for additional heptane
2. Confirm that the inlet valve on the collection vessel is in the CLOSED position
3. Add additional frozen heptane to the Hochstrom funnel
4. Using a clean hand blender, thoroughly blend the contents in the Hochstrom funnel **(Be careful not to puncture the filter paper!)**
5. OPEN the inlet valve on the collection vessel
6. Allow the mother liquor to drain into the collection vessel **(Do NOT allow the mixture to dry out between washings or channeling may occur. Once the rinsing heptane is pulled below the surface add the next wash of heptane)**
7. Once the liquid level has lowered, begin washing the isolate mixture with small amounts of frozen heptane **(Use enough frozen heptane with each wash to cover all the isolate. Ideally, you want the frozen heptane to be about 2cm above the isolate mixture. Remember, many washes with a smaller volume of frozen heptane is better than fewer washes with a larger volume of frozen heptane)**
8. Continue washing the re-crystallized isolate until you are left with pure white powder
9. Once the isolate appears completely rinsed of the mother liquor, use a clean hand blender again to mix the contents of the funnel **(Be careful not to puncture the filter paper!)**

**NOTE: If you do accidentally puncture the filter paper, immediately turn the inlet valve to the CLOSED position and notify the Shift-Lead. Then, drain the collection vessel into a large beaker. Replace the filter paper on the Hochstrom funnel and return the contents of the large beaker to the funnel and return to step 7 and continue**

1. Continue with successive washings until the isolate appears pure white and the heptane washes are quickly pulled beneath the surface of the isolate **(Allow the vacuum pump to partially dry the isolate before removing it from the Hochstrom funnel)**
2. Transfer the isolate from the funnel to a clean glass collection dish(s)
3. Create a label for the dish using the format:

BATCH# **ABC001190223**

DATE: **05/12/19**

TIME: **17:12**

TECH: **MJM**

1. Allow the Isolate to sit loosely covered with Aluminum foil for 4 hours
2. At the four-hour mark weigh the glass dish and its contents
3. Record this weight on the Re-Crystallization FORM
4. Inspect the isolate:

**If it passes COLOR and ODOR screening, cover the dish with Aluminum foil and proceed to,*****Section VIII, Part* *XII. Purging the Isolate of Residual Solvents*. Be sure to label BOTH the dish and the oven shelf with the information above. (Oven labels should be attached on the outside of the ovens, to the left of the oven door for trays at the rear and the right side for trays in the front, at the level of the shelf the dish is placed on)**

**If it does not pass COLOR and ODOR screening it must be sent back through the re-crystallization process. Return to the beginning of *Section VIII, Part I. Making a Supersaturated CBD Solution with Heptane***

1. Be sure to record your actions on the Recrystallization FORM

X. Processing the Mother Liquor

**NOTE: After approximately 24 hours in the walk-in freezer the mother liquor jars should be ready for processing**

1. Prepare your Hochstrom filtration apparatus **(See *Section VIII, Part VIII*)**
2. Using the Re-crystallization FORM, locate all the jars of mother liquor associated with your BATCH.
3. Use a long stainless steel scooper or the handle of a stainless steel spoon to loosen the crystals on the sides and bottom of the jar
4. Slowly swirl the contents of the jar to suspend the now loose crystals in the heptane
5. Gently pour the mixture into your Hochstrom funnel **(If needed use additional frozen heptane to wash all the crystals out of the jars)**
6. Continue transferring the contents of all the jars associated with your BATCH into the Hochstrom funnel being sure to leave plenty of room for additional heptane
7. Close the line valve leading to the collection vessel to stop the vacuum temporarily
8. Add clean frozen heptane to the Hochstrom funnel until you cover the isolate
9. Using the hand blender carefully blend the mixture until homogenous

**NOTE: Be careful not to puncture the filter paper. If you do puncture the filter paper, immediately close the line valve, notify a supervisor or Shift-Lead Then drain the collection vessel into a large beaker. Replace the filter paper on the Hochstrom funnel and return the contents of the large beaker to the funnel and return to step 7 and continue**

1. Open the line valve to re-establish vacuum and allow the mother liquor to drain into the collection vessel **(Do NOT allow the mixture to dry out between washings or channeling may occur. Once the rinsing heptane is pulled below the surface add the next wash of heptane)**
2. Once the liquid level has lowered, begin washing the isolate mixture with small amounts of frozen heptane **(Use enough frozen heptane with each wash to cover all the isolate. Ideally, you want the frozen heptane to be about 2cm above the isolate mixture. Remember, many washes with a smaller volume of frozen heptane is better than fewer washes with a larger volume of frozen heptane)**
3. Continue washing the isolate mixture until you are left with pure white powder
4. Once the isolate appears completely rinsed of the mother liquor, use a clean hand blender again to mix the contents of the funnel **(Be careful not to puncture the filter paper!)**
5. Continue with successive washings until the isolate appears pure white and the heptane washes are quickly pulled beneath the surface of the isolate **(Allow the vacuum pump to partially dry the isolate before removing it from the Hochstrom funnel)**
6. Transfer the isolate from the funnel to a clean glass collection dish(s)
7. Create a label for the dish using the format:

BATCH# **ABC001190223**

DATE: **05/12/19**

TIME: **17:12**

TECH: **MJM**

1. Allow the Isolate to sit loosely covered with Aluminum foil for 4 hours
2. At the four-hour mark weigh the glass dish and its contents
3. Record this weight on the Re-Crystallization FORM
4. Inspect the isolate:

**If it passes COLOR and ODOR screening, cover the dish with Aluminum foil and proceed to, *Section VIII, Part* *XV. Purging the Isolate of Residual Solvents.*** **Be sure to label BOTH the dish and the oven shelf with the information above. (Oven labels should be attached on the left of the oven door for trays at the rear, and to the right for trays in the front, at the level of the shelf the dish is placed on)**

**If it does not pass COLOR and ODOR screening it must be sent back through the re-crystallization process. Return to the beginning of *Section VIII, Part* *I. Making a Supersaturated CBD Solution with Heptane***

XI. Draining the Supernatant Liquid from the Hochstrom Apparatus

1. Remove the tubing from the base of the Hochstrom filter so that it is separated from the filtration collection vessel
2. Place the filtration collection vessel on top of a 5gal solvent barrel
3. Position another solvent barrel for use as the supernatant collection vessel at the lower drain valve on the filtration collection vessel
4. Use the dedicated stainless steel funnel to direct the supernatant fluid from the filtration collection vessel into the supernatant collection vessel
5. Once draining is complete, close the lower drain valve on the filtration collection vessel
6. Close the now filled supernatant recovery barrel using the original cap
7. Label the supernatant recovery barrel using the format:

**Saturated C7**

DATE: **4/17/19**

TECH: **MJM**

1. Move the supernatant recovery barrel to the 30L Rotovap area

XII. Heptane Recovery

1. Remove the cap of the supernatant recovery barrel and place the feed line inside, so that the line touches the bottom
2. Turn on and set the Rotovap water bath temperature to 60-70˚C
3. Turn on and set the reaction flask rotation to 120RPMs
4. Turn on the vacuum pump and confirm that any vent valves are closed
5. Confirm the Rotovap is under vacuum via the vacuum pressure gauge
6. Slowly open the Rotovap feed valve to begin adding your supernatant solution
7. DO NOT over fill the reaction flask or “bumping” will occur and any recovered heptane in the collection flask will become contaminated

**NOTE: If Bumping does occur, the collection flask must be drained and added back into the feed barrel to be Rotovaped again**

1. Adjust the Rotovap feed valve so that the condenser is able to keep up with the evaporating solvent, and a minimum amount of solvent is passing through the pump into the pump’s effluent collection jar
2. Continue feeding the reaction flask until the solvent collection flask is full

XIII. Recovered Heptane Collection

1. Slowly vent the Rotovap using the vent valve on top of the collection flask
2. Turn the vacuum pump OFF
3. Confirm that the Rotovap has returned to atmospheric pressure
4. Use a small beaker to retrieve a sample of the recovered heptane using the drain valve on the bottom of the collection flask
5. Screen the recovered heptane for ODOR **(It should not smell any different than fresh unused heptane)**
6. If the heptane passes ODOR screening, place the collection flask drain line into a designated recovered heptane storage barrel
7. If the heptane does not pass ODOR screening, place the collection flask drain line into a waste solvent storage barrel
8. OPEN the collection flask drain valve
9. Empty to collection flask into the proper storage barrel
10. Close all Rotovap vent and drain valves
11. Reengage the vacuum pump
12. Continue feeding the reaction flask until it is no more than half full, stopping occasionally to empty the solvent collection flask as described in steps 10-27

XIV. Post-Isolation Distillate Collection

1. Shut the Rotovap feed valve
2. Once solvent is no longer dripping from the condenser coils stop the rotation and confirm that no boiling is occurring in the reaction flask
3. Vent the Rotovap slowly using the vent valve on top of the collection flask
4. Disengage the vacuum pump
5. Remove the reaction flask from the body of the Rotovap
6. Wipe any water from the outside of the reaction flask
7. Confirm no water remains on the outside of the reaction flask
8. Using the large flask support stand, drain the contents of the reaction flask into tared storage vessels
9. Apply a Post-Isolation Distillate label using the Dymo label printer using the format:

TARE: **808.4g**

NET Wt.: **1782.9**

DATE: **4/16/19**

TECH: **MJM**

1. Re-attach the reaction flask to the Rotovap body
2. Close ALL vent and drain valves
3. Re-engage the vacuum pump
4. Confirm that vacuum has been re-established using the vacuum pressure gauge
5. To continue with heptane recovery, return to Section VIII, Part XII, Step 4

XV. Purging the Isolate of Residual Solvents

1. Before opening any vacuum oven(s), you must first isolate it from the vacuum pump and the second oven attached to that pump
2. Confirm that any product already inside the ovens is ready to be removed

**NOTE: Do not add material-to-be-purged to an oven if any of the product inside has been purging for more than a few hours. This will cross contaminate the semi-purged product and all product in that oven will then need to be purged for another 48 hours regardless of how much time they had already been in the vacuum oven**

1. Close the vacuum valve on the oven you plan to open
2. Slowly open the vent valve and let the pressure equalize **(Be careful not to open the valve too much, as this will cause a strong stream of air to rush in and disturb any product inside)**
3. Once the pressure inside the oven has equalized to atmosphere, you can open the oven door
4. Remove any dishes of finished isolate from the shelving inside
5. Using Isopropanol thoroughly clean the inside of the oven, paying close attention to the glass door-front and the area where the Buna seal contacts the glass **(Be extra mindful when using solvents in the vicinity of your finished isolate as cross-contamination is likely to occur)**
6. If you remove the shelves inside, be sure that the lower shelf is reattached to the temperature-probe properly when reinserting
7. Load your glass dish(s) into the oven, being sure to label the dish using the format below:

BATCH# **ABC001190223**

Isolate NET Wt. IN: **589.2g**

DATE: **05/12/19**

TIME: **17:12**

TECH: **MJM**

1. Print two identical labels using the FINAL VacOven PURGE label on the Dymo label printer
2. Attach one to the outside of the oven, to the side of the shelf the dish(s) are placed on
3. Attach the second label to the back of the Re-Crystallization FORM in the designated area(s)
4. Confirm the Vacuum Oven is set to 53˚C, or 107˚F
5. Remove the Buna gasket and rotate it 90 degrees before reinstalling
6. Shut the oven door
7. Turn your attention to the other oven that is attached to the same pump as the oven you just loaded with your product
8. Shut the vacuum vent on the other oven to protect it from the pressure change of the oven you just loaded
9. Confirm that the other oven has been isolated from the vacuum pump **(Both vacuum valves on the two ovens that are connected via the vacuum pump should be CLOSED)**
10. Confirm that the vent valve on the oven you just loaded is shut
11. Slowly open the vacuum valve on the newly loaded oven
12. Allow the vacuum pump to lower the pressure until the gauge stabilizes.
13. Allow 15-20 minutes once the gauge has stabilized before proceeding
14. Confirm that both your ovens are at their lowest pressures
15. Now, slowly reopen the vacuum valve on the second oven. **(The one you did NOT just load)**
16. Confirm that both ovens are at vacuum pressures and that the heating element of both ovens is ON and set to the correct temperature before walking away
17. Allow the product to purge under vacuum at 53˚C for 48 hours
18. After 48 hours has passed the product should be ready for removal

XVI. Removing the Final Product

1. Before opening the vacuum oven, you must first isolate it from the vacuum pump and the second oven attached to that pump
2. Confirm that any product already inside the ovens is ready to be removed

**NOTE: Do not add material to be purged to an oven if the product already inside has been purging for more than a few hours. This will cross contaminate the semi-purged product and all product in that oven will then need to be purged for another 48 hours regardless of how much time they had already been in the vacuum oven**

1. Close the vacuum valve on the oven you plan to open
2. Slowly open the vent valve and let the pressure equalize, being careful not to open the valve too much, which will cause a strong stream of air to rush in and disturb any product inside
3. Once the pressure has equalized to atmosphere you can open the door to the oven
4. Remove the purged isolate from the shelves inside
5. Record the TIME and DATE as the VacOven OUT value on the Recrystallization FORM
6. Carefully transfer your purged isolate into the “CLEAN” blender and clip the lid with the plastic pestle in place **(Be sure the lid is properly attached and clipped down)**
7. Turn the power to the blender ON
8. Set the function to “Flour Mill”
9. Press START
10. Using the plastic pestle assist the milling action by pulling the blending isolate from the outer upper edges and corners toward the center
11. Mill the isolate until its homogenous and no more “crackling” sounds can be heard
12. Press STOP
13. Turn the blender OFF
14. Remove the blender lid, and detach the pitcher from the motor base of the blender
15. Carefully transfer the milled isolate back to the same glass collection dish that it was purged in
16. Confirm that all the isolate was ground to a fine powder and no large or small chunks remain **(If any chunks or un-milled pieces are found, return just those pieces back to the blender and repeat steps 7-13)**

XVII. Sampling and Packing the Isolate

1. Combine all isolate from the BATCH into a large stainless steel mixing bowl
2. Completely homogenize the isolate using a stainless steel spoon
3. Retrieve an analytical sample vial and Analytical Request FORM
4. Weight the empty sample vial using the appropriate balance
5. Record the sample vial TARE weight on the Final Packaging FORM
6. To be sure that a representative sample is taken, sample the homogenized isolate by taking a minimum of 6 small aliquots from various areas and depths
7. Fill the sample vial to a minimum of 3.0g, but not more than 3.5g
8. Record the NET sample weight on both the Analytical Request FORM and the Final Packaging FORM, by subtracting the Gross weight of the filled sample vial from the TARE weight of the sample vial
9. Now retrieve the Final Packaging Containers and lids
10. Using the appropriate balance obtain and record the TARE weight of the container and lid together on the Final Packaging FORM
11. Fill container(s) with a minimum of 1000.2g, but no more than 1000.9g
12. Record this weight on the Final Packaging FORM
13. Package any remaining isolate that is less than 1000g
14. Record this weight on the Final Packaging FORM
15. For each container, print two identical labels using the Cannabidiol (CBD) label on the Dymo label printer
16. Attach one to the outside of the Container; being sure that the label is attached straight and in a uniform fashion **(This is IMPORTANT. This is the first impression that a customer sees when they receive our product. Take the time to do it properly)**
17. Attach the second label to the bottom/back of the Final Packaging FORM in the designated area(s)