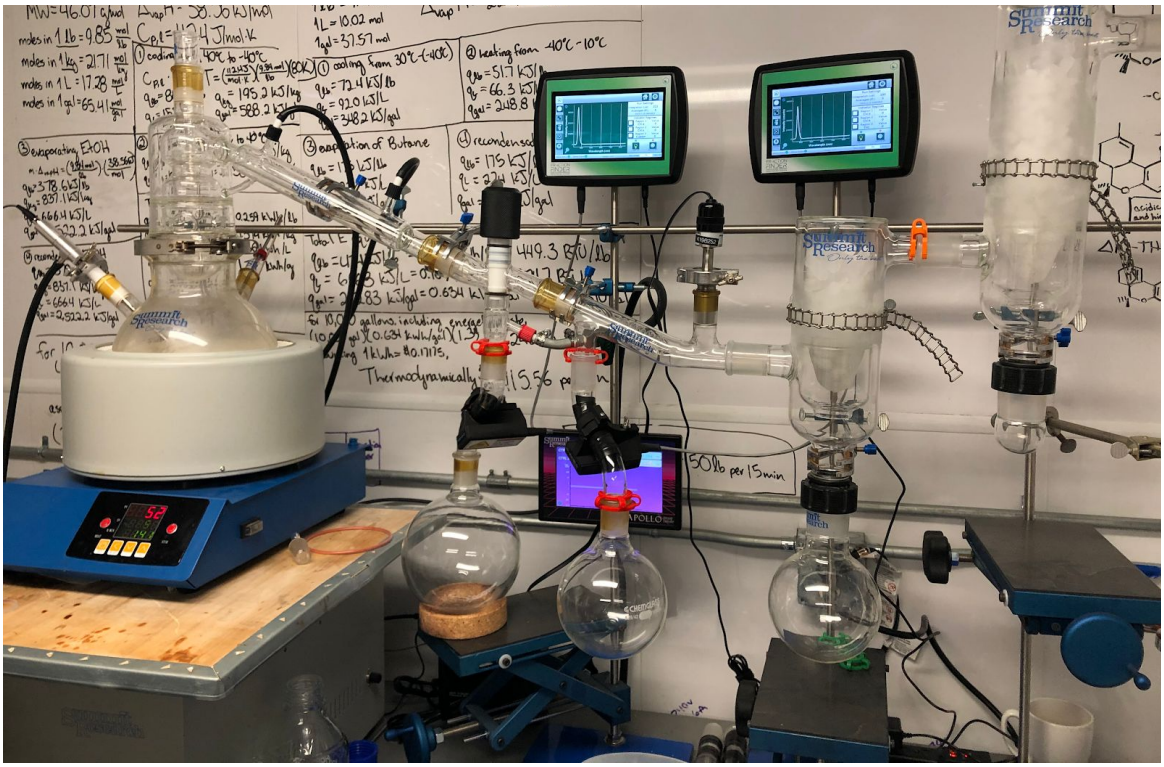
APPLICATION NOTE

HOT CONDENSED HEMP DISTILLATION WITH TWO FRACTION FINDERS



Created by

Arometrix and Prospect Farms

Purpose	Provide instructions for hot condensing Hemp crude oil with a Short Path Distillation unit that uses the Fraction Finder as a process control indicator.

Definitions	SPD Hot Tech FF nm	Short Path Distillation Hot condensing / high temperature Fraction Finder Nanometers (Wavelengths)

Equipment	● Standard Mantle-Based SPD Unit ● Qty 2 Arometrix FRACTION FINDERS ● Qty 2 Arometrix Hot Tech Adapters ● Qty 1 180° isolation valve ● Duratherm G Food-Grade HT Fluid (or other high-temp fluids)

Installation	1. One FRACTION FINDER is installed on the Hot Tech Adapter above the Distillation Collection on the left. 2. One FRACTION FINDER is installed on the Hot Tech Adapter above the Heads Collection on the right.

Fraction Finder Summary	The Fraction Finder shows a significant 410/460 nm signal during Hot Condensed SPD runs. The 410 nm peak is associated with “Heads”. The 460 nm peak is associated with “Main Body”. The 460 nm peak will slightly shift to the right when going into “Tails”.

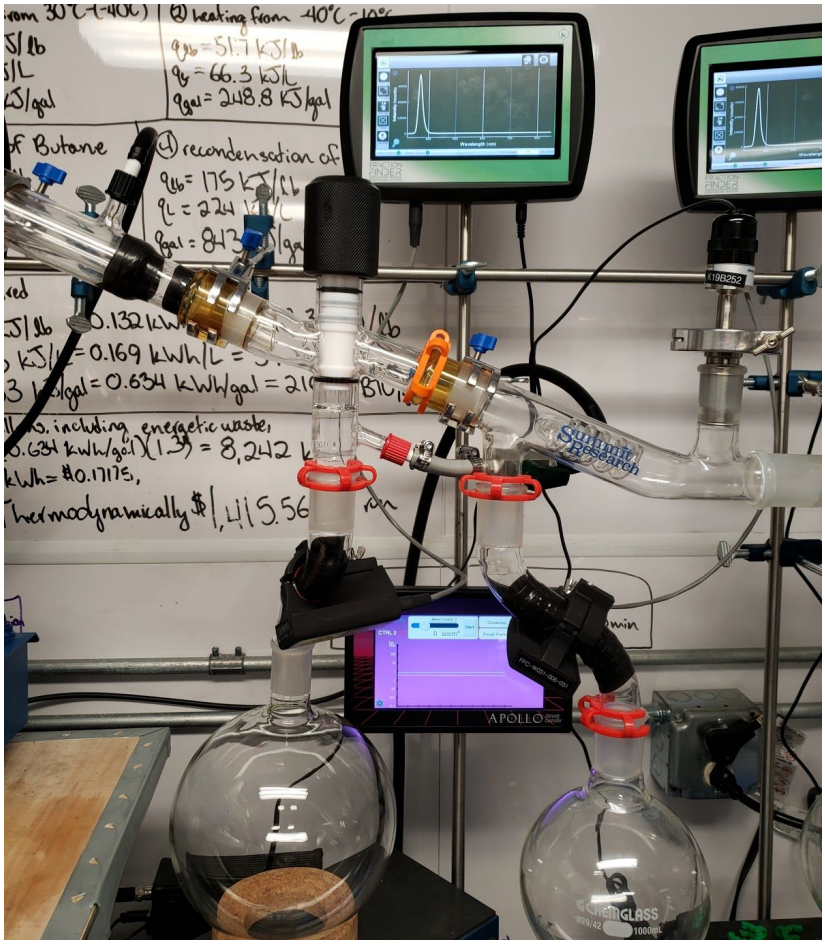
Reference Light	360-390 nm	The Reference/Excitation peak is from the sensor device and is not indicative of any distillation fractions or molecules.
Hemp Heads; “Fool’s Gold”	410-420 nm	“Fool’s Gold” is a colloquial term for a chemical component commonly seen during distillation which is golden in color and can appear to look like a cannabinoid by eye.
Hemp Main Body (CBD Indicator)	460 nm	The peak indicator associated with the presence of cannabinoids, specifically inclusive of CBD.
Hemp Tails/Degraded Cannabinoids	490 nm	The primary signifier (outside of observation) of transition to Tails from Heads, theorized to be degraded cannabinoid and/or "heavy" pigments.
Lipids	530-630 nm	Lipids are a class of molecules, and may have multiple peaks.
Chlorophyll	680 nm; 710 nm	May show 1 or 2 peaks @ 680 nm and 710 nm.

* See Table 1 in Appendix for quick reference of hard settings/values at the beginning of each major step\
** This tutorial was written using a 5 L boiling flask. The prescribed boiling flask temperatures may need to be increased for larger boiling flask volumes.

Procedure*

1. Assembly: Short Path with Hot Tech Adapters and Fraction Finders

a. Wrap both Hot Tech Adapters in Fraction Finder light-blocking tape.
b. Grease Hot Tech Adapters and any other joints. For joints in contact with high temperatures, a high viscosity grease such as Apiezon H should be used.
c. Install Hot Tech Adapter above distillation receiving flask.
d. Install Hot Tech Adapter above Tails receiving flask.
e. Fill boiling flask to 40-60% of total capacity (i.e. 2-3 L in 5 L boiling flask**) with (previously decarbed) Hemp crude oil.
f. Add dry-ice/ethanol mix to both cold traps.
g. Note that the heat-transfer fluid should be stable under high-heat (up to 200°C) and suitable for the process at hand.
    i. For this tutorial, Duratherm G was used due to food-grade necessity, but high-temperature silicon-based fluids are also a functional option.



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2. Initial Operations

a. Set Fraction Finder Settings
    i. Settings (top-right corner of display) > Wavelength Settings
    ii. Set Range 1 to 450-490 nm
    iii. Record the run. Press record (bottom-right corner of display).
b. Ensure that the isolation valve for the main's collection flask is closed (all fluid will bypass main's collection flask).
c. Apply vacuum to entire system slowly using a valve to avoid excess expansion of crude using roughing pump (volatiles pump if using a multi-pump configuration).
d. Once the system base pressure reaches below 1 Torr (preferably 4-800 mTorr depending on vapor load), begin heating/stir bar procedure (from Ambient to 150°C):
    i. Set mantle temperature first to 1-3 degrees above ambient probe temp to begin slowly and uniformly heating the entire solution.
    ii. Set stir speed slow at first to ensure actual motion from stir bar on its proper path: starting around 100 RPM if using a standard mantle.
    iii. For ramp through pre-heads temperature range (~35°C to ~130°C), increase temperature by 3-5°C to upwards of 10°C.
        1. Increase stir speed as temperature increases and viscosity of the crude in the mantle decreases.
        2. When boiling flask reaches 80°C, start heating condenser to 155°C
        3. Note that large temperature swings should only be used in the low 100°C range to avoid drastic overshooting.
        4. Above 100°C in the boiling flask, the FF Spectrum View will show a 410 nm peak which is associated with "Fool's Gold" - a molecular species of the Heads fraction (see **Figure 1** of the appendix)
e. Heat boiling flask to 155°C by increments of 1-5°C
f. Once into Heads at 155°C or greater in the boiling flask, wait for/ensure the condenser temperature has reached 155°C before proceeding
    i. The only compounds that are able to recondense in the head condenser should be cannabinoids. Heads should stay in/re-enter the vapor phase and recondense after the isolation valve/collection point for Mains.

3. Coarse Tuning Heads/Main Body Split

a. Note - This section of the run spans the approximate mantle temperature range of 155°C into the appearance of Mains, which will start can be expected to start around 165°C and reach full expression in the mid 170s°C.
    i. Confirm on Fraction Finder equipped to Heads collection:

1. That heads are present via the 410 nm peak.
2. That the 460 nm peak signifying cannabinoids first appears then increases in intensity as 170°C is approached.
3. Example spectra is shown in **Figure 2** of the appendix

ii. Temperature and stir bar speed can be slightly raised to increase the fluid vapor "momentum" or load to deposit and recondense heads further down the vapor line. This process will be referred to as "vapor loading".

b. Open isolation valve once FF spectrum view shows a strong 460 nm peak in conjunction with the 410 nm heads peak, and confirm adequate separation by:
   i. Using the overlay feature on the Heads' FF spectrum view, observe the 460 nm peak signifying cannabinoids diminishing while the 410 nm peak signifying heads persists (**Figure 3** of Appendix)
   ii. On the Mains' FF spectrum view, observe the appearance of only/mainly a 460 nm peak (**Figure 4** of Appendix)
c. **Difference on the Second Pass?** No.

4. Fine Tuning Heads/Main Body Split

a. If a strong 410 nm peak is present on the Mains' FF spectrum view:
   i. Heads are condensing with cannabinoids before the mains' collection point
   ii. The condenser temperature should be increased between 1-5°C slowly to keep heads in the vapor path until after the mains' collection point
b. If a strong 460 nm peak is on Heads' FF spectrum view AND there is not a significant 410 nm peak showing on Mains' FF spectrum view:
   i. Some cannabinoids are condensing after mains' collection point
   ii. Temperature of condenser should be decreased between 1-5°C slowly to allow for total recondensation of cannabinoids
c. If a 2nd pass will be performed:
   i. Aim to minimize the amount of cannabinoids (460 nm peak) in collected in heads
   ii. Reduce heads (410 nm peak) collected in mains' flask
      1. Do not spend too much time in pursuit of completely eliminated heads - the 2nd pass further separate these molecules.
d. **Difference on the Second Pass?** Yes.
   i. Tune condenser temperature so that the following are showing:
      1. FF on Mains' Collection Flask: Solely 460 nm signal
      2. FF on Heads' Collection Flask: Solely 410 nm signal
         a. If some 460 nm is showing that is okay

5. Main Body to Tails Transition

a. If a proper course and fine tuning procedure were performed then no action will be needed until the distillation is transitioning to tails
b. The decision to switch to Tails occurs:
    i. When 460 nm peak hits a minimum on the Wavelength View, then starts going back up. (See **Figure 5** of Appendix)
        1. To switch to this view, tap the Display button on the top-left corner of the Fraction Finder user interface.
        2. This will also correspond with the 460 nm peak shifting to the right slightly
    ii. It may also be observed that: the condensate has increased color/darkness in the distillate refluxing in the head and/or a spontaneous increase in mantle temperature
c. Close Isolation valve and collect Main body flask
d. If it is desired to collect Tails:
    i. Replace the main body flask with a new "tails flask" and collect tails
    ii. Increase boiling flask temperature to up to 200°C
    iii. Turn the condenser fluid temperature down to 70°C
    iv. See next section for what to expect during a tails collection
e. **Difference on the Second Pass?** No.

6. Tails Fraction

a. The tail fraction will initially show a 490 nm peak on the FF spectrum view (**Figure 6** of Appendix) of the tails' flask (what used to be the mains' flask).
b. The signal may shift even further to the right as the compounds such as lipids and waxes are distilled
c. **Difference on the Second Pass?** No.

Appendix

Conditions at the beginning of steps	Mains Isolation Valve Open/Closed*	Boiling Flask Temperature (°C)	Condenser Fluid Temperature (°C)	Distillation System Pressure (mTorr)
1) Assemble	Closed	Ambient	Ambient	Ambient
2) Initial Operations	Closed	Ambient	Ambient	Ambient
3) Course Tuning Split	Closed	155-165	155-165	<250
4) Fine Tuning Split	Open	175-185	155-165	<150
5) Tails Transition	Open	175-185	155-165	<150
6) Tails	Closed	195-205	155-165	<150

Table 1: *Closed position indicates that the condensed flow will bypass main body collection flask, and Open indicates that the condensed flow will be able to flow into main body collection flask

Figure 1: Example of Head's Flask Fraction Finder Spectrum View when "Fool's Gold" is first starting to appear (slight peak forming at 410 nm)

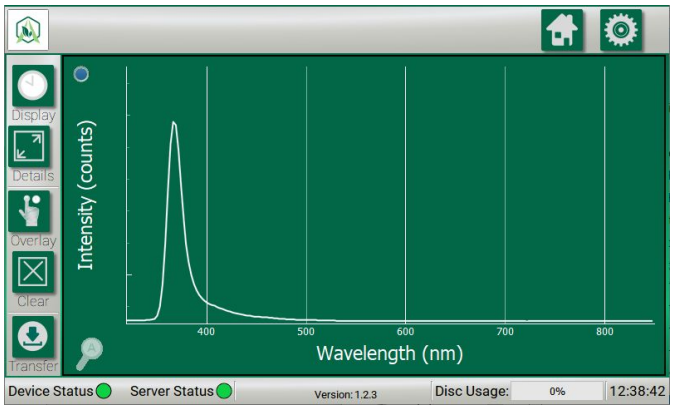

Figure 2: Example of Head's Flask Fraction Finder Spectrum View right before the isolation valve is opened to collect Main Body. Note the strong presence of both the "Fool's Gold" indicator (410 nm peak) and the cannabinoid indicator (460 nm peak).

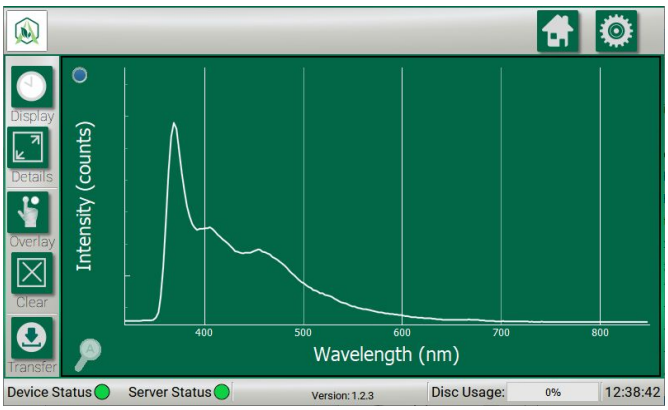

Figure 3: Example of Head's Flask Fraction Finder Spectrum View after the isolation valve is opened. Note that the majority of the signal is from the Fool's Gold indicator (410 nm peak) with a slight cannabinoid indicator.

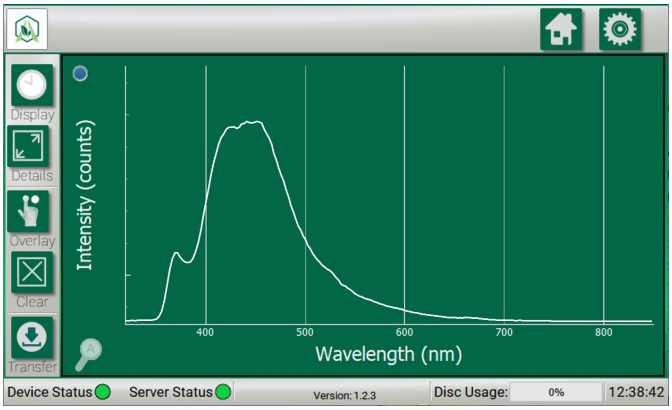

Figure 4: Example of Main's Flask Fraction Finder Spectrum View after the isolation valve is opened. Note that the majority of the signal is from the cannabinoid indicator (460 nm peak) with a minority Fool's Gold indicator.

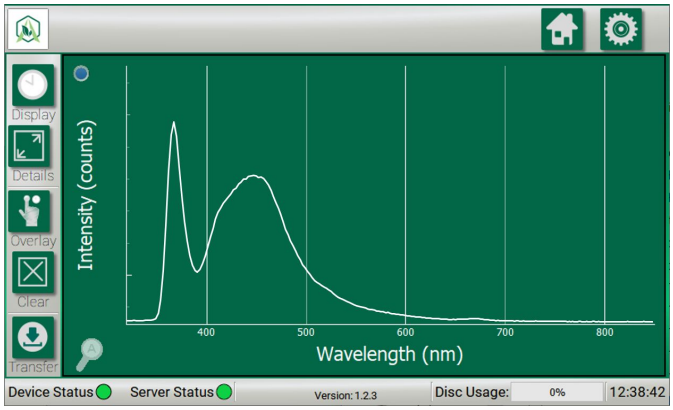

Figure 5: Example of Main's Flask Fraction Finder Wavelength View of the CBD indicator when the system is transitioning to tails, the display was cleared after all temperature tuning for the condenser and boiling flask. Note the sudden and consistent increase in the value on the right hand side of the plot, which indicates the tails transition.

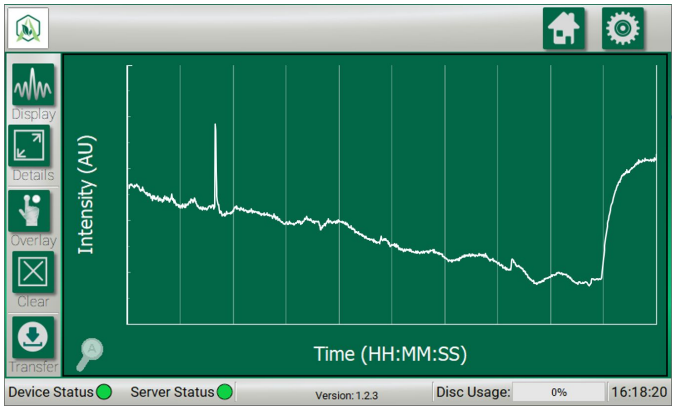

Figure 6: Example of Fraction Finder Spectrum View of tails. Note that cannabinoid indicator looks as if it is shifting to the right.