



THE IMPORTANCE OF
TEMPERATURE CONTROL



THE CANNABIS **WORKFLOW**

info@julabo.us
www.julabo.us
610-231-0250

JULABO USA, Inc.
884 Marcon Boulevard
Allentown, PA 18109

THE CHANGING TIMES

As more states legalize the recreational and medicinal use of cannabis and since the legalization for consumption in Canada, the number of processors is growing rapidly as they take advantage of the emerging market. In this growing industry, processors are using several established scientific techniques to isolate the desired cannabis components. All processes rely on standard techniques that have been used for years in the botanical, chemical and petrochemical, and distilled spirits industries. In general, cannabis processing relies primarily on first extracting the plant material and then isolating the extracts and components via evaporation/distillation. To effectively review these workflows, it is important to first understand the terminology.



The processes of success.

Extraction

During this process, a solid is placed in a solvent to remove soluble components. Making coffee or tea are common examples of extraction.

Distillation

In this process, a liquid component is isolated by selective heating, vaporization and condensation. Distilleries use this to make spirits from a mash mixture of fermented grains.

Vacuum Distillation

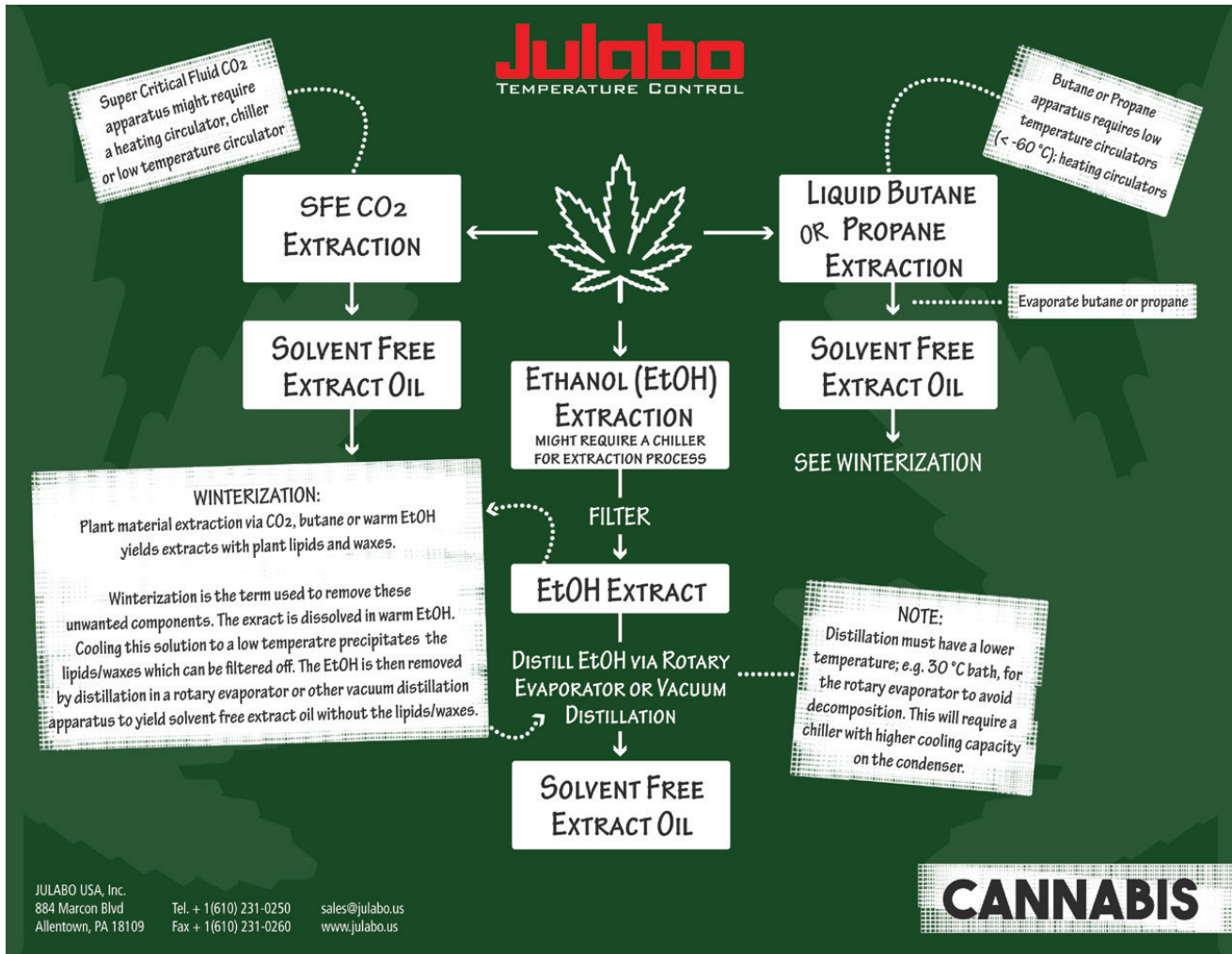
This process uses distillation under reduced pressure. Placing a liquid mixture under vacuum enables the distillation process to occur at a lower temperature. This lowers the liquid boiling point, increases the rate of distillation and reduces exposure of temperature-sensitive components (this eliminates unwanted degradation due to high heat exposure). A simple example of the difference pressure makes to the boiling point: Water at sea level boils at 212°F (100°C); in Denver, CO, it boils at 203°F (95°C) due to lower atmospheric pressure.

Decarboxylation

Enrichment of THC or CBD content is typically performed. Quantities of non-active THCA or CBDA convert to THC and CBD by exposure to heat. Heat facilitates decarboxylation (a chemical rearrangement which expels carbon dioxide) transforming THCA/CBDA to THC/CBD. For dry plant matter this is typically accomplished by taking chopped plant material and heating to 100 – 150 °C for enough time to facilitate the decarboxylation. For extracts, the decarboxylation process takes place after winterization by heating the extract oil to 100 - 160 °C.

CANNABIS EXTRACTION METHODOLOGIES

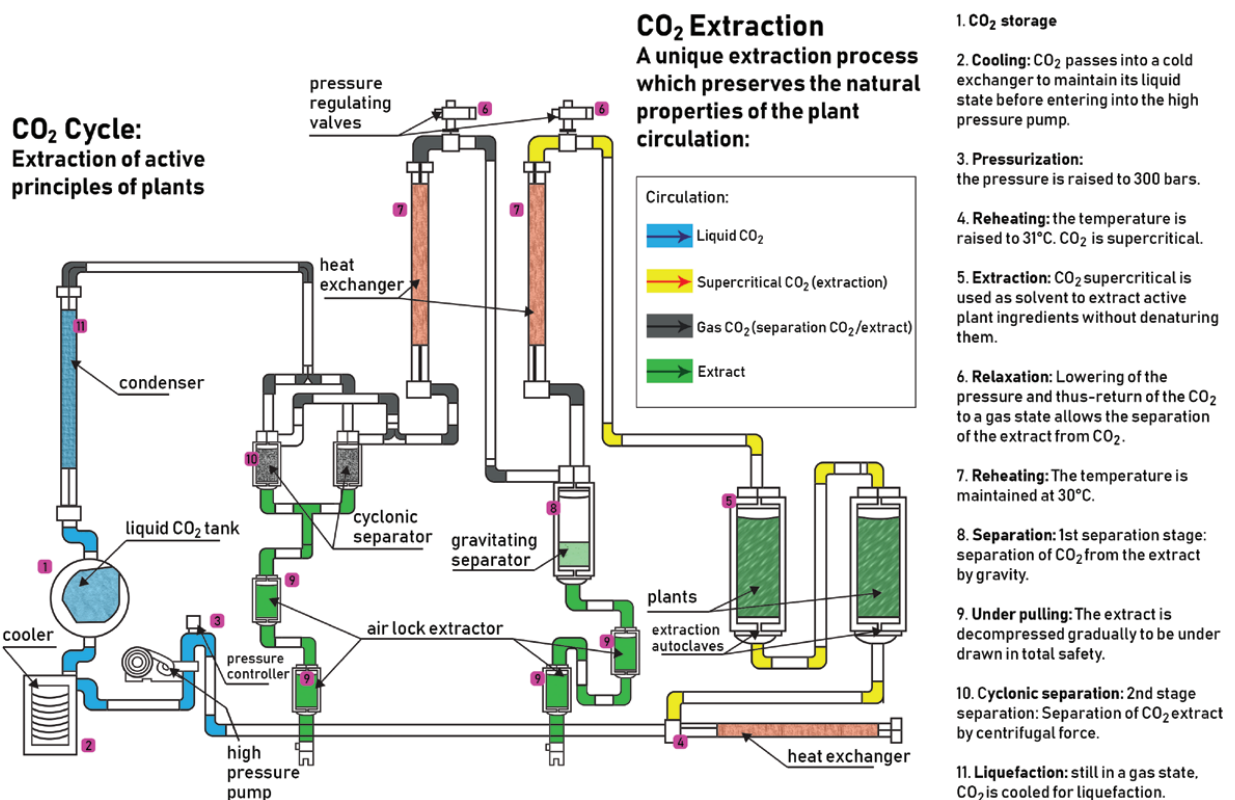
The cannabis market uses three main solvent extraction techniques. In all processes, the plant material is subjected to a solvent to remove active compounds from the plant matter and filtered to yield a solution of the solvent with plant extracts.



SUPER CRITICAL CARBON DIOXIDE

Systems that utilize carbon dioxide (CO₂) pressurize the CO₂ to a sub-critical or supercritical state. The CO₂ stream passes through a chamber containing cannabis material. The distillate can be isolated easily by reducing the pressure which evaporates the CO₂, leaving a cannabis extract with no solvent. Tweaking the temperature and pressure affords CO₂ systems the ability to yield extracts with a complete terpene profile. Sophisticated extraction apparatus can also incorporate fractionation, which enables process tuning to isolate desired components. Refrigerated chillers that are integrated into these systems facilitate recycling of the CO₂ by condensing the gas back to a liquid state. Recirculating heaters in the evaporator provide heating to 30°C to assist in removal of CO₂ from the extract. Consistent, accurate temperature control of these components is crucial to the operation of the process.

THE SUPERCRITICAL EXTRACTION PROCESS



LIQUEFIED HYDROCARBONS

Systems pressurize butane, propane or other low molecular weight hydrocarbons to a liquid state (like what you see in a butane lighter). The liquid hydrocarbon passes through a bed of cannabis material and filter, yielding an extract solution of hydrocarbon and plant extract. Like the CO₂ method, a reduction in pressure evaporates the hydrocarbon liquid, yielding a solvent-free plant extract. This method requires great attention to safety due to the flammability of the hydrocarbon used. Maintaining the pressurized hydrocarbon in the liquid state requires low temperatures. Recirculating temperature control units (TCUs) that provide cooling to -60°C (-76°F) and below facilitate this process. Heating circulators are also incorporated to increase the liquid butane evaporation when isolating the extract prior to recycling of the butane. In this process, it is crucial that the cooling and heating power of the TCUs meet the capacity requirements for the size of the application.



ETHANOL EXTRACTION

Food grade or USP grade ethanol is used as a solvent to extract plant material. This method varies from vessels to reactors, filter reactors, spinning vessels to barrels. A popular process has the ethanol chilled to $\leq -20^{\circ}\text{C}$ (-4°F) either in a cold room or freezer and then pumped into a container of cannabis. If a jacketed vessel is used to cool ethanol for the extraction process, a low temperature TCU acts as the cooling source. After a soak period, the ethanol solution is either filtered or the plant material removed in a 'tea bag' fashion. The resultant ethanol extract mother liquor is then concentrated by removing the ethanol. Typical distillation apparatus used to remove the ethanol include rotary evaporators, falling film evaporators or a batch vacuum distillation system.

EXTRACTION PROCESS RESIDUES AND WINTERIZATION

All extraction methodologies described above yield an oil once the solvent has been removed. This oil contains plant waxes, lipids, possibly chlorophyll, terpenes, THC/CBD and other cannabinoids. Additional processing to remove unwanted plant components is necessary to produce a more desirable extract product. Winterization is the term used to describe the process of removing the plant waxes, lipids and other undesirable components. Dissolution of the extraction oil in ethanol and chilling to temperatures $< -20^{\circ}\text{C}$ causes the unwanted components to precipitate. This cooling process is conducted in cold rooms, freezers or with jacketed vessels or jacketed filter reactors. Cooling jacketed vessels to $< -20^{\circ}\text{C}$ can be achieved with recirculating chillers. Filtration of this cold solution removes the unwanted components. The resulting ethanol solution is concentrated via vacuum distillation. The solvent-free, purified extract can be dried on trays in a vacuum oven to yield a yellow to amber extract. This product is typically formulated into products for the recreational cannabis market. The winterized residue must undergo further processing to yield high purity a high purity isolate for use in medicinal applications or various recreational formulations.

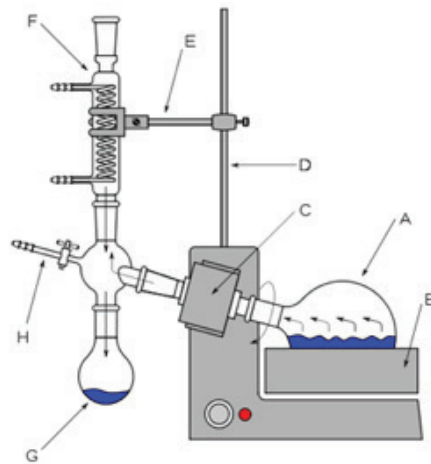
ROTARY EVAPORATION

Since its invention in 1950, the rotary evaporator has been a ubiquitous scientific tool for the use of solvent removal. The rotary evaporator enables the removal of solvent in a controlled manner under vacuum. Sizes range from bench top (to 5 L flasks) to pilot scale (20 L and up). Reducing the pressure in the rotary evaporator by a vacuum pump lowers the boiling point of the solvent to be removed; specifically, ethanol in the case of cannabis extract processing. Typically, the distilling flask (A) is filled to 50% volume. The water bath (B) is heated to 30-40°C. The condenser temperature (F), controlled by a recirculating chiller, is set to -10°C to 0°C. Once the water bath and condenser have reached the set points, the distillation flask is rotated from 150-200 rpm. This creates a thin film on the upper surface of the round glass flask, which increases the solution surface area and enhances the solvent evaporation rate. Applying an appropriate vacuum to the system (H) lowers the boiling point.

To achieve a recommended target, set the vacuum to achieve an ethanol vapor temperature of 15-20°C. As the ethanol evaporates, it will condense and collect into the distillate flask (G). Optimization of the parameters allows for easy reproducibility.

THINGS TO WATCH FOR: Increasing the evaporation rate by lowering the vacuum and/or increasing the water bath temperature can lead to capacity overload on the condenser. The evaporation rate can exceed the condensation capacity of the recirculating chiller. In this case, ethanol vapor will pass through the condenser and into the vacuum pump. Cannabis extracts require lower water bath temperatures to minimize thermal decomposition. Thus, the condenser temperature of -10°C to 0°C will require a chiller with adequate cooling capacity at those low temperatures.

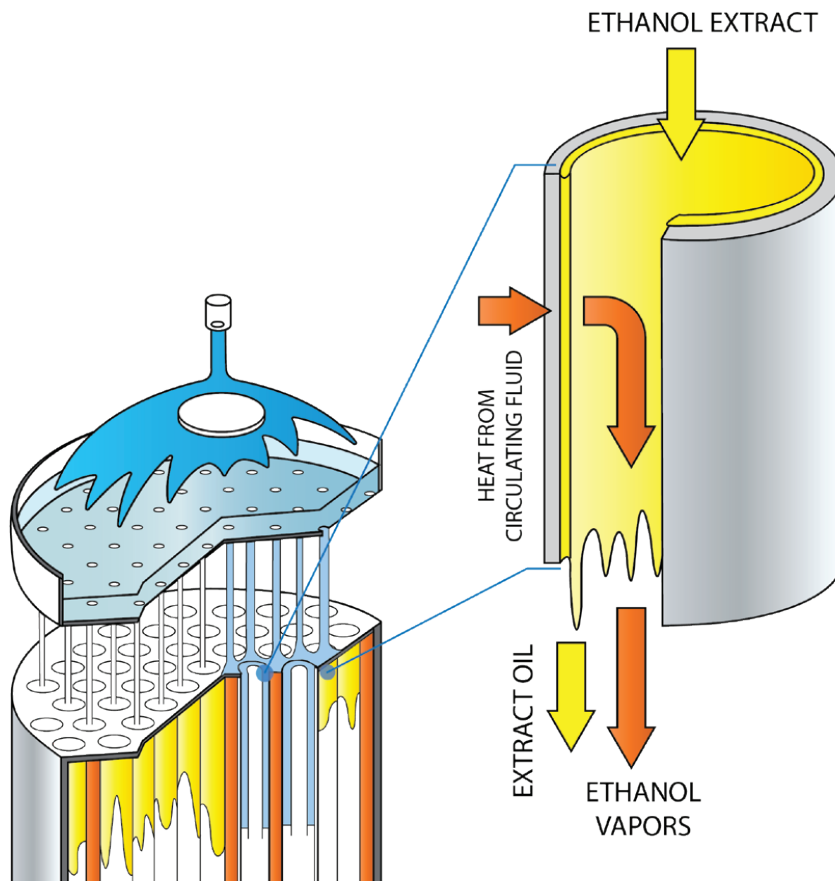
Rotary evaporator manufacturers have multiple options for automatic vacuum control and refilling accessories (manual and automated) to increase throughput.



Components of a rotary evaporator

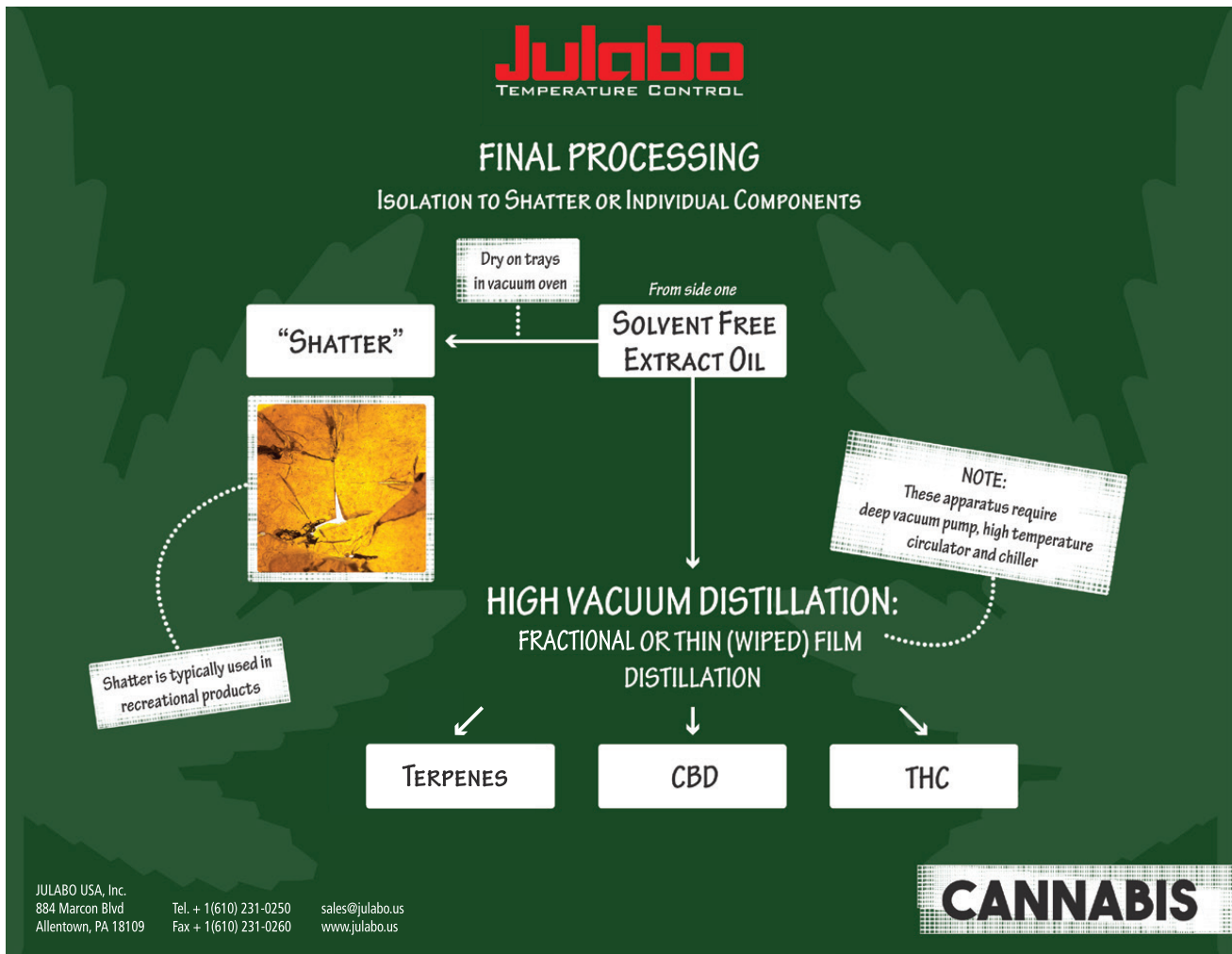
FALLING FILM EVAPORATION

Large volumes of cannabis ethanol extract can be concentrated utilizing a falling film evaporator. Falling film evaporators are essentially vertical shell and tube heat exchangers. While placed under vacuum the ethanol solution flows down an externally heated tube or tubes causing the ethanol to vaporize. The ethanol vapor collects in a condenser / cold trap while the higher boiling cannabis extract flows down the walls of the tube into a collection vessel. This method provides a high capacity evaporation rate with a short heat exposure time to the extract. Benefits of this evaporation method include limited heat exposure to the extract and the possibility of continuous operation. This apparatus requires properly sized heating circulators to facilitate the ethanol vaporization and chillers to condense the ethanol vapors.



VACUUM DISTILLATION

Isolation of high purity THC or CBD for medicinal and recreational applications requires additional processing. For CBD various cannabis plant strains now produce higher amounts of CBD or hemp can be used as the starting material. Reduction in THC content is important to eliminate the psychoactive high effect to yield a product rich in CBD for anti-inflammatory, anti-seizure and other indications. Distillation methods can isolate enriched component fractions of extracts. Since terpenes, THC and CBD have high boiling points (156-250°C; 312-482°F) distillation under atmospheric conditions is undesirable. Exposure to oxygen at these high temperatures can promote oxidation and prolonged heat exposure leads to thermal decomposition. By applying a vacuum, the boiling points are lowered. Vacuum conditions remove oxygen, thus eliminating product oxidation while lowering the boiling point temperature to lessen heat exposure.



VACUUM DISTILLATION



SHORT-PATH DISTILLATION:

A short path distillation apparatus with a multi-position receiver facilitates component isolation. The oil is heated in a flask under vacuum (typically with a magnetically stirred hot plate) with a short path distillation attachment. The condenser is cooled with a recirculating chiller to condense the component vapors. As the vapor temperature increases, indicating a new compound/mixture fraction, the multi-position receiving flask is adjusted to isolate the different fractions of terpenes, THC and CBD.



FRACTIONAL DISTILLATION:

To improve results over the short path method, a longer fractionating column can be installed between the vessel with heated oil and the condenser apparatus. This can consist of various types of columns (Vigreux, Oldershaw, etc.), enabling finer separation of the components. The added length of the fractionating column with protrusions, trays or packing material causes the vapor to equilibrate with the liquid state, thus providing a refined separation of the components.



WIPED FILM DISTILLATION:

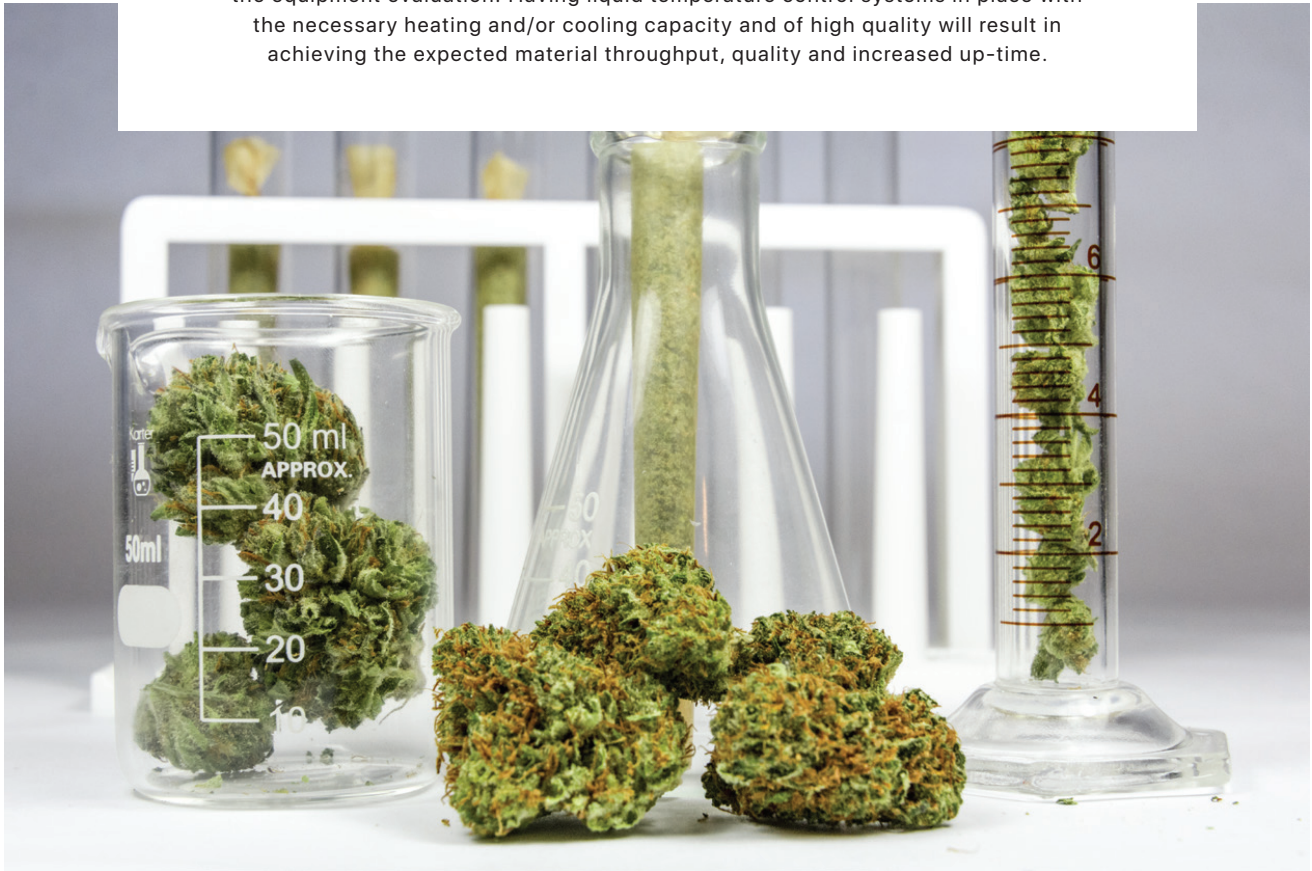
This variation of short path distillation can operate in batch or continuous modes. While under vacuum, the oil is introduced to the top of a heated vertical cylinder. As the oil enters the cylinder, it encounters rotating wipers or rollers that create a thin film on the heated surface. A long, slender condenser in the middle of the apparatus, cooled with recirculating fluid, condenses the vapor. Receiving vessels collect the condensate and the high temperature residue at the bottom.

Reduced exposure time of the oil to high temperature conditions is the key benefit of this technique. It can also increase productivity if the apparatus is configured for continuous mode operation. A recirculating heater provides temperature control of the feed container and outer jacketed wiped film body. Refrigerated circulators cool the condenser and vacuum trap. Process optimization of the feed rate, vacuum and temperatures must be conducted to yield the desired component composition in the distillate.

All vacuum distillation fractions might require further refinement by repeating the distillation process to achieve the desired extract purity and composition.

CONCLUSION

Temperature control plays a vital role in the cannabis extraction workflow. Attention to detail and optimization of the processing conditions from extraction to component isolation remain critical to maximization of yield and purity. Consultation with liquid temperature control equipment providers can alleviate future problems by understanding the method and scale used in the process to recommend the proper products from the beginning. Important: consider temperature from the beginning of the equipment evaluation! Having liquid temperature control systems in place with the necessary heating and/or cooling capacity and of high quality will result in achieving the expected material throughput, quality and increased up-time.



NEED HELP?

The JULABO team is here to assist with all your extraction, processing and heating/cooling needs.

[Click here](#) to speak with a Temperature Control specialist now.