WHITEPAPER

The Cannabis Workflow and the IMPORTANCE OF TEMPERATURE CONTROL

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As more states legalize the recreational and medicinal use of cannabis in the United States and 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. In order to effectively review these workflows, it is important to first understand the terminology.

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 or 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:

Prior to extraction, plant matter is often enriched to increase THC content. Quantities of non-psychoactive Δ9-tetrahydrocannabinolic acid (THCA) in the plant matter can be converted to THC by exposing it to heat. The heating causes the THCA to decarboxylate (a chemical rearrangement which expels carbon dioxide) forming THC. Similarly, heat treatment of CBD-A rich material decarboxylates forms CBD for medicinal purposes. For dry plant matter, this is typically accomplished by taking chopped plant material and heating to 100-150°C for a sufficient time to facilitate the decarboxylation. For extracts, the decarboxylation process takes place after winterization by heating the extract oil to 100-140°C.

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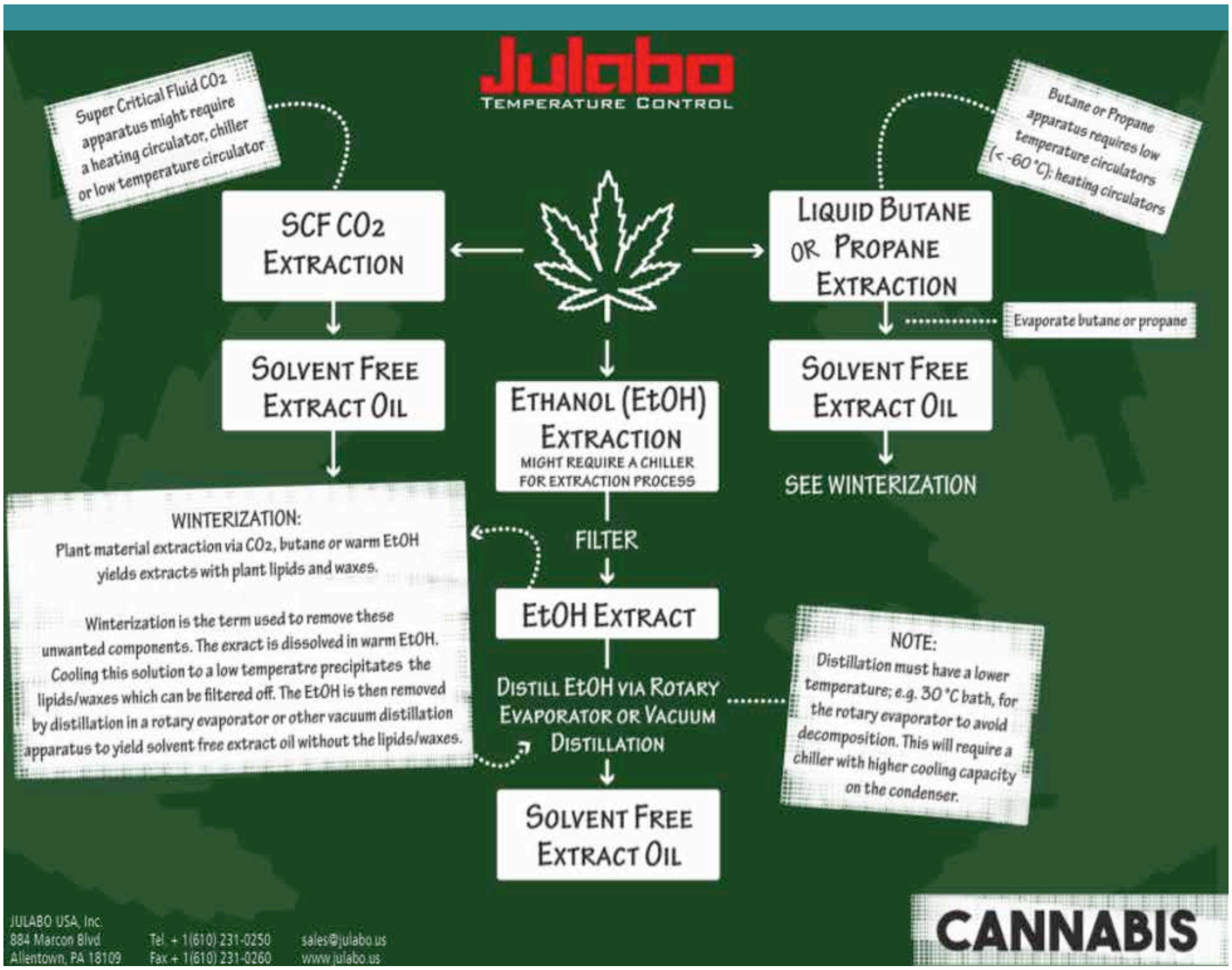
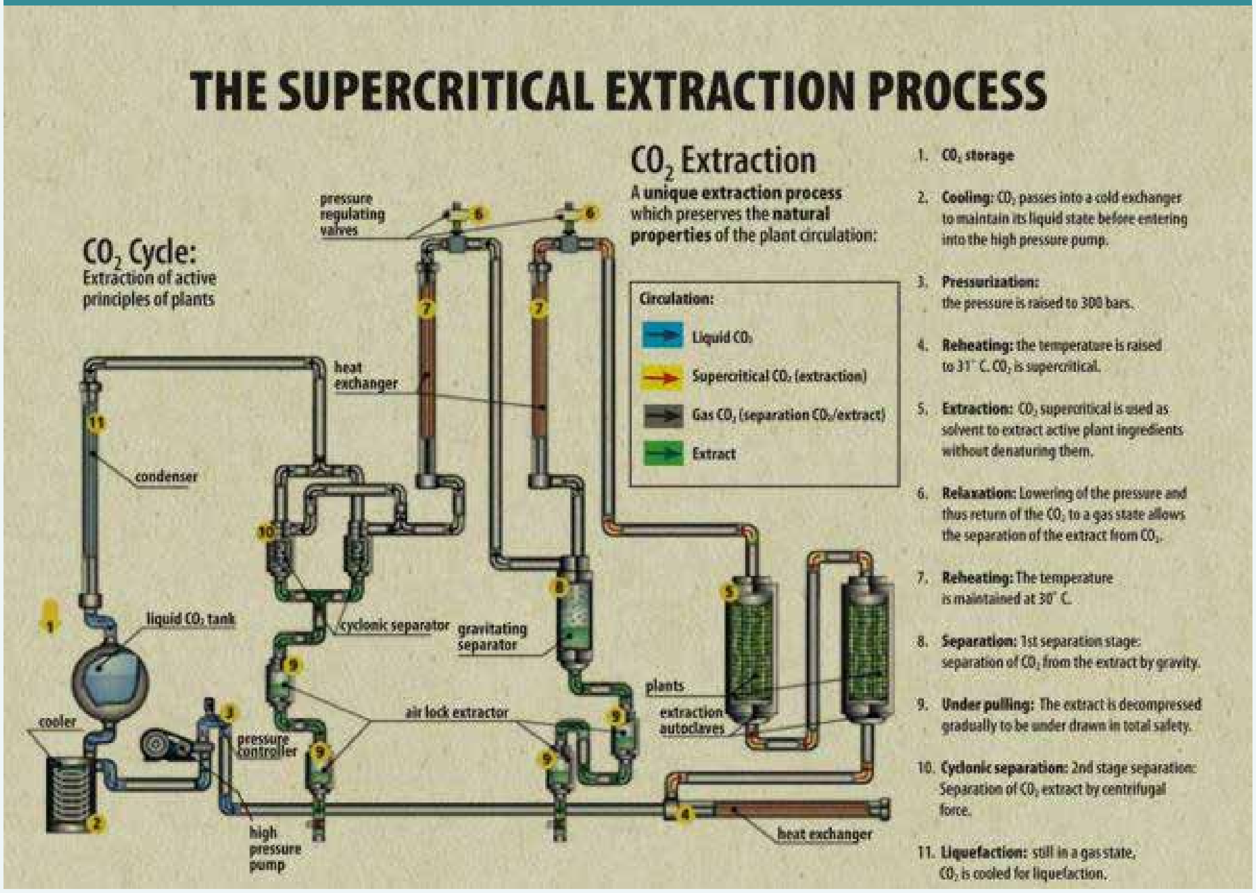
Cannabis Extraction Methodologies

The cannabis market uses three main 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 its supercritical state. This converts CO2 into a liquid that passes through a chamber containing cannabis material. The filtrate can be isolated easily by reducing the pressure that evaporates the CO2, leaving a cannabis extract with no solvent. Sophisticated extractors on the market can also incorporate fractionation, which enables process tuning to isolate desired components. Refrigerated chillers that are integrated into these systems recycle the CO2 by condensing the gas back to a liquid state. Recirculating heaters provide reheating to 30°C. Consistent, accurate temperature control of these components is crucial to the operation of the process.



Super Critical CO2 Extraction Example.

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 that can provide cooling to -60°C (-76°F) and below facilitates this process. Heating circulators are also incorporated to increase the liquid butane evaporation to isolate the extract and recycle the butane. In this process, it is crucial that the cooling and heating power of the liquid temperature control circulators meet the capacity requirements for the size of the application.

Cannabis Extract From The Liquefied Butane Process.

Ethanol Extraction:

Food grade or USP grade ethanol is used as a solvent to extract plant material. This can be used in a variety of vessels from reactors to barrels. A popular process has the ethanol chilled to <-20°C (-4°F) either in a cold room or freezer and then pumped into a container of cannabis. After a soak period, the ethanol solution is either filtered or the plant material removed in a 'tea bag' fashion. The resultant mother liquor of ethanol and extract is then concentrated by removing the ethanol. Typical distillation apparatus used to remove the ethanol include rotary evaporators or a vacuum distillation system. If a jacketed vessel or jacketed filter reactor is used to cool ethanol for the extraction process, a recirculating chiller acts as the cooling source.

Extraction Process Residues and Winterization

All extraction methodologies described above yield an oil once the solvent has been removed. This oil contains plant lipids, possibly chlorophyll, waxes, fats, terpenes, THC and other cannabinoids. Additional processing to remove the plant lipids and waxes is necessary to produce a more desirable extract product. **Winterization is the term used to describe the process of removing the plant lipids, fats and waxes**. Dissolution of the extraction oil in ethanol and chilling to temperatures <-20°C causes the lipids, fats and waxes to precipitate. This cooling process is conducted in cold rooms, freezers or with jacketed vessels or jacketed filter reactors. Cooling jacketed vessels to <-20°C can be achieved with recirculating chillers. Filtration of this cold solution removes the lipids, fats and waxes. The resulting ethanol solution is concentrated via vacuum distillation (batch process or rotary evaporator). The solvent-free, purified extract can be dried on trays in a vacuum oven to yield a yellow to amber crystal-like material called shatter. Shatter contains terpenes, THC and other cannabinoids. This shatter product is typically formulated into products for the recreational cannabis market. The winterized residue must undergo further processing for use in medicinal applications.

Rotary Evaporation

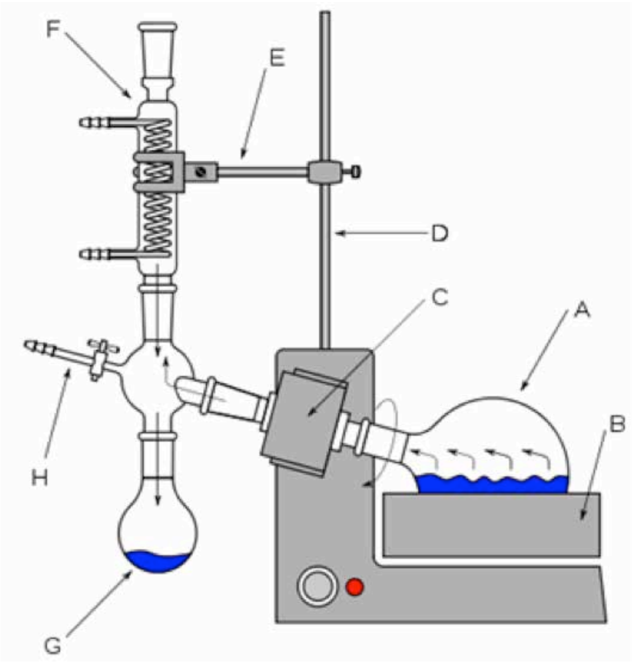
Since its invention in 1950, the rotary evaporator has been a ubiquitous scientific tool for the use of solvent removal. The rotary evaporator enables the removal of solvent in a controlled manner under vacuum. Sizes range from bench top (to 5 L flasks) to pilot scale (20 L and up).

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 glass cylinder, 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.

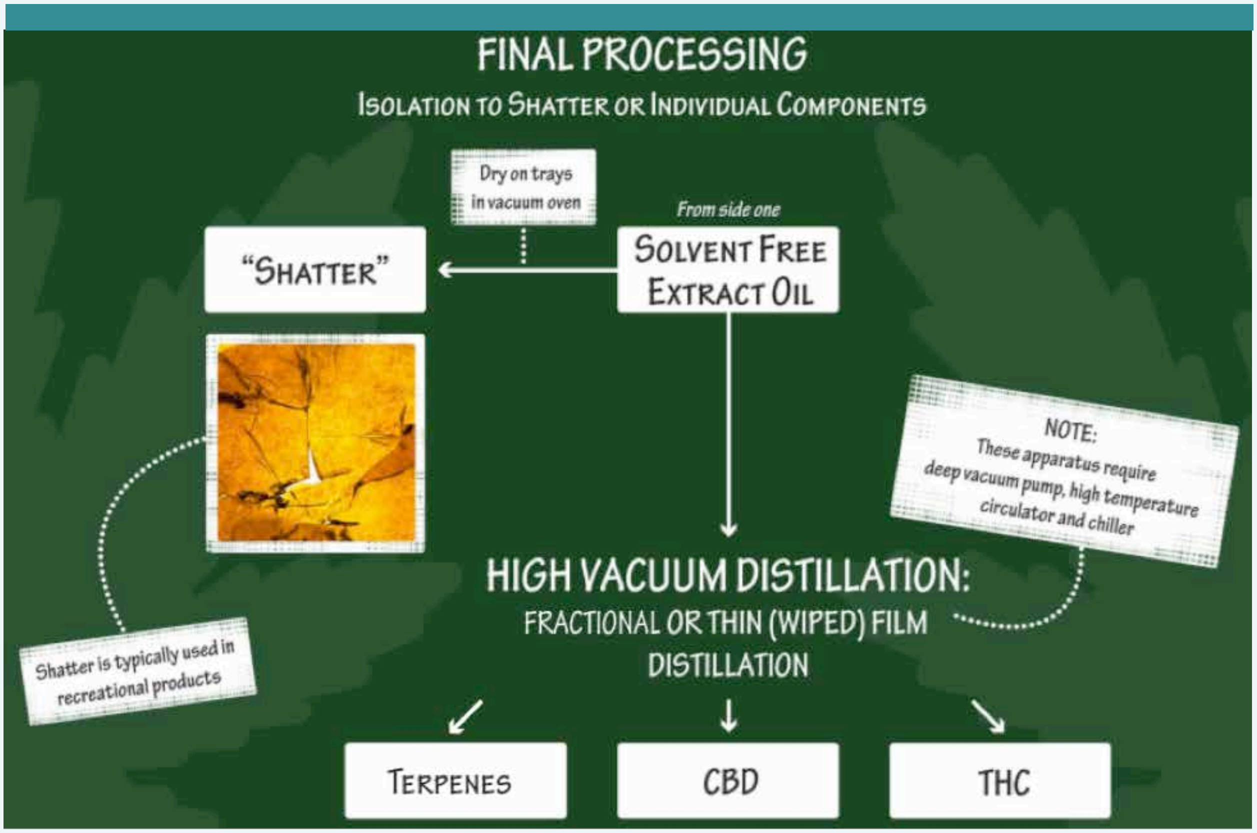
Things to watch for:

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.



Components of a rotary evaporator.

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



Vacuum Distillation

Isolation of CBD for medicinal applications requires additional processing to remove terpenes and lower THC content. Various marijuana 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.

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 provide cooling for condensation of 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.

Bench scale short path vacuum distillation set up with 3-position cow receiver.

Bench scale fractional distillation apparatus with reflux and Vigreux column.

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.

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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.

Short path wiped film evaporator.
Photo courtesy of Asahi Glassplant Inc.

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 to operate in a continuous mode. 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 purity and composition.

Creating The Right Systems To Maximize Quality, Throughput

Consistent, accurate temperature control plays a vital role in creating the most productive cannabis extraction workflow. Attention to detail and optimization of the processing conditions from extraction to component isolation is critical when maximizing yield and purity. Consultation with liquid temperature control equipment providers can alleviate future problems by helping processors understand the method and scale used in the process as well as recommending the proper products from the beginning. Having high-quality liquid temperature control systems in place with the necessary heating and/or cooling capacity helps processors achieve expected material throughput, quality and increased uptime standards.

NEED HELP?

The J-Team is here to assist with all your extraction, processing and heating/cooling needs. Click here to speak with a **Temperature Control specialist** now.

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