



extraction Magazine

EXTRACTIONMAGAZINE.COM

ISSUE 1 FEB/MAR 2018

2018

Brings Dramatic Evolution
of Extraction Business Model

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TO TERPENE EXTRACTION

SOLUTION TO POLLUTION
IS NOT DILUTION

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LETTER FROM THE EDITOR



There are few times when you get to participate in changing the course of history. There are few times when life is stranger than fiction. But it would be hard to write this script and have anyone believe it. Extraction Magazine will be actively involved in this rare opportunity. The goal is to stay current, stay readable, stay in peer-reviewed science with an informal style that doesn't compromise facts. We will endeavor to provide readers the information about all forms of extraction -- solvent and non-solvent-based technologies, as well as hybrid mixtures of solvents or modes.

The goal of Extraction Magazine is to interrupt and disrupt what people have believed is true without asking for peer-reviewed journals to support the evidence.

What is the BEST extraction? It is not about your preferred mode, but rather about what the company is formulating for their customers.

- B** Botanical Material's integrity maintained to assure the extraction of desired components in natural form
- E** Extraction efficacy, efficiency, effectiveness, and economics must be held in pre-defined boundaries and goals
- S** Safety and health of everyone throughout the workflow is essential criteria with goal of exceeding regulatory standards
- T** Testing with modern technology in every facet (e.g. hardware, software, and standard operating procedures, etc.)

It is from this platform that we will provide you relevant and timely information that you can use to maximize the quality of products.

We will explore the scale from nanograms to kilograms. Sample preparation before analysis in lab is also extraction, for example.

So, don't be surprised to find articles exploring sample preparation for analytical labs being explored as well as purification for isolates. Both of those are clear examples of extraction.

Business of science is critical to the success of moving the ball forward in this industry as much as any other.

This month we explore importance of density, solvent-centric focus on choices, tips on distillation, as well as the pressing of plant for rosin.

May you extract the best from your day,

John A. MacKay, PhD



CREATIVE

CONTENT EDITOR
JOHN A. MACKAY, PHD

CREATIVE DIRECTOR
RÉGIS SUDD

WRITING

Ben Amira; Yahav Blaicher; Jack Bohannan; Rob Brown; Christopher Cortina; Siobhan Danger Darwish; Connor Fitzhugh; Matthew Gates; Stephen Goldman; Hary Resin; Dr.

COPY EDITOR
STACY SIDLINE

PHOTOGRAPHY
MARK RUTHERFORD

WEB DESIGN
AAMIR IQBAL

BUSINESS

PUBLISHER MACE MEDIA

MEDIA ADVERTISING EXECUTIVES
MARK NOCKELS
CASEY WALSH

SOCIAL MEDIA
GINA BOULANGER

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28



32



35

CONTENTS

MARKET

2018 Brings Dramatic Evolution of Extraction Business Models 06

Asking what is the best extraction instrument for your business is the WRONG question 12

TECH

A Solventless Approach to Terpene Extraction 18

ASK THE DOC

Extraction of Cars from Peninsula 22

TIPS AND TRICKS

3 Common Distillation Mistakes 26

Solution to Pollution is NOT Dilution 30

3 Easy Steps to Using Heat and Pressure Technique for High Quality "Rosin" 36

Density: Supercritical Carbon Dioxide's Most Overlooked Variable 40

2018 Brings Dramatic Evolution of Extraction Business Models

John A. MacKay, PhD

THREE SIGNIFICANT CHANGES IN THE CANNABIS EXTRACTION MARKET 2017:

1. The number of companies that are directly or indirectly involved in the extraction segment had significant growth.
2. The vast diversity of products and accessories that supported multiple solvent choices increased in number. The new products ranged from simpler to complex.
3. The number of business model choices customers had for partnering with extraction companies or third-party companies expanded.

Cataloging the number of new companies and the diversity of new innovations would take weeks to describe and most likely be out date within an hour of starting to write the document. However, the number of ways that companies can take advantage of these innovations has also grown.

The business model five years ago was simple. You chose an instrument that met your needs, then agreed on a specification and price. After sending the vendor down payment (typically half of price), the vendor started to build the instrument. Next you waited until alerted that the instrument was built. At that time, you sent the other half of the money and received the instrument on the back dock. The vendor supplied a few days of installation and familiarization and you were on your own. On your own met with varying results and success. It depended a lot on the operator of the instrument more than the instrument itself. There were less

than 10 manufacturers at the time and uncountable number of “homemade” systems. Whether hydrocarbons, alcohols, supercritical, fluorocarbons, etc., there were only a few choices.

The business model started evolving in the past 18 months, both in scope and number of providers. I have broken it down into five general market segments that are relevant enough to discuss. The segments have broadened as the innovations and regulations are happening at a rapid pace. Customers are looking for ways to minimize having an instrument that is obsolete by the time it arrives. Some companies are finding ways to address the customer's concerns with some innovation in financing.

1. Purchase and sale agreement
2. Lease-to-own
3. Rental
4. Lease with shared risk/reward between vendor and company
5. Full service model based on shared risk/reward between vendor and company

The first three are easy to describe. The first, purchase, involves a straight transaction. While the customer might have different ways to secure the financing, the vendor just sees the money. The second, the vendor or the vendor with a financial partner, shares part of the financial risk of securing the financing.

The third, rental, is a more complex contract, as the number of months that the instrument is to be used and how to assure the delivery and subsequent pick up needs are very well defined to meet the expecta-



tions. The maintenance and use of the instrument falls on the user, and the vendor must have a price that allows for the depreciation and restocking of the instrument.

The fourth model merges a bit of the leasing and rental models in that there is a base price of the instrument that must be covered, and then an agreed-on price and a variable based on amount of oil produced, or on usage either in runs or time in use, for example, \$5 per gram of crude oil or \$400 per hour of usage. To assure that the vendor is making money on the instrument, there is a standard base price per month. For example, it might be \$20,000 per month or the variable of time would be a base of 50 hours per month that the instrument is in use. The variable of amount collected would be 4,000 grams (4 Kg) of crude oil.

The fifth model, is an enhancement of the fourth. At a minimum the vendor supplies an extraction instrument. However, more models are considering a full solution from plant to formulation. Here the risk and significant reward is shared for the productivity. The vendor is now very interested in the customer's success. The amount of potential that a plant can provide needs to be maximized for both parties to profit.

SO HOW DO YOU PICK WHICH SOLUTION MAKES SENSE FOR YOUR BUSINESS TODAY?

1. Always start with your served market – your patients/customers. What are the needs that you are satisfying? What is the formulation

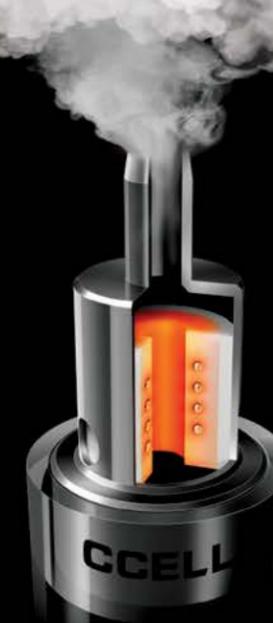
that you are going to provide them based on local regulations of manufacture in distributions? Write them down, including the recipes.

2. What is your realistic scaling for your operations? This is based on so many parameters that only your company can know. Is it 300 pounds per month of starting material or 300 pounds oil? Those are two very different solutions.
3. What are your current human resources needs to be up and running at maximum capacity?

THREE COMMON SCENARIOS:

1. If you already have been in the business for years and have a stable organization with experienced operators and standard operating procedures (SOP), and you may be looking to expand at your current production, then one of the first three choices may be optimal. You would not have a reason to need more help. If, however, you are expanding by opening a second facility locally or in another state, then having more local help at those locations would bring in four and five at least for the short term.
2. An expansion of the first is if you were exploring other solvents or non-solvent choices, e.g., CO2 to hydrocarbon or ethanol or the expansion of the pre- or post- extraction technologies to add productivity to process. This would be adding innovations in terpene

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extraction or enhancing decarboxylation yield before extraction.

- Starting a new facility and wanting to transition more quickly from small scale to large scale with the help of the vendor provides more than just instrumentation but also on-site, long-term assistance without the large capital expenditures for the first two years.

The market is filled with examples that are expanding to the first four segments. One is ekstraktLAB. Dr. Jon Thompson, president of the company, has been adjusting his financing options to serve the extraction market. "We have customers in each category. Some have completed rental agreements along with operating contracts and others just buy the equipment outright via cash or financing. We remain flexible." Thompson notes that the recent infusion of investment capital has changed the way most of his customers want to purchase the equipment. "We see 10-20 kg output oil per day with our popular extract 140 mid-size machines, so the extractor pays for itself in a matter of months. Investors do the math and generally want to own the equipment."

ekstraktLAB has also evolved its offerings beyond extractor equipment to eliminate bottlenecks in the entire process. "A typical financing package includes all the equipment to manufacture oils, not just extractors," said Conor Corrigan, sales manager for the company. "When newcomers to the industry think extraction, they typically don't realize that they need more equipment than just an extractor. Downstream and upstream equipment is required to be successful. Because our extractors are high throughput, we have had to develop new products to eliminate the bottlenecks. We found that they were struggling with filtration throughput, lower grade carbon black, pesticide contamination, and remediation. Our suite of products such as the ekstraktLAB, DrainDroyd, CarbonX, pure99 and igwLAB along with consulting, customer service and maintenance service help to produce an integrated, data-driven operation."

Most companies offer leasing, but not rentals. Andy Joseph President and CEO of APEKs Super-

critical, has offered that as an option for a couple years. He observed that given the "rental option, though it initially looked appealing, most customers opted for the lease to purchase. With the nearly 75 customers that took advantage of leasing, there were only a couple of companies that could not meet obligations of the lease. Compared to the early days when our company was funding it, there are now many more choices for third party leasing with better terms and conditions."

For true on-site extraction service Marij Pharmaceutical has fully equipped trailers that can be parked on site to help with extraction. Kim Edwards Vice President / COO at Acacia Diversified Holdings, Inc. has stated that there are customers that have set up permanent power for their returns, as well as some that are just permanently at hemp sites. The flexibility of having an asset move from one location to the next is important for planning as well as reducing the cost of having that service during parts of the year that it would not be idle.

Mike Citron, vice president of XtractNOW, said, "we are focused on just the fifth segment of the business. The customer cannot purchase the instrument. We provide the instrument and the resources to run the instrument and expand into the pre-extraction SOPs to help customers truly maximize their productivity. It is a win-win situation. With the shared reward, I am not focused on the risk. XtractNOW believes we can make a significant difference in someone's business within a week."

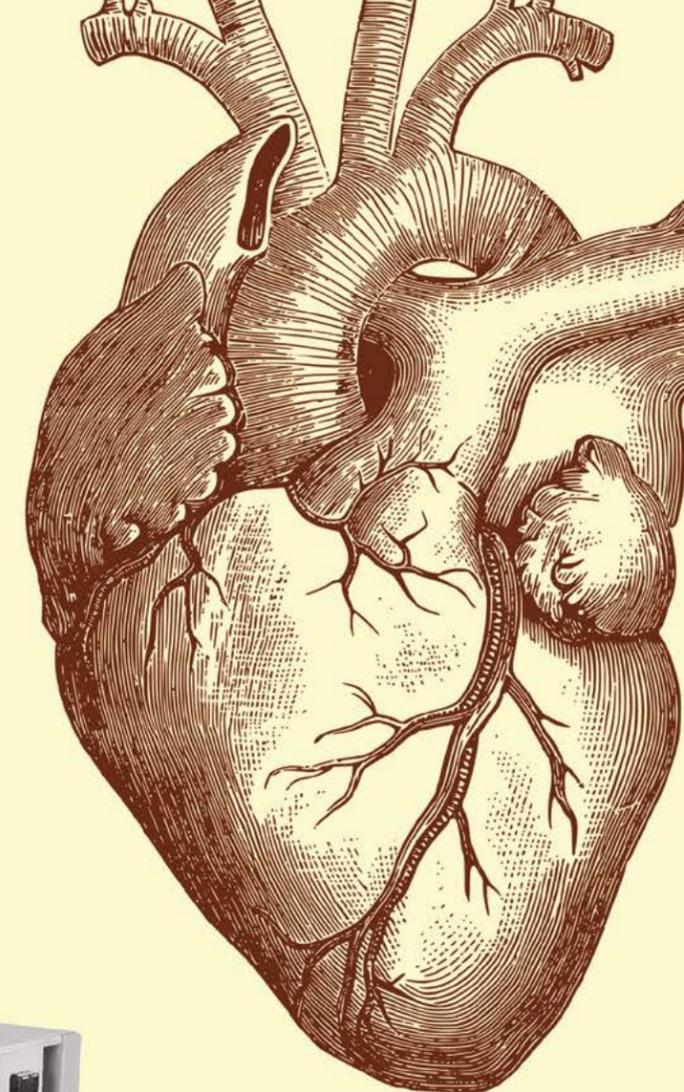
While carbon dioxide is the highest initial capital expenditure, the other modes of operation also provide more financing and on-site aid.

CONCLUSION:

The innovations and changes are happening quickly in the extraction processes. Companies are responding to the changes to help customers have a choice in their business models.

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Asking what is the best extraction instrument for your business is the **WRONG** question

David McGhee CEO TamiE inc
Inventor/Owner of the Tamisium Extractor Patents

When people ask about the best technique for extraction, the question they really SHOULD be asking is “what solvents should I consider for the formulations I want to provide patients?” The instruments that are invented are then “made for purpose” and certified by the individual state’s professional engineers¹ certified in that respective state. If an instrument has passed the rigorous testing for the design and material of construction, then you can be assured of its safety when used according to the manufacturer.

Simply put, in order of importance: what are the formulations producing; what are the compounds for formulation; and finally what is the cannabis chemotype (or chemovar)² to provide these in the quality and volume needed. If you don’t have these nailed down, then your chances of being successful are compromised. The material you use to extract compounds (and not extract – e.g., leave behind) determine your solvents, not the other way around. Another important consideration is the scaling of your business over the next few years.

While ethanol, steam distillation and carbon dioxide have been covered in many peer-reviewed articles, books, and on the web for many plant derived compounds, such as antioxidants, perfumes, vanilla, cocoa, decaffeinated coffee and hops, they are not the only option for cannabis, so I would like to discuss the facets of other volatile

hydrocarbon choices that meet the criteria above. Just as carbon dioxide isn’t the only way to decaffeinate coffee, CO₂ is NOT the only way to safely and effectively extract terpenes, terpenoids, and cannabinoids, etc., from cannabis chemovars. In fact, many times volatile hydrocarbons are significantly more effective and productive.

Some of the attributes of ethanol, acetone, or something similar in polarity range, are solvents that do not focus on a specific target and are not as volatile. They instead focus on a range of targets. I would say they do well at getting some of everything out, but not a lot of one thing.

Hydrocarbons are excellent solvents to consider in some formulations’ workflow. While sometimes these solvents are ignored or unduly maligned in the cannabis applications, there are some reasons why many companies choose them for this important part of their business.

1. The non-polar aspect of hydrocarbons matches the polarity of the compounds you want to extract and leaves behind the polar compounds you do not want.
 - a. Though it seems like a complex question involving chemistry and physics, it is not complex. Think about why you put a compound on your car that is a “wax”

(non-polar material) to protect against “water” (polar material). The decarboxylated cannabinoids and the volatile mono- and di-terpenes are like the wax – they are more non-polar than the natural carboxylated compounds that the plant synthesizes. So warm ethanol is polar, water is polar, n-butane is non-polar, liquid carbon dioxide is non-polar.

- b. Polar compounds like water also contribute to growth of mold, for example, which is why it is important to know the moisture of cannabis chemovar.
2. The hydrocarbons do not typically react to the composition of the cannabis chemovar.
 - a. If a solvent is going to degrade some of the compounds you want, or if it is reacting with other compounds to make a toxin, or if it’s toxic to humans or animals on its own, then it is not a viable candidate. Remember that it wasn’t long ago that coffee was decaffeinated only with chemicals like ethyl acetate and methylene chloride versus CO₂ as it is today. But there are still many processes that safely use methylene chloride³.
 3. Hydrocarbons have a lower boiling point than the cannabinoids and terpenes.





- a. A typical rule of thumb is that a solvent must be 100 degrees Fahrenheit lower than your target's boiling point. To avoid boiling your target away when removing your solvent, this number must be known and seriously considered. The easiest example is the flavor and fragrance market. The fragrance market would be destroyed if you were losing the volatile compounds by boiling it away when removing solvent in this manner.
- 4. Volatile hydrocarbons are commonly used and have well-documented regulations and procedures for where and how used.
 - a. Another misconception is that most solvents that match the polarity (non-polar) of most targets (non-polar) are flammable, so flammability is not a factor in choosing one over another. Labs and extractors are chosen based on their ability to deal safely with flammable solvents.
 - b. Recovery of volatile hydrocarbons is less complex to manage regarding when it is used to extract and when it is time to recover it. There are choices that allow it to be removed without the aid of electrical reciprocating pumps. A solvent boiling point that is used for removal is also the temperature it condenses back into a liquid. If too low, you will be forced to use a pump to compress the solvent because it would be impractical to use distillation due to the low temps the solvent would require for it to be condensed without the aid of a compressing pump.
- 5. In the event you have multiple targets with different target polarities, requiring dif-

ferent solvents, determine which solvent uniformly mixes well with more solvents to expand your targeting ability. If they don't mix, they will not uniformly pass through the plant material at the same time.

- 6. Hydrocarbon extraction requires minimal special pre-treatment of your material.
 - a. You may remove a lot of your target before you even start or create unwanted molecular changes with heat if applied during drying. Further oxidation may occur if time and air are used. Does heat time and air during drying alter the molecular structure of your target making excessive drying something you want to avoid?

It is also important to acknowledge your targets are volatile, so you don't want a system that is too low a pressure rating prohibiting you from using low-pressure volatile solvents, which may require a nominal pressure rating. The solvents to consider are butane (32F) isobutane (11F) or R134a (-15F).

You will most likely lean toward the lower pressure solvent n-butane⁴ see figure 1. N is for Normal, which means it is a straight chain without

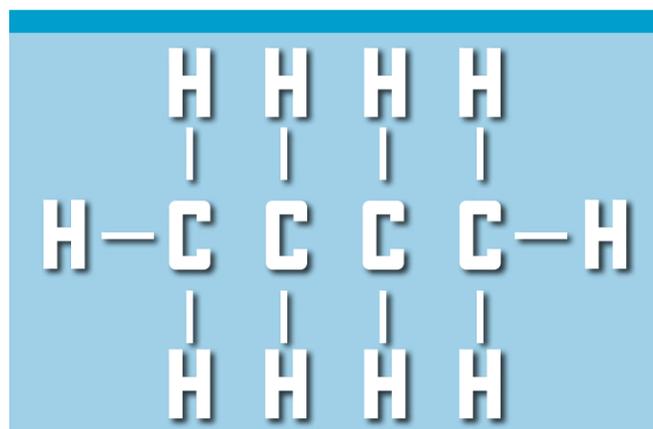
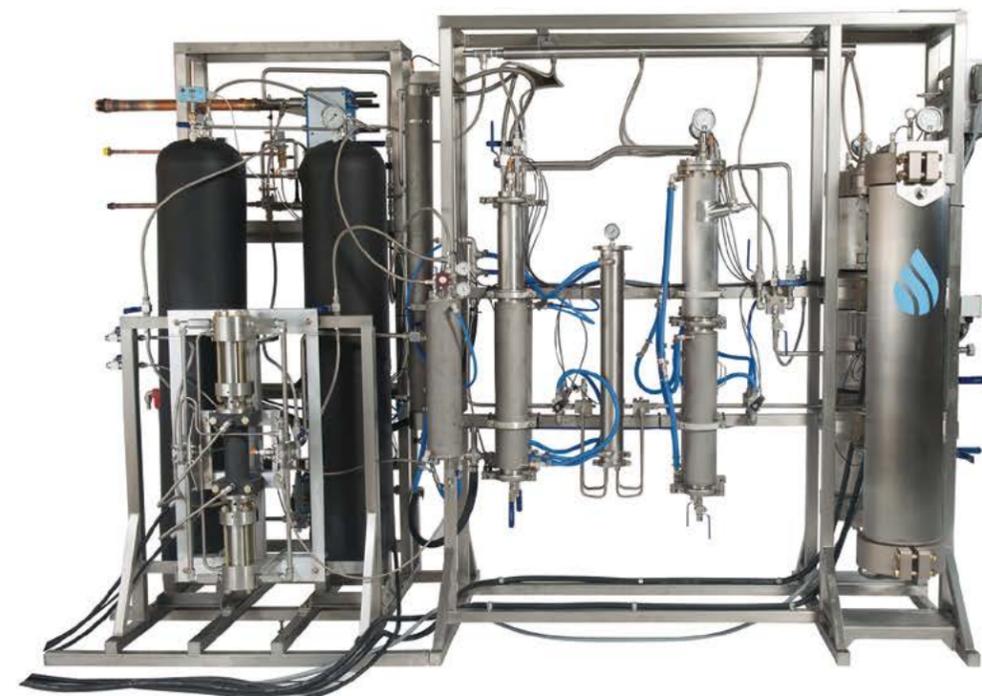


Figure 1 n-butane has 4 carbons and 10 hydrogens and is in a straight chain configuration

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any of the branching butane isomers. This distinction is important to make sure and ask about in your solvent choices.

All of which, by the way, are also flammable and mix well with butane and would be the same co-solvents or accompanying solvents that are mixed with any solvent such as CO2 to expand its targeting ability. Volatility of a solvent is important because it is imperative that your solvent's boiling point be much lower than your target. I have found 100 degrees Fahrenheit boiling point difference to be enough between your target and solvent to ensure that very little of your target evaporates when boiling your solvent away during solvent removal/recovery.

The general public often mistakes volatile⁵ to mean flammable⁶. Volatile simply means to vibrate faster and, in the case of evaporation, vibrate enough to float away as a vapor, making it more susceptible to flammability if it is combustible. Carbon dioxide is very volatile, but not combustible. The more volatile, the lower the boiling point, but also the higher the pressure requirement to contain it at all the temperature ranges you may want to use it. Scaling solvents by volatility forms a potential range of target requirements and how solvents meet or fail to meet those requirements.

Until those tests are considered or even regulated, these contaminants would not be evident. Although this could be prevented by distilling the

solvent, high-pressure, volatile solvents do not allow distillation easily or economically adding even more cost to an already costly process. This result encourages a solvent choice more favorable toward low-pressure solvents that can be easily recovered using distillation.

Right now, the only required test is for known contaminants, such as pesticides. As other contaminants are discovered expect testing requirements to change, which can include polyaromatic hydrocarbons⁷ (PAH, or PNA or PCA) that have toxic limits in the parts per billion ranges. At that time, the way solvents are obtained or transported will change your allowed solvent choices once again. The source matters for even carbon dioxide. Is it from plants or fossil fuel? What are the actual residual solvents in the CO2 tank? Solvents that are more easily cleaned using low-temperature distillation are going to be the least affected by these changes.

Standard testing does not find every known chemical, toxin or carcinogen. Testing labs need to have a target to look for. Testing for a full array can be costly and avoided if not required.

The industry as a whole will continue to evolve, and solvent choices and extraction abilities are an important part of that process. Opinion will lead the way shaping many if not all other facets of the industry. The extraction lab is the first place it is all gathered, sorted, weighed and distributed in a standardized way.

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A Solventless Approach to Terpene Extraction

Cannabis Sativa plants produce and accumulate terpene-rich resin within the secretory cells of glandular trichomes. Monoterpenes and sesquiterpenes are important components of Cannabis resin as they contribute to the unique attributes of different Cannabis strains. Terpenes are responsible for the plant's aroma and flavor. They possess specific medical properties and may act synergistically with cannabinoids. The extraction of terpenes and cannabinoids from Cannabis is a function of their solubility in different organic solvents. Solvents like methanol, ethanol, butane, and hexane are commonly used in Cannabis extractions. However, aside from safety considerations, extracts produced with such solvents are considered "one-pot extractions"; no selectivity between cannabinoids and terpenes can be achieved. This is problematic for two reasons. First, during post-processing, thermally liable compounds such as terpenes undergo degradation reactions. Second, isolation of terpenes from these solvents proves to be difficult because of similar vapor pressures. Among the various extraction techniques, we explore the use of supercritical fluids as a solvent for the targeted extraction of terpenes from cannabinoids in Cannabis.

Data suggests that the interaction between cannabinoids and terpenes affects the pharmacological properties of different cannabis strains. This relationship is commonly referred to as the "entourage effect." There are several promising applications based on the combined use of cannabinoids and terpenes, such as combining CBD with the monoterpenes limonene, linalool and pinene to treat acne or adding caryophyllene, linalool and myrcene to 1:1 CBD/THC extracts to treat sleeping disorders.

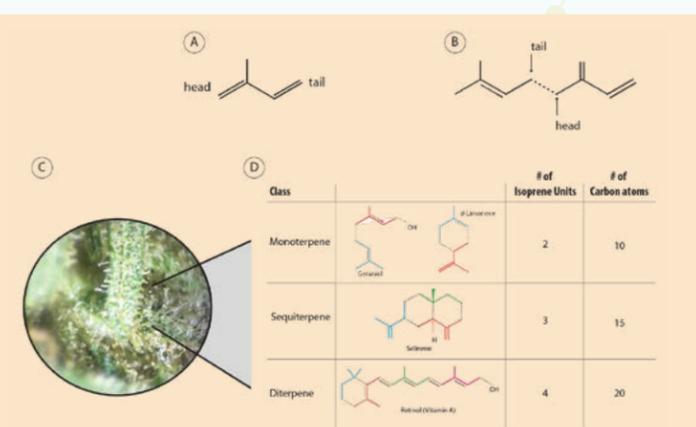


Figure 1. (A) Five carbon isoprene unit (B) head-to-tail linkage of two isoprene units to form monoterpane Myrcene (C) terpene-rich resin within the secretory cells of glandular trichomes (D) classification of terpenes based on the number of isoprene units

The smallest and most volatile terpenes are monoterpenes, which are biosynthesized by the head-to-tail addition of two isoprene units (Figure 1). An isoprene unit is a 5-carbon molecule on which terpenes are built. Combining three isoprene units forms a class of compounds called sesquiterpenes, which are less volatile than monoterpenes. The largest and least volatile terpenes are biosynthesized by the joining of 4 or more isoprene units.

Terpenes are both volatile and thermolabile compounds; they easily oxidize or hydrolyze depending on their respective structures. The environment in which Cannabis is stored and the methods used in processing will affect the chemical composition of these isoprene contain compounds. Post-processing methods that require heat lead to the accelerated degradation of terpenes.

For example, distillation of crude cannabis oil presents two problems. First, delicate monoterpenes readily thermally degrade. Second, organic solvents co-elute with the terpene fraction.

“Any remaining vapors which escape both internal and external condensers and which may contain small amounts of solvents, water or lighter terrene components, are collected in a cold trap....”

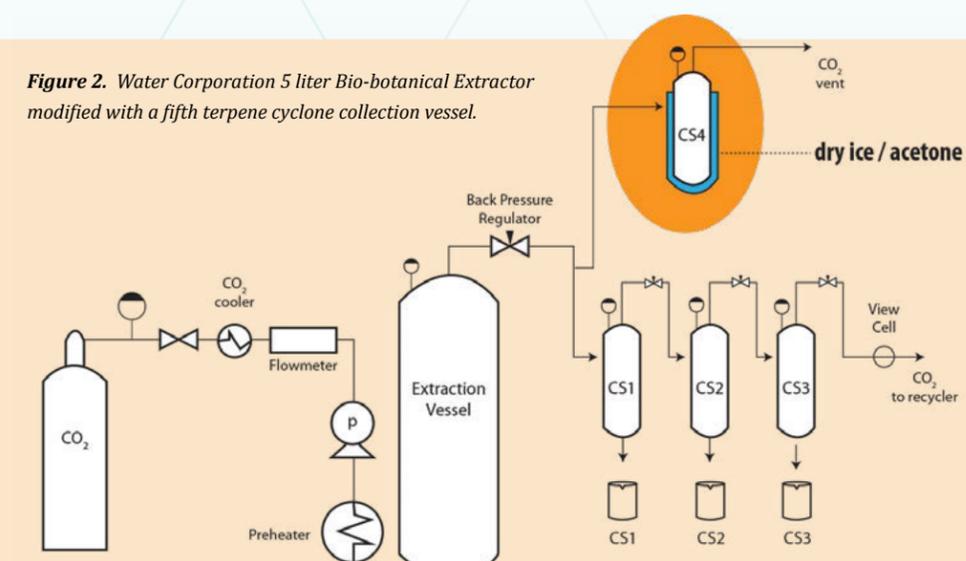
If this terpene fraction is used in Cannabis oil formulation, these organic solvents will contaminate the final product.

Supercritical fluid extraction is an effective method for the separation of monoterpenes from sesquiterpenes, their alcohol derivatives, and cannabinoids. The main supercritical (SC) solvent used is carbon dioxide (CO₂). CO₂ is inexpensive and is a GRAS (generally recognized as safe) solvent. CO₂ reaches supercritical state at conditions of 31 C and 74 bar and returns to a gas state once exposed to ambient conditions. This allows for simple solute recovery and results in a solvent-free extract. By modifying the pressure and temperature of the CO₂ system, the strength of the solvent can be adjusted, enabling a selective extraction of cannabinoids and terpenes.

SUPERCRITICAL EXTRACTION EQUIPMENT AND TERPENE EXTRACTION PROCEDURE

A terpene extraction optimization experiment was conducted using a Waters Company 5L Bio-Botanical Extraction System modified to include a 4th

Figure 2. Water Corporation 5 liter Bio-botanical Extractor modified with a fifth terpene cyclone collection vessel.



terpene collection vessel (CS4). This system has a maximum operating pressure up to 600 Bar; the extraction pressure is maintained by an automatic backpressure regulator (ABPR). An electrical jacket controls the extraction vessel temperature. There are three collection vessels (CS1-CS3), each independently heated and provided with manual pressure control. The high-pressure pump delivers a maximum mass flow of 200 g/min. The solvent flows through a heat exchanger to bring the liquid CO₂ to supercritical state before entering into the extractor vessel. The supercritical stream dissolves the target components from the botanical material and is directed from the extraction vessel to the appropriate cyclone separators. The optimized conditions are summarized in **Table 1**.

Compounds within the class of cannabinoids and

terpenes have varying solubility in SC CO₂. The extraction of terpene-rich hemp is divided into 3 stages. The density profile of SC-CO₂ is increased at each stage to target different compounds.

To extract monoterpenes the flow of SC-CO₂ is directed into CS4 bypassing the cyclone separators 1 through 3. Without the addition of CS4, volatile monoterpenes evacuate with SC-CO₂ from SC3 into the CO₂ recycler. With the addition of CS4, it is possible to create conditions that allow for the recovery of volatile monoterpenes. When CS4 is at ambient temperature, volatile monoterpenes evacuate with gas CO₂ and terpenes are lost. To remedy this, CS4 is cooled to -78 with a solvent mixture of acetone and dry ice. Once chilled, the extraction vessel is pressurized to 70 bar and extraction occurs at 50 C. Mono-

terpenes have high solubility at these conditions, while sesquiterpenes have mild to low solubility. In Stage 2, both temperature and pressure is increased to target less volatile terpenes or sesquiterpenes. During this stage, the flow of SC-CO₂ is directed into CS1 and continues through CS3. After 45 minutes, sesquiterpenes are col-

lected from CS3. In stage 3 of the extraction, the pressure parameter is increased to 300 bar for cannabinoid extraction. Total extraction time for monoterpenes was 45 minutes, extraction time for cannabinoids was 5.5 hours, and the total extraction efficiency was 92%.

	STAGE 1	STAGE 2	STAGE 3
Extraction Temperature (C)	50	55	55
Extraction Pressure (bar)	70	150	300
Flow Rate (g/min)	100	150	200
Extraction time (min)	45	45	45
CS1 Pressure (bar) / Temperature (C)	N/A	103 / 50	103 / 50
CS2 Pressure (bar) / Temperature (C)	N/A	65 / 45	65 / 45
CS3 Pressure (bar) / Temperature (C)	N/A	51 / 35	51 / 35
SC4 Pressure (bar) / Temperature (C)	ambient / -78	N/A	N/A

Extraction Time Terpenes 45 min
Extraction Time Cannabinoids 5.5 hours
Extraction Efficiency 92%

Table 1. Optimized Supercritical CO₂ extraction conditions for terpenes found in terpene rich Hemp.

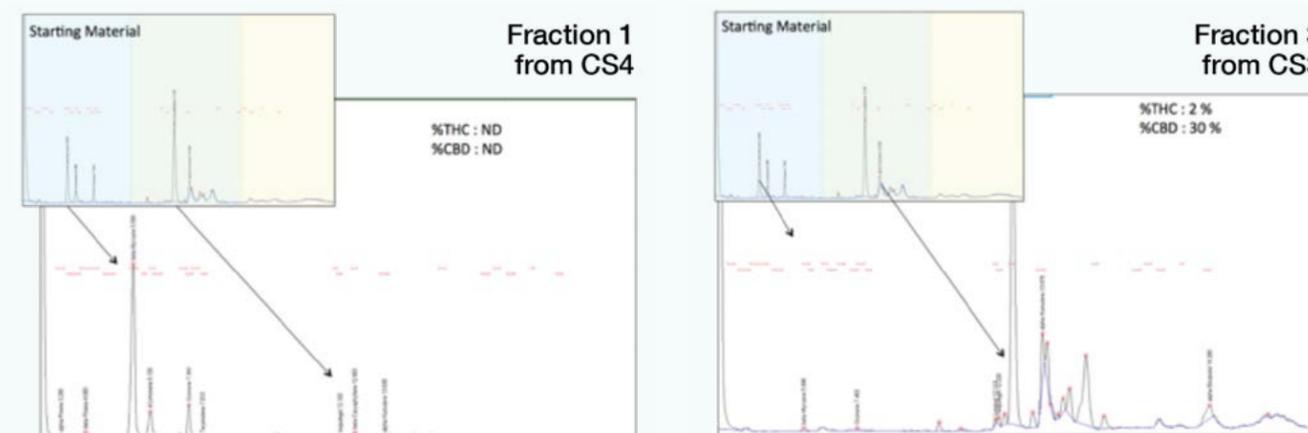


Figure 3. Left chromatogram: Monoterpene fraction obtained at extraction conditions of 70 bar and 50C. Right chromatogram: Sesquiterpenes obtained at extraction conditions of 150bar and 55C.

GENERAL CONCLUSIONS

- #1. With the addition of SC4, Supercritical CO₂ is an effective solvent for the extraction of terpenes from Cannabis. The ability of SC-CO₂ to return to a gas state once exposed to ambient conditions allows for simple terpene recovery and results in no detectable residual solvents.
- #2. Monoterpene fractions are obtained from CS4 with high purity and no detectable cannabinoids. Plant waxes and cannabinoids co-elute with sesquiterpenes, which are collected from CS3. Due to the

more robust nature of sesquiterpenes, these compounds can undergo post-processing methods such as winterization with minimal to no degradation.

- #3. The ability of separate monoterpenes from sesquiterpenes and cannabinoids allows for post processing of cannabinoids without the danger of terpene degradation. Once cannabinoids are purified, both cannabinoid and terpene fraction can be reintroduced yielding a full-plant, high-terpene extract.

EXTRACTION OF CARS FROM PENINSULA



What is extraction? Where do the compounds come from? How do you know when you are completed?

- A. Separation of one compound from at least one other
- B. Mostly from trichomes and most in globular trichomes
- C. You monitor your starting material and extracted material and account for all the compounds

All of these great questions are difficult to answer in less than a 90-minute lecture with photos, charts, complex equations and discussions about which is the best mode.

First, mode does not change the completeness or speed of the extraction. Pick any extraction mode and the process is the same, the results may vary, and the overall process is common.

So, I am going to take significant liberties with chemistry, physics and mathematics as well as most attributes associated with common sense to consider these questions.

Problem at hand: You need to evacuate the peninsula as soon as possible. While there are many types of vehicles that need to be evacuated, you are only interested in the evacuation of three similar cars. They will be distinguished by color: red, blue and green.

Some assumptions to my emergency car evacuation (extraction):

- There are 10,000 vehicles on the peninsula.
- They are in 100 identical parking garages that are located on the crossroads of the avenues that run east-west and the streets that run north-south.
- The blocks are all one-tenth mile squares.



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- There is a single bridge connecting the peninsula to the main land.
- 30 percent are red cars, 20 percent are blue cars, and 10 percent are green cars, and are uniformly distributed in each parking garage. Therefore, based on 10,000 total number of cars there are:
 - o 3,000 red cars
 - o 2,000 blue cars
 - o 1,000 green cars
- Based on some obscure rule (my game, my rules):
 - o ONLY a maximum of 20 red cars can go across bridge per minute
 - o ONLY a maximum of 10 blue cars can go across bridge per minute
 - o ONLY a maximum of 4 green cars can go across bridge per minute

What would the evacuation look like? Based on the information above the rate of evacuation would be constant for each color vehicle until they are all across the bridge. Chart 1 demonstrates this process.

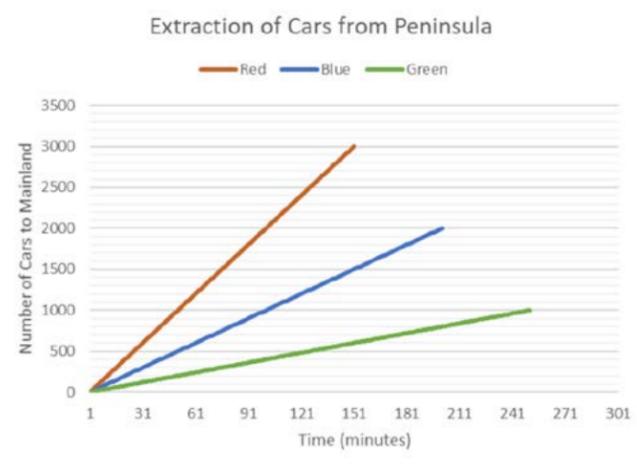


Chart 1. Rate of Extraction of Vehicles from Peninsula to Mainland

So, based on this model, even though there were three times as many red cars and green cars, the red cars were off the peninsula first. That is because the rate that the cars were coming off the island was five times faster. The evacuation time was 150, 200 and 250 minutes respectively for the red, blue and green cars.

WHAT HAS THIS GOT TO DO WITH EXTRACTION OF CANNABIS?

Based on my criteria there was a difference in the rate of extraction between the cars. This is called

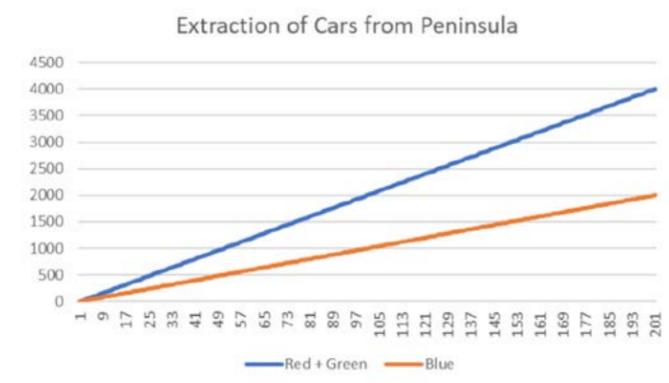


Chart 2. Evacuation of Cars from Peninsula After Conversion of Green to Red

kinetics. It is based on many factors, but mostly how much of a compound is soluble in a specific solvent.

Think of iced tea. If you have tea and put ice in it AND then add sugar, what happens to the sugar? Sure, it falls straight to the bottom. Well, not ALL of it, because in that solvent (hopefully water), the sugar is a little soluble.

Think of hot tea. If you have tea in boiling water AND then add sugar, what happens to the sugar? Sure, it goes right into solution.

Let's change the rules.

We are going to convert the green cars to red cars. This change now increases the number of red cars from 3,000 to 4,000. The number of blue cars will remain the same.

The total evacuation has been reduced from 250 minutes to 200 minutes. Of course, it takes more time to remove the red cars (150 minute to 200 minutes), but the total time of the evacuation is reduced by 50 minutes (reduction of 20%).

Alright let's compare and ask some other questions.

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3 COMMON DISTILLATION MISTAKES

Lilibel de la Puente



Every day, operators must make sure their equipment is running at peak performance levels. Although fine-tuned advanced skill is of ultimate importance, so too is the basic maintenance of a system. I've put together 3 common and basic distillation mistakes that I made and learned from.

1. VACUUM SELECTION

Proper vacuum pump selection for distillation is paramount. Lower vacuum levels are necessary to avoid temperatures that decompose cannabinoids and other compounds. True "molecular distillation" happens below 0.01 torr¹, and few operators out there are distilling at this level due to the gas loads put on their system by the cannabis oil. However, one may achieve a perfectly adequate distillation level by knowing and caring for their vacuum pump appropriately.

First, understanding the following components when selecting a pump:

- It should be a mechanical rotary vane or scroll pump. Scroll pumps do not require changing of vacuum oil and some are chemical resistant. Most people use rotary vane pumps, and I will discuss the finer points of these.
- Look for the following when selecting pumps: CFM, Ultimate Vacuum, Gas and Ballast settings. CFM= Cubic feet per minute (ft³/min). This tells one how fast the vacuum can remove gas load (terpenes/solvents) from the system. The better "de-gassed" one's material, the less CFM one may need.
- Gas Ballast settings: Vacuum manufacturers

post graphs showing how ultimate vacuum changes in relation to having the Gas Ballast on. One way to mitigate a large gas load on one's system is to open the Gas Ballast. Opening the Gas Ballast will evacuate volatiles through the exhaust port of the pump. However, vacuum levels are sacrificed.

- Another setting on some pumps is the High Throughput vs. High Vacuum settings. High Throughput is suggested for long distillations. One must use their own expertise and judgement to optimize their vacuum settings.
- Use a diffusion pump in series with a mechanical pump. This will help achieve deeper vacuum levels. The diffusion pump may not be used above 400mtorr. Generally speaking, a well-maintained empty vacuum system will achieve 95-120mtorr on mechanical pump, and down to 5mtorr with diffusion on.

2. IMPROPER INSULATION

The heating and insulation of the distillation apparatus will make or break one's distillation process. Even the best heating mantle will become inefficient if one does not insulate properly. The first step to insulation is to recognize that the fluid will not stay in the gas state unless encouraged to. Although the heating mantle may be at the correct temperature, if one is using a simple pot still, a hemispherical mantle will greatly decrease unwanted reflux to the distillation head. Wrap heat tape around the distillation head and make sure it's HOT. Aluminum foil is a friend! Use it to cover the small areas where the heat tape and mantle don't reach. Continue to increase the temperatures as you increase the mantle temperature.

For fractional distillation, keep in mind that this is like doing a simple distillation hundreds of times over. One may or may not insulate the column depending on what one is distilling. If the fractional distillation appears to be proceeding normally (the high-boiling point material is condensing), insulation could potentially prevent that material condensing and it would come over with the lighter components. However, if one is distilling substances with higher boiling points, insulation may help the vapors reach the top of the column and separate.

If the process is taking place on a wiped-film evaporator, make sure that all oil entering the still body is thoroughly pre-heated prior to contact with the heating bands or oil jacket. This serves two purposes: making the fluid less viscous for better wiping along the body surface; and degassing any remaining volatiles, which can splatter onto the internal condenser, causing streaking of dark material.

3. MAINTENANCE AND CLEANING MISTAKES

Clean the distillation apparatus after every run. A "run" can be defined as more than just a batch process! If one has several gallons of material, then re-loading the distillation apparatus when the feed becomes low is okay, with some exceptions: if there was an accident such as "bumping," which caused material to flow to the condenser and has contaminated the product; if the vacuum level is not sufficient due to volatiles getting into the oil. In that case, change the vacuum oil.

If one is only running the same material over and over (continuous mode), then schedule a routine maintenance every few days to keep the still in prime operating mode.

The glassware is the first thing people think of when cleaning. Make sure all of the glassware is squeaky clean. Vacuum pump oil should also be changed whenever there is a decrease in performance. Buy lots of varying sizes and lengths of bottle brushes for the difficult angles, and spray out scrubbed areas with a solvent wash bottle.

If one is using a wiped-film evaporator, it includes taking apart pieces that don't normally get touched, such as the external condenser side-arms. Remove terpene build-up and re-grease all joints. Remove the internal condenser and make sure it is free of collected solids. Running a high-boiling point vegetable oil and solvent through the apparatus with the wipers on will help remove particles on the still body walls. It's worth the money to buy two internal condensers and two still bodies to keep in rotation. A quick swap-out of the internal condenser saves time that would be wasted on producing distillate.

Each one of these points could easily be a book. I've picked out some of the basic highlights that I think are a good start for an operator seeking direction. As one grows in their profession, these points are polished to a fine point, and must be continually refined each and every day.

Lilibel is the COO of Beaker & Wrench, a professional engineering consultancy based in Los Angeles, CA. lilibel@beakerandwrench.com

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1. Vogel, Arthur I. Textbook of Practical Organic Chemistry. 5th ed., Longman Scientific & Technical, 1989.



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Solution to Pollution is NOT Dilution

Don't Introduce Contaminates to Your Cannabis While Adding Terpenes

Katie Schnur, BS Chemistry,
Product Manager, Ricca Chemical

Lance Green, BS, MS Chemistry,
Associate Product Manager, Ricca Chemical

The reintroduction of extracted terpenes post extraction has gained popularity as the market moves toward meeting consumer demands. There are two major reasons for the addition to a formulation: potential therapeutic and consumer preferences. If these two factors were placed on the opposite ends of the spectrum, it could be a percentage of both. There is growing evidence that the terpenes in cannabis,

and other plants, have a potential therapeutic on their own.¹ This result is in addition to the proven benefits on top of the centuries of documented anecdotal benefits.²

As a commercial product formulation by adding a specific terpene blend or strain, such as Blue Dream or Berry Gelato, to your cannabis or hemp product you can create a unique product scent

and effect to help you build brand loyalty.

The addition of the terpenes, which are extracted by distillation from natural plant materials, is not without its considerations.

There are some common solvents found in commercially available terpenes, most at levels that are not considered harmful to consumers. The plant from

which the terpenes are derived will absorb substances present in the soil or air in the environment in which the plant is grown. In some cases, solvents will be introduced into the terpenes during their processing. In many of the high-quality terpenes in the market, you can clearly identify the level of remaining solvents by reviewing the Residual Solvent results on the Certificate of Analysis (C of A) that should accompany your terpenes from the manufac-

turer. If there is not one, then that is your first reason for concern about the number of other potential contaminants or homogeneity of the samples.

As part of the legalization process, states have established testing limits for a number of solvents used during the terpene extraction process.

Cannabis and hemp products offered for sale to consumers must be tested for residual solvents to ensure that contaminants are not present in levels that could potentially harm consumers' health. Just as you strive to reduce contamination during your extraction and post-extraction processes, doing the same as you introduce terpenes is critical to the potential contamination content of your final product. One must consider the additive accumulation of the total amount of these contaminants, which could originate from the cannabis/hemp starting material, the extraction and/or post extraction process or the terpenes you introduce to your product.

A sample of a commercially available Limonene Terpene Isolate, was tested in its concentrated form:



RESIDUAL SOLVENT ANALYSIS

ANALYTE	RESULT	ACTION LIMIT	ANALYTE	RESULT	ACTION LIMIT
N-butane	Not Detected	N/A	Isobutane	361.578 PPM	N/A
Acetone	2143.577 PPM	N/A	Methanol	355.854 PPM	N/A
Ethanol	Not Detected	N/A	Isopropanol	Not Detected	N/A
Benzene	1170.640 PPM	N/A	Toluene	173.965 PPM	N/A
Pentane	Not Detected	N/A	Hexane	Not Detected	N/A
Heptane	Not Detected	N/A	Ethyl Acetate	365.271 PPM	N/A
2-Methylpentane	Not Detected	N/A	Tetrahydrofuran	56.712 PPM	N/A
Cyclohexane	Not Detected	N/A	1,4-Dioxane	Not Detected	N/A
2-Ethoxethanol	Not Detected	N/A	Ethyl Benzene	Not Detected	N/A
O-Xylene	57.690 PPM	N/A	Cumene	Not Detected	N/A
Propane	Not Detected	N/A	Acetonitrile	Not Detected	N/A
3-MethylPentane	Not Detected	N/A	m-p-Xylene	Not Detected	N/A



By comparing the results to the state limits in which you intend to sell your products, you can access the terpene content for any potential nonconformities. For example, in Oregon the Action Limit for Benzene is 2 ug/g. Based on this data, you could choose to use this terpene in your product and attempt to dilute away the potential issue. In this example, you would need to dilute to less than 0.01% of the end product's total volume. Depending on the product, vape, edible or dab, you would also need to consider the uniformity within the product of the terpenes to ensure the potential risk has been eliminated. Alternatively, choosing a source for your terpenes that demonstrates lower levels of harmful residual solvents can reduce your risk of potential issues in failing laboratory testing or worse.

Being aware of the components of everything you put into your product will help ensure the success of your product. The old adage is definitely at work here, quality in ...quality out. Choosing consistent, high-quality terpenes from a reliable source and reviewing the test data with each lot will help you offer safe, consistent and enjoyable product to your customers.

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3 EASY STEPS

to Using Heat and Pressure
Technique for High Quality "Rosin"

Pressure and heat have been used for decades for the extraction of high-value compounds from plants, with documentation on high pressure as early as 1914. There are many "personal techniques" with products that have been repurposed for heated plus pressure extraction such as clothing irons, parchment papers, razor blades and small mesh screens. Like the early use of butane purchased at hardware stores, these techniques are not recommended for the new world of regulations and good manufacturing practices without some guidance. At the Emerald Cup in Santa Rosa in December 2017, I tested a cannabis chemovar that was



Total potential D9THC **19.3%**
Total THC **21.9%**

THCA	20.6%
D9THC	1.3%
CBGA	ND
CBD	ND
CBDA	ND
CBN	ND



THCA	80.0%
D9THC	7.7%

pressed on a system exhibiting there. The potency went from just over 20% potential THC (THCA + THC) to just over 80% for the resin. Of course, I couldn't test for anything else based on just the technology of the Orange Photonics Light Lab Cannabis Analyzer, but anything else on the plant could have made up the other 20% of the concentrate, such as lipids, cellulose, contaminants, etc.

The extraction of potentially active phytocannabinoids that are concentrated in trichomes can use pressure to break open the cell walls with pressure versus the use of solvents. The use of higher pressure up to 600 bar can be accomplished with many different tools that are commercially available for industrial use. These methods can be learned from many early cannabis books, such as *Beyond Buds: Marijuana Extracts: Hash, Vaping, Dabbing, Edibles and Medicines* by Ed Rosenthal (author) and David Downs (contributor), that describe extraction products and methods for providing a quality amount of concentrated cannabinoids. Another suggested peer reviewed article is *Advances in Extraction of Natural Ingredients by High Pressure Extraction Technology* written by Chung-Yi Wang, et. al. in 2013.¹

Of course, many people have seen rosin products when used for the bows on stringed instruments and on the pitcher's mound in baseball. Rosin most commonly describes the product that comes from a combination of heat (160C) and low-pressure sap of pine trees, for example. It is also being used to describe the oil from cannabis chemovars using the same mode of extraction. With such a potentially easy technique to concentrate the trichomes in cannabis, for example, 20% cannabinoid chemovar to 70 or 80% depending on the method used, it is compelling to try.

A SOLVENT FREE EXTRACTION TECHNIQUE

One of the reasons this technique has been so popular is that it is a very simple, safe and affordable method to producing high-quality results in a relatively short amount of time. Essentially, with the high-pressure technique, you're separating the oil from your cannabis material. Yields are usually like other extraction methods, with anywhere from 10% to 15% yield. With dry sift or Kief, the yields can be even higher. It also will extract all other components that are on the flower, including contaminants such as pesticides and chlorophyll, as it not discriminating in its products.

THREE CRUCIAL TOOLS YOU NEED FOR EXTRACTIONS

You can make high-grade oil in seconds. Unlike other extraction methods, you don't need expensive equipment or chemicals. There are commercial tools available that provide reproducible and programable conditions:

1. Commercially available system of a simple source for heat and pressure that has been developed and approved for application or for personal use, and has been used by many people, such as a clothes iron.
2. Parchment paper, due to its surface that can withstand some heat and pressure.
3. A filtering source, such as a .25 micron metal screen to allow the oil to move from the press to the parchment paper for further purification and/or formulations.

THREE STEPS FOR BEST EXTRACTION FOR THE SMALL SCALE:

1. Carefully prepare your material to be processed.
 - a. Knowing the history of the material, as contaminants such as pesticides, mold, etc. will also be concentrated.
 - b. Chopping or grinding the material provides a greater available surface area and more sample can be extracted at each time. This step is not essential as the whole plant can be pressed.
 - c. Having a reproducible moisture content will reduce post processing oxidation.
2. Prepare your instrument and area around it to assure it is clean to eliminate any cross-con-

tamination of extracted material. The instrument will have a feedback on the conditions, with some that are able to do methods with different step gradient of pressures.

3. Prepare for the post-extraction if needing to dewax or distill for greater purity.

For the small or personal batch sizes, there are books and sites that describe extraction using common appliances and hand or mechanical presses and screens. Make sure they are made with safe material and appliances are not compromised and do not present an unintended consequence.

References

- 1 Food Industry Research and Development Institute, No. 569, Sec. 2, Bo-Ai Rd., Chiayi, 60060, Taiwan (Tel.: D886 5 2918922; fax: D886 5 2861590; e-mails: cywang@firdi.org.tw)



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DENSITY:

Supercritical Carbon Dioxide's Most Overlooked Variable

By: Mark June-Wells, Ph.D.

Cannabis sativa is a plant with a complex suite of medicinally important solutes. Besides the cannabinoids, which show promise in the treatment and prevention of many diseases, there are other important pharmaceutical compounds including terpenes, flavonoids, and carotenoids. In the rapidly evolving legal marijuana market the development of unique products that comply with stringent quality standards is of the utmost importance. Organic solvent systems such as butane are static systems that effectively remove solutes from cannabis material and deposit all extracted materials in a single collection stream. Furthermore, organic solvents are difficult to remove from the extract. It takes time, care, and know-how to meet the increasingly stringent state standards for residual solvents when using organic solvents. Carbon dioxide has the potential to provide extractors the ability to create a variety of products due to its manipulability and avoid costly production delays because there is no concern about residual solvents. Most articles fo-

cus on the safety of carbon dioxide; fewer articles have discussed the separation power of carbon dioxide and how its properties could be leveraged to create unique cannabis products. Density is the key to targeted extraction, separation, and product development.

There are two features of carbon dioxide important to the discussion of its flexibility as a solvent: 1) Crossover Phenomenon and 2) Retrograde Solubility. Before diving in to those discussions, it is valuable to review how pressure, temperature, and density are related. Carbon dioxide density is a product of pressure and temperature in the supercritical state (i.e., pressure/temperature >73Bar/31C). In general, as more pressure is applied density will increase and as more heat is applied density will decrease; those two variables determine the density of supercritical carbon dioxide. So, what is crossover phenomenon? Simply, the crossover phenomenon is the relationship between a solute's solubility and carbon dioxide pressure under isothermal (i.e., constant temperature) conditions where a solute's solubility increases exponentially at a critical pressure (i.e., density). Below that density, a solute's solubility is low; above that density, a solute's solubility is high. The most important part of the crossover phenomenon is that it is solute-specific. This means that by using extraction parameters in concordance with a solute's specific solubility profile, it can be extracted preferentially from a mixture. Retrograde solubility can be viewed as the inverse of the crossover phenomenon. Retrograde solubility is the process by which solutes are separated from a mixture of many solutes dissolved in supercritical carbon dioxide. Simply described, numerous solutes are extracted at high carbon dioxide density; carbon dioxide density is then reduced in a sequential manner via separation vessels (i.e., different temperature/pressure settings) to isolate specific solutes. So, how do these two phenomena apply to cannabis?





The pharmacologically important compounds that are part of the cannabis plant vary in molecular weight, volatility, polarity, and number of functional groups, which means that each solute has individual solubility profiles. Most cannabis manufacturers are focused on extracting the cannabinoids and terpenes from plant material. However, there are other bioactive compounds that have medicinal value including carotenoids and flavonoids. All of the extractable compounds in the cannabis plant have different properties that allow for targeted extraction or separation through retrograde isolation. While much research is still necessary to refine the solubility data of cannabis extractables, the proceeding will present estimated ranges for crossover solubility densities for important cannabis compounds. Terpenes are a large and diverse group that are classified based on the number of carbons (i.e., monoter-

penes (C10)/sesquiterpenes (C20)). These plant compounds are responsible for the flavors and smells of cannabis; furthermore, they are volatile and extracted at low carbon dioxide pressures and temperatures. In general, terpenes have low crossover solubility where most desirable terpenes are dissolved in carbon dioxide at low density (i.e., 0.25 – 0.30g/ml). Cannabinoids are the primary solute of interest to cannabis extractors; there are over 100 individual cannabinoids that range in molecular weight from 250 – 350g/mol. These solutes vary in physical state, polarity, and specific crossover solubility profiles. In general, cannabinoids exhibit crossover densities for carbon dioxide between 0.65 and 0.75g/ml. Flavonoids are a family of polyphenolic compounds of which there are 5,000 or more known to naturally occur. The molecular weight and polarity of these compounds vary, but their likely crossover sol-

ubility densities in supercritical carbon dioxide is between 0.65 and 0.80g/ml. Carotenoids are another important medicinal compound present in cannabis. There are 600+ known carotenoids that exhibit very high molecular weights (C40). Some well-known carotenoids include lutein and lycopene; the crossover solubility density in supercritical carbon dioxide for carotenoids has not been refined, but likely lies between 0.85 and 0.90g/ml. So, what do all of these data mean to extraction companies?

The reader likely noted that there was no description of the crossover density for chlorophyll in the previous paragraph. While chlorophyll is a nutraceutical in some industries, in the cannabis industry it is not a desirable solute to extract from cannabis material. Fortunately, chlorophyll is a very large molecule that is not easily extracted from plant material using carbon dioxide. The crossover density for chlorophyll is between 0.90 and 1.00g/ml., which makes it easy to avoid

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it is important to input carbon dioxide density as part of your internal analytical processes. Many supercritical fluid extractors do not do real-time density calculations, but a technician can obtain a determination of carbon dioxide density if their temperature and pressure readings are reliable. By incorporating density into the analytical protocols, a company can better refine their extraction process through statistical modeling because of the solute specific density solubility relationships. Thirdly, technicians with an extractor that has one collection vessel (i.e., single stream) can use the crossover phenomenon to target specific compounds; then by modifying extraction conditions, they can target other extractable compounds. This sequential pressure modification process (i.e., increasing density over time) effectively fractions the mixture of solutes present in cannabis sativa. Fourthly, if a supercritical fluid extractor has separation capability, the technician can take advantage of

retrograde solubility by modifying carbon dioxide density in a sequential manner through a series of collection vessels and separate specific compounds from a mixture of solutes that are dissolved in supercritical carbon dioxide.

Carbon dioxide is a manipulable solvent; pressure and temperature determine solvent density, which can be used as a variable to refine extraction and separation processes because of the unique, density-dependent solute solubility characteristics. Data analytics are an important part of developing an extraction/separation process; that requires that an extraction company purchase a machine capable of determining carbon dioxide temperature and pressure accurately. If data are collected in an efficacious manner, the extraction process can be tailored to create a wide variety of products that meet the increasingly stringent quality standards of the US marijuana industry.



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