n-Butane Extraction of Cannabis Oils

Steven F. Baugh, Golden Extractors, LLC
Linn D. Havelick, Havelick & Associates, LLC

Abstract:

Extraction of oils from the Cannabis plant provides benefits in both the medical and recreational arenas. Botanical products have high variability in biologically active ingredients while extracts can be prepared at consistent levels for reproducible dosing. Additionally, extracts can be processed into a wider variety of medical products than cannabis flowers; including pills, capsules, gel-caps, skin creams and trans-dermal patches. Various extraction solvents have been used over time, and these solvents are also employed in food and pharmaceutical industries. The use of Butane as an extractant to recover biologically active molecules from plant material such as marijuana has been shown to effective and advantageous in comparison with other extractants. While Butane is a flammable gas, proper use and safety has already been demonstrated in other industries around the world.

Key Words:

Butane, n-butane, iso-butane, cannabis, extraction, flow, pressure, recovery, standardization, LEL, UEL, TWA, STEL, Standard Operating Procedures (SOPs), building codes, Regulatory Compliance

Background:

Several methods have been used to recover oils and biologically active compounds from Cannabis plant material. These include use of extractants such as ethanol, carbon dioxide (both as liquid and supercritical fluid forms), glycerin, Butane, isopropanol, naphtha and other hydrocarbon solvents. Controlled use of solvents can produce products with reproducible cannabinoid and biochemical profiles. Standardized extraction procedures allow for consistent dosing in both medical and recreational products. Water processes do not actually dissolve the material so solvent is not a proper term, it is a physical separation controlled by the porosity of the filters/bags employed.

The use of Butane as a solvent for marijuana product has been shown to be effective in recovery of cannabinoids from plant material, with recovery of THC exceeding 95% depending on solvent contact time, number of washes and plant feed material. Butane extraction has been used for foods and flavors over the past several decades, and was approved by international committee in 1981 for extraction of food.

Butane exists in two different isomers or structures, n-butane and isobutane, which are very similar in physical properties except for boiling point and vapor pressure. N-butane generates vapor pressure of approximately 20 psig at 75F. The temperature/pressure profile of n-butane results in relatively low operating pressures in normal operating conditions. This low operating pressure has led to n-butane being preferred for use in indoor cooking appliances over liquid petroleum gas (LPG) which has room temperature operating pressures in excess of 100 psi. The same lower operating pressure improves the safety profile for the use of closed-loop pressurized Butane extractors. In the context of this paper, the use of "Butane" refers to butane in general, n-butane and iso-butane are used when specificity is important, such as illustrating the differences.

While this paper discusses many of the important points regarding butane extraction, it is not intended to be a legal guide to building codes, electrical codes, fire codes or regulatory compliance. The State of Colorado has enacted rules and regulations founded on internationally recognized building fire and electrical codes, including construction and use permitting, and requiring review and approvals by recognized engineering and safety professionals. This paper is based upon those recommendations and requirements. Your local building, electrical and fire codes may vary.

Extract Properties and Use

Cannabis sativa is a plant with a long history of medical and recreational use, with well documented medical extracts and tinctures, making it one of the best examples of a traditional botanical drug or even dietary supplement by legal definition. Extracts of cannabis, both recent and historical, have concentrated the cannabinoids, the class of compounds named for the plant in which they were discovered. There are multiple, different cannabinoid receptors in the body. These receptors interact with different cannabinoids that result in intoxication and/or medical effects.

One of the cornerstones of modern medical practice is consistent dosing. Dosage of medically-necessary compounds are typically adjusted for age, weight and overall medical status. Consistent levels of medication are necessary to treat a patient and maintain therapeutic control. Plant material in general, and cannabis plants in particular, have the disadvantage of having biologically active compound content vary significantly by sub-species, growing season, growing media (soil, inert substrates, hydroponics), grow temperature and humidity and a host of other variables that engineered grows are only now discovering.

Natural medicinal products that prove useful are quickly standardized, typically by extraction. Standardization is commonly parallel to bioactivity guided fractionation, where a standardized product that shows medical activity is dissected, broken down into smaller and smaller fractions until the medical activity can be attributed to a compound, or class of compounds. Then these compounds and strategic analogs are synthesized, further controlling the purity of the drug. Cannabis has followed this path as well, with single cannabinoid drugs like THC and CBD investigated around the world.

Beyond product standardization and reproducible dosing there is avoidance to smoking. Smoking may be necessary for patients with nausea who cannot tolerate swallowing a pill, but there are other alternatives. Trans-dermal delivery, edibles and vaporization avoid many of the hazards associated with smoking cannabis, and extracts are preferred in these delivery systems.

Butane: Physical Properties

Butane is a small hydrocarbon molecule (C_4H_{10}), present as a clear, colorless and mostly odorless gas at room temperature. There are different isomers of butane, including normal butane (n-butane) and iso-butane, and they have slightly different physical properties. N-Butane is commonly used as a propellant in aerosol products such as deodorant, hair spray and pressurized lubricants. N-Butane is also a refrigerant, R600, seeing increasing use in domestic refrigeration with the demise of chlorofluorocarbons. N-Butane generates vapor pressure of approximately 20 psig at 75°F. Iso-butane has a vapor pressure nearly twice that of n-butane, so it is important to confirm which butane is being used, especially with extractors incorporating glass components.

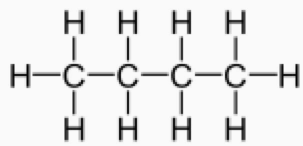
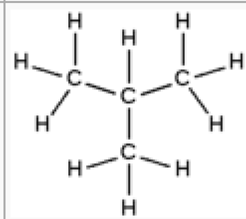
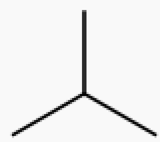
Common name	**normal butane** **unbranched butane** ***n*-butane**	**isobutane** ***i*-butane**
IUPAC name	**butane**	**2-methylpropane**
Molecular diagram		
Skeletal diagram		

Table 1: Butane Isomers and Structures (from Wikipedia)

Butane is also a significant component of LPG or CNG. In the case of LPG or CNG Propane makes up a majority of the weight and contributes to a vapor pressure of greater than 100 psig at room temperature. Because the rate of a gas leak is driven by the pressure of the liquid/gas, Butane is the fuel of choice for indoor cooking appliances (20 psig) versus LPG (>100 psig) which is commonly used outside and prohibited from indoor use.

Hazardous Materials Management

The flammable and potentially explosive nature of Butane presents occupational and property hazards. The successful management of these hazards is possible with the use of designed, engineered closed-loop systems that prevent the release of Butane into open air.

In the underground, illegal markets for marijuana butane extraction of oils was usually performed by open “blasting”. Blasting involved the emptying of a canister of butane through a marijuana-packed tube with an open collection bowl that collected the butane/oil mixture as it drained out of the tube. The butane was separated from the extracted oil by evaporation into the open air around the extractor. This typically resulted in the person performing the extraction sitting in a cloud of butane vapor well within the lower explosive level (LEL) and upper explosive level (UEL)[i]. In recent years, the use of blasting techniques by users and illegal processors has resulted in numerous fires, burns and deaths due to unexpected ignition of these butane vapor clouds.

The use of well designed butane closed-loop systems in the commercial marketplace, and in facilities appropriate for these systems, is expected to expand. When these systems are used in conjunction with facilities designed to meet building, fire and safety codes, and with appropriate ventilation, engineering controls and work process safeguards, the rate and number of fires, explosions and injuries will decline. This decline will likely closely track the legalization and commercialization of the marijuana industry.

Modern electrical and building codes already exist to cover compressed flammable gases in homes and the work place. In the context of the use of designed Butane closed-loop systems, the management of these hazards become an occupational health and safety management responsibility. Appropriate limits, procedures and engineering controls can improve the safe use of these extraction systems.

The International Fire Code (IFC) states that a Maximum Allowable Quantity of a liquefied flammable gas (such as Butane) per Fire Control Area is 30 gallons on the first floor/grade of a building. These quantities may increase if the area is protected by a fire sprinkler system and/or the storage is in an approved gas cabinet or exhausted enclosure. The quantities may decrease if the use is below grade in basements or in upper floors of a building. The IFC Maximum Allowable Quantity (MAQ) is established for buildings constructed to standard industrial ratings. If the MAQ is to be exceeded, the building or space must be constructed to an “H” or High Hazard Group Specification.

A well-designed closed-loop Butane extraction system is capable of processing as much as 15 pounds per day of dried marijuana plant matter per eight-hour shift while keeping the total amount of Butane below the IFC 30-gallon MAQ. Thus, with proper precautions, a relatively large industrial production facility can operate a Butane extraction system without the additional expense of a high-hazard building construction.

Those proper precautions include but are not limited to the following:

- limitations on the amount of Butane in use and storage in an area
- appropriate ventilation to prevent gas concentrations above lower explosive limits
- workspace and equipment design to avoid inadvertent ignition points
- equipment materials of construction
- flammable gas monitoring systems with automatic shutdown of Butane tanks
- standard operating procedures and daily leak checks
- proper personal protective equipment
- training of personnel
- area signage.

In addition to flammability and explosion hazards, Butane can act as an asphyxiant and rapidly evaporating liquid can cause frostbite or burns. If flammability hazards are well controlled to prevent Butane air concentrations from approaching its Lower Explosive Limit (LEL), asphyxiation is not a hazard. Avoiding open evaporation of Butane (such as in open blasting extraction) can also minimize the potential for burns or frostbite.

Butane residue in extracts manufactured using a well-designed closed-loop extraction system and with proper post-extraction processing is typically very low. The use of vacuum systems to remove trace residues of Butane solvent from the product is very effective. Laboratory verification of the remaining Butane levels in products is recommended and required by State of Colorado regulations.

Why Butane Extractors?

Alcohol and glycerine extractions are often used to produce tinctures and skin products that can be consumed orally or applied directly by the consumer. Alcohol can be removed from the extract, however this requires additional equipment and time to recover the alcohol and meet various laws and liquor codes. Butane and carbon dioxide are both gases at room temperature and can be removed from the extract with minimal effort.

Carbon dioxide supercritical fluid extractors are also popular for extraction of cannabis. It appears that supercritical fluid extracts are more selective, and yield a less viscous extract without some of the waxes and oils necessary to create some consumer products. These extracts appear to be well suited for blending in beverages, edibles and packaging in disposable vaporizer refill cartridges. Also carbon dioxide presents additional hazards beyond simple asphyxiation due to the physiology of carbon dioxide and high pressures that can displace the air in a room very quickly. Carbon dioxide has an occupational exposure limit of 5,000 ppm (one-half percent in air over an eight hour shift). A concentration of 30,000 ppm (three percent) is considered immediately dangerous to life and health.

Butane extracts contain oils, waxes and other plant compounds in addition to cannabinoids. Discussions have surfaced regarding “winterizing” Butane extracts to remove the waxes, presumably to mimic supercritical extracts and not plug vaporizers in the cold. Other blogs discuss the use of waxes to be added to supercritical extracts and other processing to yield a final product that can be handled like Butane extracts. While these endeavors can be appealing, especially given equipment costs, it is much like putting a square peg through a round hole. Butane extracts and supercritical fluid extracts are different, and both should be considered if necessary versus investing in the technology and time to make them similar.

The most commonly used technique for butane extraction in black markets and consumer processing is known as “Open Blasting.” In this procedure, an open pipe or tube is packed with marijuana and a can of butane (usually obtained from a camping goods store) is blasted through the tube. The butane liquid leaches out the THC and oils and is drained into an open pan. The butane is allowed to evaporate into the open air leaving an oily reside behind in the pan. The unfortunate operator is left in a cloud of highly explosive butane-air mixture.

While “Open Blasting” was the standard practice for butane extractions (often in legalized industrial settings), the open discharge of butane under uncontrolled conditions with untrained personnel has been a major source of risk, injury and property damage. A whole industry segment was developed around clandestine butane extracts, including open blasting systems sold directly to consumers. The professional closed loop extractors on the market today are attempts to provide a safe working environment for production of these products with high customer demand without forcing these dangerous activities back underground where they cannot be regulated or managed.

Closed Loop Butane Extractors:

A closed loop Butane extractor is much like a refrigerator or air conditioner that also happens to extract plant material. There is a low pressure side (cold), and a high pressure side or compression side (hot). This is also the design of a heat pump, with one side taking heat from the environment and providing cooling by evaporation, while the other provides heat through compression. In the case of the closed loop extractor, heat is supplied to maintain the recovery (evaporation) of Butane rather than allowing the effects of evaporative cooling to slow the recovery process.

Figure 1 is a flow chart of the butane going through a common closed-loop extractor design. The plant material is dried, loaded into an extraction vessel, and liquid Butane is passed through the plant matter. The liquid extract is then collected and the Butane is recovered through the vapor phase, allowing collection of the extract residue from the container. A pump is often used to recover the Butane, creating a vacuum on the inlet side to pull the butane vapors in, and compressing the vapor into liquid and into the recovery tank on the outlet side.

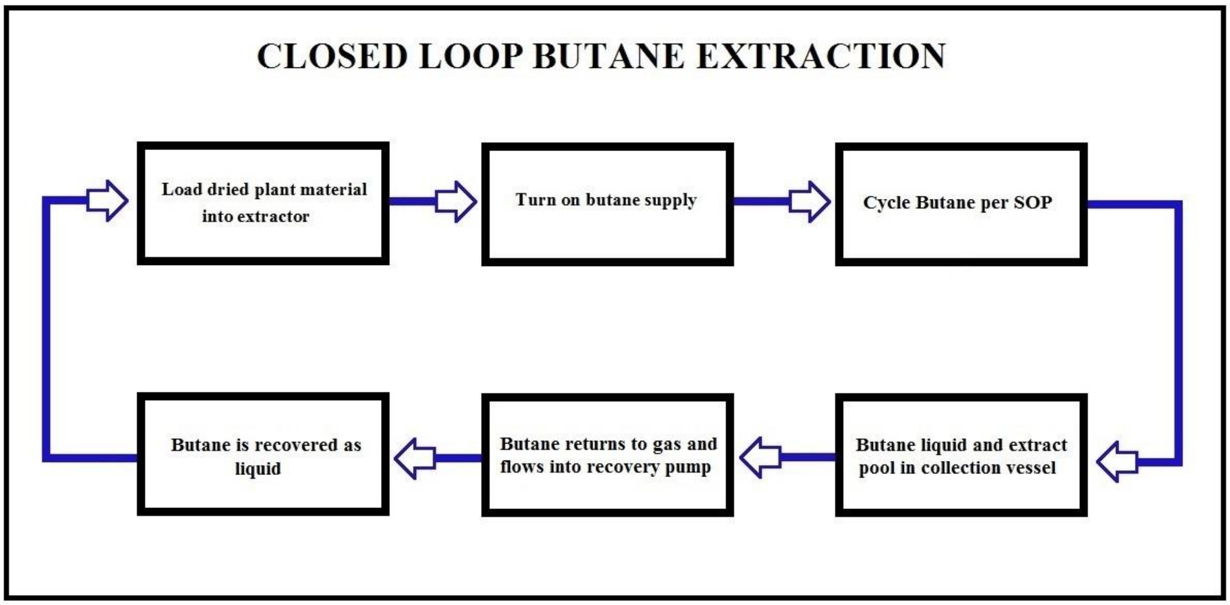


Figure 1: Butane Extraction System Flow Chart

When only Butane is recovered the pressure in the recovery tank will be slightly above the vapor pressure of the Butane at the temperature of the tank (high pressure side). Additional Butane vapor that is forced into the recovery tank is compressed into a liquid in the process, keeping the pressure of the tank at the vapor pressure of the Butane. During the extraction air is introduced into the system, primarily through the empty spaces in the dry plant material loaded in the extractor and any leaks under vacuum. This air does not compress into liquid at 20 psig and contributes to increasing pressure in the recovery or process tank.

Excess air pressure can be easily removed from the recovery tank. N-Butane boils at 32°F, so ice water will cool Butane down to the point where it doesn't boil and contribute significant vapor pressure inside the tank. Venting the tank while below 32°F allows the trapped air to be released thereby significantly reducing the pressure in the tank without the weight of butane decreasing. For this reason it is also useful to track Butane tank levels and recovery progress by weight and not pressure as most people are used too with compressed gases such as air. A full butane tank will be at 20 psig and so will a nearly empty butane tank.

Not all closed-loop extractor designs are the same. Some have the butane flow through the plant material while others soak the plant material in the butane during recovery. Beyond those differences there are three main types of closed-loop butane extractors, which can be classified as described below:

Passive Extractors- These devices are more a consumer class of extractor rather than a commercial machine. These extractors have no pump, and drive the butane back and forth by a temperature gradient. The source tank is heated and the recovery tank cooled to drive the butane through the plant material. The temperatures are reversed to recover the butane and dry the product. These extractors typically have small capacities given the time to process the butane thermally at reasonable temperatures. Recently heating tapes have shown up on popular models to speed the butane recovery.

Assisted Extractors- These extractors approximate the original blasting process, with the extraction being driven by the vapor pressure of the butane alone (20 psig) on the low pressure side. A pump is used to recover the butane on the high pressure side, and a recovery tank is used that is separate from the source tank to prevent the pressure from rising in the extractor over time. These systems can use glass components in the extractor section given the low operating pressures of the n-Butane. Air in the recovery tank must be vented before putting into use as the supply tank to avoid over-pressuring the extractor.

Powered Extractors- Closed-loop systems with pump assisted butane recovery, but normally one process butane tank. These extractors are typically made of stainless steel components with pressure ratings above 125 psig. These extractors continually recycle and re-use butane, with the pressure building in the gas cylinder during the process due to entrained air. These pressures are transferred within the single tank to the liquid butane supply outlet, creating higher extraction pressures and more rapid/violent flow through the plant material. This can mean less contact time with the liquid butane if the design does not pool the butane in the plant material. Some of the historically passive extractor models are being upgraded to powered extractors to provide more rapid recovery of the Butane and to shorten process times.

Design Considerations:

Large industrial scale extraction systems that use large quantities of butane must be housed in a building constructed to a “H” high-hazard occupancy code standard. A system at this scale could process hundreds of pounds of plant material per day-well beyond the capacity of almost any legal growing operation today.

In order to meet the needs of the medium-sized business an extraction system capable of processing 5 to 15 pounds of plant material per day is needed in much of today’s market. Systems in this size category can be designed to fit within a standard industrial classification building (F) rather than a high-hazard structure. Certainly, factors such as ventilation, sources of ignition, materials of construction, Butane quantities, and personal protective equipment must be considered even in a space with an industrial classification.

Safety starts with code compliance; including proper materials selection, sealed switches, solid state relays, induction motors and fans, and an LEL/solenoid on the gas supply with multiple sensors. Additional features such as high temperature limit controllers and submerged heating elements as used in the LPG industries help to complete the package. Propane use in vehicles has spawned readily available safety equipment, demonstrating that compressed flammable gases can be used safely.

Standard Operating Procedures

Safety elements such as operator training, daily leak checks, engineering controls and safe practices should be outlined in a comprehensive safety program, with written policies and Standard Operating Procedures (SOPs). In addition, conditions and procedures for making the extract should be clearly outlined to allow effective training, minimize the impact of employee turnover, increase corporate value and to standardize the manufacturing process.

Key extraction variables to control during the extraction include pre-treatment of the plant material, extraction pressure and flow, total amount of butane, pooling time and temperature. Different strains and growing conditions yield different product colors and textures, and specific conditions can be developed for each particular strain if desired. Glass offers several advantages for low pressure extractors, allowing a visual endpoint for all extractions versus strain and growing-specific extraction conditions. This is particularly useful for simplifying standard operating procedures, managing process changes/control and increasing productivity.

The Standard Operating Procedure is the culmination of everything discussed here; how the above issues are addressed in each extractor design, business or state can be different, but must be documented to create a reproducibly safe and productive workplace. Safety, Productivity and Quality are key aspects of the cannabis extraction process, and all facets need to be addressed to create the safest environment possible, while producing high quality extract of know potency and consistency.

Relevant Websites

Golden Extractors: http://www.GoldenExtractors.com

Havelick & Associates LLC: http://www.HavelickIndustrialHygiene.com

Colorado MED Regulations: http://www.colorado.gov/cs/Satellite/Rev-MMJ/CBON/1251592984795

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[i] Below the LEL there is not enough fuel to support explosion, and above the UEL there is not enough oxygen to support an explosion.