

Technical Data of Cannabinoids

| Chemical | Formula | FW (g/mol) | Boiling Point (°C) | Boiling Point (°F) | Melting Point (°C) | Density (g/mL) | Solubility in Water (g/100 g) | Flash Point (°C) |
|--------------------------------|----------|------------|--------------------|--------------------|--------------------|----------------|-------------------------------|------------------|
| Solvents | | | | | | | | |
| Isopropyl alcohol (2-propanol) | C3H8O | 88.15 | 82.4 | 180.32 | -88.5 | 0.785 | Miscible | 12 |
| methanol | CH4O | 32.04 | 64.6 | 148.28 | -98 | 0.791 | Miscible | 12 |
| acetone | C3H6O | 58.079 | 56.05 | 132.89 | -94.7 | 0.7845 | Miscible | -20 |
| ethanol | C2H6O | 46.07 | 78.5 | 173.3 | -114.1 | 0.789 | Miscible | 13 |
| heptane | C7H16 | 100.2 | 98 | 208.4 | -90.6 | 0.684 | 0.01 | -4 |
| 710 Spirits | Mixture | Mixture | 80 - 98 | 176 - 208.4 | -91 | 0.797 - 0.791 | Miscible | 17 |
| Cannabinoids | | | | | | | | |
| THC | C21H30O2 | 314.2 | 157 | 314.6 | | | | |
| THCA | C22H30O4 | 358.47 | 105 | 220 | | | | |
| CBD | C21H30O2 | 314.22 | 160 - 180 | 320-356 | | | | |
| CBDA | C22H30O4 | 358.47 | 316 - 531 | 600.8-987.8 | | | | |
| CBN | C21H26O2 | 310.19 | 185 | 365 | | | | |
| CBC | C21H30O2 | 314.22 | 185 | 365 | | | | |
| CBG | C21H32O2 | 314.22 | 105 | 220 | | | | |
| 8-THC | C21H30O2 | 314.16 | 175-178 | 347-352 | | | | |
| THCV | C19H26O2 | 286.41 | 220 | 428 | | | | |
| Terpenes | | | | | | | | |
| B-myrcene | C10H16 | 136.23 | 166-168 | 330.8-246.2 | | | | |
| B-caryophyllene | C15H24 | 204.35 | 119 | 246.2 | | | | |
| d-Limonene | C10H16 | 136.23 | 177 | 350.6 | | | | |
| Linalool | C10H18O | 154.25 | 198 | 388.4 | | | | |
| pulegone | C10H16O | 152.23 | 224 | 435.2 | | | | |
| 1,8-cineole | C10H18O | 154.25 | 176 | 348.8 | | | | |
| a-pinene | C10H16 | 136.23 | 156 | 312.8 | | | | |
| a-terpineol | C10H18O | 154.25 | 218 | 424.4 | | | | |
| terpineol-4-ol | C10H18O | 154.25 | 209 | 408.2 | | | | |
| p-cymene | C10H14 | 134.22 | 177 | 350.6 | | | | |
| Flavonoids | | | | | | | | |
| apigenin | C15H10O5 | 270.24 | 178 | 352.4 | | | | |
| quercetin | C15H10O7 | 302.23 | 250 | 482 | | | | |
| cannflavin A | No data | No data | 182 | 359.6 | | | | |
| B-sitosterol | C29H50O | 414.71 | 134 | 273.2 | | | | |

Data from: <https://comptox.epa.gov/dashboard>

Each cannabinoid, terpene, and flavonoid have different boiling points that are generally higher than the solvents used to extract them. If higher temperatures are used to evaporate off solvents, the boiling points of the desired compounds should be high enough that they will be safe from evaporation or degradation. Different variables such as run time or pressure can be adjusted to compensate for products that are heat sensitive.

710 Spirits

710 Spirits is a blended product of high purity heptane and ethanol. The boiling point to start evaporating off the solvent is a range from 80 °C to 98 °C to account for each solvent. Since this is a range, different factors can be adjusted to ensure the solvent is evaporated off appropriately. Time, temperature and pressure can affect the evaporation rate and how much solvent is evaporated off.

Heptane

Heptane is a high purity, nonpolar, organic solvent. It has a higher boiling point at 98 °C and creates the higher boiling end point for the 710 Spirits. According to the Certificate of Analysis for 710 Spirits, the heptane does evaporate off and follows regulations for the end products. Different variables can affect the rate and volume of heptane removed, such as pressure, temperature, and time.

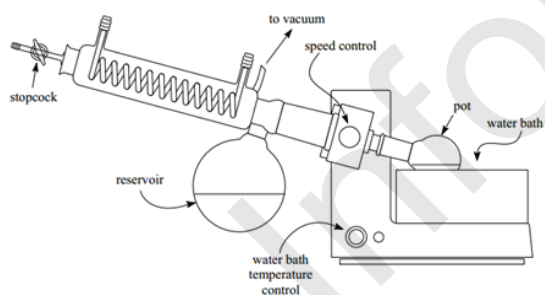
Ethanol

Ethanol is a food grade, polar, organic solvent. It has a lower boiling point at 80 °C, so it can start boiling and evaporating off faster than n-heptane. This solvent contributes to the lower end of the boiling point range for 710 Spirits. Evaporating off the solvent will require lower temperatures but again can be effected by temperature, pressure and time.

Rotary Evaporation

Rotary evaporation removes solvent from a higher boiling point mixture. This type of evaporation can handle larger volumes of solvent and has faster evaporation times for lower boiling point solvents. The flask is under vacuum while spinning in a water bath. The rotation increases the surface area of the solvent to be removed, increasing the rate of evaporation. The vacuum reduces the boiling point of the solvent and provides the means to separate the solvent from the compound of interest. Pressures, temperatures, and run time can be adjusted to achieve solvent evaporation in a quick and efficient timeframe. **When the pressure above a liquid is reduced, the vapor pressure needed to induce boiling is also reduced, and the boiling point of the liquid decreases.**

Rotary evaporation can separate solvent from different types of mixtures and materials but it is crucial that the desired compound has a higher boiling point than the solvent. Lower boiling point solvents work best, but rotary evaporation is more commonly used to remove water (b.p. 100 °C). Higher boiling point solvents (DMF - b.p. 153 °C, DMSO, etc.) would be easier to remove using other techniques, but with the correct vacuum pump and conditions, higher boiling point solvents can be removed using rotary evaporation.



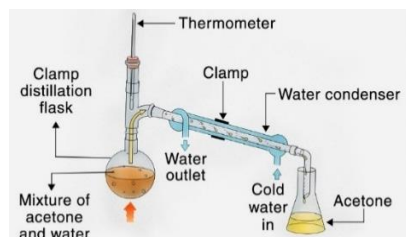
<http://mirandamusic.com/mpnorganic/rotovap.html>

Rotary evaporation is a popular setup in the cannabis industry. It is favorable as it can handle large volumes of samples and evaporate off solvent quickly. Solvent recovery is also possible in large quantities depending on the capacity of the rotary evaporator. Further processing of the cannabis product may be necessary to remove any residual solvent or separate unwanted compounds from the final product.

Distillation

Simple distillation is a procedure where two liquids with different boiling points can be separated. Preferably there is at least fifty degrees difference in the boiling points of the two liquids. As the liquid starts to heat, the vapors that form will be richest in the liquid that boils at the lowest temperature. The temperature will continue to increase until the boiling

point of the next lowest boiling compound is approached. The process can be repeated until all fractions of the original mixture have been separated.



<https://sciencestruck.com/types-of-distillation>

Steam distillation

Like vacuum distillation (see below), steam distillation is a method for distilling compounds which are heat-sensitive. The temperature of the steam is easier to control than the surface of a heating element, and allows a high rate of heat transfer without heating at a very high temperature. This process involves bubbling steam through a heated mixture of the raw material. The vapor mixture is cooled and condensed, usually yielding a layer of oil and a layer of water.

Steam distillation of various aromatic herbs and flowers can result in two products; an essential oil as well as a watery herbal distillate. The essential oils are often used in perfumery and aromatherapy while the watery distillates have many applications in aromatherapy, food processing and skin care.

Vacuum distillation

Some compounds have very high boiling points. To boil such compounds, it is often better to lower the pressure at which such compounds are boiled instead of increasing the temperature. Once the pressure is lowered to the vapor pressure of the compound (at the given temperature), boiling and the rest of the distillation process can commence. This technique is referred to as vacuum distillation and it is commonly found in the laboratory in the form of the **rotary evaporator (see above)**.

This technique is also very useful for compounds which boil beyond their decomposition temperature at atmospheric pressure and which would therefore be decomposed by any attempt to boil them under atmospheric pressure.

Short path distillation

Short path distillation is a distillation technique that involves the distillate travelling a short distance, often only a few centimeters, and is normally done at reduced pressure. A classic example would be a distillation involving the distillate travelling from one glass bulb to another, without the need for a condenser separating the two chambers. This technique is often used for compounds which are unstable at high temperatures or to purify small amounts of product. The advantage is that the heating temperature can be considerably lower (at reduced pressure) than the boiling point of the liquid at standard pressure, and the distillate only has to travel a short distance before condensing. A short path ensures that little compound is lost on the sides of the apparatus and degradation is decreased.

In cannabis extraction, after winterization the product is passed through heated lines into an evaporation chamber that is spinning in order to create a thin film on the wall. The desired product passes down the walls into a separate flask while the solvent is condensed down a coil in the center of the chamber into a different flask. The distillation path is shorter than traditional apparatuses in this set up and can reduce processing time.



<https://www.youtube.com/watch?v=UWXu35jaK60>

The above picture is a short path distillation set up by Root Sciences. The winterized product is in the flask on the left while the short path set up with the evaporation chamber is on the right. The product is separated into two flasks at the bottom.

Fractional Distillation

Fractional distillation is a separation of a liquid mixture into fractions differing in boiling point (and hence chemical composition) by means of distillation, typically using a fractionating column. It can incorporate as many fractionating columns as necessary depending on the complexity of the starting mixture and what end products are desired.

This type of distillation seems popular in extraction of essential oils since the goal is to separate out various terpenes or flavonoids and each have different boiling points. A few cannabis places used this type of distillation to enhance the flavor of their end product since the more complex system can distill out the many desired terpenes more easily.

Extractions

Liquid-liquid Extraction

Liquid-liquid extraction separates mixtures based on their relative solubilities in two immiscible liquids. It is chosen over distillation for separation applications that would not be cost effective or possible with distillation. In a liquid-liquid extraction a liquid stream containing the component to be recovered (solute) is fed into an extraction where it contacts a solvent. The two liquids need to be immiscible or slightly miscible to allow them to form a dispersion, with one liquid dispersed as droplets in the other.

Many cannabis extraction techniques known in the industry today are different varieties of a liquid-liquid extraction.

Solvent Extraction

A method for separating compounds based on their relative solubilities in two immiscible liquids. Substance is treated with a solvent and the substance separates into more and less soluble compounds. Polar molecules dissolve in polar solvents and nonpolar molecules dissolve in nonpolar solvents.

Supercritical Solvent Extraction

Also known as Supercritical Fluid Extraction (SFE). The process of separating one component from another using supercritical fluids as the extraction solvent. Extraction is usually from a solid matrix but can be from liquids. Carbon dioxide is the most common supercritical fluid used. Extraction conditions are above critical temperatures and pressures. Modifiers such as ethanol or methanol can be added but will alter the conditions of the extraction. The

addition of polar solvents can help dissolve polar compounds in the starting mixture. The properties of the supercritical fluid can be modified by altering the temperature and pressure allowing for selective extraction. Generally, increasing the pressure can increase solubility. **A higher pressure is necessary to keep the carbon dioxide in a supercritical fluid state.**

The system requires higher pressures which can increase costs and also must have a pump for the carbon dioxide and a means of maintaining pressure in the system. Once the material is dissolved into the supercritical fluid the material is pushed into a separator at lower pressure where the extracted material settles out.

Solid-Liquid Extraction

This type of extraction is similar to liquid-liquid extraction except that there is a solid matrix that comes into contact with a liquid solvent. The desired components are extracted into the liquid phase and then the sample is filtered to remove any solid particles that are leftover. The solid sample is then discarded or used for other purposes.

Microwave Assisted Process/Extraction (MAP/MAE)

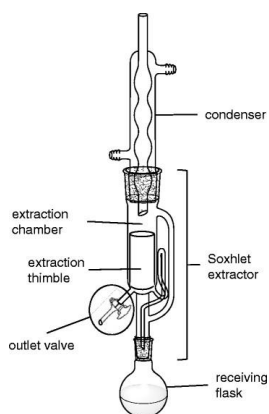
A process for extracting a particular analyte or compound from a source matrix using a microwave. The sample is in the form of a solid material, most commonly a source plant material. The sample is heated using microwaves in a controlled environment (closed-vessel or open-vessel) at certain temperatures, pressures, and various time intervals. The target for heating in dried plant material is the minute traces of moisture that occur in plant cells. The heating of this moisture inside the plant cell results in evaporation and creates tremendous pressure on the cell wall. The cell wall is pushed from the inside and eventually ruptures. The active constituents from inside the cell wall are released and can be collected. The yield from the plant samples can be enhanced if the sample is soaked in solvents suitable for this type of extraction. Higher yields can also be obtained by increasing temperature which allows for faster penetration of the solvent into the cell walls.

The benefits of this type of extraction is that the technique requires less solvent volume, has high and fast extraction performance and can protect heat sensitive constituents. Time of extraction, temperature, and microwave power are all factors that can affect the performance of the extraction and determine the amount of yield on each run.

Different types of solvents are more suitable for this type of extraction, as with any other extraction technique. Solubility of the desired analyte and the ability of the solvent to absorb microwaves are two major factors when adding a solvent to this type of extraction. The selected solvent should have a high selectivity towards the analyte of interest than the other components. Solvents that are transparent (hexane) to microwaves do not heat up under microwave and those with good microwave absorbing capacity (ethanol) get heated up faster and enhance the extraction process. Optimum extraction results can be achieved by using mixtures of high and low microwave absorbing solvents but that is determined by the desired end product of the extraction.

Soxhlet Extraction

Soxhlet extraction is a continuous solid-liquid extraction with the use of a solvent. A solid containing the material that is to be extracted is placed into a thimble which is then placed in the Soxhlet extractor. The desired solvent is then heated to boiling and the rising vapor is then condensed down into the Soxhlet by a condenser. The condensed solvent passes through the thimble containing the solid sample and fills the Soxhlet until it reaches capacity and drains back down into the solvent flask. This process happens over and over until the material to be extracted is out of the sample and is extracted into the organic solvent. The solvent is then evaporated off. A picture of a typical Soxhlet set up can be seen below:



https://www.researchgate.net/figure/Modified-Soxhlet-extractor-for-the-extraction-of-PCBs-in-soil_fig7_272365906

Typically, a Soxhlet extraction is used when the desired compound has a limited solubility in a solvent and the impurities are insoluble in the solvent. If the desired compound has high solubility in the desired solvent a simple filtration can be used to separate out the compound from the insoluble material.

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