

Improving The Quality And Efficiency Of Terpene Extraction From Cannabis Plant

Highly efficient microwave processing of terpenes with the Milestone ETHOS X

Introduction

As the cannabis industry matures, it creates additional opportunities for cannabis processors and growers for both medicinal and recreational use. Originally cannabis processors focused on designing efficient extraction processes to maximize the THC and CBD extraction, while other key molecules were not considered, such as the strain-specific terpenes in the plant. Among the multitude of different technologies available the most common techniques used in the cannabis market are light hydrocarbon, ethanol, or supercritical fluid extraction. Many cannabis extract producers understand that during their extraction procedures terpenes are lost or degraded, losing a pivotal component of the plant. Terpenes and terpenoids are small molecules synthesized by the cannabis plant, well known for their characteristic taste and smell. These molecules are predominantly used for flavoring in edible and recreational products.

Terpenes are volatile molecules and they might be lost in the drying step, fundamental for most ethanol and supercritical fluid extractions. To address the limitations of the conventional extraction techniques, Milestone has developed and patented (European patents EP 1 439 218, EP 1 618 798 and EP 1 629 725) the **ETHOS X** for the Microwave Green Extraction of Natural Products.

The **ETHOS X** takes advantage of the unique microwave selective heating mechanism. The internal heating of the water within the sample distends its cells and leads to rupturing of the glands and oleiferous receptacles. This process is extremely fast and terpenes are completely evaporated by the in-situ water of the plant material without any previous sample preparation typically required for other extraction approaches. The ETHOS X process leads to the efficient and fast production of terpenes, providing a complete terpene profile with superior quality over all the other extraction techniques. Cannabis extract producers can integrate Milestone's ETHOS X into their process, maximizing the extraction.



This technical note describes how the cannabis plant can be processed using Milestone's ETHOS X to produce high quality flavoring terpenes, while preserving the quality of the cannabis plant material for the further processing for THC and CBDs extraction with conventional techniques.

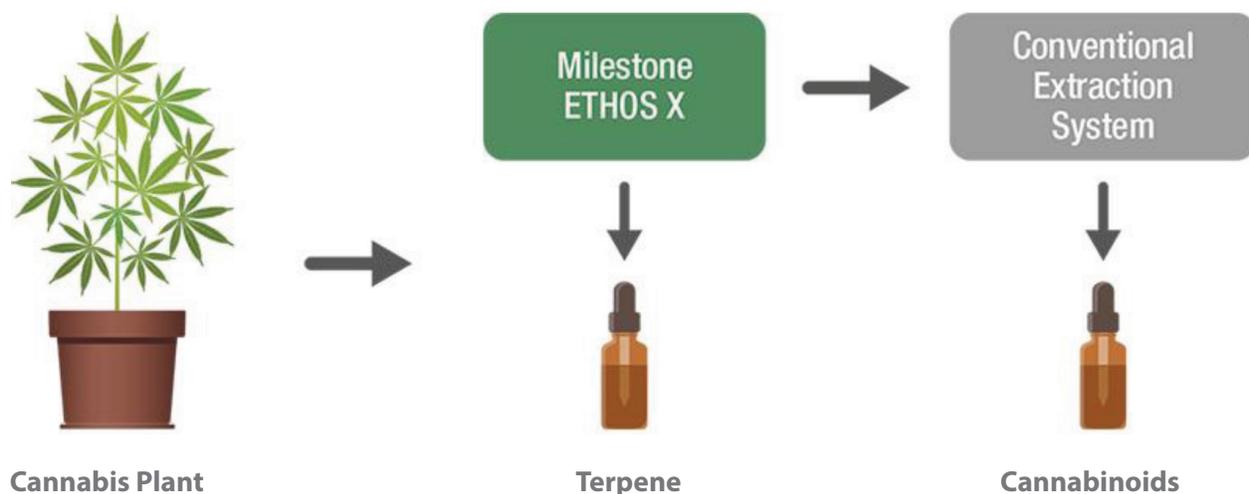


Figure 1 - Cannabis processing diagram with Milestone ETHOS X

Experimental

Instrument

The ETHOS X meets the requirements of the growing demand for production of high-quality terpenes expressing the complete terpene profile of the cannabis plant, thanks to its unique benefits, such as:

- Solvent-Free Extraction for Highest Quality
- Shorter Run-Times Combined with User-Friendly Platform
- Flexible Configuration for the Widest Range of Lab Needs
- Market-Leading Innovation, Safety, and Technology

Developed and patented by Milestone, the ETHOS X is the unique benchtop microwave extraction system able to provide production scale capability in the shortest processing time. It has been specifically designed for production, with the stainless-steel distillation module for easy use and handling.





The ETHOS X, with its high sample capacity, up to 12 L, is simply the most productive, fastest, easiest and most efficient extraction system for terpene extraction ever made!

Sample And Reagents

- Cannabis sample: OGKB 2.0 strain
- Distilled water.

Typical Procedure

1. Cannabis sample weight will vary based on the ETHOS X reactor volume, see the table below for detailed information:
 - a. Fresh material, weight directly in the 5 L ETHOS X glass extraction reactor
 - b. Fresh frozen material, weight directly in the 5 L ETHOS X glass extraction reactor, wait 30 min to thaw before proceeding with the extraction
 - c. Dry material, the plant material has to be moistened prior to starting the microwave extraction
2. Close the reactor with its glass cover, place it inside the ETHOS X, assemble the stainless-steel distillation module and connect to the water chiller.
3. Run the microwave program as shown in table 1.
4. Pure terpene products are collected into the stainless-steel distillation module.

| Reactor Size | Sample Amount | Microwave Power |
|--------------|---------------|-----------------|
| Small (21) | Up to 400 g | 400W |
| Medium (41) | Up to 1500 g | 1500W |
| Large (121) | Up to 3000 g | 1800W |

Table 1 -ETHOS X suggested working conditions



Results And Discussion

The aim of the study was to prove that the ETHOS X will efficiently extract the complete terpene profile of cannabis without degradation of the cannabis material, allowing for further processing to obtain the cannabinoids (including THC and CBD) by conventional extraction techniques.

In this specific work, terpene extraction was performed on fresh frozen OGKB 2.0 strain working on 1 kg of cannabis with the 5 L ETHOS X reactor. The sample was microwave-treated at constant power for 40 min. After 10 min, the terpene fraction started to be collected in the distillation module, above the water layer. The extraction was completed after 40 min when the terpenes were completely extracted. At the end of the extraction, the terpene fraction was collected from the glass distillation module, frozen for 30 min to remove extra water and then stored in a fridge at 4 °C.



The ETHOS X provided a superior terpene fraction with characteristic fragrance and flavor profile of the original fresh cannabis plant. This was possible thanks to the ability of the ETHOS X to quickly process fresh material, protecting the valuable molecules from the oxidation processes. The terpene fraction was analyzed via GC-MS providing the fingerprint of the extract. .

| ETHOS X EXTRACT - Total Yield* 1.59% | |
|--------------------------------------|-------------------------|
| δ -Limonene | <i>trans</i> -Nerolidol |
| β -Myrcene | β -Pinene |
| Linalool | <i>cis</i> -Nerolidol |
| α -Humulene | Camphene |
| α -Pinene | <i>cis</i> -Nerolidol |

Table 2 -List of the main terpenes in the ETHOS X extract (from OGKB 2.0 strain)

*by weight

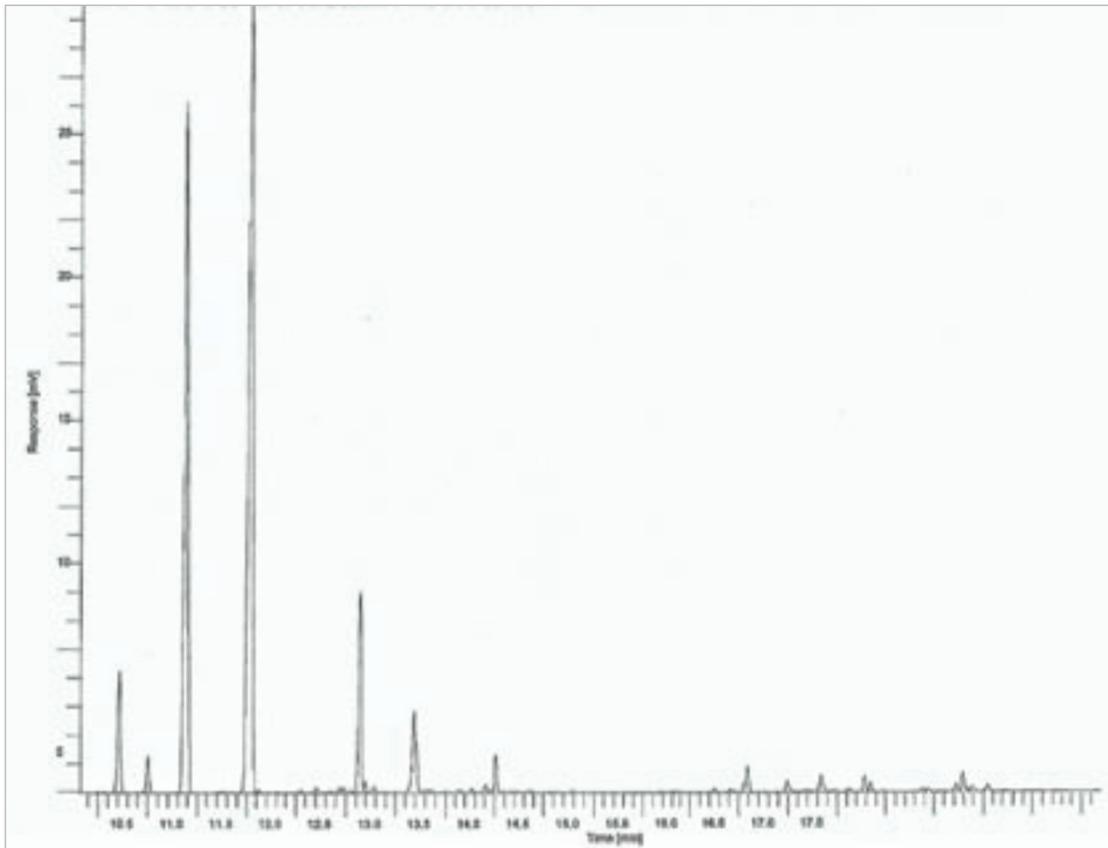


Figure 2 -GC-MS profile of the ETHOS X extract





To conclude the evaluation, the cannabis material has been extracted before and after the ETHOS X treatment with closed-loop hydrocarbon extraction to collect the cannabinoids.

| | Plant Raw Material ^b | ETHOS X extract | ETHOS X processed plant material ^d |
|---------------------------------|---------------------------------|-----------------|---|
| Terpenes ^a | 1.4 | 1.6 | 0.1 |
| Total Cannabinoids ^a | 78 | c | 77 |

Table 3 -Recovery data of terpene and total cannabinoids from the described processing step in Figure 1

a Data are calculated in yield by weight

b Extracted with close-loop hydrocarbon extraction system.

c. Not detected (<LOO)

d.Cannabis plant material extracted with close-loop hydrocarbon extraction system after Ethos X process.

The potency analysis (Table 3) shows that the ETHOS X did not affect the yield or quality of the cannabinoids (78% in the raw material vs 77% in the processed material). Moreover, the ETHOS X extract is free of cannabinoids, avoiding the need for a purification step. The very low content of terpenes in the ETHOS X processed plant material confirms the ability of the ETHOS X to efficiently extract the terpene molecules in a 40 min process.

This provides a better solution for cannabis processors to extract their cannabis derived terpenes and then continue the extraction for cannabinoids with any of the traditional methods.

Conclusions

The data shown in this work, demonstrates that the ETHOS X maximizes the extraction of terpenes from cannabis material, without compromising the quality of the THC and other cannabinoids extracted in the following process. Its ability to work with fresh material produces a superior terpene profile that is unmatched by conventional extraction techniques. Moreover, the fast processing times ensures the integrity of all cannabinoids, enhancing the overall efficiency in the cannabis processing industry and ensuring fast return on investment.

Milestone's ETHOS X is proven to be a unique and beneficial tool for cannabis processors, allowing them to obtain terpenes with unmatched quality and completeness, ready to use for flavoring in edible and recreational products without effecting the cannabinoid quality and extraction process.

Acknowledgement

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About Milestone

Established in 1998, Milestone is headquartered in Italy and has offices in Germany, Switzerland, the United States, China, Japan and Korea. We operate worldwide through a network of over 100 exclusive distributors, all providing our customers premium application and service support. Milestone's mission is to help chemists by offering them the most advanced instrumentation for sample preparation and direct mercury analysis in the world. Our industry-leading technology, in combination with fast, responsive service and applications support, allows Milestone to support our goal of providing you the highest return on investment possible.

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