

Sample Preparation & Processing

Evaluation of Evaporative Techniques in the Extraction and Preparation of Cannabis Oil

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California Gold Extractions specialises in the extraction and refinement of Tetrahydrocannabinol (THC) oil for the medical marijuana market in California. As part of the refinement process, the oil must be dissolved in a greater volume of ethanol and subsequently filtered. In order to then remove the ethanol, a controlled evaporation process is employed. To date, the most common method of removal has been the use of a rotary evaporator; however, as volumes increase, the time and skill required to effectively remove the ethanol also increases.

The Rocket 4D Synergy, Genevac Ltd, (Figure 1) was evaluated as a potential tool in the process of production scale up. The system provides bump free, automated evaporation as well as greater and more flexible throughput than other available evaporators. The system was tested and compared against a rotary evaporator to see if the Rocket, with these additional benefits, would match the performance of the industry standard rotary evaporator.

Method

Raw cannabis extract is obtained by subjecting cannabis plant material to supercritical fluid CO₂ (SFC) extraction, which yields raw cannabis extract. This extract contains, among other things, THC and other cannabinoids as well as a variety of fats and waxes. In order to remove these inert materials and concentrate the active ingredient(s) a refinement process, winterisation, is employed. As part of this process, the raw extract is dissolved in multiple volumes of ethanol and filtered. Following filtration, the ethanol must be removed to yield refined THC oil.

Equal volumes (2L) of winterised, dissolved extract were used in each evaporator. The temperature was set at 50°C for both evaporators. The rotary evaporator uses a water bath, while the Rocket controls heat energy input by generating low pressure, temperature controlled steam.

The rotary evaporator required some attention during the process to maintain an appropriate evaporation rate. Both the depth of the evaporation flask in the bath and the rotational rate were adjusted and the cold trap required monitoring and filling. In addition, the system had to be paused once to drain the solvent collection flask and manually stopped at end of run.

The Rocket has built-in methods that vary the operational parameters based on the solvents present. For this test the Medium Boiling Point (Med BP) method was selected. Once the extract was loaded and method selected, the system was started and no other user input was required. The Rocket controlling pressure, temperature and monitoring parameters throughout the run, stopping automatically once the process was complete.

Dried samples were analysed for residual ethanol by headspace-gas chromatography.



Figure 1. Rocket 4D Synergy Evaporator

Results

See Table 1. The Rocket took approximately 60 min to complete evaporation, while the rotary evaporator took about 80 min. Both systems recovered roughly equal volumes of ethanol, approximately 1.4 L. In addition, both systems recovered roughly 200 mL of refined extract. (Figure 2)

Residual ethanol data for both samples can also be seen in Table 1. Overall, the Rocket sample had a slightly greater concentration of residual ethanol, however, this was within operational limits. It may be possible, by making changes to user defined parameters in the Rocket method, such as final drying time, to further optimise residual solvent going forward.



Figure 2. Refined THC oil extract: Left, from rotary evaporator; Right, from Rocket 4D Synergy evaporator.

Table 1. Comparison of results: residual ethanol by headspace-gas chromatography, process time, and final extract volume.

	Residual Ethanol (ppm)	Process time (min)	Extract Recovery (mL)
Rotovap	2.44%	80	200
Rocket	2.72%	60	200

Conclusion

Despite the slight increase in residual ethanol, the Rocket was found to provide an overall more efficient drying solution. The whole of the evaporation process took less time and provided for much more consistent operation that did not require monitoring or additional user input following system start up. This allowed for other tasks to be completed concurrent with evaporation, as well as allowing for solvent removal to take place overnight or other times when no one is onsite.

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