
# CANNABIS - WHITEPAPER COLLECTION

## Recovery of THC Aerosol using “preheated Cartridge” method

CERULEAN

a coesia company

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# Introduction

A method had been previously developed for volatilising the very viscous cannabis liquids present in some vaping devices predominantly containing the lipid (-)-trans-delta-9-tetrahydrocannabinol which is commonly referred to as THC. In order to successfully volatilise the cannabis liquid the cartridge had to be moderately pre-heated in an oven before vaping started. It was noted that the process of vaping, if sufficiently frequent, would mildly “self-heat” the liquid allowing aerosol formation throughout an experiment.

This mild heating method, although not essential for the current Colorado State metal recovery regulations, anticipates wider toxicological analysis of THC aerosol that may be required as knowledge and legislation regarding cannabis vapour develops. The method used a gentle heating method as opposed to overdriving the internal heater for specific reasons:

The internal heating system might be device regulated to a maximum power or heating temperature, thus preventing heating to a high level at initiation.

Overheating could initiate thermal degradation of components within the liquid and falsely indicate the presence of toxins, such are carbonyls, that may only be generated during misuse.

Gentle heating is expected not to preferentially volatilise some components of the cannabis extract and so change any toxicological delivery profile

To partially explore the usefulness of the method an experiment was conducted to determine the recovery rate from the cartridge using physical capture methods.

# Experimental

Using the Cerulean CETI1 aerosol generation machine, an experiment was performed on a Select vaporising system coupled with an Essential Berry Gelato flavour cartridge. The liquid was labelled as 92.1% of total Cannabinoids in a 1g cartridge. The product was selected for the extreme viscosity the THC liquid presented and was considered a “difficult” product to generate aerosol from.

Before use the cartridge was heated to approximately 50°C in an oven. Between puff blocks the cartridge was returned to the oven for 20 minutes.

Puff blocks were defined as being 40 puffs of 3 second duration, volume 55ml with a square shaped profile. Puffs were taken on a 30 second cycle. It was observed that the noticeably warm cartridge was still warm at the end of the puff block through heating from the coil.

For the vaping experiment the device was angled at 30° to the horizontal to mimic user preferred use patterns.

For each puff block a freshly charged battery pack was used. It had been noted in an earlier experiment that the battery packs could deliver between 80 and 120 puffs on this puffing regime before starting to show signs of discharge. Consequently, it was decided that the lower 40 puff cycle would ensure that no experimental artefacts would be introduced through low battery power.

The CETI1 aerosol generation machine was manufactured and supplied by Cerulean, Rockingham Drive, Milton Keynes UK and consists of a programmable puff engine with a maximum sweep volume of 70ml based around a stepper driven controlled precision aluminium bore syringe. The primary capture pad was a glass fibre pad supplied by Whatman, specified to capture 99.9% of all particles with a diameter of 0.3 micrometres or greater in a Cambridge Filter Holder (CFH). The secondary capture pad was a sealed “Helipet” type filter.

The device under test was activated by flow and was fitted with a square shaped mouthpiece. This was connected to the CFH labyrinth seals, a set of thin silicone seals that allow a seal to be formed gently around test products with cylindrical symmetry, via a short length of silicone tubing that pushed into the CFH seal.

The experimental arrangement is shown in Figure 1:

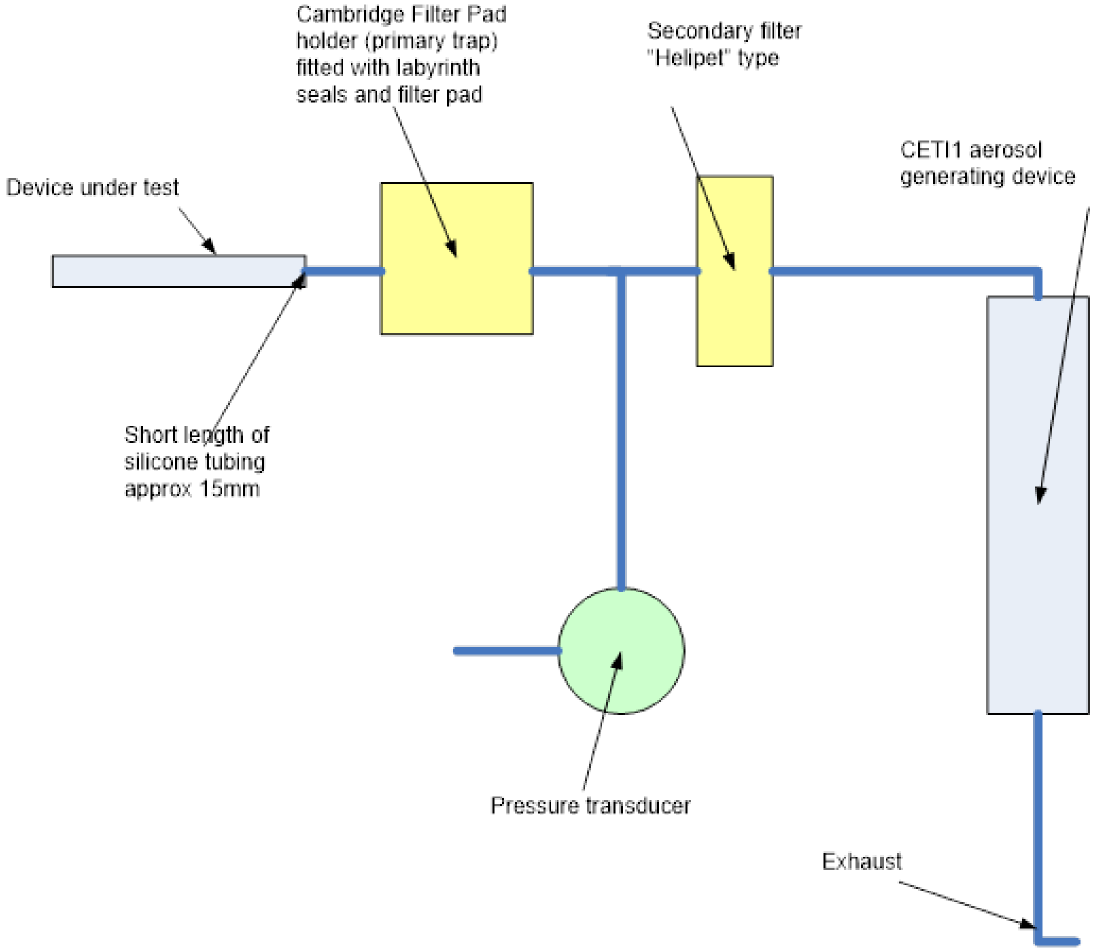


*Figure 1: Schematic of experimental apparatus*

A Baratron transducer was placed in the circuit beyond the primary capture pad to allow measurement of the pressure drop / flow in the puffing circuit during the puff blocks. This had a two-fold purpose; firstly to observe puff shape distortion through increased pressure drop through resistance to flow in either the capture pad or device and secondly to ensure that pressure drop was not rising and so restricting flow.

A 0.1mg resolution balance was used for mass balance calculations.

The experimental protocol required that the cartridge containing the cannabis liquid was weighed and then heated prior to use. The primary and secondary capture pads were both weighed. The primary capture pad was routinely "capped" to prevent any aerosol remaining within the CFH from escaping.

Once the cartridge was considered to have reached oven temperature – no precise measurements were made – the cartridge was weighed, and a fresh battery pack was fitted to the device.

This was then fitted onto the CFH which had been weighed and pre-loaded onto the CETI1 aerosol generating machine.

Just prior to analysis the puffing block cycle would be started as would the data logging software for the pressure drop / flow device.

At the end of the puff block the cartridge would be weighed and then returned to the oven. The CFH, properly capped, would be weighed as would the secondary capture pad.

This process was repeated for all puff blocks.

Percentage recovery was calculated from the loss in weight of the cartridge and the total weight gained by the two pads.

# Results

The problems inherent in the processes of vaping these very viscous liquids was observed in the second puff block. Here during the puff block the nominal 110mmWG pressure drop (see figure 2) increased rapidly to >500mmWG (see figure 3. Note the CETI1 puff engine did not stall even at this large pressure drop) and the flow through the vaping device dropped dramatically such that the activation indication device on the battery was illuminated only intermittently or for a very short period.

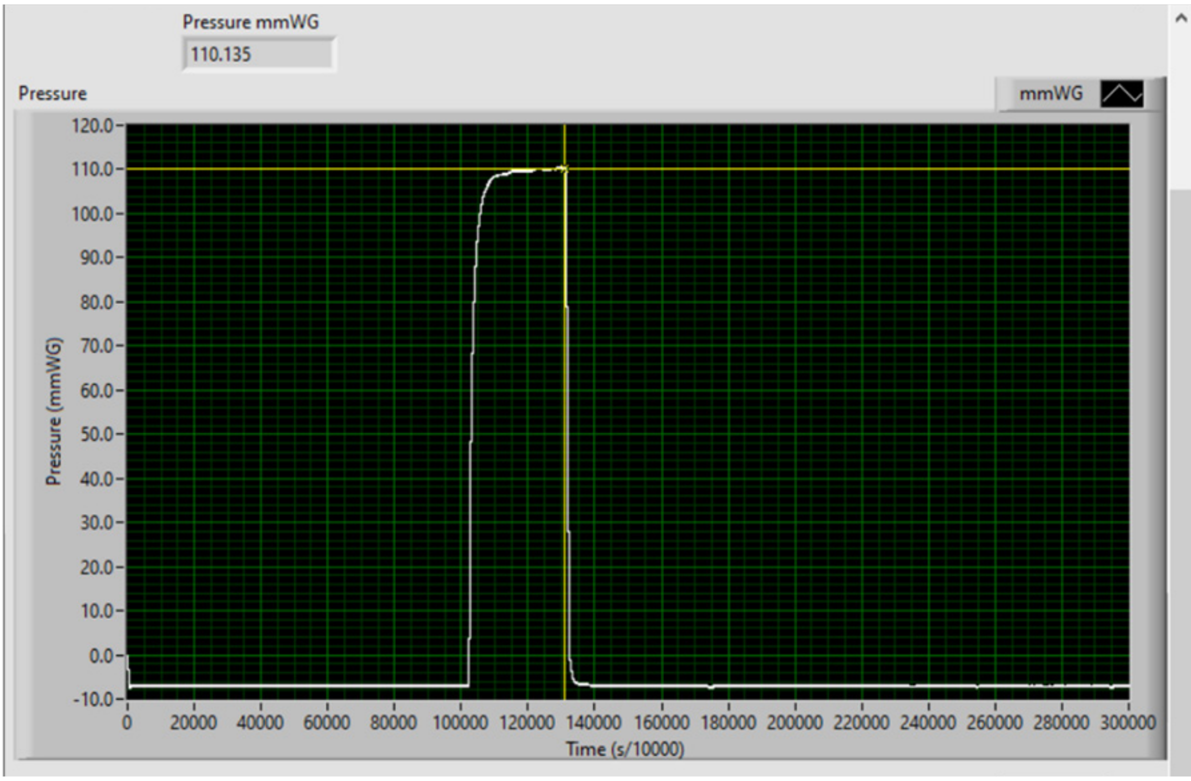


*Figure 2: Pressure drop trace as captured by the Baratron transducer and logging system. Trace is for the Select vaping system with high cannabinoid content cannabis oil. The pressure drop of 110mmWg is typical of a correctly operating device that is producing aerosol. Note slight distortion/rounding of leading edge of the nominally square wave form as a slight vacuum is produced in the system before aerosol is generated.*

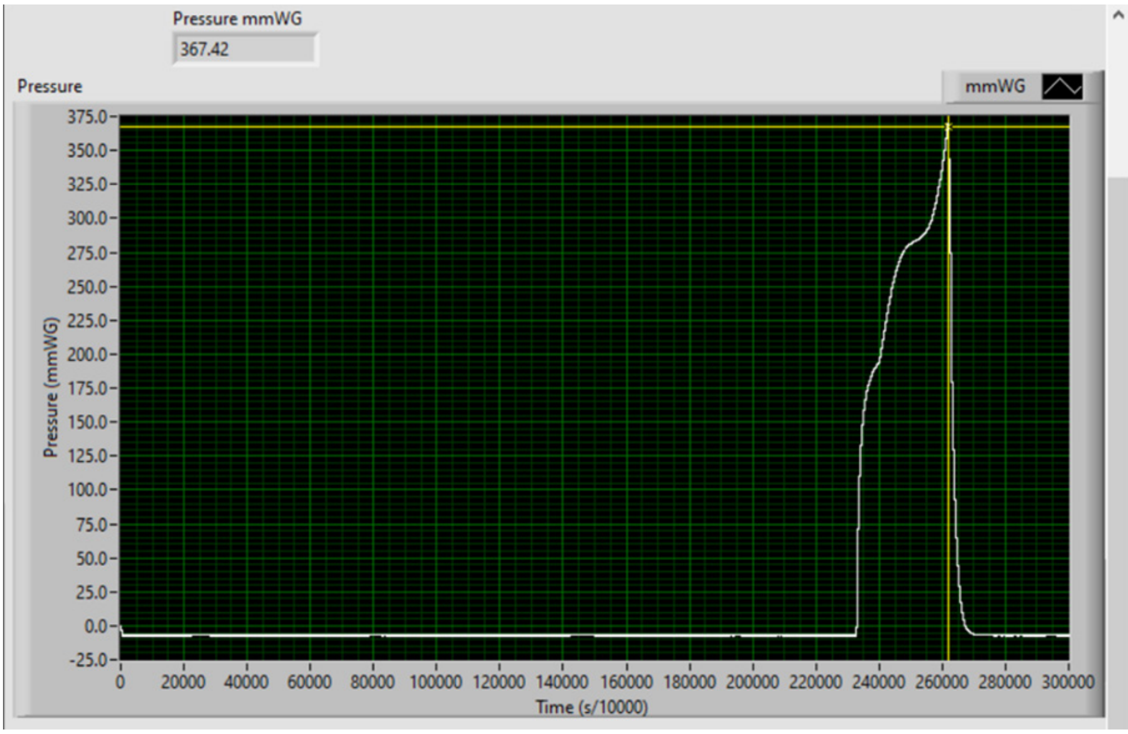


*Figure 3: Highly distorted pressure drop profile caused by resistance to draw of the cannabis oil under test. This distortion increases as the cannabis oil cools or as the battery pack begins o fail, reducing the heating effect of the coil. Eventually the waveform with over range at 500mmWg indicating a total failure to produce aerosol.*

This puff block was terminated prematurely after 35 puffs and it was observed that the cartridge was cold and had several bubbles frozen within the cartridge (see figure 4). It was postulated that these had formed when pulling air through the liquid and then "set" in place when cooled. These had produced a temporary blockage to flow, causing further cooling of the liquid.

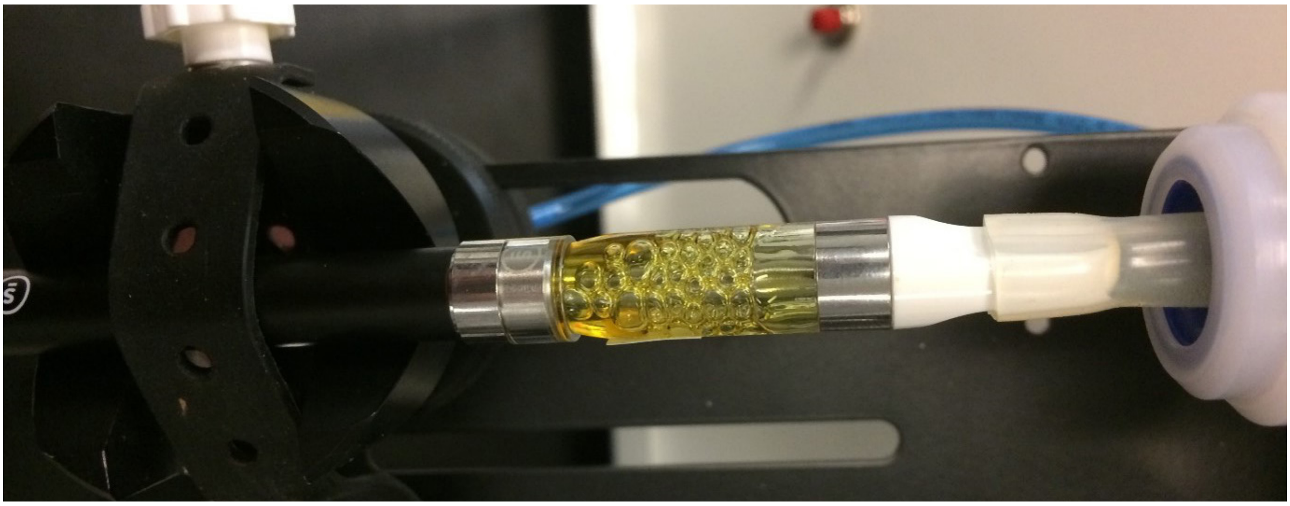

*Figure 4: Cannabis vape cartridge showing "frozen" liquid bubbles within the cartridge. Returning the cartridge to an oven for 30 minutes restored some fluidity to the liquid although not all bubbles disappeared.*

Returning the cartridge to the oven for 30 minutes restored some fluidity to the cannabis liquid and the test could be resumed. Thereafter the pressure drop of the system was carefully monitored to ensure that no further blocks to flow occurred.

The results of the mass balance experiment is shown in table 1
(all mass units are grams)

| Puff block | Puffs | Cartridge mass loss | Primary CFH mass gain | Secondary filter mass gain | Total mass recovered | Total recovery % | Primary recovery % |
|---|---|---|---|---|---|---|---|
| 1 | 40 | 0.20719 | 0.2052 | 0 | 0.2057 | 99% | 99% |
| 2 | 35 | 0.08711 | 0.0845 | 0 | 0.0846 | 97% | 97% |
| 3 | 40 | 0.2278 | 0.2240 | -0.0001 | 0.2242 | 98% | 98% |
| 4 | 40 | 0.1201 | 0.1162 | 0.0008 | 0.1181 | 98% | 97% |
| 5 | 40 | 0.0264 | 0.0246 | -0.0011 | 0.0215 | 81% | 93% |
| Total | | 0.6686 | 0.6545 | -0.0004 | 0.6541 | 98% | 98% |

*Table 1: recovery weights from Select Essential Berry Gelato cannabis cartridge*

As the cannabis liquid is essentially hydrophobic in nature the mass balance is not expected to be unduly influenced by the laboratory humidity. This was not controlled nor were there any conditioning steps taken for the capture pads used. The small loss in weight of the secondary filter during the final stage of the experiment could have been some drying of this filter by the drawing of air through the pad.

Post experiment observation of the capture pad showed that the aerosol was captured in a well distributed manner without any “breakthrough” on the pad or hotspots for capture for example directly in line with the mouthpiece. See figure 5.

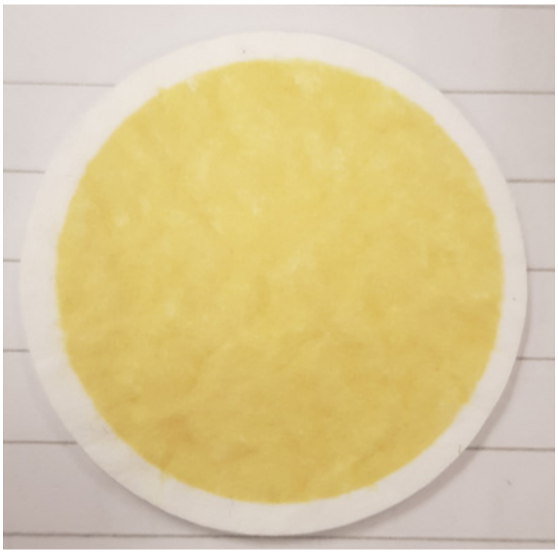

*Figure 5: Fibre pad after 40 puffs of select essential berry gelato THC aerosol had been captured. Note even distribution of condensate.*

# Discussion

The very high recovery rates for the aerosol generated are pleasing, particularly given the extreme difficulty in getting the liquid into an acceptable aerosol in the first place. Moreover, as can be seen from the secondary capture device the vast majority of the aerosol is captured by our primary capture system with recovery rates consistently greater than 90%.

The difficulty in maintaining the slight warming of the cartridge was evident from the second puff block where some cooling resulted in an increase in pressure drop and problems with vaporisation. An improved scheme would be to provide an indirect heat source that could be controlled for the cartridge in situ whilst puffing.

This indirect heating rather than direct heating within the liquid is proposed to ensure that experimental artefacts from overheating the liquid are not introduced; whilst this is not specifically required for metals analysis as mandated by the Colorado State regulator, it may well be extremely important when future toxicological studies are performed.

Overheating and decomposition products, particularly of terpenes possibly degrading to harmful carbonyls, are of course a function of the device heating and control, however it could be difficult to decouple the effects of a device being used to vaporise a cannabis liquid and the liquid itself, so exercising caution at the stage of method development would be prudent.

In this experiment capture of the aerosol was effected by the use of a glass fibre pad. It is a moot point if this is a suitable capture method for metals analysis as the glass fibre pad could contain trace metals and performing a blank analysis of the pad may not be sufficient to provide an analysis with the required precision or limits of detection and quantification. By analogy tobacco smoke methods do not use pad methods for metals analysis. Instead they would use impinger methods when analysing Mercury but would typically use alternate systems for other trace metals such as an electrostatic precipitator trap[1]. Other methods for hydrophobic aerosol collection have been developed by the CDC and presumably could be adapted for cannabis products[2] . Tobacco smoke, and nicotine vaping aerosol, does have the distinct advantage of being easy to transport without condensation so recovery rates using these methods are good. It is feared that that in practice when vaping cannabis oils much of the volatilised oil will not reach the capture area for analysis.

The method of capture will in part be determined by the LOQ/LOD of subsequent measurement apparatus and also by the threshold for regulatory requirement – if this is sufficiently high that a "blank" analysis would be well below any regulatory threshold then the glass fibre pad could be used where the contribution from the liquid under test would need to be significant to cross any threshold.

One observation is that the capture method should be in close proximity to the device under test as condensation of aerosol occurs rapidly. The short silicone tube was examined at the end of the experimental procedure (this length of tube was considered part of the cartridge for the mass balance experiments) and condensate was observed very close to the mouthpiece.

Any capture system with long pathlengths to for example impingers risks a low recovery rate. Heating the transfer tubing may be an option to improve transport but a fine balance will need to be achieved between aerosol transport versus decomposition.

This readiness to condense could be exploited if the capture system was for instance comprised of quartz wool, loosely packed in the CFH or modified CFH so as not to impede flow from the device, that is situated in a cooling medium that would promote condensation.

# Conclusions

The method of preheating cannabis cartridges containing THC to a modest 50°C before commencing vaping has the desired effect of allowing reasonable aerosol deliveries of between 2 and 5mg per puff (cf. typical nicotine vaping recovery per puff of 2mg).

The recovery rate of the aerosol has been shown to be better than 90% (93% to 99% with an average of 98%) through the use of a simple glass fibre capture pad situated in close proximity to the device under test.

The applicability of gentle external heating to vapour production for metals analysis as mandated by the Colorado regulator is clear.

*1 " Determination of Ni, Pb, Cd, Cr, As and Se in mainstream Tobacco smoke". Health Canada Method T-109 1999.*

*2 Halstead, M.; Gray, N.; Gonzalez-Jimenez, N.; Fresquez, M.; Valentin-Blasini, L.;Watson, C.; Pappas, R.S. Analysis of Toxic Metals in Electronic Cigarette Aerosols Using a Novel Trap Design. J. Anal. Toxicol. 2020, 44, 149–155.*

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