PharmaHemp d.o.o. | Koprska 106 c | SI-1000 Ljubljana | Slovenia
+386 1 42 39 790 | info@pharmahemp.si | www.pharmahemp.com

REPORT
Laboratory testing of cannabis smoke filtration through cone-shaped filter tips

Dr. Bostjan Jancar, Mag. Valentina Malin, Mag. Janja Ahej
Pharmahemp Laboratories, PharmaHemp d.o.o., Koprska ulica 106c, Ljubljana, Slovenia

Date: October 23, 2024

ABSTRACT
This study evaluated the impact of different filter types on the cannabinoid content in cannabis smoke, specifically focusing on cannabidiol (CBD) and delta-9-tetrahydrocannabinol (THC). Three filter types—MTIP (cardboard shell), CTIP (aluminium shell), both supplied by I-nvention Delft, Netherlands, and a classic rolled cardboard filter—were tested using a smoking simulator under conditions resembling casual human smoking. The analysis of the smoke showed no significant differences in the concentrations of CBD and THC between the filter types. Approximately 40-50% of cannabinoids were lost during the smoking process, which was attributed to the burning characteristics of the cannabis rolls. The results indicate that the choice of filter does not significantly affect the amount of cannabinoids in the smoke.

* **In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com**

TABLE OF CONTENTS

1. INTRODUCTION

Conical filter tips and pre-rolled paper cones containing filter tips were supplied by I-nvention, Delft, Netherlands. Filter tips were of two types designated MTIP and CTIP, with the first based on cardboard shell and latter based on aluminium shell. The interiors of both filter tips consisted of spherical particles with approximate diameter of 2.3 mm.

The aim was to determine the impact of both filter types on the cannabinoid content in the smoke exiting the filter tip under conditions resembling regular smoking. For simulation of human smoking a smoking simulator with a controllable puffing cycle was used. The control of puff volume, puff duration and puff interval enable a replication of conditions occurring during casual human smoking. To evaluate the influence of filter tips on chemistry of the exiting smoke different methods have been applied, mostly aimed to research of tobacco smoke. Most widely applied are methods involving Cambridge filter pads in combination of specially suited Cambridge filter holder or gas washers containing appropriate combination of solvents for capturing particles and dissolving chemicals in the smoke. In this study we used laboratory-scale gas washers equipped with glass frit for creating tiny bubbles of gas stream and thus enhancing the interaction with the solvent. After each smoking experiment the solvent was analyzed and cannabinoids quantified, which enabled comparison of influence of filter tips.

2. MATERIALS AND METHODS

Flowers and leaves of legally cultivated Cannabis sativa L. plants were ground to approximate particle size of 0.5 to 1mm. The mixture was sampled and analyzed in 5 parallels using High-Performance Liquid Chromatography (HPLC – Agilent 1260 Infinity II with double diode array detector) applying an in-house validated method, the results are shown in Table I. From the ground material exact amounts were weighed (varying from 797 to 1186 mg) using analytical scale (Mettler Toledo XPE 204M) which were used for in-situ rolling of conically shaped rolls and filling of pre-rolled cones. Both variants were equipped with MTIP, CTIP and classic rolled cardboard filter tips.

A laboratory setup was assembled, comprising two gas washers with glass frits and a smoking simulator (Fei Hong Instrument Equipment Co.), connected via silicone tubing (Fig. 1).Conically shaped roll with filter was placed in front of the gas washers connected to the smoke simulator. Gas washers were filled with 200ml of a 80 : 20 mixture of n-Hexane : Absolute Ethanol to ensure solubility of neutral and acidic forms of cannabinoids. Smoke simulator was tested at several different puff volumes, durations and intervals to find optimal smoking conditions resembling actual human smoking. After each smoking experiment solvent mixtures in both gas washers were sampled and quantified for cannabinoids using validated HPLC method. These, together with the results from the analysis of cannabis before smoking, were used to calculate recoveries of two major cannabinoids cannabidiol (CBD) and delta-9-tetrahydrocannabinol (THC).

Table I: Concentration of cannabinoids in 5 samples of ground Cannabis sativa L. material used in experiments.

Cannabinod	$w_{Cannabis\_1}$ [%]	$w_{Cannabis\_2}$ [%]	$w_{Cannabis\_3}$ [%]	$w_{Cannabis\_4}$ [%]	$w_{Cannabis\_5}$ [%]	$w_{Cannabis\_Avg}$ [%]
CBDV	0,112	0,100	0,110	0,117	0,114	0,111
CBDA	4,420	4,440	4,360	4,420	4,490	4,426
CBGA	0,083	0,083	0,082	0,083	0,083	0,083
CBG	0,139	0,135	0,132	0,142	0,142	0,138
CBD	4,050	4,040	3,960	4,130	4,080	4,052
THCV	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
CBN	0,042	0,042	0,042	0,043	0,043	0,042
D-9-THC	0,154	0,156	0,150	0,156	0,155	0,154
D-8-THC	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
CBL	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
CBC	0,186	0,187	0,183	0,191	0,190	0,187
D-9-THCA	0,019	0,019	0,020	0,020	0,020	0,020
CBV	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
CBCA	0,099	0,101	0,096	0,098	0,099	0,099
CBT	0,033	0,032	0,038	0,039	0,037	0,036
CBE	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

After each experiment gas washers were sequentially washed with isopropanol, acetone, Milli-Q water and again with isopropanol. Glass frit from the gas washer closest to cannabis containing burning roll was soaked in 0.1M NaOH and subsequently washed as gas washers.

Three filters of each type, after randomly chosen experiment, were soaked in 12 ml of 80 : 20 of Hexane : Absolute Ethanol solution and ultrasonicated. The solutions were quantified for cannabinoids and the results were used to determine recovery at each filter type.

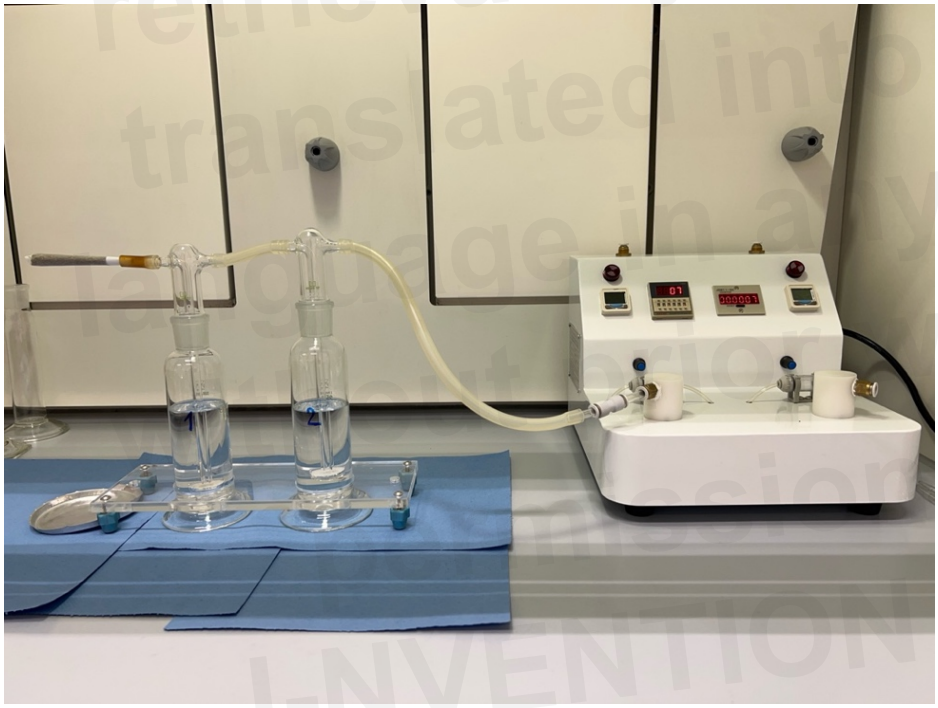

Fig. 1: Laboratory apparatus with gas washers and smoking simulator – before the experiment.

3. RESULTS AND DISCUSSION

Initial experiments showed that the optimal setting of the smoking simulator for a steady burn and keeping the solvent in the second gas washer (Solvent_2) clear was 3s puff duration with a 10s puff interval. HPLC quantification of cannabinoids with a Limit of Quantification (LOQ) of 3 ppm was used to analyze the solution in the second gas washer. At the set parameters of the smoking simulator the analysis always shows that the concentrations of all 16 tested cannabinoids were below LOQ after randomly chosen experiments. Under these conditions the concentration of cannabinoids in ash left after burning of the roll was also below LOQ. These findings imply that most of the cannabinoids in such experimental setup are dissolved in the solvent contained in the first gas washer (Solvent_1), lost with burning and/or retained by the filter tip.

After each experiment Solvent_1 was dark yellow and Solvent_2 was clear (Fig. 2). The HPLC quantification of Solvent 1 enabled calculation of CBD and THC recovery for each roll-filter combination. Results are shown in Tables II-IV.

Fig. 2: Laboratory apparatus with gas washers and smoking simulator – after the experiment.

Table II: CBD and THC recovery (%) in Solvent_1 for "conventional" cardboard rolled filters (Reference in CoAs found in appendix: CON).

Cannabinod	1	2	3	C_1	C_2	C_3	C_4	C_5	C_6	Average
CBD	45	44	45	34	42	41	48	36	35	41
D-9-THC	52	53	60	44	51	53	74	41	39	52

Table III: CBD and THC recovery (%) in Solvent_1 for MTIP filters (Reference in CoAs found in appendix: MTIP).

Cannabinod	2	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8	Average
CBD	44	33	47	42	42	39	52	42	43	43
D-9-THC	45	41	67	47	50	57	68	54	56	54

Table IV: CBD and THC recovery (%) in Solvent_1 for CTIP filters (Reference in CoAs found in appendix: CTIP).

Cannabinod	2	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8	Average
CBD	49	31	39	39	42	43	39	38	44	40
D-9-THC	60	33	43	46	46	60	44	44	55	48

The cannabis plant material used in experiments apart from neutral CBD and THC also contains acidic forms the canabidiolic acid (CBDA) and delta-9- tetrahydrocannabinolic acid (THCA). The recoveries shown in Tables II-IV were calculated based on assumption that decarboxylation of acidic forms only leads to neutral forms, which means that 0.877 molar mass ratio was applied in calculation. Comparison of values shown in Tables II-IV indicate that there are no significant differences between recoveries of CBD and THC regardless of the filter type used. Furthermore Tables II-IV indicate that 40-50% of CBD and THC is lost during the smoking experiment with all three tested filter tip types.

Tables V-VII show recoveries calculated based on analyses of filter tips after smoking experiments. The results shown indicate that 4-12% of CBD and THC can be retained by the filter tip during a smoking experiment. The most consistent recoveries were obtained for aluminium CTIP filter type. Further experiments are needed to improve statistics.

Table V: CBD and THC recovery (%) in filter tip for "conventional" cardboard rolled filters (Reference in CoAs found in appendix: CON).

Cannabinod	C_1	C_2	C_3	Average
CBD	5	4	8	6
D-9-THC	8	5	11	8

Table VI: CBD and THC recovery (%) in filter tip for MTIP filters (Reference in CoAs found in appendix: MTIP).

Cannabinod	C_3	C_7	2	Average
CBD	4	6	6	6
D-9-THC	8	11	12	10

Table VII: CBD and THC recovery (%) in filter tip for CTIP filters (Reference in CoAs found in appendix: CTIP).

Cannabinod	C_1	C_2	C_3	Average
CBD	5	5	4	5
D-9-THC	7	7	4	6

The results of the experiments indicate that there are no significant differences between the three tested filter types regarding concentration of cannabinoids CBD and THC in the smoke that exist the filter during smoking of cannabis-filled rolls.

Figs. 3-6 show graphical presentation of results in the form of diagrams.

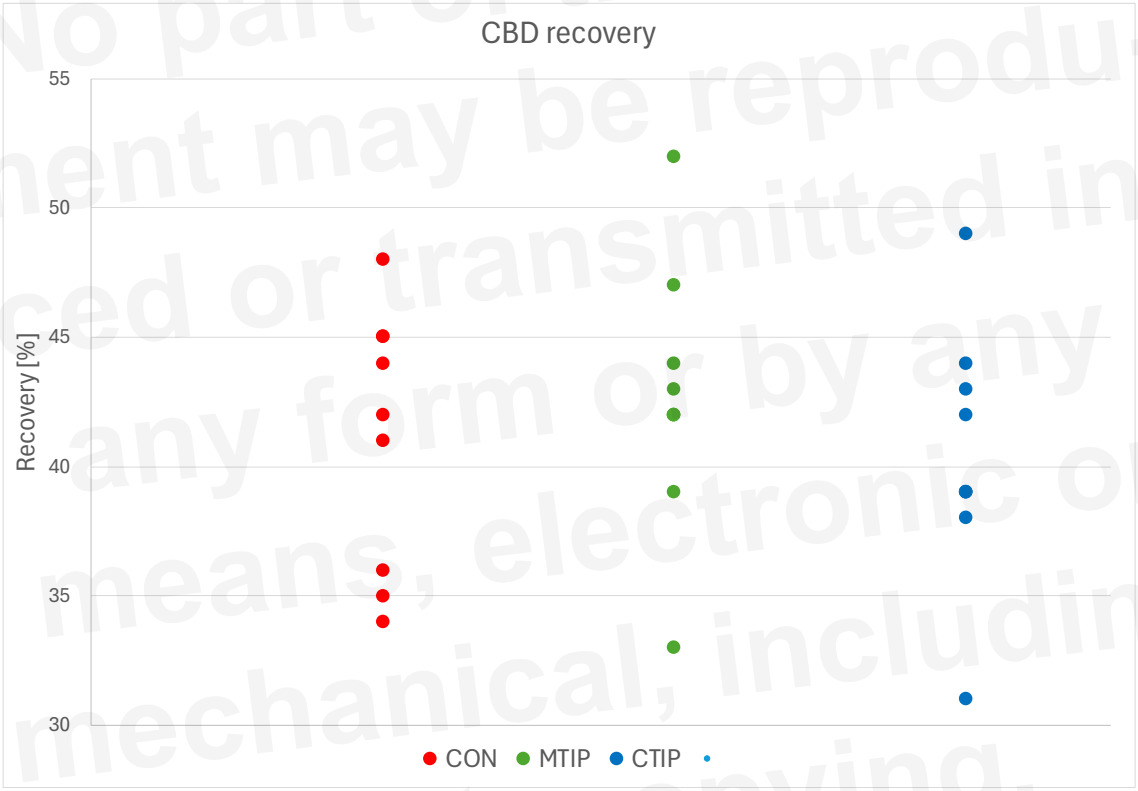

Fig. 3: Recovery of CBD in Solvent_1 for conventional cardboard rolled filters, MTIP and CTIP.

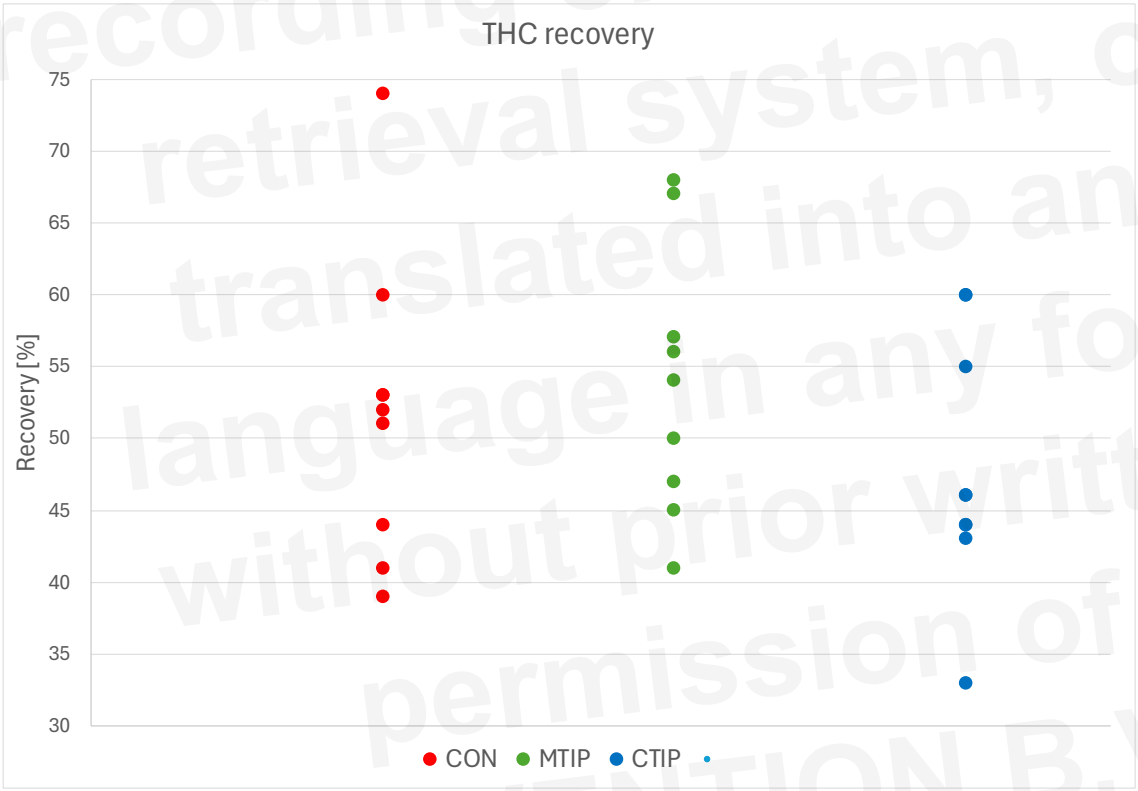

Fig. 4: Recovery of THC in Solvent_1 for conventional cardboard rolled filters, MTIP and CTIP.

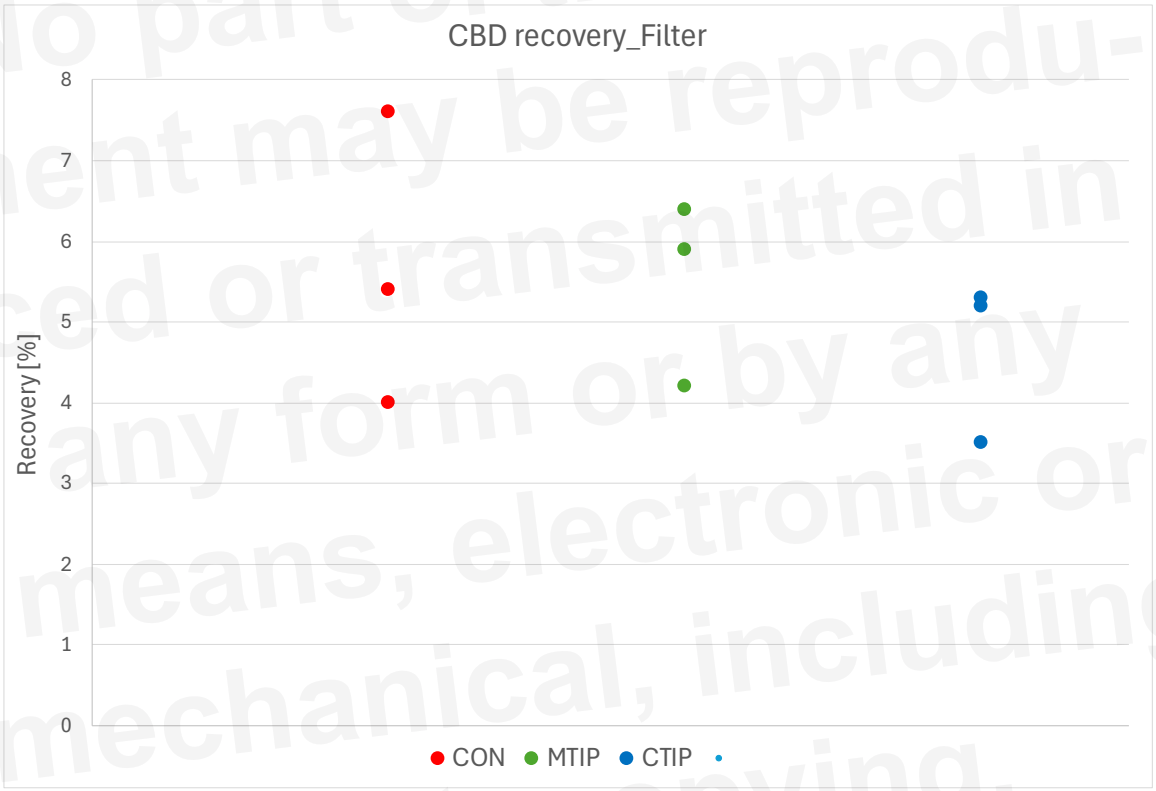

Fig. 5: Recovery of CBD in filters for conventional cardboard rolled filters, MTIP and CTIP.

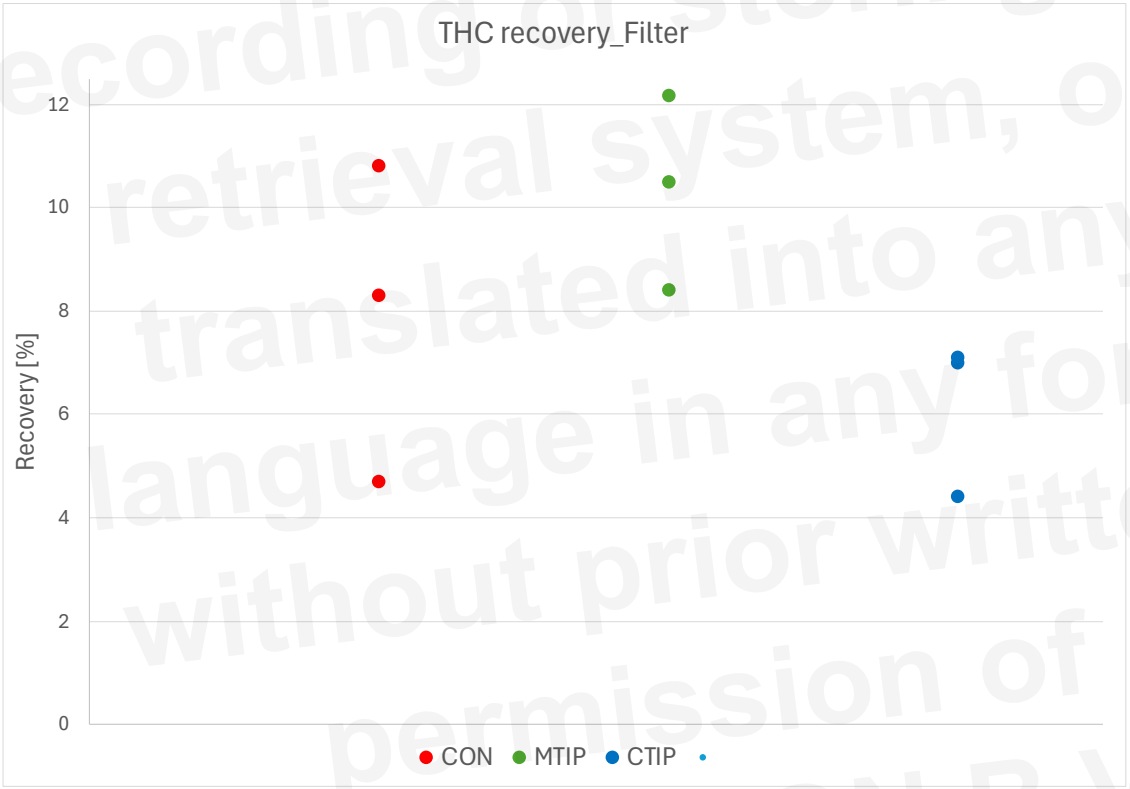

Fig. 6: Recovery of THC in filters for conventional cardboard rolled filters, MTIP and CTIP.

4. CONCLUSIONS

This study demonstrates that there are no significant differences between the tested filter types (MTIP, CTIP provided by I-vention, Delft, Netherlands, and classic rolled cardboard filter) regarding the levels of CBD and THC in the smoke. All filters allowed similar amounts of cannabinoids to pass through. Additionally, around 40-50% of the cannabinoids were lost during the smoking process, likely due to the burning characteristics of the cannabis rolls rather than the filter type. The results indicate that the choice of filter does not significantly impact the cannabinoid content in the smoke.

5. SUPPLEMENTARY ANALYSIS

Date: December 12, 2024

Based on the results and conclusions described in chapters 3 and 4 of this report, additional analyses of cannabinoid content in cannabis smoke, focusing on CBD and THC, were performed at the request of I-nvention Delft, Netherlands. Five different types of pre-rolled paper cones containing distinct types of conical cardboard filters were used for the analyses. The cones were named as follows: *, AND * (Fig. 7).

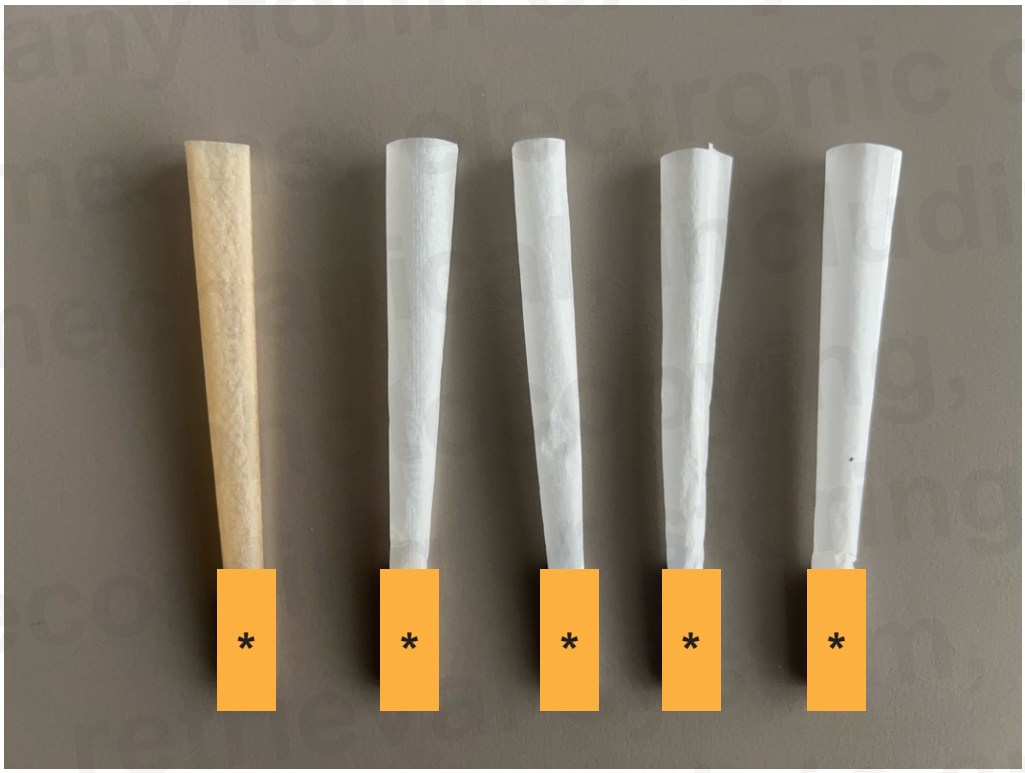

Fig. 7. Pre-rolled paper cone types used in experiments. From left to right: *, *.

The equipment and methods used in supplementary analyses were identical to those described in the previous experiments in chapter 2: Materials and Methods. The same batch of ground flowers and leaves of legally grown Cannabis sativa L. plants, the same analytical equipment, the same laboratory setup, and the same puffing machine with identical settings were used in these experiments.
The results of HPLC quantification and subsequent calculation of CBD and THC recovery in Solvent_1 for the five pre-rolled cone types are presented in Tables VIII-XII.

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com

Table VIII: CBD and THC recovery (%) in Solvent_1 for * pre-rolled cones (Reference in CoAs found in appendix: *.

Cannabinod	C_1	C_2	C_3	C_4	Average
CBD	36	27	31	33	32
D-9-THC	34	39	35	36	36

Table IX: CBD and THC recovery (%) in Solvent_1 for * pre-rolled cones (Reference in CoAs found in appendix: *.

Cannabinod	C_1	C_2	C_3	C_4	Average
CBD	36	29	42	36	36
D-9-THC	36	32	51	41	40

Table X: CBD and THC recovery (%) in Solvent_1 for * pre-rolled cones (Reference in CoAs found in appendix: *.

Cannabinod	C_1	C_2	C_3	C_4	Average
CBD	33	38	34	36	35
D-9-THC	28	45	40	37	38

Table XI: CBD and THC recovery (%) in Solvent_1 for * pre-rolled cones (Reference in CoAs found in appendix: *.

Cannabinod	C_1	C_2	C_3	C_4	Average
CBD	40	42	37	40	40
D-9-THC	36	51	32	43	41

Table XII: CBD and THC recovery (%) in Solvent_1 for * pre-rolled cones (Reference in CoAs found in appendix: *.

Cannabinod	C_1	C_2	C_3	C_4	Average
CBD	41	44	39	41	41
D-9-THC	38	48	42	40	42

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com

As in the previous experiments, the recoveries were calculated based on the assumption that decarboxylation of acidic forms results solely in neutral forms (using a 0.877 molar mass ratio for calculation). The results presented in Tables VIII-XII indicate recoveries of CBD and THC in the range of 35% to 42% for all pre-rolled cone types. Among the tested cones, * and * demonstrated the highest and most consistent recoveries. These are comparable to but slightly lower than the previously tested MTIP, CTIP, and conventional cardboard rolled filter tips.

Graphical comparison of recoveries of CBD and THC in Solvent_1 for all tested filter tips is shown in Figs. 8 and 9.

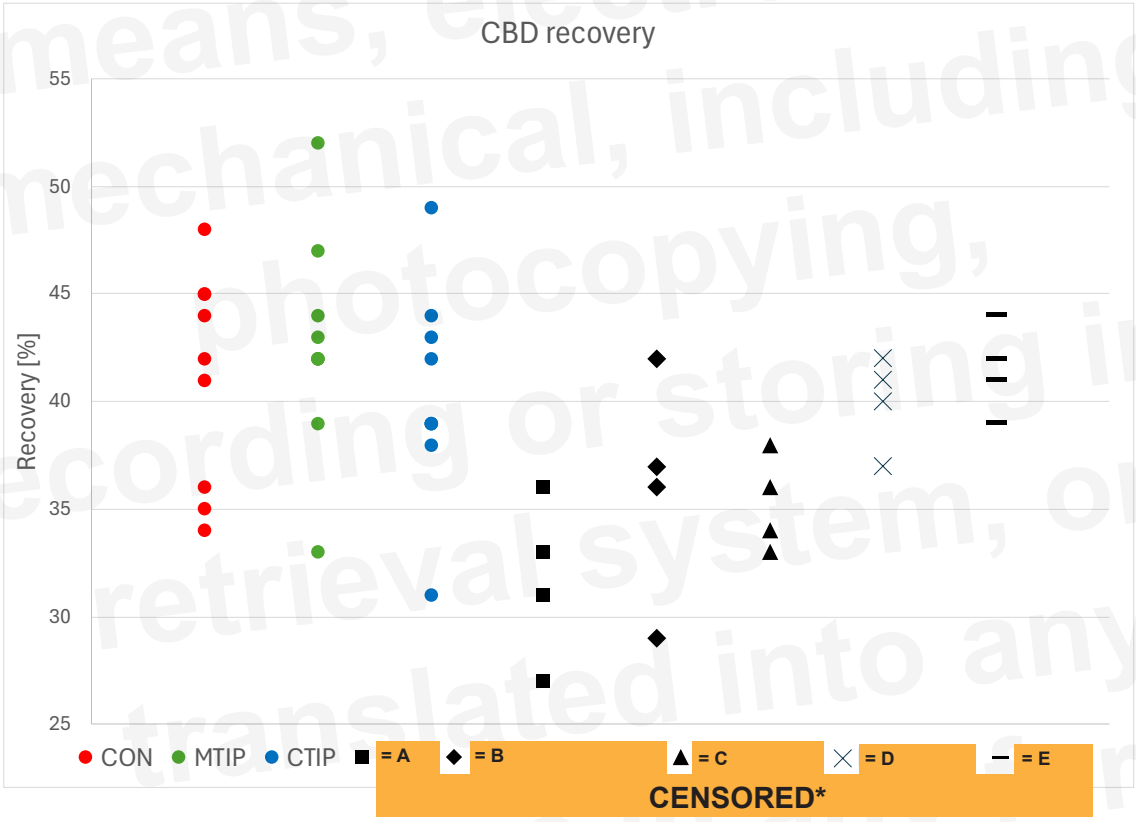

Fig. 8: Recovery of CBD in Solvent_1 for all filter types tested.

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com

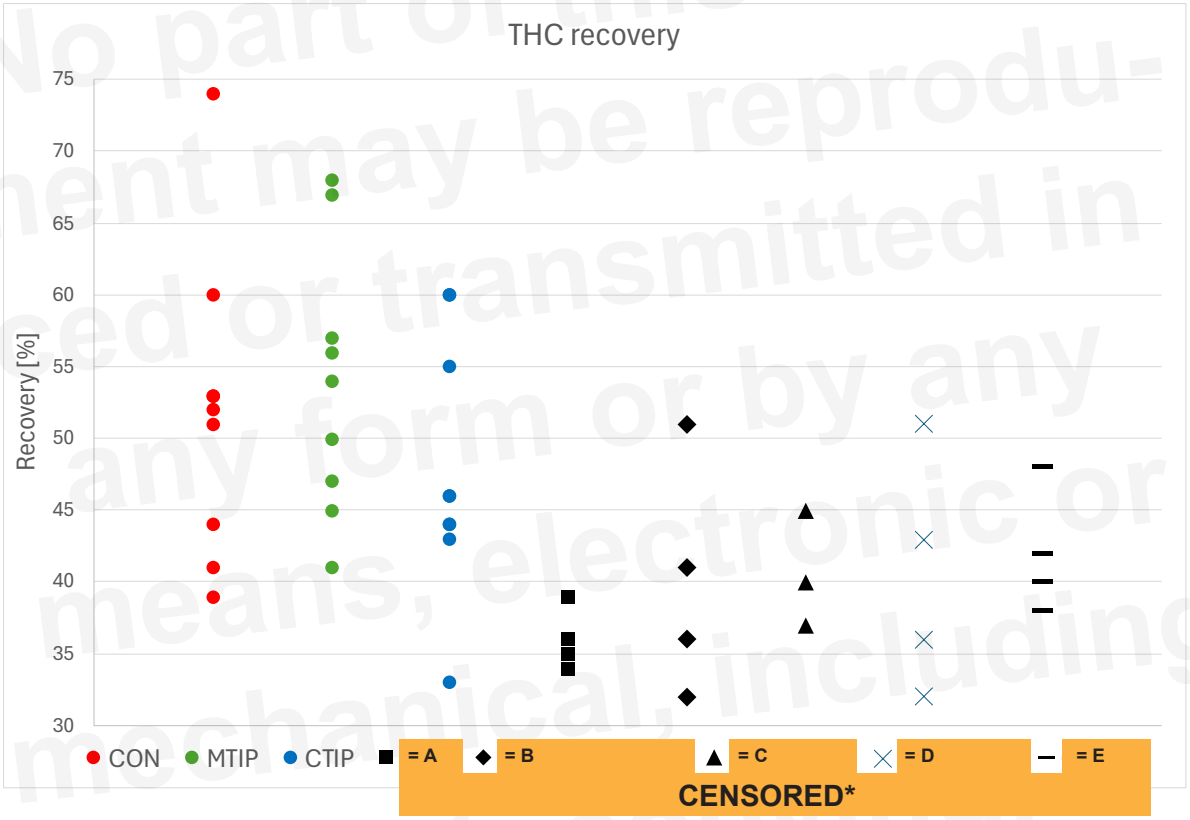

Fig. 9: Recovery of THC in Solvent_1 for all filter types tested.

As with previous experiments, the supplementary analysis also involved determining recoveries based on the analysis of filter tips according to the procedure described in chapter 2 – Materials and Methods. The calculated recoveries are presented in Tables XIII-XVII. Consistent with prior experiments conducted on conventional, CTIP, and MTIP filters, the findings suggest that 4-12% of CBD and THC might be retained by the filter tip during smoking trials. This is depicted graphically in Figs. 10 and 11.

Table XII: CBD and THC recovery (%) in filter tip for * filters (Reference in CoAs found in appendix: *).

Cannabinod	C_1	C_2	C_3	Average
CBD	5	4	3	4
D-9-THC	8	6	6	7

Table XIV: CBD and THC recovery (%) in filter tip for * filters (Reference in CoAs found in appendix: *).

Cannabinod	C_1	C_2	C_3	Average
CBD	8	4	6	6
D-9-THC	12	5	10	9

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com

Table XV: CBD and THC recovery (%) in filter tip for * filters (Reference in CoAs found in appendix: *).

Cannabinod	C_1	C_2	C_3	Average
CBD	4	7	5	5
D-9-THC	6	10	9	8

Table XVI: CBD and THC recovery (%) in filter tip for * filters (Reference in CoAs found in appendix: *).

Cannabinod	C_1	C_2	C_3	Average
CBD	7	7	6	7
D-9-THC	11	10	9	10

Table XVII: CBD and THC recovery (%) in filter tip for * filters (Reference in CoAs found in appendix: *).

Cannabinod	C_1	C_2	C_3	Average
CBD	9	7	7	8
D-9-THC	12	11	13	12

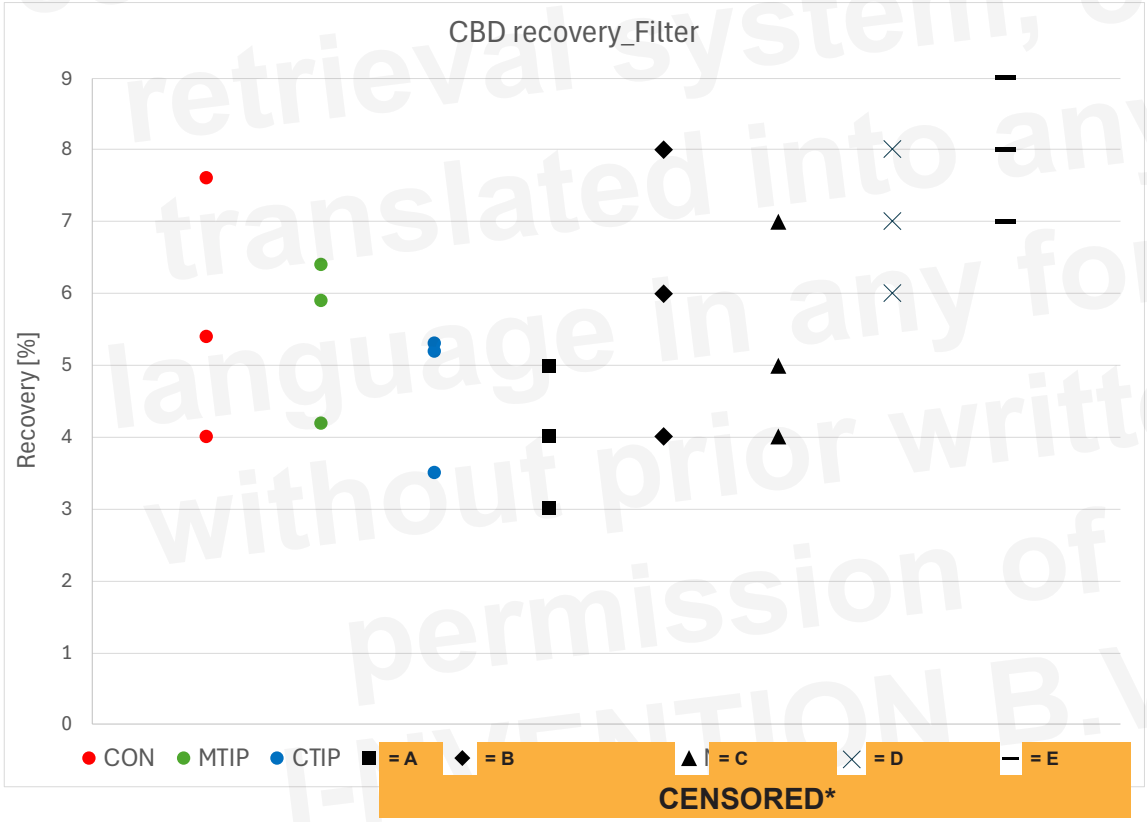

Fig. 10: Recovery of CBD in filters for all filter types tested.

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com

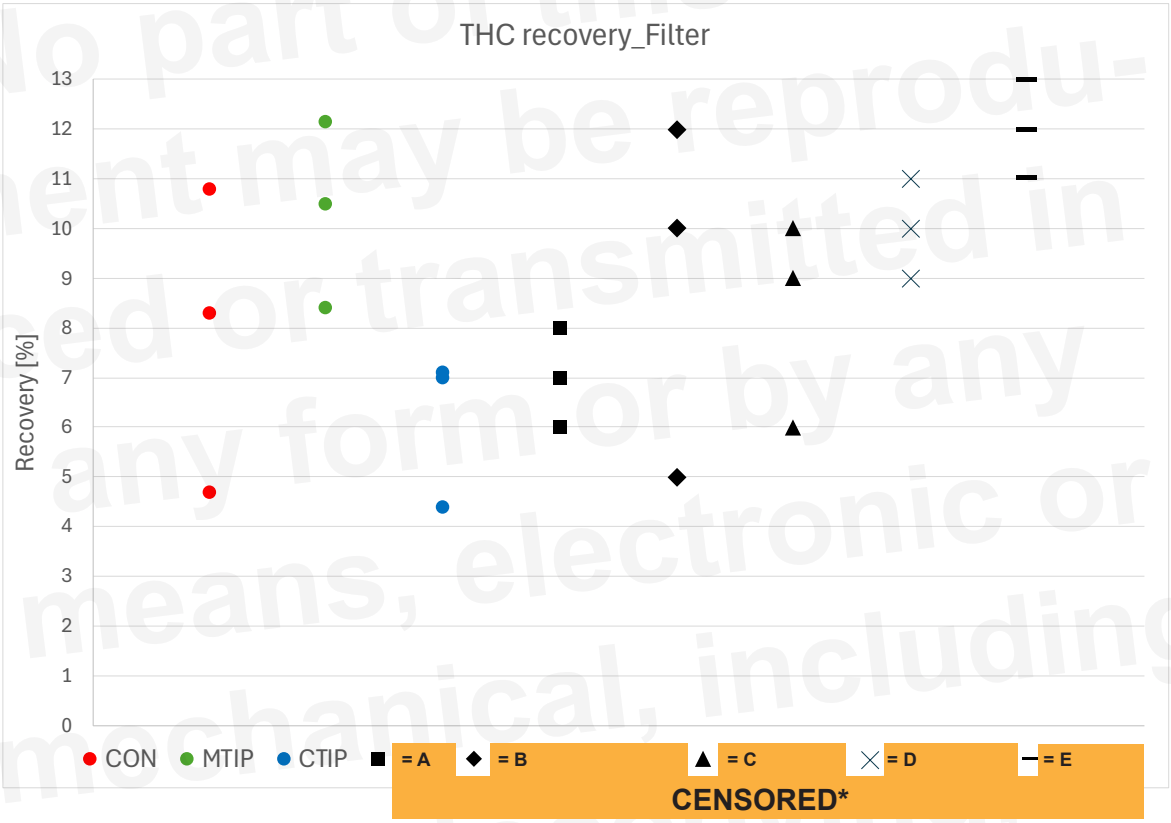

Fig. 11: Recovery of CBD in filters for all filter types tested.

The comparison between the supplementary analysis of pre-rolled cones (* * and *) and the previously tested CTIP (aluminum shell) and MTIP (cardboard shell) filters revealed similar cannabinoid recovery patterns. Cannabinoid recoveries in Solvent 1 for CTIP and MTIP filters ranged from 40% to 50%, while pre-rolled cones exhibited recoveries between 35% and 42%, with * showing the highest and most consistent results most comparable to CTIP and MTIP. Regarding cannabinoid retention within the filters, both CTIP and MTIP filters retained 4-12% of cannabinoids, with CTIP demonstrating more consistent performance. Similarly, pre-rolled cones showed comparable retention rates.. These results suggest that while CTIP and MTIP filters offer slightly higher cannabinoid recoveries, the differences are minor. Overall, the findings indicate that filter material and design play a limited role in cannabinoid recovery, with burn characteristics being the primary factor influencing cannabinoid loss during smoking.

* In this document the actual brandnames are censored. We have chosen the market leading brands to compare with our products. For those of you that would like to discuss the uncensored verion of the report, please contact: legal@i-nvention.com