Cannabidiol Oil Analysis with the Pyroprobe

Application Note
Pharmaceutical - Cannabis

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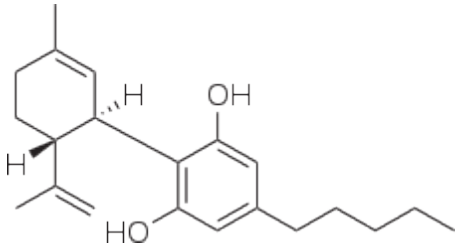
Abstract

Thermal Analysis of CBD oil using a CDS Model 6150 Pyroprobe.

With promise for treating a variety of ailments, cannabidiol (CBD oil) derived from the cannabis plant, has a growing interest in the pharmaceutical industry. The non-intoxicating extract is being credited with helping to treat many medical issues. Cut with hemp oil prior to use, it is a complex natural product, containing many volatile and non-volatile constituents. Successive thermal treatments using the Pyroprobe can clarify ingredients in natural materials such as CBD oil by separating ingredients based on their volatility, then pyrolyzing the non-volatile portion, like the oil itself.

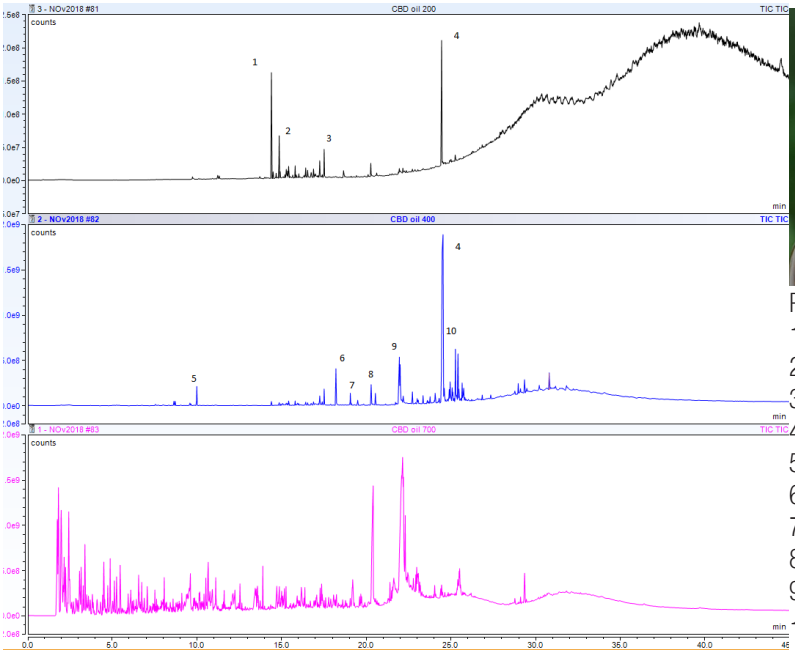


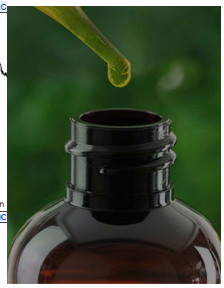
Cannabidiol

About 500 micrograms of 5% CBD in organic hemp oil was placed into a quartz DISC tube, and run on a CDS Model 6150 Pyroprobe interfaced to a GC-MS. The Pyroprobe was programmed to heat first to 200°C, and then to 400°C to look at semi-volatiles in the oil; then finally to 700°C to examine pyrolysis products. Before each of these heating steps, the Pyroprobe waits for the GC to become ready, then starts the GC as the heating begins.

Chromatograms of all 3 temperature runs are shown in Figure 1. At 200°C, some of the oil starts to vaporize, exhibiting as an unresolved mixture at the end of the chromatogram. Along with this, active compounds in the oil, like the sesquiterpene α-Bisabolol (with anti-irritant, anti-inflammatory, and anti-microbial properties), α-Caryophyllene (often found in aromatic plants), and the cannabinoid, CBD (active ingredient) vaporize. Then, at 400°C some fatty acids, alcohols like olivetol and phytol (a diterpene alcohol), and more cannabinoids evolve. Finally, at 700°C, the remainder of the oil pyrolyzes, breaking down the remaining triglycerides of the oils into long chain alkenes, alkanes, alkynes, aldehydes, and alcohols. A more detailed list of peak search results are found in Tables 1-3.

Performing multi-step thermal analysis on both pharmaceuticals and natural products which make up pharmaceuticals, can help an analyst find distinct components in their chemical make-up, providing valuable information for competitive analysis and product development.





Peak #	Identification
1	Caryophyllene
2	Humulene
3	a-Bisabolol
4	Cannabidiol
5	1,3,8-p-Menthatriene
6	Olivetol
7	Phytol
8	n-Hexadecanoic acid
9	Linoleic acid
10	δ1-Tetrahydrocannabinol

Figure 1. 5% CBD oil in hemp oil, 200°C (top), 400°C, and 700°C (bottom).

Experimental Parameters

The sample was pyrolyzed in a DISC tube, using a CDS Model 6150 Pyroprobe.

Pyrolysis:	200, 400, 700°C 30s
Interface:	300°C
Transfer Line:	325°C
Valve Oven:	300°C

GC/MS

Column:	5% phenyl (30m x 0.25mm)
Carrier:	Helium, 50:1 split
Injector:	320°C
Oven:	40°C for 2 minutes 10°C/min to 300°C hold 15 minutes
Ion Source:	230°C
Mass Range:	35-600amu

Table 1: 200°C Library Search Results

Ret.Time (min)	Top Hit
9.74	Linalool
11.23	a-Terpineol
11.32	Decanal
13.71	a-ylangene
14.40	Caryophyllene
14.50	a-Bergamotene
14.64	a-ylangene
14.68	cis-ß-Farnesene
14.86	Humulene
14.92	Alloaromadendrene
15.05	ß-copaene
15.20	Patchoulene
15.28	g-Selinene
15.66	Patchoulene
15.80	a-Bisabolene
15.86	ß-Guaiene
15.95	Columbin
16.02	Formic acid, 3,7,11-trimethyl-1,6,10-dodecatrien-3-yl ester
16.08	N,N'-Bis(Carbobenzyloxy)-lysine methyl(ester)
16.42	Caryophyllene oxide
16.54	5-Azulenemethanol, 1,2,3,4,5,6,7,8-octahydro-a,a,3,8-tetramethyl-
16.69	Ethyl iso-allocholate
16.89	ß-Guaiene
17.00	a-acorenol
17.18	Pregan-20-one, 2-hydroxy-5,6-epoxy-15-methyl-
17.33	Columbin
17.51	a-Bisabolol
17.82	5,8,11,14-Eicosatetraynoic acid
18.66	Phenanthrene
20.27	n-Hexadecanoic acid
21.39	Ethyl iso-allocholate
22.00	7,8-Epoxylanostan-11-ol, 3-acetoxy-
22.26	Astaxanthin
22.88	Arenobufagin
24.45	Cannabidiol
24.51	7,8-Epoxylanostan-11-ol, 3-acetoxy-
24.58	Cinobufotalin
32.21	Lycoxanthin

Table 2: 400°C Library Search Results

Ret.Time (min)	Top Hit
5.02	Hexane, 2,4-dimethyl-
5.25	2-Octene
6.48	2-Methyl-1-hepten-3-yne
6.78	Heptanal
7.54	2-Heptenal, (Z)-
7.72	2,6-Dimethyl-1,3,5,7-octatetraene, E,E-
8.05	Cyclohexene, 5-methyl-3-(1-methylethenyl)-, trans-(-)-
8.64	2,6-Dimethyl-1,3,5,7-octatetraene, E,E-
8.72	D-Limonene
9.59	Cyclohexene, 1-methyl-4-(1-methylethylidene)-
9.65	Benzene, 1-methyl-4-(1-methylethenyl)-
9.85	13-Heptadecyn-1-ol
10.00	p-Mentha-1,5,8-triene
10.18	Cyclopentene,1-hexyl-
11.06	6-Dodecene, (Z)-
14.40	Caryophyllene
14.96	5-Hexadecyne
15.06	9,17-Octadecadienal, (Z)-
15.28	b-Selinene
15.36	a-Selinene
15.42	ß-Bisabolene
15.80	a-Bisabolene
15.86	ß-Guaiene
15.95	3,5,11-Eudesmatriene)-
16.42	Caryophyllene oxide
17.00	Aromadendrene oxide-(2)
17.05	Caryophylla-4(12),8(13)-dien-5a-ol
17.11	a-acorenol
17.33	Isoaromadendrene epoxide
17.40	ß-Guaiene
17.51	a-Bisabolol
17.90	1-Hexadecanol, 2-methyl-
18.21	1,3-Benzenediol, 5-pentyl-
18.46	Z,Z-3,15-Octadecadien-1-ol acetate
18.61	Pregan-20-one, 2-hydroxy-5,6-epoxy-15-methyl-
19.00	1-Heptatriacotanol
19.06	Neophytadiene
19.11	2-Pentadecanone, 6,10,14-trimethyl-
19.30	Ethanol, 2-(9-octadecenyloxy)-, (Z)-
19.84	1-Heptatriacotanol
20.29	n-Hexadecanoic acid
20.33	Ethyl iso-allocholate
20.38	13-Heptadecyn-1-ol
20.54	Cannabicyclol
21.49	17-Pentatriacontene
21.73	Phytol
21.79	Gamolenic acid
21.95	(Z)-18-Octadec-9-enolide
22.00	cis-Vaccenic acid
22.18	Octadecanoic acid
22.37	Ethyl iso-allocholate
22.72	Uvaol
23.01	Ethanol, 2-(9,12-octadecadienyloxy)-, (Z,Z)-
23.06	Z,Z-3,15-Octadecadien-1-ol acetate
23.35	.DELTA.8-Tetrahydrocannabinol
23.48	7,8-Epoxylanostan-11-ol, 3-acetoxy-
24.51	Cannabidiol
24.53	Resorcinol, 2-p-mentha-1,8-dien-3-yl-5-pentyl-, (-)-(E)-
25.19	11-Acetoxy-d8-tetrahydrocannabinol
25.26	Dronabinol
25.32	Hydroxy-d 9-tetrahydrocannabinol, 8-a
26.41	7,8-Epoxylanostan-11-ol, 3-acetoxy-
26.59	Ethyl iso-allocholate
26.75	Docosanoic acid, 1,2,3-propanetriyl ester
28.97	Tocopherol
29.34	Astaxanthin
29.44	Rhodopin
30.80	Sitosterol

Table 3: 700°C Library Search Results

Ret.Time (min)	Top Hit
1.71	Carbon dioxide
2.12	1,3-Pentadiene
2.24	Bicyclo[2.1.0]pentane
2.44	1-Hexene
2.71	(Z),(Z)-2,4-Hexadiene
2.85	1,4-Cyclohexadiene
2.97	(Z),(Z)-2,4-Hexadiene
3.07	Benzene
3.16	1,3-Cyclopentadiene, 1-methyl-
3.24	trans-1,4-Hexadiene
3.29	Bicyclo[3.1.0]hexane
3.39	1-Heptene
3.94	1-Methylcyclohexa-2,4-diene
4.31	3,4-Heptadiene
4.88	1-Octene
5.03	Hexane, 2,4-dimethyl-
5.26	4-Octene, (E)-
5.38	Cyclopentene, 3-propyl-
5.70	Tricyclo[3.2.1.0(1,5)]octane
5.89	1,4-Heptadiene, 3-methyl-
5.94	2,4-Octadiene
6.04	E,Z-4-Ethylidenecyclohexene
6.07	2,4-Octadiene
6.10	Ethylbenzene
6.19	E,Z-4-Ethylidenecyclohexene
6.56	2-Nonene
6.65	3-Nonene, (E)-
6.74	Cyclooctene, (Z)-
7.07	1,3-Nonadiene, (E)-
8.07	1-Decene
9.38	3-Heptenoic acid
9.61	1-Undecene
13.89	1-Hexadecanol
20.37	n-Hexadecanoic acid
21.95	(Z)-18-Octadec-9-enolide
22.19	9,12-Octadecadienoic acid (Z,Z)-
24.40	9,12-Octadecadienoic acid (Z,Z)-, 2,3-dihydroxypropyl ester

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READINGS:

CDS Application Note #184a Pyrolysis GC-MS of Pharmaceutical Packaging

CDS Application Note #105a Cracking Products of Oleic Acid and Olive Oil

Sam, K. American Lab Multistep Thermal Characterization of Liquid-Filled Capsules and Medication Packaging Using GC/MS. Am. Lab, April 2018.