

**Date :** June 25, 2020

**CERTIFICATE OF ANALYSIS – GC PROFILING**

**SAMPLE IDENTIFICATION**

**Internal code :** 20F15-PTH04

**Customer identification :** Org Black Pepper - India - BS0103912R

**Type :** Essential oil

**Source :** *Piper nigrum*

**Customer :** Plant Therapy

**ANALYSIS**

**Method:** PC-MAT-007 - Analysis of the composition of an essential oil or other volatile liquide by FAST GC-FID (in French); identifications validated by GC-MS.

**Analyst :** Sylvain Mercier, M. Sc., Chimiste

**Analysis date :** June 23, 2020

Checked and approved by :

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Alexis St-Gelais, M. Sc., chimiste 2013-174

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*PHYSICOCHEMICAL DATA*

**Physical aspect:** Faintly yellow liquid

**Refractive index:**  $1.4802 \pm 0.0003$  (20 °C; method PC-MAT-016)

*CONCLUSION*

No adulterant, contaminant or diluent has been detected using this method.

## ANALYSIS SUMMARY – CONSOLIDATED CONTENTS

New readers of similar reports are encouraged to read table footnotes at least once.

Identification	%	Class
Ethanol	tr	Aliphatic alcohol
Isovaleral	tr	Aliphatic aldehyde
2-Methylbutyral	tr	Aliphatic aldehyde
Toluene	0.01	Simple phenolic
Tricyclene	0.01	Monoterpene
$\alpha$ -Thujene	1.37	Monoterpene
$\alpha$ -Pinene	12.83	Monoterpene
Camphene	0.36	Monoterpene
$\alpha$ -Fenchene	0.01	Monoterpene
meta-Cymene	0.02	Monoterpene
Sabinene	11.33	Monoterpene
$\beta$ -Pinene	10.64	Monoterpene
Myrcene	1.91	Monoterpene
$\alpha$ -Phellandrene	1.26	Monoterpene
Pseudolimonene	0.04	Monoterpene
Menthatriene isomer I	0.01	Monoterpene
$\Delta^3$ -Carene	9.46	Monoterpene
$\alpha$ -Terpinene	0.08	Monoterpene
ortho-Cymene	0.03	Monoterpene
para-Cymene	0.68	Monoterpene
Limonene	14.95	Monoterpene
$\beta$ -Phellandrene	1.60	Monoterpene
1,8-Cineole	0.04	Monoterpenic ether
(Z)- $\beta$ -Ocimene	0.02	Monoterpene
(E)- $\beta$ -Ocimene	0.16	Monoterpene
$\gamma$ -Terpinene	0.18	Monoterpene
cis-Sabinene hydrate	0.17	Monoterpenic alcohol
Isoterpinolene	0.10	Monoterpene
Terpinolene	0.36	Monoterpene
para-Cresol	0.01	Simple phenolic
para-Cymenene	0.02	Monoterpene
$\alpha$ -Pinene oxide	0.01	Monoterpenic ether
trans-Sabinene hydrate	0.10	Monoterpenic alcohol
Unknown	0.02	Unknown
Linalool	0.29	Monoterpenic alcohol
Verbenol analog?	0.01	Monoterpenic alcohol
Unknown	0.01	Oxygenated monoterpene
trans-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
cis-Limonene oxide	0.01	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.04	Monoterpenic alcohol
trans-para-Menth-2-en-1-ol	0.01	Monoterpenic alcohol
trans-Limonene oxide	0.01	Monoterpenic ether
trans-Verbenol	0.02	Monoterpenic alcohol
1,4-Dimethyl-4-acetylcyclohexene	tr	Monoterpenic ketone
meta-Mentha-4,6-dien-8-ol	tr	Monoterpenic alcohol

Pinocarvone	0.01	Monoterpenic ketone
<i>cis</i> -Sabinol	0.02	Monoterpenic alcohol
Terpinen-4-ol	0.29	Monoterpenic alcohol
<i>trans</i> -2-Caren-4-ol	0.01	Monoterpenic alcohol
meta-Cymen-8-ol	0.04	Monoterpenic alcohol
Unknown	0.01	Unknown
$\alpha$ -Terpineol	0.07	Monoterpenic alcohol
<i>cis</i> - $\alpha$ -Phellandrene epoxide (IPP vs Me)	0.03	Monoterpenic ether
Verbenone	0.06	Monoterpenic ketone
Unknown	0.01	Unknown
<i>trans</i> -Carveol	0.03	Monoterpenic alcohol
<i>cis</i> -Carveol	0.02	Monoterpenic alcohol
Cuminal	0.04	Monoterpenic aldehyde
Carvone	0.01	Monoterpenic ketone
Unknown	0.03	Unknown
<i>trans</i> -Ascaridole glycol	0.01	Monoterpenic alcohol
Bornyl acetate	tr	Monoterpenic ester
Unknown	0.05	Unknown
Methyl geranate	0.01	Monoterpenic ester
Bicycloelemene	0.01	Sesquiterpene
$\delta$ -Elemene isomer	0.01	Sesquiterpene
$\delta$ -Elemene	1.38	Sesquiterpene
$\alpha$ -Cubebene	0.17	Sesquiterpene
Cyclosativene I	0.08	Sesquiterpene
Cyclosativene II	0.02	Sesquiterpene
$\alpha$ -Copaene	2.67	Sesquiterpene
<i>cis</i> - $\beta$ -Elemene	0.01	Sesquiterpene
$\beta$ -Cubebene	0.27	Sesquiterpene
$\beta$ -Elemene	0.33	Sesquiterpene
Unknown	0.02	Unknown
Isocaryophyllene	0.01	Sesquiterpene
$\alpha$ -Gurjunene	0.07	Sesquiterpene
$\beta$ -Caryophyllene	19.19	Sesquiterpene
$\beta$ -Copaene	0.14	Sesquiterpene
<i>trans</i> - $\alpha$ -Bergamotene	0.08*	Sesquiterpene
$\alpha$ -Guaiene	[0.08]*	Sesquiterpene
Unknown	0.01	Sesquiterpene
$\alpha$ -Humulene	0.91	Sesquiterpene
Unknown	0.02	Unknown
allo-Aromadendrene	0.02	Sesquiterpene
( <i>E</i> )- $\beta$ -Farnesene	0.02	Sesquiterpene
$\beta$ -Santalene	0.06	Sesquiterpene
$\gamma$ -Gurjunene	0.01	Sesquiterpene
<i>trans</i> -Cadina-1(6),4-diene	0.02	Sesquiterpene
$\gamma$ -Muurolole	0.09	Sesquiterpene
Germacrene D	0.22	Sesquiterpene
$\beta$ -Selinene	0.29	Sesquiterpene
<i>trans</i> -Muurolole-4(15),5-diene	0.03	Sesquiterpene
$\alpha$ -Selinene	0.23	Sesquiterpene
epi-Cubebol	0.09	Sesquiterpenic alcohol
Bicyclogermacrene	0.04	Sesquiterpene
Viridiflorene	0.06	Sesquiterpene

α-Muurolene	0.29	Sesquiterpene
β-Bisabolene	0.72	Sesquiterpene
Cubebol	0.14	Sesquiterpenic alcohol
γ-Cadinene	0.07	Sesquiterpene
7-epi-α-Selinene	0.04	Sesquiterpene
δ-Cadinene	0.62	Sesquiterpene
<i>trans</i> -Calamenene	0.06	Sesquiterpene
( <i>E</i> )-γ-Bisabolene	0.01	Sesquiterpene
α-Calacorene	0.03	Sesquiterpene
( <i>E</i> )-α-Bisabolene	0.03	Sesquiterpene
Isocaryophyllene epoxide B	0.04	Sesquiterpenic ether
Germacrene B	0.04	Sesquiterpene
( <i>E</i> )-Nerolidol	0.04	Sesquiterpenic alcohol
Spathulenol	0.05	Sesquiterpenic alcohol
Caryophyllene oxide isomer	0.16	Sesquiterpenic ether
Caryophyllene oxide	0.67	Sesquiterpenic ether
Humulene epoxide I	0.02	Sesquiterpenic ether
Humulene epoxide II	0.04	Sesquiterpenic ether
α-Corocalene	0.01	Sesquiterpene
Alismol	0.19	Sesquiterpenic alcohol
Caryophylladienol II	0.04	Sesquiterpenic alcohol
τ-Cadinol	0.01	Sesquiterpenic alcohol
τ-Muurolol	0.02	Sesquiterpenic alcohol
α-Muurolol	0.09	Sesquiterpenic alcohol
<i>cis</i> -Calamenen-10-ol	tr	Sesquiterpenic alcohol
<i>trans</i> -Calamenen-10-ol	0.02	Sesquiterpenic alcohol
(3 <i>Z</i> )-Caryophylla-3,8(13)-dien-5β-ol	0.01	Sesquiterpenic alcohol
Dehydrojinkoh-eremol	tr	Sesquiterpenic alcohol
meta-Camphorene	0.02	Diterpene
para-Camphorene	0.03	Diterpene
<b>Consolidated total</b>	<b>99.05%</b>	

\*: Individual compounds concentration could not be found due to overlapping coelutions on columns considered [xx]: Duplicate percentage due to coelutions, not taken into account in the consolidated total

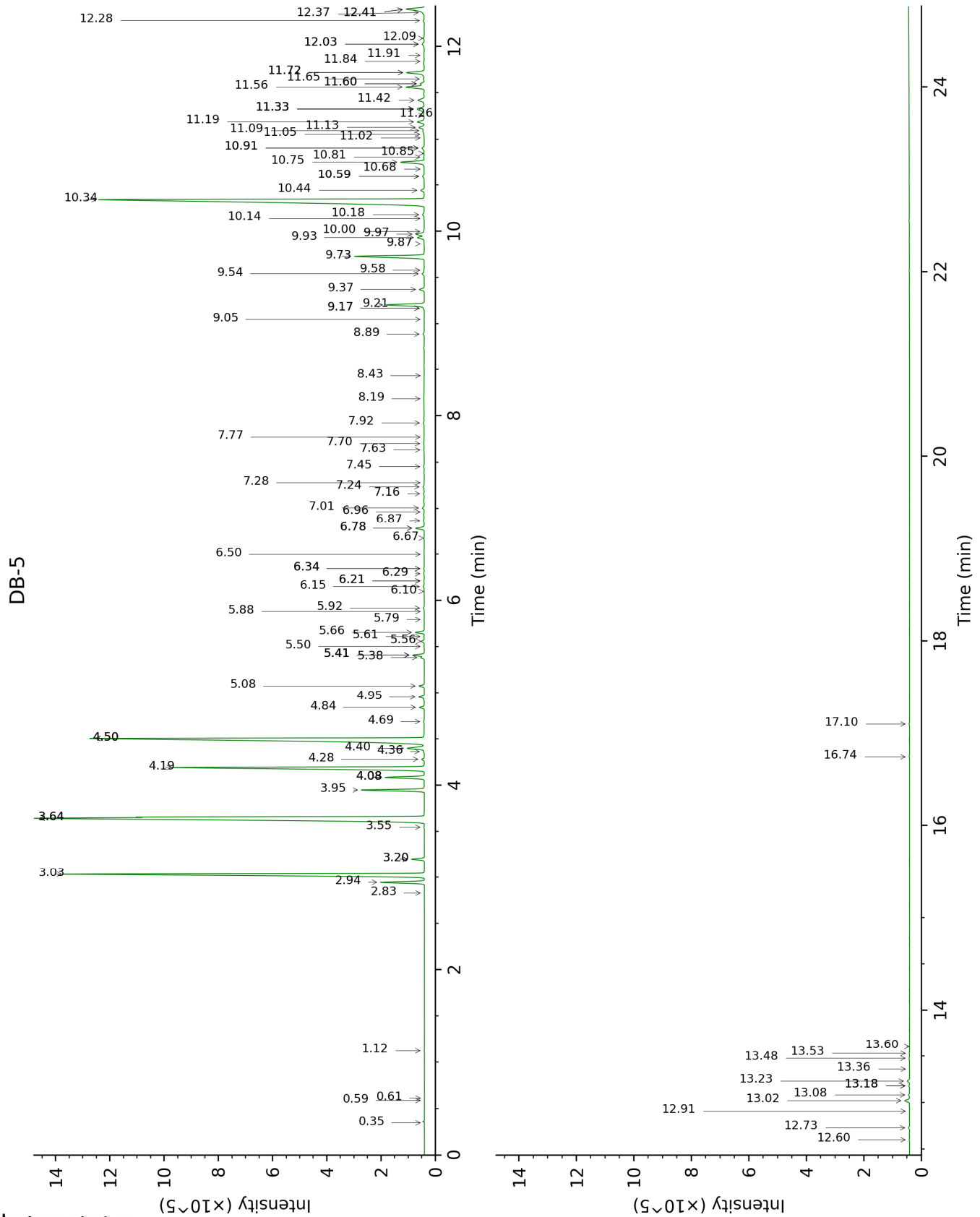
tr: The compound has been detected below 0.005% of total signal.

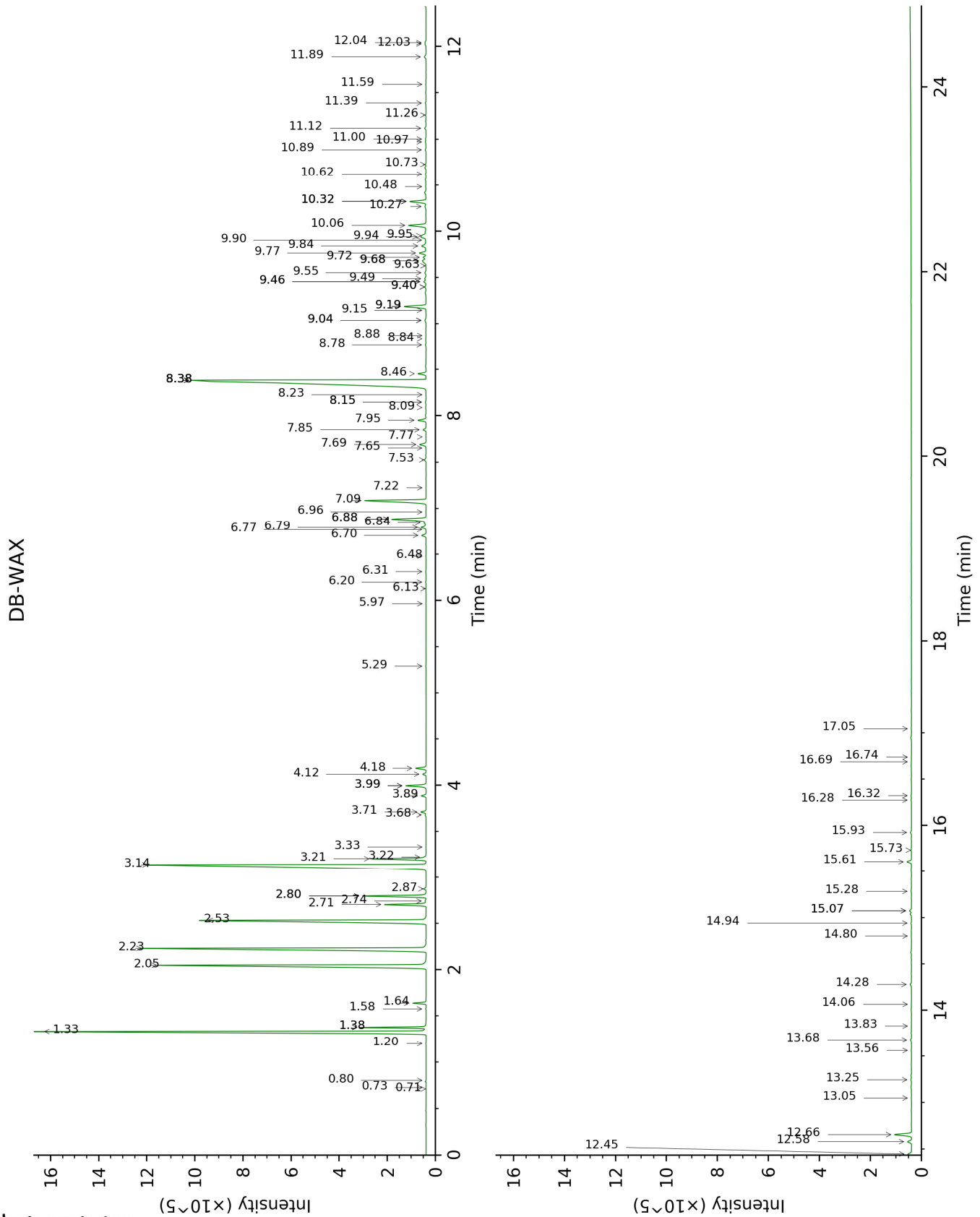
Note: no correction factor was applied

**About "consolidated" data:** The table above presents the breakdown of the sample volatile constituents after applying an algorithm to collapse data acquired from the multi-columns system of PhytoChemia into a single set of consolidated contents. In case of discrepancies between columns, the algorithm is set to prioritize data from the most standard DB-5 column, and smallest values so as to avoid overestimating individual content. This process is semi-automatic. Advanced users are invited to consult the "Full analysis data" table after the chromatograms in this report to access the full untreated data and perform their own calculations if needed.

**Unknowns:** Unknown compounds' mass spectral data is presented in the "Full analysis data" table. The occurrence of unknown compounds is to be expected in many samples, and does not denote particular problems unless noted otherwise in the conclusion.

This page was intentionally left blank. The following pages present the complete data of the analysis.







FULL ANALYSIS DATA

Identification	Column DB-5			Column DB-WAX		
	R.T	R.I	%	R.T	R.I	%
Ethanol	0.35	522	tr	0.80	906	tr
Isovaleral	0.59	641	tr	0.73	884	tr
2-Methylbutyral	0.61	651	tr	0.71	878	tr
Toluene	1.12	762	0.01	1.38*	1000	1.42
Tricyclene	2.83	918	0.01	1.20	971	0.01
$\alpha$ -Thujene	2.94	926	1.37	1.38*	1000	[1.42]
$\alpha$ -Pinene	3.03	932	12.83	1.33	993	12.68
Camphene	3.20*	942	0.38	1.64	1026	0.36
$\alpha$ -Fenchene	3.20*	942	[0.38]	1.58	1020	0.01
meta-Cymene	3.55	965	0.02	2.80*	1132	1.93
Sabinene	3.64*	972	22.14	2.23	1085	11.33
$\beta$ -Pinene	3.64*	972	[22.14]	2.05	1066	10.64
Myrcene	3.95	992	1.91	2.80*	1132	[1.93]
$\alpha$ -Phellandrene	4.08*	1001	1.29	2.70	1125	1.26
Pseudolimonene	4.08*	1001	[1.29]	2.74	1128	0.04
Menthatriene isomer I	4.08*	1001	[1.29]	3.33	1175	0.01
$\Delta$ 3-Carene	4.19	1008	9.46	2.53	1112	9.39
$\alpha$ -Terpinene	4.28	1013	0.08	2.87	1138	0.08
ortho-Cymene	4.36	1018	0.03	3.99*	1225	0.71
para-Cymene	4.40	1021	0.68	3.99*	1225	[0.71]
Limonene	4.50*	1027	16.66	3.14	1159	14.95
$\beta$ -Phellandrene	4.50*	1027	[16.66]	3.20	1164	1.60
1,8-Cineole	4.50*	1027	[16.66]	3.22	1166	0.04
(Z)- $\beta$ -Ocimene	4.69	1039	0.02	3.68†	1202	0.22
(E)- $\beta$ -Ocimene	4.84	1048	0.16	3.89	1217	0.16
$\gamma$ -Terpinene	4.95	1056	0.18	3.71†	1204	[0.22]
cis-Sabinene hydrate	5.08	1063	0.17	6.79	1425	0.21
Isoterpinolene	5.38	1083	0.10	4.12	1235	0.12
Terpinolene	5.41*	1085	0.38	4.18	1239	0.36
para-Cresol	5.41*	1085	[0.38]	13.83	2014	0.01
para-Cymenene	5.41*	1085	[0.38]	6.20	1381	0.02
$\alpha$ -Pinene oxide	5.50	1090	0.01	5.29	1316	0.01
trans-Sabinene hydrate	5.56	1094	0.10	7.85	1505	0.11
Unknown [m/z 109, 43 (65), 95 (54), 119 (50), 91 (47)... 149 (8)...]	5.61	1097	0.02	5.97	1364	0.01
Linalool	5.66	1100	0.29	7.95	1513	0.30
Verbenol analog?	5.80	1109	0.01	8.23	1534	0.01
Unknown [m/z 41, 67 (75), 69 (59), 79 (55), 81 (44), 71 (41)... 150 (5)]	5.88	1115	0.01	6.13	1376	0.01
trans-para-Mentha-2,8-dien-	5.92	1117	0.03	8.78	1576	0.03

1-ol						
<i>cis</i> -Limonene oxide	6.10	1129	0.01	6.31	1390	0.02
<i>cis</i> -para-Mentha-2,8-dien-1-ol	6.15	1132	0.04	9.40*	1626	0.06
<i>trans</i> -para-Mentha-2-en-1-ol	6.21*	1136	0.02	8.84	1582	0.01
<i>trans</i> -Limonene oxide	6.21*	1136	[0.02]	6.48	1402	0.01
<i>trans</i> -Verbenol	6.29	1141	0.02	9.40*	1626	[0.06]
1,4-Dimethyl-4-acetylcyclohexene meta-Mentha-4,6-dien-8-ol	6.34*	1145	0.02	7.22	1458	tr
	6.34*	1145	[0.02]	9.19*	1609	0.91
Pinocarvone	6.50	1155	0.01	7.77	1498	0.01
<i>cis</i> -Sabinol	6.67	1166	0.02	10.73	1736	0.02
Terpinen-4-ol	6.78*	1173	0.32	8.46	1552	0.29
<i>trans</i> -2-Caren-4-ol	6.78*	1173	[0.32]	7.65	1490	0.01
meta-Cymen-8-ol	6.87	1179	0.04	11.39	1792	0.03
Unknown [m/z 43, 135 (73), 59 (46), 93 (39), 91 (35), 81 (32)...]	6.96	1185	0.01			
$\alpha$ -Terpineol	7.01	1188	0.07	9.68*†	1649	0.37
<i>cis</i> - $\alpha$ -Phellandrene epoxide (IPP vs Me)	7.16	1198	0.03	10.89	1750	0.04
Verbenone	7.24	1203	0.06	9.49†	1634	[0.17]
Unknown [m/z 93, 71 (74), 92 (35), 41 (24), 69 (23), 136 (21)...]	7.28	1206	0.01			
<i>trans</i> -Carveol	7.45	1218	0.03	11.26	1781	0.02
<i>cis</i> -Carveol	7.63	1230	0.02	11.59	1810	0.01
Cuminal	7.70	1235	0.04	10.48	1715	0.02
Carvone	7.77	1240	0.01	9.90	1667	0.03
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.92	1250	0.03	11.00	1759	0.03
<i>trans</i> -Ascaridole glycol	8.18	1268	0.01	14.06	2036	0.01
Bornyl acetate	8.43	1286	tr	8.15*	1528	0.04
Unknown [m/z 111, 126 (93), 43 (90), 71 (60)...]	8.89	1313	0.05	15.07*†	2135	0.13
Methyl geranate	9.05	1324	0.01	9.63	1645	0.02
Bicycloelemene	9.17*	1333	0.03	6.96	1438	0.01
$\delta$ -Elemene isomer	9.17*	1333	[0.03]	6.77	1424	0.01
$\delta$ -Elemene	9.21	1335	1.38	6.88*	1432	1.37
$\alpha$ -Cubebene	9.37	1347	0.17	6.70	1419	0.18
Cyclosativene I	9.54	1359	0.08	6.84	1429	0.08

Cyclosativene II	9.58	1362	0.02	6.88*	1432	[1.37]
$\alpha$ -Copaene	9.73	1372	2.67	7.09	1447	2.66
<i>cis</i> - $\beta$ -Elemene	9.86	1382	0.01	8.15*	1528	[0.04]
$\beta$ -Cubebene	9.93	1386	0.27	7.69	1492	0.26
$\beta$ -Elemene	9.97	1389	0.33	8.38*	1546	19.49
Unknown [m/z 71, 100 (92), 111 (79), 69 (46), 109 (45)...]	10.00	1391	0.02	17.05	2339	0.01
Isocaryophyllene	10.14	1401	0.01	8.09	1523	0.02
$\alpha$ -Gurjunene	10.18	1404	0.07	7.53	1480	0.08
$\beta$ -Caryophyllene	10.34	1416	19.19	8.38*	1546	[19.49]
$\beta$ -Copaene	10.44	1424	0.14	8.38*	1546	[19.49]
<i>trans</i> - $\alpha$ -Bergamotene	10.59*	1435	0.08	8.38*	1546	[19.49]
$\alpha$ -Guaiene	10.59*	1435	[0.08]	8.38*	1546	[19.49]
Unknown [m/z 139, 69 (60), 41 (51), 43 (47), 119 (41)... 204 (1)]	10.68	1441	0.01			
$\alpha$ -Humulene	10.75	1447	0.91	9.19*	1609	[0.91]
Unknown [m/z 109, 110 (33), 43 (30), 95 (23), 71 (20), 41 (19)...]	10.81	1451	0.02			
allo-Aromadendrene	10.85	1454	0.02	8.88	1584	0.01
( <i>E</i> )- $\beta$ -Farnesene	10.91*	1458	0.11	9.46*†	1631	0.17
$\beta$ -Santalene	10.91*	1458	[0.11]	9.04*	1597	0.07
$\gamma$ -Gurjunene	11.02	1466	0.01	9.04*	1597	[0.07]
<i>trans</i> -Cadinene-1(6),4-diene	11.05	1469	0.02	9.15	1606	0.02
$\gamma$ -Muuroolene	11.09	1472	0.09	9.46*†	1631	[0.17]
Germacrene D	11.13	1475	0.22	9.68*†	1649	[0.37]
$\beta$ -Selinene	11.19	1479	0.29	9.77	1656	0.28
<i>trans</i> -Muurolo-4(15),5-diene	11.26	1484	0.03	9.72†	1652	[0.37]
$\alpha$ -Selinene	11.33*	1489	0.42	9.84	1662	0.23
epi-Cubebol	11.33*	1489	[0.42]	11.89	1836	0.09
Bicyclogermacrene	11.33*	1489	[0.42]	9.94†	1670	0.37
Viridiflorene	11.33*	1489	[0.42]	9.55	1639	0.06
$\alpha$ -Muuroolene	11.42	1496	0.29	9.95†	1671	[0.37]
$\beta$ -Bisabolene	11.56	1507	0.72	10.06	1680	0.74
Cubebol	11.60*	1510	0.15	12.45	1886	0.14
$\gamma$ -Cadinene	11.60*	1510	[0.15]	10.27	1697	0.07
7-epi- $\alpha$ -Selinene	11.65	1514	0.04	10.32*	1702	0.67
$\delta$ -Cadinene	11.72*	1520	0.69	10.32*	1702	[0.67]
<i>trans</i> -Calamenene	11.72*	1520	[0.69]	11.12	1769	0.06
( <i>E</i> )- $\gamma$ -Bisabolene	11.84	1529	0.01	10.32*	1702	[0.67]
$\alpha$ -Calacorene	11.91	1534	0.03	12.03	1849	0.04
( <i>E</i> )- $\alpha$ -Bisabolene	12.03*	1544	0.11	10.62	1727	0.03
Isocaryophyllene epoxide B	12.03*	1544	[0.11]	12.04	1850	0.04

Germacrene B	12.09	1549	0.04	10.97	1757	0.04
(E)-Nerolidol	12.28	1564	0.04	13.68	1999	0.05
Spathulenol	12.37	1570	0.05	14.28	2057	0.06
Caryophyllene oxide isomer	12.41*	1573	0.84	12.58	1898	0.16
Caryophyllene oxide	12.41*	1573	[0.84]	12.66	1905	0.67
Humulene epoxide I	12.60	1589	0.02	13.05	1941	0.01
Humulene epoxide II	12.73	1599	0.04	13.25	1959	0.03
$\alpha$ -Corocalene	12.91	1613	0.01	13.56	1988	0.01
Alismol	13.02	1623	0.19	15.61	2189	0.18
Caryophylladienol II	13.08	1628	0.04	15.93	2221	0.04
$\tau$ -Cadinol	13.18*	1636	0.02	14.80	2108	0.01
$\tau$ -Muurolol	13.18*	1636	[0.02]	14.94	2122	0.02
$\alpha$ -Muurolol	13.23	1640	0.09	15.07*†	2135	[0.13]
<i>cis</i> -Calamenen-10-ol	13.36	1651	tr	16.32	2263	0.02
<i>trans</i> -Calamenen-10-ol	13.48	1660	0.02	16.69	2301	0.01
(3Z)-Caryophylla-3,8(13)-dien-5 $\beta$ -ol	13.53	1665	0.01	16.74	2306	0.01
Dehydrojinkoh-eremol	13.60	1671	tr	16.28	2258	0.02
meta-Camphorene	16.74	1951	0.02	15.28	2156	0.03
para-Camphorene	17.10	1984	0.03	15.74	2201	0.02
<b>Total identified</b>		<b>99.17%</b>			<b>98.70%</b>	
<b>Total reported</b>		<b>99.35%</b>			<b>98.75%</b>	

\*: Two or more compounds are coeluting on this column

[xx]: Duplicate percentage due to coelutions, not taken into account in the consolidated total

†: Peaks apexes were resolved, but peaks overlapped and were summed for analysis

tr: The compound has been detected below 0.005% of total signal.

Note: no correction factor was applied

R.T.: Retention time (minutes)

R.I.: Retention index