

**Date :** March 18, 2021

**CERTIFICATE OF ANALYSIS – GC PROFILING**

*SAMPLE IDENTIFICATION*

**Internal code :** 21C11-PTH01


**Customer identification :** Black Pepper - India - B40107208R

**Type :** Essential oil

**Source :** *Piper nigrum*

**Customer :** Plant Therapy

*ANALYSIS*

**Method:** PC-MAT-014  - Analysis of the composition of an essential oil or other volatile liquid by FAST GC-FID (in French); identifications validated by GC-MS.

**Analyst :** Seydou Ka, M. Sc.

**Analysis date :** March 17, 2021

Checked and approved by :

---

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

*Notes: This report may not be published, including online, without the written consent from Laboratoire PhytoChemia. This report is digitally signed, it is only considered valid if the digital signature is intact. The results only describe the samples that were submitted to the assays.*

### PHYSICOCHEMICAL DATA

**Physical aspect:** Faintly yellow liquid

**Refractive index:** 1.4830 ± 0.0003 (20 °C; method PC-MAT-016)

### ISO 3061:2008 - OIL OF BLACK PEPPER - INDIA

Compound	Min. %	Max. %	Observed %	Complies?
α-Pinene	3	12	11	Yes
β-Pinene	5	12	9	Yes
Sabinene	6	15	10	Yes
Δ <sup>3</sup> -Carene	3	15	8	Yes
Limonene	10	17	11	Yes
δ-Elemene	0.5	3.5	1.5	Yes
α-Copaene	0.5	4.5	2.0	Yes
β-Caryophyllene	12	29	29	Yes
Germacrene D		2.0	0.3	Yes
β-Selinene	0.5	3.5	0.9	Yes
α-Selinene		3.0	0.6	Yes
Caryophyllene oxide		1.0	0.7	Yes
<b>Refractive index</b>	1.4780	1.4870	1.4830	Yes

### CONCLUSION

No adulterant, contaminant or diluent has been detected using this method. The oil complies with the ISO standard for Indian black pepper oil.

## ANALYSIS SUMMARY – CONSOLIDATED CONTENTS

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

Identification	%	Class
Isovaleral	0.01	Aliphatic aldehyde
2-Methylbutyral	tr	Aliphatic aldehyde
Toluene	tr	Simple phenolic
Hashishene	0.01	Monoterpene
Tricyclene	0.02	Monoterpene
$\alpha$ -Thujene	1.00	Monoterpene
$\alpha$ -Pinene	10.76	Monoterpene
Camphene	0.32	Monoterpene
$\alpha$ -Fenchene	0.02	Monoterpene
Thuja-2,4(10)-diene	tr	Monoterpene
meta-Cymene	0.01	Monoterpene
$\beta$ -Pinene	9.18	Monoterpene
Sabinene	9.51	Monoterpene
6-Methyl-5-hepten-2-one	0.02	Aliphatic ketone
Dehydro-1,8-cineole	0.01	Monoterpenic ether
Myrcene	1.47	Monoterpene
2-Carene	0.01	Monoterpene
$\alpha$ -Phellandrene	1.42	Monoterpene
Pseudolimonene	0.04	Monoterpene
$\Delta^3$ -Carene	8.27	Monoterpene
$\alpha$ -Terpinene	0.18	Monoterpene
ortho-Cymene	0.08	Monoterpene
para-Cymene	0.64	Monoterpene
Limonene	11.49	Monoterpene
1,8-Cineole	0.98*	Monoterpenic ether
$\beta$ -Phellandrene	[0.98]*	Monoterpene
(Z)- $\beta$ -Ocimene	0.02	Monoterpene
(E)- $\beta$ -Ocimene	0.09	Monoterpene
Unknown	0.02	Monoterpene
$\gamma$ -Terpinene	0.33	Monoterpene
cis-Sabinene hydrate	0.13	Monoterpenic alcohol
Isoterpinolene	0.11	Monoterpene
Terpinolene	0.39	Monoterpene
para-Cymenene	0.02	Monoterpene
trans-Sabinene hydrate	0.09	Monoterpenic alcohol
Unknown	0.01	Unknown
Linalool	0.44	Monoterpenic alcohol
Verbenol analog?	0.01	Monoterpenic alcohol
Unknown	0.01	Unknown
Unknown	tr	Oxygenated monoterpene
trans-para-Mentha-2,8-dien-1-ol	0.04	Monoterpenic alcohol
cis-Limonene oxide	0.01	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
trans-para-Menth-2-en-1-ol	0.04	Monoterpenic alcohol
trans-Verbenol	0.01	Monoterpenic alcohol

meta-Mentha-4,6-dien-8-ol	0.01	Monoterpenic alcohol
Pinocarvone	0.03	Monoterpenic ketone
Borneol	0.01	Monoterpenic alcohol
cis-Sabinol	0.01	Monoterpenic alcohol
Terpinen-4-ol	0.71	Monoterpenic alcohol
meta-Cymen-8-ol	0.01	Monoterpenic alcohol
Cryptone	0.02	Normonoterpenic ketone
para-Cymen-8-ol	0.02	Monoterpenic alcohol
Unknown	0.01	Unknown
α-Terpineol	0.07	Monoterpenic alcohol
Myrtenal	0.01	Monoterpenic aldehyde
Methyl salicylate	0.03	Phenolic ester
trans-Isopiperitenol	0.03	Monoterpenic alcohol
Unknown	0.03	Oxygenated monoterpene
Verbenone	tr	Monoterpenic ketone
Unknown	0.01	Unknown
Car-2-en-4-one?	0.02	Monoterpenic ketone
trans-Carveol	0.02	Monoterpenic alcohol
cis-Carveol	0.01	Monoterpenic alcohol
Cuminal	0.01	Monoterpenic aldehyde
Carvone	0.01	Monoterpenic ketone
Car-3-en-2-one	0.01	Monoterpenic ketone
Unknown	0.02	Unknown
Methyl citronellate	0.01	Monoterpenic ester
trans-Ascaridole glycol	0.01	Monoterpenic alcohol
Cuminol	0.01	Monoterpenic alcohol
Unknown	0.01	Oxygenated monoterpene
Car-3-en-5-one	0.02	Monoterpenic ketone
Methyl geranate	0.01	Monoterpenic ester
Bicycloelemene	0.05	Sesquiterpene
δ-Elemene	1.53	Sesquiterpene
α-Cubebene	0.20	Sesquiterpene
Unknown	0.03	Sesquiterpene
Cyclosativene I	0.07	Sesquiterpene
Cyclosativene II	0.04	Sesquiterpene
α-Ylangene	0.02	Sesquiterpene
α-Copaene	1.98	Sesquiterpene
cis-β-Elemene	0.03	Sesquiterpene
β-Cubebene	0.16	Sesquiterpene
β-Elemene	0.66	Sesquiterpene
Isocaryophyllene	0.08	Sesquiterpene
α-Gurjunene	0.18	Sesquiterpene
β-Caryophyllene	28.71	Sesquiterpene
β-Copaene	0.17	Sesquiterpene
γ-Elemene	0.01	Sesquiterpene
trans-α-Bergamotene	0.01	Sesquiterpene
α-Guaiene	0.19	Sesquiterpene
Unknown	0.02	Unknown
Unknown	0.01	Sesquiterpene
α-Humulene	1.29	Sesquiterpene
allo-Aromadendrene	0.02	Sesquiterpene
β-Santalene	0.04	Sesquiterpene

(E)-β-Farnesene	0.05	Sesquiterpene
γ-Gurjunene	0.02	Sesquiterpene
trans-Cadina-1(6),4-diene	0.04	Sesquiterpene
γ-Muurolene	0.05	Sesquiterpene
Germacrene D	0.30	Sesquiterpene
β-Selinene	0.93	Sesquiterpene
trans-Muurolo-4(15),5-diene	0.06	Sesquiterpene
α-Selinene	0.64	Sesquiterpene
epi-Cubebol	0.08	Sesquiterpenic alcohol
Viridiflorene	0.11	Sesquiterpene
Epizonarene	0.02	Sesquiterpene
α-Muurolene	0.33	Sesquiterpene
β-Bisabolene	0.28	Sesquiterpene
Cubebol	0.13	Sesquiterpenic alcohol
(3E,6E)-α-Farnesene	0.05	Sesquiterpene
7-epi-α-Selinene	0.06	Sesquiterpene
trans-Calamenene	0.04	Sesquiterpene
δ-Cadinene	0.81	Sesquiterpene
trans-Cadina-1,4-diene	0.05	Sesquiterpene
(E)-γ-Bisabolene	0.01	Sesquiterpene
α-Calacorene	0.02	Sesquiterpene
(E)-α-Bisabolene	0.03	Sesquiterpene
Isocaryophyllene epoxide B	0.05	Sesquiterpenic ether
Germacrene B	0.06	Sesquiterpene
(E)-Nerolidol	0.04	Sesquiterpenic alcohol
Spathulenol	0.04	Sesquiterpenic alcohol
Caryophyllene oxide	0.66	Sesquiterpenic ether
Unknown	0.01	Oxygenated sesquiterpene
Humulene epoxide I	0.04	Sesquiterpenic ether
Humulene epoxide II	0.04	Sesquiterpenic ether
α-Corocalene	0.02	Sesquiterpene
Alismol	0.16	Sesquiterpenic alcohol
Caryophylladienol I?	0.02	Sesquiterpenic alcohol
Caryophylladienol II	0.01	Sesquiterpenic alcohol
τ-Muurolol	0.04	Sesquiterpenic alcohol
α-Muurolol	0.12	Sesquiterpenic alcohol
(3Z)-Caryophylla-3,8(13)-dien-5β-ol	0.02	Sesquiterpenic alcohol
Dehydrojinkoh-eremol	0.01	Sesquiterpenic alcohol
meta-Camphorene	0.02	Diterpene
<b>Consolidated total</b>	<b>99.39%</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.

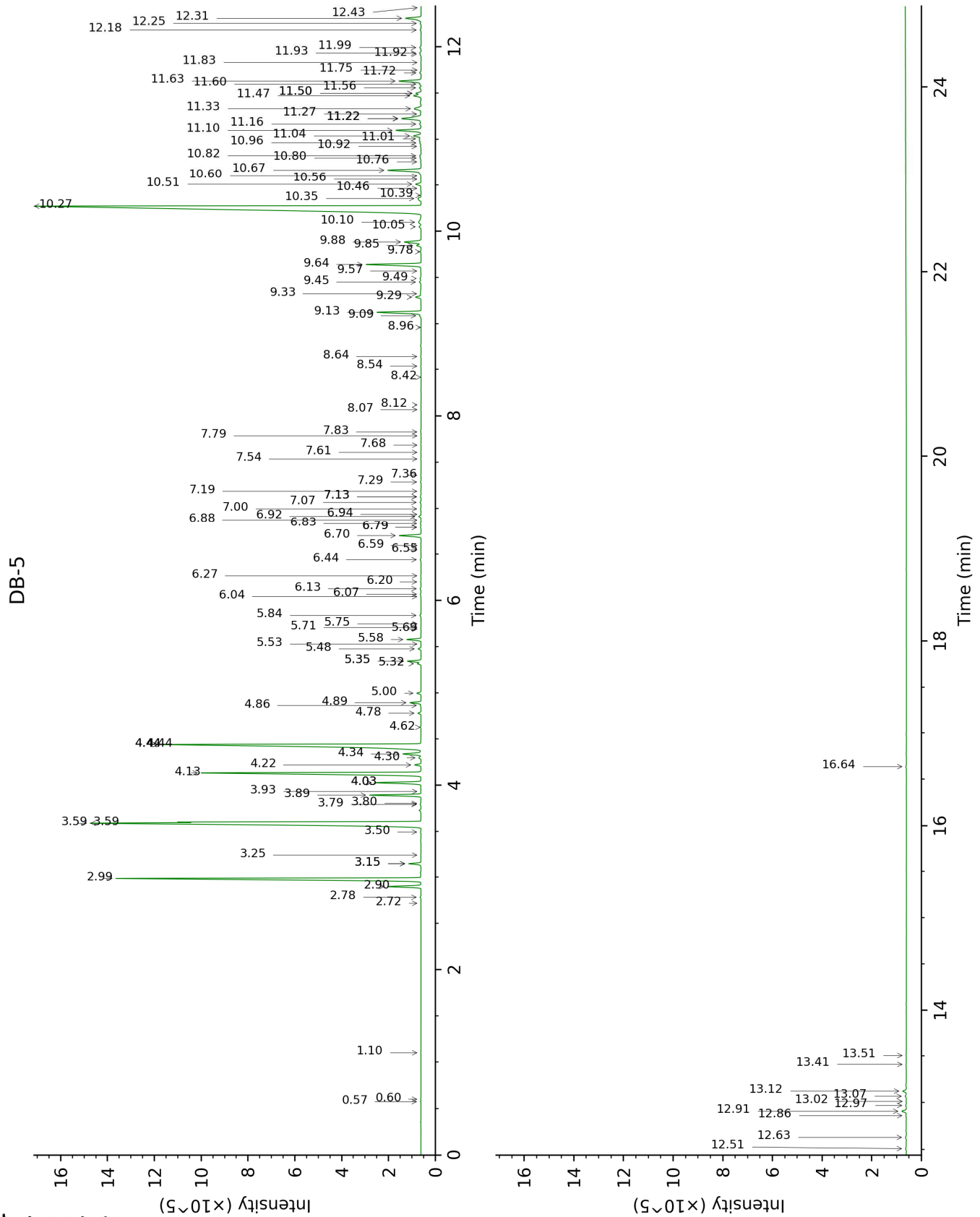
Note: no correction factor was applied

**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.

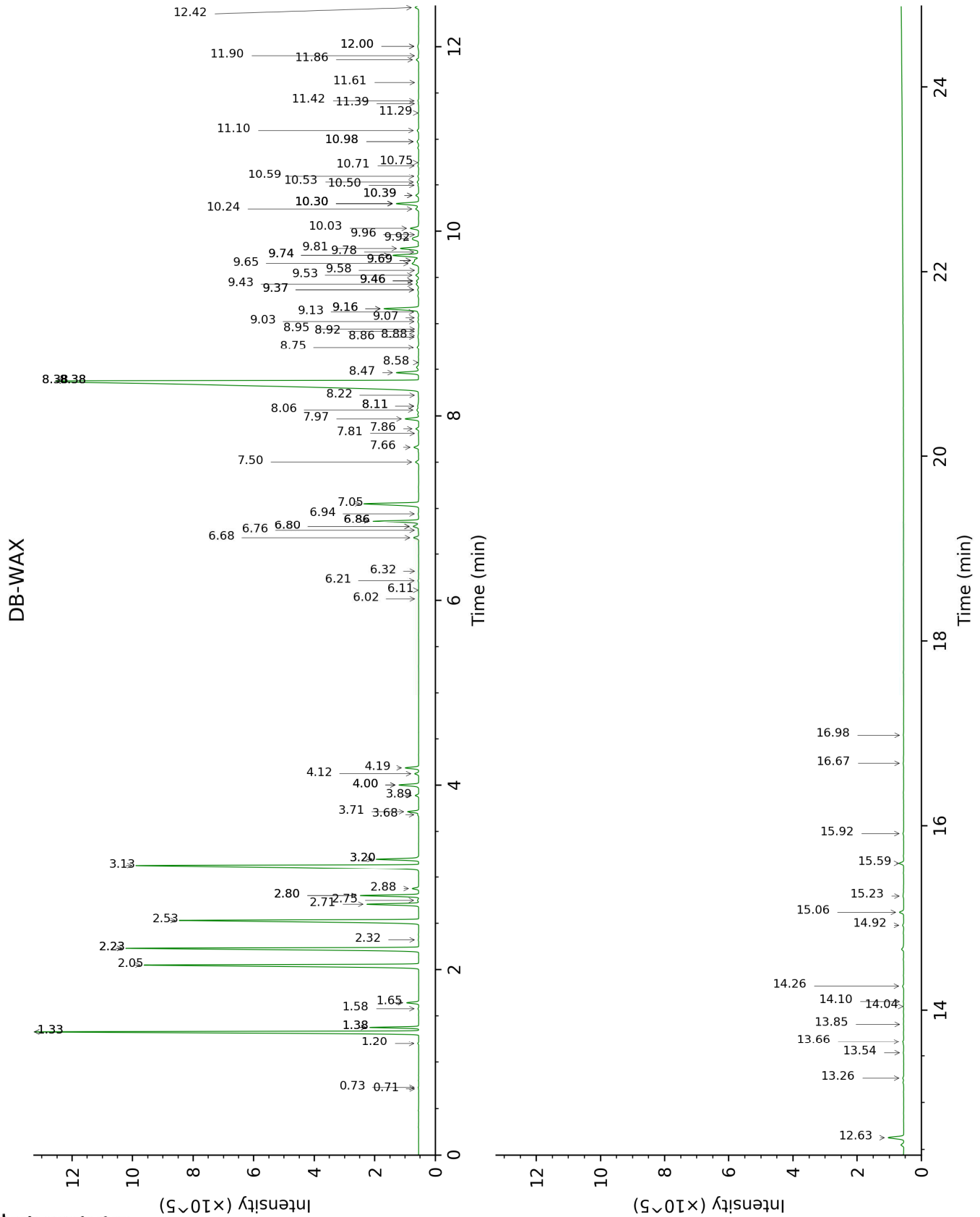
**Unknowns:** Unknown compounds' mass spectral data is presented in the "Full analysis data" table. The occurrence of unknown compounds is to be expected in many samples, and does not denote particular problems unless noted otherwise in the conclusion.



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	%
Isovaleral	0.57	642	0.01	0.73	887	0.01
2-Methylbutyral	0.60	652	tr	0.71	881	tr
Toluene	1.10	758	tr	1.38*	1001	1.01
Hashishene	2.72	913	0.01	1.33*	994	10.73
Tricyclene	2.78	917	0.02	1.20	972	0.02
$\alpha$ -Thujene	2.90	925	1.00	1.38*	1001	[1.01]
$\alpha$ -Pinene	2.99	931	10.76	1.33*	994	[10.73]
Camphene	3.15*	942	0.34	1.64	1028	0.32
$\alpha$ -Fenchene	3.15*	942	[0.34]	1.58	1021	0.02
Thuja-2,4(10)-diene	3.25	948	tr	2.23*	1086	9.54
meta-Cymene	3.50	965	0.01	2.80*	1134	1.54
$\beta$ -Pinene	3.59*†	972	18.68	2.05	1068	9.18
Sabinene	3.59*†	972	[18.68]	2.23*	1086	[9.54]
6-Methyl-5-hepten-2-one	3.79	985	0.02			
Dehydro-1,8-cineole	3.80	986	0.01			
Myrcene	3.89	992	1.47	2.80*	1134	[1.54]
2-Carene	3.93	995	0.01	2.32	1096	0.01
$\alpha$ -Phellandrene	4.03*	1001	1.42	2.71	1127	1.42
Pseudolimonene	4.03*	1001	[1.42]	2.75	1130	0.04
$\Delta^3$ -Carene	4.13	1008	8.27	2.53	1113	8.26
$\alpha$ -Terpinene	4.22	1014	0.18	2.88	1140	0.18
ortho-Cymene	4.30	1018	0.08	4.00*	1228	0.61
para-Cymene	4.34	1021	0.64	4.00*	1228	[0.61]
Limonene	4.44*	1028	12.47	3.13	1161	11.49
1,8-Cineole	4.44*	1028	[12.47]	3.20*	1166	1.18
$\beta$ -Phellandrene	4.44*	1028	[12.47]	3.20*	1166	[1.18]
(Z)- $\beta$ -Ocimene	4.62	1039	0.02	3.68	1204	0.03
(E)- $\beta$ -Ocimene	4.78	1049	0.09	3.89	1220	0.10
Unknown [m/z 41, 69 (97), 95 (33), 67 (31), 53 (18)...]	4.86	1054	0.02			
$\gamma$ -Terpinene	4.89	1056	0.33	3.71	1207	0.34
cis-Sabinene hydrate	5.00	1063	0.13	6.80*	1430	0.22
Isoterpinolene	5.32	1084	0.11	4.12	1237	0.12
Terpinolene	5.35*	1085	0.41	4.19	1242	0.39
para-Cymenene	5.35*	1085	[0.41]	6.21	1387	0.02
trans-Sabinene hydrate	5.48	1094	0.09	7.86	1510	0.09
Unknown [m/z 109, 43 (65), 95 (54), 119 (50), 91 (47)... 149 (8)...]	5.53	1097	0.01	6.02	1372	tr
Linalool	5.58	1100	0.44	7.97	1518	0.43

Verbenol analog?	5.69	1108	0.01	8.22	1538	0.01
Unknown [m/z 94, 59 (83), 43 (81), 95 (56), 109 (50), 79 (50), 91 (40)...]	5.71	1109	0.01			
Unknown [m/z 41, 67 (75), 69 (59), 79 (55), 81 (44), 71 (41)... 150 (5)]	5.75	1111	tr	6.11	1379	tr
<i>trans</i> -para-Mentha-2,8-dien-1-ol	5.84	1117	0.04	8.75	1579	0.06
<i>cis</i> -Limonene oxide	6.04	1130	0.01	6.32	1394	0.01
<i>cis</i> -para-Mentha-2,8-dien-1-ol	6.07	1132	0.03	9.37*	1629	0.04
<i>trans</i> -para-Mentha-2-en-1-ol	6.13	1136	0.04	8.88	1589	0.01
<i>trans</i> -Verbenol	6.20	1140	0.01	9.37*	1629	[0.04]
meta-Mentha-4,6-dien-8-ol	6.27	1145	0.01	9.13	1609	0.02
Pinocarvone	6.44	1156	0.03	7.81	1506	0.01
Borneol	6.55	1163	0.01	9.69*	1654	0.13
<i>cis</i> -Sabinol	6.59	1166	0.01	10.75	1743	0.02
Terpinen-4-ol	6.70	1173	0.71	8.47	1557	0.71
meta-Cymen-8-ol	6.79*	1179	0.02	11.39	1797	0.01
Cryptone	6.79*	1179	[0.02]	9.07	1604	0.02
para-Cymen-8-ol	6.83	1182	0.02	11.42	1800	0.02
Unknown [m/z 43, 135 (73), 59 (46), 93 (39), 91 (35), 81 (32)...]	6.88	1184	0.01			
$\alpha$ -Terpineol	6.92	1187	0.07	9.69*	1654	[0.13]
Myrtenal	6.94	1188	0.01	8.58	1566	0.01
Methyl salicylate	7.00	1192	0.03	10.39*	1712	0.10
<i>trans</i> -Isopiperitenol	7.07	1197	0.03	10.24	1700	0.13
Unknown [m/z 109, 91 (100), 81 (88), 94 (75), 119 (74), 96 (73), 41 (63)... 150 (2)]	7.13*	1201	0.03	10.72	1740	0.03
Verbenone	7.13*	1201	[0.03]	9.46*	1636	0.08
Unknown [m/z 93, 71 (74), 92 (35), 41 (24), 69 (23), 136 (21)...]	7.19	1205	0.01			
Car-2-en-4-one?	7.29	1212	0.02	9.46*	1636	[0.08]
<i>trans</i> -Carveol	7.36	1216	0.02	11.28	1789	0.01
<i>cis</i> -Carveol	7.54	1228	0.01	11.61	1818	0.01

Cuminal	7.61	1233	0.01	10.50	1722	0.02
Carvone	7.68	1239	0.01	9.96	1677	0.01
Car-3-en-2-one	7.79	1246	0.01	10.30*	1705	0.80
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.83	1248	0.02	10.98*	1762	0.07
Methyl citronellate	8.07	1265	0.01	8.11*	1529	0.05
<i>trans</i> -Ascaridole glycol	8.12	1268	0.01	14.04	2041	0.01
Cuminol	8.42	1289	0.01	14.10	2046	0.01
Unknown [m/z 43, 93 (66), 91 (44), 41 (38), 69 (35)... 152? (1)]	8.54	1297	0.01			
Car-3-en-5-one	8.64	1304	0.02	11.90	1843	0.01
Methyl geranate	8.96	1324	0.01	9.58	1646	0.01
Bicycloelemene	9.09	1332	0.05	6.86*	1435	1.56
$\delta$ -Elemene	9.13	1335	1.53	6.86*	1435	[1.56]
$\alpha$ -Cubebene	9.29	1347	0.20	6.68	1421	0.17
Unknown [m/z 95, 147 (61), 96 (39), 93 (37), 94 (37)... 204 (4)]	9.33	1349	0.03	6.80*	1430	[0.22]
Cyclosativene I	9.45	1358	0.07	6.76	1427	0.02
Cyclosativene II	9.49	1361	0.04	6.86*	1435	[1.56]
$\alpha$ -Ylangene	9.57	1367	0.02	6.94	1441	0.02
$\alpha$ -Copaene	9.64	1372	1.98	7.05	1449	2.00
<i>cis</i> - $\beta$ -Elemene	9.78	1381	0.03	8.11*	1529	[0.05]
$\beta$ -Cubebene	9.85	1386	0.16	7.66	1494	0.17
$\beta$ -Elemene	9.88	1389	0.66	8.38*	1550	29.81
Isocaryophyllene	10.05	1400	0.08	8.06	1526	0.08
$\alpha$ -Gurjunene	10.10	1404	0.18	7.50	1482	0.12
$\beta$ -Caryophyllene	10.27	1417	28.71	8.38*	1550	[29.81]
$\beta$ -Copaene	10.35	1423	0.17	8.38*	1550	[29.81]
$\gamma$ -Elemene	10.39	1426	0.01	8.92	1592	0.01
<i>trans</i> - $\alpha$ - Bergamotene	10.46	1431	0.01	8.38*	1550	[29.81]
$\alpha$ -Guaiene	10.51	1435	0.19	8.38*	1550	[29.81]
Unknown [m/z 41, 97 (78), 69 (77), 43 (71), 125 (67), 55 (56)... 168 (39)]	10.56	1439	0.02	16.98	2341	0.01
Unknown [m/z 139, 69 (60), 41 (51), 43 (47), 119 (41)... 204 (1)]	10.60	1442	0.01			
$\alpha$ -Humulene	10.67	1446	1.29	9.16*	1612	1.27
allo- Aromadendrene	10.76	1453	0.02	8.86	1588	0.03

β-Santalene	10.80	1456	0.04	8.95	1594	0.01
(E)-β-Farnesene	10.82	1458	0.05	9.43	1634	0.11
γ-Gurjunene	10.92	1466	0.02	9.03	1601	0.03
trans-Cadina-1(6),4-diene	10.96	1468	0.04	9.16*	1612	[1.27]
γ-Muurolene	11.01	1472	0.05	9.46*	1636	[0.08]
Germacrene D	11.04	1474	0.30	9.65	1652	0.38
β-Selinene	11.10	1479	0.93	9.74*	1659	0.94
trans-Muurola-4(15),5-diene	11.16	1484	0.06	9.78	1662	0.02
α-Selinene	11.22*	1488	0.95	9.82	1665	0.64
epi-Cubebol	11.22*	1488	[0.95]	11.86	1839	0.08
Viridiflorene	11.22*	1488	[0.95]	9.53	1641	0.11
Epizonarene	11.27	1492	0.02	9.74*	1659	[0.94]
α-Muurolene	11.33	1496	0.33	9.92	1674	0.36
β-Bisabolene	11.47	1507	0.28	10.03	1683	0.29
Cubebol	11.50*	1509	0.17	12.42	1890	0.13
(3E,6E)-α-Farnesene	11.50*	1509	[0.17]	10.39*	1712	[0.10]
7-epi-α-Selinene	11.56	1514	0.06	10.30*	1705	[0.80]
trans-Calamenene	11.60	1517	0.04	11.10	1773	0.06
δ-Cadinene	11.63	1519	0.81	10.30*	1705	[0.80]
trans-Cadina-1,4-diene	11.72	1526	0.05	10.53	1724	0.05
(E)-γ-Bisabolene	11.75	1528	0.01	10.30*	1705	[0.80]
α-Calacorene	11.83	1535	0.02	12.00*	1852	0.06
(E)-α-Bisabolene	11.92	1542	0.03	10.59	1730	0.03
Isocaryophyllene epoxide B	11.93	1543	0.05	12.00*	1852	[0.06]
Germacrene B	11.99	1548	0.06	10.98*	1762	[0.07]
(E)-Nerolidol	12.18	1563	0.04	13.66	2004	0.03
Spathulenol	12.25	1568	0.04	14.26	2062	0.04
Caryophyllene oxide	12.31	1573	0.66	12.63	1908	0.56
Unknown [m/z 161, 105 (84), 43 (80), 119 (72), 93 (62), 121 (54)... 204 (38), 222 (2)]	12.42	1582	0.01	13.85	2023	0.01
Humulene epoxide I	12.51	1588	0.04			
Humulene epoxide II	12.63	1598	0.04	13.26	1968	0.03
α-Corocalene	12.86	1617	0.02	13.54	1993	0.01
Alismol	12.91	1621	0.16	15.59	2195	0.15
Caryophylladienol I?	12.97	1626	0.02			
Caryophylladienol II	13.02	1630	0.01	15.92	2229	0.02
τ-Muurolol	13.07	1634	0.04	14.92	2127	0.03
α-Muurolol	13.12	1639	0.12	15.06	2141	0.15

(3Z)-Caryophylla-3,8(13)-dien-5β-ol	13.41	1663	0.02	16.67	2308	0.02
Dehydrojinkoh-eremol	13.51	1670	0.01			
meta-Camphorene	16.64	1950	0.02	15.23	2159	0.01
<b>Total identified</b>		<b>99.30%</b>			<b>99.28%</b>	
<b>Total reported</b>		<b>99.45%</b>			<b>99.33%</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