

**Date :** February 05, 2021

**CERTIFICATE OF ANALYSIS – GC PROFILING**

*SAMPLE IDENTIFICATION*

**Internal code :** 21B04-PTH02


**Customer identification :** Organic Black Pepper - BS01052011R

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

**Analysis date :** February 05, 2021

Checked and approved by :

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

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### PHYSICOCHEMICAL DATA

**Physical aspect:** Faintly yellow liquid

**Refractive index:** 1.4841 ± 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	7	Yes
β-Pinene	5	12	7	Yes
Sabinene	6	15	8	Yes
Δ3-Carene	3	15	13	Yes
Limonene	10	17	12	Yes
δ-Elemene	0.5	3.5	3.6	No
α-Copaene	0.5	4.5	2.9	Yes
β-Caryophyllene	12	29	28	Yes
Germacrene D		2.0	0.2	Yes
β-Selinene	0.5	3.5	1.2	Yes
α-Selinene		3.0	0.9	Yes
Caryophyllene oxide		1.0	1.0	Yes
<b>Refractive index</b>	1.4780	1.4870	1.4841	Yes

### 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
Isovaleral	0.01	Aliphatic aldehyde
Toluene	0.01	Simple phenolic
Hashishene	0.02	Monoterpene
$\alpha$ -Thujene	0.24	Monoterpene
$\alpha$ -Pinene	7.45	Monoterpene
Camphene	0.12	Monoterpene
$\alpha$ -Fenchene	0.02	Monoterpene
Thuja-2,4(10)-diene	0.01	Monoterpene
meta-Cymene	0.04	Monoterpene
Sabinene	7.83	Monoterpene
$\beta$ -Pinene	7.36	Monoterpene
Dehydro-1,8-cineole	0.02	Monoterpenic ether
Myrcene	1.19	Monoterpene
2-Carene	0.01	Monoterpene
$\alpha$ -Phellandrene	1.69	Monoterpene
Pseudolimonene	0.03	Monoterpene
$\Delta^3$ -Carene	12.97	Monoterpene
$\alpha$ -Terpinene	0.08	Monoterpene
ortho-Cymene	0.06	Monoterpene
para-Cymene	0.90	Monoterpene
Limonene	11.84	Monoterpene
$\beta$ -Phellandrene	0.41	Monoterpene
(Z)- $\beta$ -Ocimene	0.01	Monoterpene
(E)- $\beta$ -Ocimene	0.04	Monoterpene
$\gamma$ -Terpinene	0.10	Monoterpene
cis-Sabinene hydrate	0.01	Monoterpenic alcohol
meta-Cymenene	0.01	Monoterpene
Isoterpinolene	0.19	Monoterpene
para-Cymenene	0.04	Monoterpene
Terpinolene	0.52	Monoterpene
$\alpha$ -Pinene oxide	0.02	Monoterpenic ether
trans-Sabinene hydrate	0.02	Monoterpenic alcohol
Linalool	0.55	Monoterpenic alcohol
trans-para-Mentha-2,8-dien-1-ol	0.02	Monoterpenic alcohol
cis-Limonene oxide	0.02	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
trans-para-Menth-2-en-1-ol	0.03	Monoterpenic alcohol
trans-Verbenol	0.02	Monoterpenic alcohol
Pinocarvone	0.03	Monoterpenic ketone
cis-Sabinol	0.02	Monoterpenic alcohol
Terpinen-4-ol	0.09	Monoterpenic alcohol
meta-Cymen-8-ol	0.04	Monoterpenic alcohol
para-Cymen-8-ol	0.05	Monoterpenic alcohol
$\alpha$ -Terpineol	0.01	Monoterpenic alcohol
Myrtanal	0.11	Monoterpenic aldehyde

Unknown	0.05	Oxygenated monoterpene
Verbenone	0.07	Monoterpenic ketone
Car-2-en-4-one?	0.05	Monoterpenic ketone
<i>trans</i> -Carveol	0.03	Monoterpenic alcohol
<i>cis</i> -Carveol	0.02	Monoterpenic alcohol
Cuminal	0.03	Monoterpenic aldehyde
Carvone	0.02	Monoterpenic ketone
Car-3-en-2-one	0.01	Monoterpenic ketone
Unknown	0.05	Unknown
Methyl citronellate	0.01	Monoterpenic ester
Bornyl acetate	0.01	Monoterpenic ester
Unknown	0.02	Oxygenated monoterpene
Car-3-en-5-one	0.05	Monoterpenic ketone
para-Menth-5-en-1,2-diol isomer III	0.07	Monoterpenic alcohol
Unknown	0.02	Oxygenated monoterpene
$\delta$ -Elemene isomer	0.10	Sesquiterpene
Bicycloelemene	0.04	Sesquiterpene
$\delta$ -Elemene	3.56	Sesquiterpene
$\alpha$ -Cubebene	0.24	Sesquiterpene
Cyclosativene I	0.02	Sesquiterpene
Cyclosativene II	0.03	Sesquiterpene
$\alpha$ -Copaene	2.91	Sesquiterpene
<i>cis</i> - $\beta$ -Elemene	0.03	Sesquiterpene
$\beta$ -Cubebene	0.12	Sesquiterpene
$\beta$ -Elemene	0.99	Sesquiterpene
Isocaryophyllene	0.04	Sesquiterpene
$\alpha$ -Gurjunene	0.16	Sesquiterpene
$\beta$ -Caryophyllene	27.70	Sesquiterpene
<i>cis</i> - $\alpha$ -Bergamotene	0.07	Sesquiterpene
$\beta$ -Copaene	0.02	Sesquiterpene
<i>trans</i> - $\alpha$ -Bergamotene	0.23*	Sesquiterpene
$\alpha$ -Guaiene	[0.23]*	Sesquiterpene
Unknown	0.03	Unknown
Unknown	0.04	Sesquiterpene
$\alpha$ -Humulene	1.60	Sesquiterpene
allo-Aromadendrene	0.04	Sesquiterpene
( <i>E</i> )- $\beta$ -Farnesene	0.08	Sesquiterpene
$\beta$ -Santalene	0.04	Sesquiterpene
$\gamma$ -Muurolene	0.18	Sesquiterpene
Germacrene D	0.20	Sesquiterpene
<i>trans</i> -Muurola-4(15),5-diene	0.05	Sesquiterpene
ar-Curcumene	0.03	Sesquiterpene
$\beta$ -Selinene	1.19	Sesquiterpene
$\alpha$ -Selinene	0.87	Sesquiterpene
epi-Cubebol	0.02	Sesquiterpenic alcohol
Bicyclogermacrene	0.06	Sesquiterpene
$\alpha$ -Muurolene	0.11	Sesquiterpene
Cubebol	0.02	Sesquiterpenic alcohol
$\beta$ -Bisabolene	0.34	Sesquiterpene
$\gamma$ -Cadinene	0.08	Sesquiterpene
7-epi- $\alpha$ -Selinene	0.04	Sesquiterpene
$\delta$ -Cadinene	0.79	Sesquiterpene

<i>trans</i> -Calamenene	0.07	Sesquiterpene
( <i>E</i> )- $\gamma$ -Bisabolene	0.01	Sesquiterpene
<i>trans</i> -Cadina-1,4-diene	0.01	Sesquiterpene
$\alpha$ -Calacorene	0.02	Sesquiterpene
( <i>E</i> )- $\alpha$ -Bisabolene	0.02	Sesquiterpene
Isocaryophyllene epoxide B	0.11	Sesquiterpenic ether
Germacrene B	0.07	Sesquiterpene
( <i>E</i> )-Nerolidol	0.02	Sesquiterpenic alcohol
Spathulenol	0.01	Sesquiterpenic alcohol
Caryophyllene oxide isomer	0.25	Sesquiterpenic ether
Caryophyllene oxide	1.05	Sesquiterpenic ether
Unknown	0.01	Oxygenated sesquiterpene
Humulene epoxide I	0.04	Sesquiterpenic ether
Humulene epoxide II	0.06	Sesquiterpenic ether
Alismol	0.27	Sesquiterpenic alcohol
Caryophylladienol I	0.01	Sesquiterpenic alcohol
Caryophylladienol II	0.05	Sesquiterpenic alcohol
$\alpha$ -Muurolol	0.02	Sesquiterpenic alcohol
$\alpha$ -Cadinol	0.02	Sesquiterpenic alcohol
<i>cis</i> -Calamenen-10-ol	0.01	Sesquiterpenic alcohol
<i>trans</i> -Calamenen-10-ol	0.01	Sesquiterpenic alcohol
(3 <i>Z</i> )-Caryophylla-3,8(13)-dien-5 $\beta$ -ol	0.01	Sesquiterpenic alcohol
$\alpha$ -Bisabolol	0.01	Sesquiterpenic alcohol
Unknown	0.01	Oxygenated sesquiterpene
<b>Consolidated total</b>	<b>98.98%</b>	

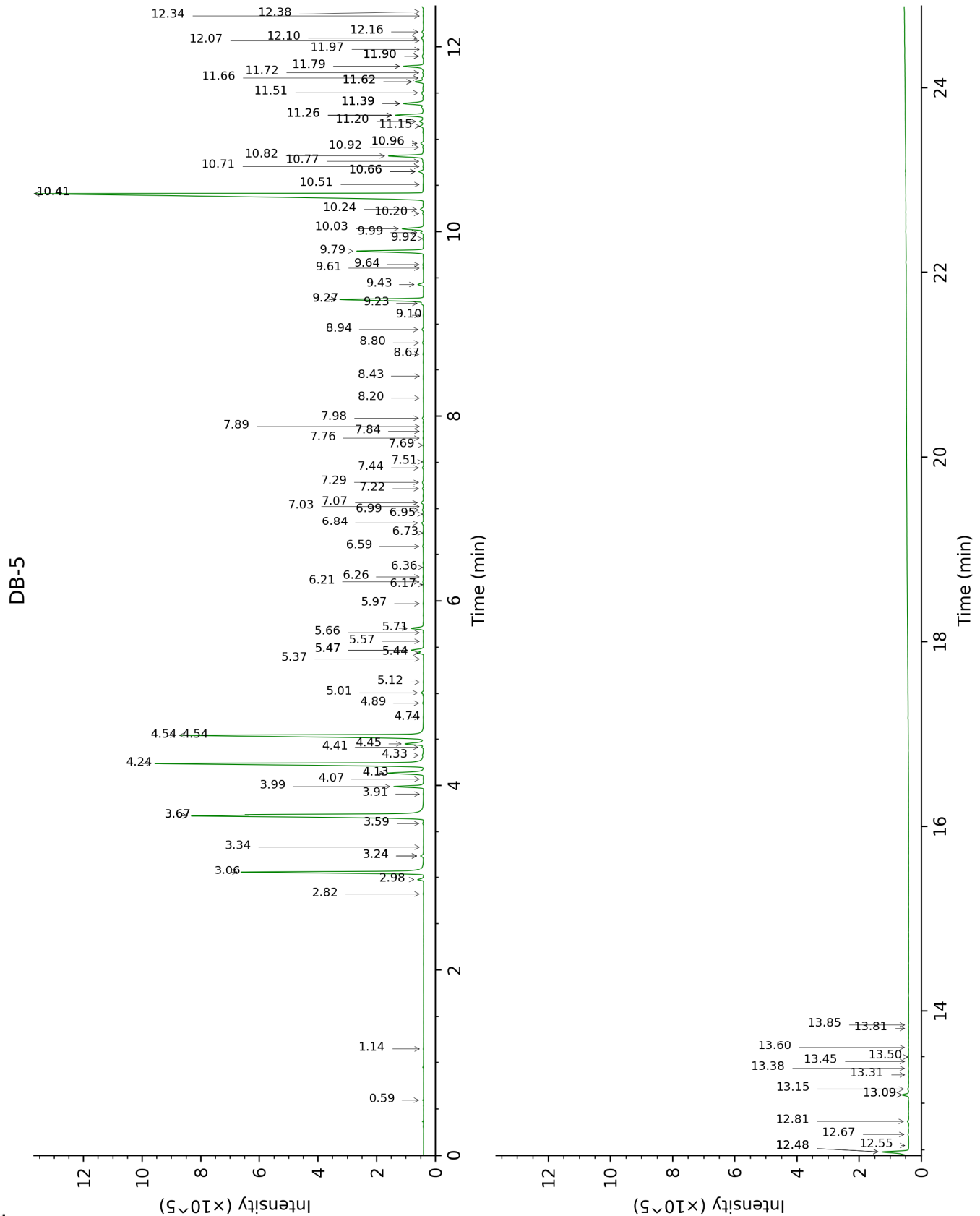
\*: Individual compounds concentration could not be found due to overlapping coelutions on columns considered  
[xx]: Duplicate percentage due to coelutions, not taken into account in the consolidated total

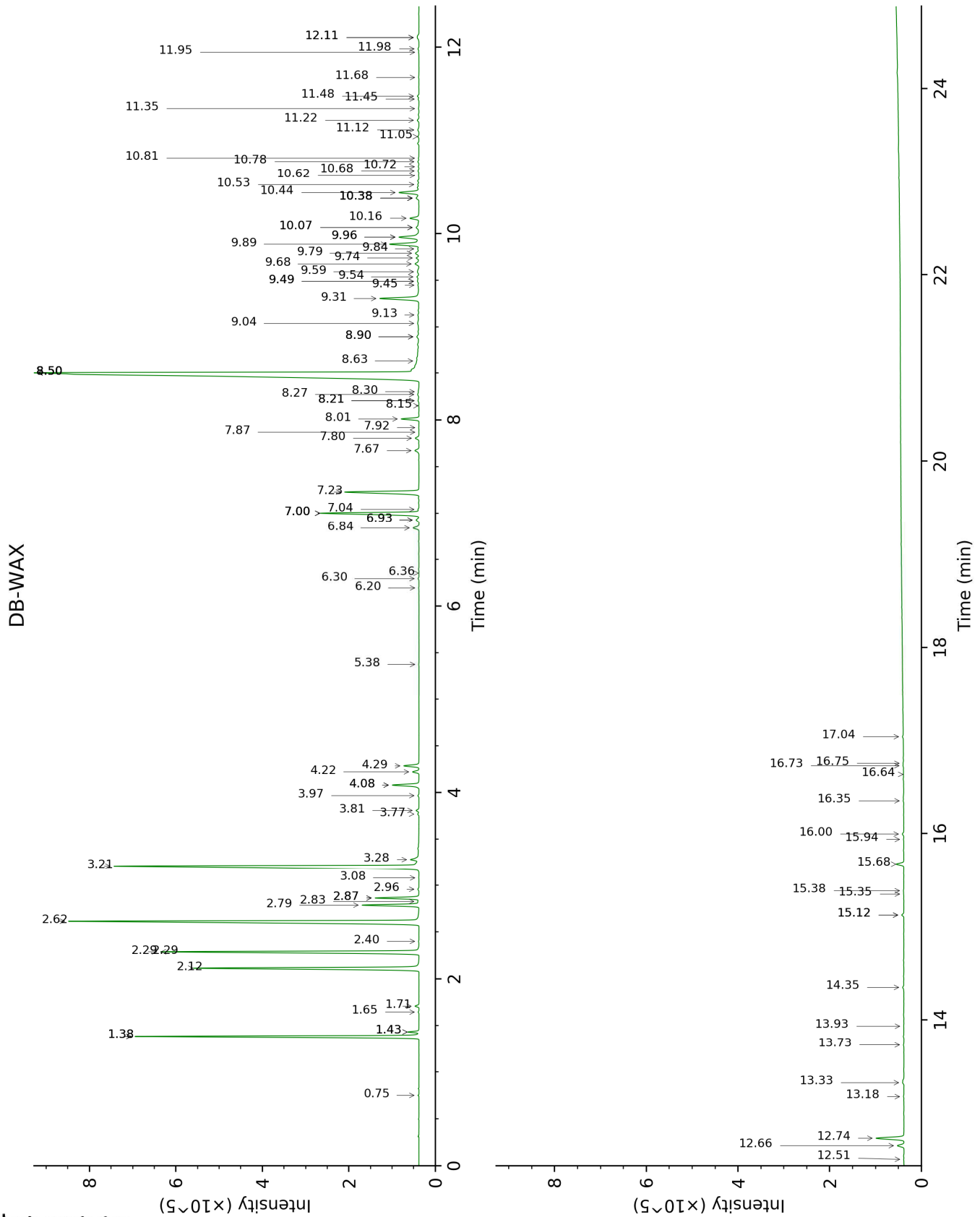
Note: no correction factor was applied

**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	0.60	640	0.01	0.75	887	0.01
Toluene	1.14	764	0.01	1.43*	1003	0.28
Hashishene	2.82	916	0.02	1.38*	997	7.53
$\alpha$ -Thujene	2.98	926	0.24	1.43*	1003	[0.28]
$\alpha$ -Pinene	3.06	931	7.45	1.38*	997	[7.53]
Camphene	3.24*	943	0.15	1.71	1030	0.12
$\alpha$ -Fenchene	3.24*	943	[0.15]	1.64	1024	0.02
Thuja-2,4(10)-diene	3.34	949	0.01	2.29*	1087	8.02
meta-Cymene	3.59	966	0.04	2.86*	1136	1.23
Sabinene	3.67*†	972	15.19	2.29*	1087	[8.02]
$\beta$ -Pinene	3.67*†	972	[15.19]	2.12	1070	7.36
Dehydro-1,8-cineole	3.91	987	0.02	3.08	1152	0.01
Myrcene	3.99	993	1.19	2.86*	1136	[1.23]
2-Carene	4.07	998	0.01	2.40	1098	0.02
$\alpha$ -Phellandrene	4.14*	1002	1.70	2.79	1130	1.69
Pseudolimonene	4.14*	1002	[1.70]	2.83	1133	0.03
$\Delta^3$ -Carene	4.24	1009	12.97	2.62	1116	13.07
$\alpha$ -Terpinene	4.33	1014	0.08	2.96	1143	0.04
ortho-Cymene	4.41	1020	0.06	4.08*	1230	0.94
para-Cymene	4.45	1022	0.90	4.08*	1230	[0.94]
Limonene	4.54*	1028	12.24	3.21	1163	11.84
$\beta$ -Phellandrene	4.54*	1028	[12.24]	3.28	1168	0.41
(Z)- $\beta$ -Ocimene	4.74	1040	0.01	3.77	1207	0.05
(E)- $\beta$ -Ocimene	4.89	1050	0.04	3.97	1222	0.04
$\gamma$ -Terpinene	5.01	1057	0.10	3.81	1210	0.10
cis-Sabinene hydrate	5.12	1064	0.01	6.93*	1436	0.13
meta-Cymenene	5.37	1080	0.01	6.20	1382	0.01
Isoterpinolene	5.44	1084	0.19	4.22	1240	0.22
para-Cymenene	5.47*	1086	0.57	6.30	1389	0.04
Terpinolene	5.47*	1086	[0.57]	4.29	1245	0.52
$\alpha$ -Pinene oxide	5.56	1092	0.02	5.38	1323	0.01
trans-Sabinene hydrate	5.66	1098	0.02	7.92	1510	0.01
Linalool	5.70	1101	0.55	8.01	1517	0.55
trans-para-Mentha-2,8-dien-1-ol	5.97	1118	0.02	8.90*	1586	0.07
cis-Limonene oxide	6.18	1132	0.02	6.36	1393	0.01
cis-para-Mentha-2,8-dien-1-ol	6.21	1134	0.03	9.45	1630	0.04
trans-para-Mentha-2-en-1-ol	6.26	1137	0.03	8.90*	1586	[0.07]
trans-Verbenol	6.36	1144	0.02	9.49*	1633	0.08
Pinocarvone	6.59	1159	0.03	7.87	1506	0.01

<i>cis</i> -Sabinol	6.73	1168	0.02	10.82	1743	0.02
Terpinen-4-ol	6.84	1175	0.09	8.50*	1555	28.47
meta-Cymen-8-ol	6.94	1182	0.04	11.45	1797	0.03
para-Cymen-8-ol	6.99	1185	0.05	11.48	1800	0.06
$\alpha$ -Terpineol	7.03	1187	0.01	9.74	1654	0.09
Myrtenal	7.07	1190	0.11	8.63	1565	0.15
Unknown [m/z 109, 91 (100), 81 (88), 94 (75), 119 (74), 96 (73), 41 (63)... 150 (2)]	7.22	1200	0.05	10.78	1740	0.04
Verbenone	7.29	1204	0.07	9.54	1637	0.07
Car-2-en-4-one?	7.44	1215	0.05	9.49*	1633	[0.08]
<i>trans</i> -Carveol	7.51	1220	0.03	11.34	1788	0.02
<i>cis</i> -Carveol	7.69	1232	0.02	11.68	1817	0.02
Cuminal	7.76	1237	0.03	10.53	1718	0.04
Carvone	7.84	1242	0.02	9.96*	1672	0.89
Car-3-en-2-one	7.89	1246	0.01	10.38*	1706	0.13
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.98	1252	0.05	11.05	1763	0.04
Methyl citronellate	8.20	1267	0.01	8.15	1528	0.01
Bornyl acetate	8.43	1284	0.01	8.21*	1532	0.05
Unknown [m/z 43, 93 (66), 91 (44), 41 (38), 69 (35)... 152? (1)]	8.67	1300	0.02			
Car-3-en-5-one	8.80	1304	0.05	11.98	1844	0.04
para-Menth-5-en-1,2-diol isomer III	8.94	1314	0.07	15.12*	2140	0.08
Unknown [m/z 91, 79 (94), 77 (72), 41 (37), 93 (31)... 152 (1)]	9.10	1325	0.02			
$\delta$ -Elemene isomer	9.23	1334	0.10	6.93*	1436	[0.13]
Bicycloelemene	9.27*	1337	3.63	7.04	1444	0.04
$\delta$ -Elemene	9.27*	1337	[3.63]	7.00*	1441	3.61
$\alpha$ -Cubebene	9.43	1348	0.24	6.84	1429	0.24
Cyclosativene I	9.61	1361	0.02	7.00*	1441	[3.61]
Cyclosativene II	9.64	1363	0.03	7.00*	1441	[3.61]
$\alpha$ -Copaene	9.79	1374	2.91	7.23	1458	2.89
<i>cis</i> - $\beta$ -Elemene	9.92	1383	0.03	8.30	1540	0.04
$\beta$ -Cubebene	9.99	1388	0.12	7.80	1501	0.15
$\beta$ -Elemene	10.03	1391	0.99	8.50*	1555	[28.47]
Isocaryophyllene	10.20	1402	0.04	8.21*	1532	[0.05]
$\alpha$ -Gurjunene	10.24	1406	0.16	7.67	1491	0.18
$\beta$ -Caryophyllene	10.41*	1418	27.77	8.50*	1555	[28.47]
<i>cis</i> - $\alpha$ -Bergamotene	10.41*	1418	[27.77]	8.27	1537	0.07
$\beta$ -Copaene	10.51	1426	0.02	8.50*	1555	[28.47]
<i>trans</i> - $\alpha$ -Bergamotene	10.66*	1436	0.23	8.50*	1555	[28.47]

α-Guaiene	10.66*	1436	[0.23]	8.50*	1555	[28.47]
Unknown [m/z 41, 97 (78), 69 (77), 43 (71), 125 (67), 55 (56)... 168 (39)]	10.71	1440	0.03	17.04	2338	0.03
Unknown [m/z 139, 69 (60), 41 (51), 43 (47), 119 (41)... 204 (1)]	10.77	1445	0.04			
α-Humulene	10.82	1449	1.60	9.31	1618	1.60
allo-Aromadendrene	10.92	1456	0.04	9.04	1597	0.07
(E)-β-Farnesene	10.96*	1459	0.13	9.59	1642	0.08
β-Santalene	10.96*	1459	[0.13]	9.13	1604	0.04
γ-Murolene	11.15	1473	0.18	9.68	1649	0.17
Germacrene D	11.20	1476	0.20	9.79	1658	0.15
trans-Muurolo-4(15),5-diene	11.26*	1482	1.25	9.84	1662	0.05
ar-Curcumene	11.26*	1482	[1.25]	10.68	1731	0.03
β-Selinene	11.26*	1482	[1.25]	9.89	1666	1.19
α-Selinene	11.39*†	1491	1.08	9.96*	1672	[0.89]
epi-Cubebol	11.39*†	1491	[1.08]	11.95	1841	0.02
Bicyclogermacrene	11.39*†	1491	[1.08]	10.07*	1680	0.17
α-Muurolole	11.51	1500	0.11	10.07*	1680	[0.17]
Cubebol	11.62*	1509	0.36	12.51	1891	0.02
β-Bisabolene	11.62*	1509	[0.36]	10.16	1688	0.34
γ-Cadinene	11.66	1512	0.08	10.38*	1706	[0.13]
7-epi-α-Selinene	11.72	1516	0.04	10.38*	1706	[0.13]
δ-Cadinene	11.79*	1522	0.89	10.44	1711	0.79
trans-Calamenene	11.79*	1522	[0.89]	11.22	1778	0.07
(E)-γ-Bisabolene	11.90*	1530	0.07	10.38*	1706	[0.13]
trans-Cadina-1,4-diene	11.90*	1530	[0.07]	10.62	1727	0.01
α-Calacorene	11.97	1536	0.02	12.10*	1855	0.13
(E)-α-Bisabolene	12.07	1543	0.02	10.72	1735	0.02
Isocaryophyllene epoxide B	12.10	1546	0.11	12.10*	1855	[0.13]
Germacrene B	12.16	1551	0.07	11.12	1769	0.06
(E)-Nerolidol	12.34	1564	0.02	13.73	2004	0.02
Spathulenol	12.38	1568	0.01	14.35	2064	0.06
Caryophyllene oxide isomer	12.48*	1576	1.38	12.66	1904	0.25
Caryophyllene oxide	12.48*	1576	[1.38]	12.74	1912	1.05
Unknown [m/z 161, 105 (84), 43 (80), 119 (72), 93 (62), 121 (54)... 204 (38), 222 (2)]	12.55	1581	0.01	13.93	2024	0.01
Humulene epoxide I	12.67	1590	0.04	13.18	1953	0.02

Humulene epoxide II	12.81	1601	0.06	13.33	1966	0.05
Alismol	13.09*	1625	0.28	15.68	2195	0.27
Caryophylladienol I	13.09*	1625	[0.28]	15.94	2223	0.01
Caryophylladienol II	13.16	1630	0.05	16.00	2229	0.05
$\alpha$ -Muurolol	13.31	1642	0.02	15.12*	2140	[0.08]
$\alpha$ -Cadinol	13.38	1648	0.02	15.38	2166	0.02
<i>cis</i> -Calamenen-10-ol	13.45	1654	0.01	16.35	2266	0.02
<i>trans</i> -Calamenen-10-ol	13.50	1658	0.01	16.75	2308	0.02
(3 <i>Z</i> )-Caryophylla-3,8(13)-dien-5 $\beta$ -ol	13.60	1667	0.01	16.73	2305	0.02
$\alpha$ -Bisabolol	13.81	1684	0.01	15.35	2162	0.02
Unknown [m/z 43, 108 (62), 93 (51), 41 (42), 109 (37), 69 (36)...]	13.85	1687	0.01	16.64	2295	0.01
<b>Total identified</b>	<b>99.07%</b>			<b>98.80%</b>		
<b>Total reported</b>	<b>99.30%</b>			<b>98.94%</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

Note: no correction factor was applied

R.T.: Retention time (minutes)

R.I.: Retention index