

Date : April 08, 2022

CERTIFICATE OF ANALYSIS – GC PROFILING

SAMPLE IDENTIFICATION

Internal code : 22D05-PTH02


Customer identification : Black Pepper ORGANIC - Sri Lanka - BS0107R

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 : Pamela Lavoie, M.Sc., Chimiste

Analysis date : April 08, 2022

Checked and approved by :

Alexis St-Gelais, Ph. D., 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Notes: This report may not be published, including online, without the written consent from Laboratoire PhytoChemia.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: Clear liquid

Refractive index: 1.4815 ± 0.0003 (20 °C; method PC-MAT-016)

ISO 3061:2008 - OIL OF BLACK PEPPER - SRI LANKA

Compound	Min. %	Max. %	Observed %	Complies?
α-Pinene	10	16	10	Yes
β-Pinene	9	12	9	Yes
Sabinene	10	17	11	Yes
Δ3-Carene	5	11	8	Yes
Limonene	13	16	14	Yes
δ-Elemene	0.5	3.0	2.3	Yes
α-Copaene	1.5	4.0	3.2	Yes
β-Caryophyllene	12	21	24	No
Germacrene D		1.0	0.3	Yes
β-Selinene		2.0	0.1	Yes
α-Selinene	0.5	2.0	0.1	No
Caryophyllene oxide		1.0	0.3	Yes
Refractive index	1.4750	1.4900	1.4815	Yes

CONCLUSION

No adulterant, contaminant or diluent has been detected using this method. The oil marginally does not comply with the ISO standard for black pepper oil from Sri Lanka.

ANALYSIS SUMMARY – CONSOLIDATED CONTENTS

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

Identification	%	Class
Isovaleral	tr	Aliphatic aldehyde
2-Methylbutyral	tr	Aliphatic aldehyde
Toluene	tr	Simple phenolic
Tricyclene	0.01	Monoterpene
α -Thujene	1.11	Monoterpene
α -Pinene	9.93	Monoterpene
Camphene	0.29	Monoterpene
α -Fenchene	0.01	Monoterpene
3,7,7-Trimethylcyclohepta-1,3,5-triene	0.01	Monoterpene
β -Pinene	8.98	Monoterpene
Sabinene	10.98	Monoterpene
Dehydro-1,8-cineole	0.01	Monoterpenic ether
Myrcene	1.96	Monoterpene
2-Carene	0.01	Monoterpene
Pseudolimonene	0.03	Monoterpene
α -Phellandrene	1.59	Monoterpene
Δ^3 -Carene	7.74	Monoterpene
Unknown	tr	Monoterpene
α -Terpinene	0.16	Monoterpene
meta-Cymene	0.02	Monoterpene
para-Cymene	0.43	Monoterpene
Limonene	14.20	Monoterpene
β -Phellandrene	2.17	Monoterpene
(Z)- β -Ocimene	0.03	Monoterpene
(E)- β -Ocimene	0.21	Monoterpene
Unknown	0.03	Monoterpene
γ -Terpinene	0.30	Monoterpene
cis-Sabinene hydrate	0.18	Monoterpenic alcohol
Isoterpinolene	0.12	Monoterpene
Terpinolene	0.47	Monoterpene
para-Cymenene	0.02	Monoterpene
trans-Sabinene hydrate	0.11	Monoterpenic alcohol
Linalool	0.37	Monoterpenic alcohol
Verbenol analog?	0.01	Monoterpenic alcohol
trans-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
cis-Limonene oxide	0.02	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.02	Monoterpenic alcohol
trans-Limonene oxide	0.01	Monoterpenic ether
trans-Verbenol	0.02	Monoterpenic alcohol
meta-Mentha-4,6-dien-8-ol	0.01	Monoterpenic alcohol
1,4-Dimethyl-4-acetylcyclohexene	0.01	Monoterpenic ketone
Sabinaketone	0.01	Normonoterpenic ketone
Pinocarvone	0.01	Monoterpenic ketone
Borneol	0.01	Monoterpenic alcohol
cis-Sabinol	0.01	Monoterpenic alcohol

Terpinen-4-ol	0.38	Monoterpenic alcohol
meta-Cymen-8-ol	0.02	Monoterpenic alcohol
para-Cymen-8-ol	0.01	Monoterpenic alcohol
α -Terpineol	0.07	Monoterpenic alcohol
Myrtenal	0.01	Monoterpenic aldehyde
<i>cis</i> - α -Phellandrene epoxide (iPr vs Me)	0.01	Monoterpenic ether
Unknown	0.01	Oxygenated monoterpene
Verbenone	0.01	Monoterpenic ketone
Car-2-en-4-one?	0.02	Monoterpenic ketone
<i>trans</i> -Carveol	0.01	Monoterpenic alcohol
<i>cis</i> -Carveol	0.01	Monoterpenic alcohol
Nerol	0.01	Monoterpenic alcohol
Citronellol	0.01	Monoterpenic alcohol
Unknown	0.02	Unknown
Methyl citronellate	0.01	Monoterpenic ester
Bornyl acetate	0.01	Monoterpenic ester
Unknown	0.01	Oxygenated monoterpene
Myrtenyl acetate	0.01	Monoterpenic ester
Methyl geranate	0.01	Monoterpenic ester
δ -Elemene	2.32	Sesquiterpene
Bicycloelemene	tr	Sesquiterpene
α -Cubebene	0.23	Sesquiterpene
Cyclosativene I	0.11	Sesquiterpene
Cyclosativene II	0.02	Sesquiterpene
α -Ylangene	0.02	Sesquiterpene
α -Copaene	3.21	Sesquiterpene
β -Cubebene	0.30	Sesquiterpene
β -Elemene	0.32	Sesquiterpene
Isocaryophyllene	0.02	Sesquiterpene
α -Gurjunene	0.08	Sesquiterpene
Unknown	0.01	Sesquiterpene
<i>cis</i> - α -Bergamotene	0.01	Sesquiterpene
β -Caryophyllene	24.37	Sesquiterpene
β -Copaene	0.20	Sesquiterpene
γ -Elemene	0.01	Sesquiterpene
<i>trans</i> - α -Bergamotene	0.06*	Sesquiterpene
α -Guaiene	0.06*	Sesquiterpene
Unknown	0.02	Unknown
Unknown	0.05	Sesquiterpene
α -Humulene	1.11	Sesquiterpene
Unknown	0.01	Unknown
allo-Aromadendrene	0.02	Sesquiterpene
(<i>E</i>)- β -Farnesene	0.12	Sesquiterpene
β -Santalene	0.02	Sesquiterpene
γ -Gurjunene	0.02	Sesquiterpene
<i>trans</i> -Cadina-1(6),4-diene	0.05	Sesquiterpene
γ -Muurolene	0.07	Sesquiterpene
Germacrene D	0.35	Sesquiterpene
β -Selinene	0.13	Sesquiterpene
ar-Curcumene	0.01	Sesquiterpene
<i>trans</i> -Muurola-4(15),5-diene	0.07	Sesquiterpene
α -Selinene	0.10	Sesquiterpene

epi-Cubebol	0.10	Sesquiterpenic alcohol
Viridiflorene	0.06	Sesquiterpene
Bicyclogermacrene	0.01	Sesquiterpene
α -Muurolene	0.53	Sesquiterpene
β -Bisabolene	0.83	Sesquiterpene
γ -Cadinene	0.04	Sesquiterpene
Cubebol	0.17	Sesquiterpenic alcohol
7-epi- α -Selinene	0.06	Sesquiterpene
<i>trans</i> -Calamenene	0.05	Sesquiterpene
δ -Cadinene	0.91	Sesquiterpene
<i>trans</i> -Cadina-1,4-diene	0.05	Sesquiterpene
α -Calacorene	0.01	Sesquiterpene
(<i>E</i>)- α -Bisabolene	0.02	Sesquiterpene
Isocaryophyllene epoxide B	0.04	Sesquiterpenic ether
α -Elemol	0.03	Sesquiterpenic alcohol
(<i>E</i>)-Nerolidol	0.06	Sesquiterpenic alcohol
Spathulenol	0.04	Sesquiterpenic alcohol
Caryophyllene oxide	0.27	Sesquiterpenic ether
Caryophyllene oxide isomer	0.06	Sesquiterpenic ether
Globulol	0.01	Sesquiterpenic alcohol
Humulene epoxide I	0.01	Sesquiterpenic ether
Ledol	0.01	Sesquiterpenic alcohol
Humulene epoxide II	0.02	Sesquiterpenic ether
α -Corocalene	0.01	Sesquiterpene
Alismol	0.08	Sesquiterpenic alcohol
Caryophylladienol II	0.01	Sesquiterpenic alcohol
Caryophylladienol I?	0.01	Sesquiterpenic alcohol
τ -Muurolol	0.04	Sesquiterpenic alcohol
α -Muurolol	0.13	Sesquiterpenic alcohol
Phytone	0.01	Terpenic ketone
Consolidated total	99.44%	

*: 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

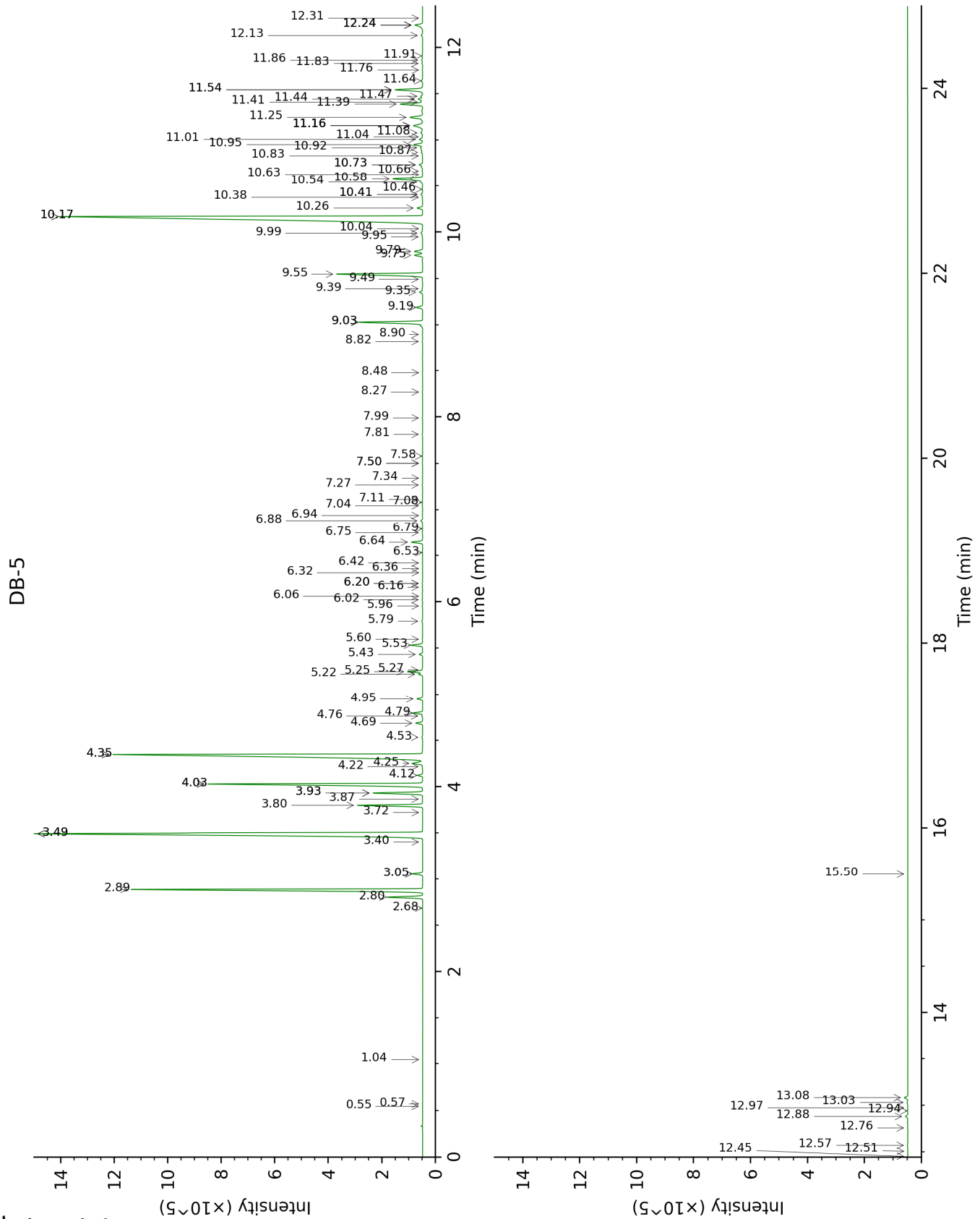
tr: The compound has been detected below 0.005% of total signal.

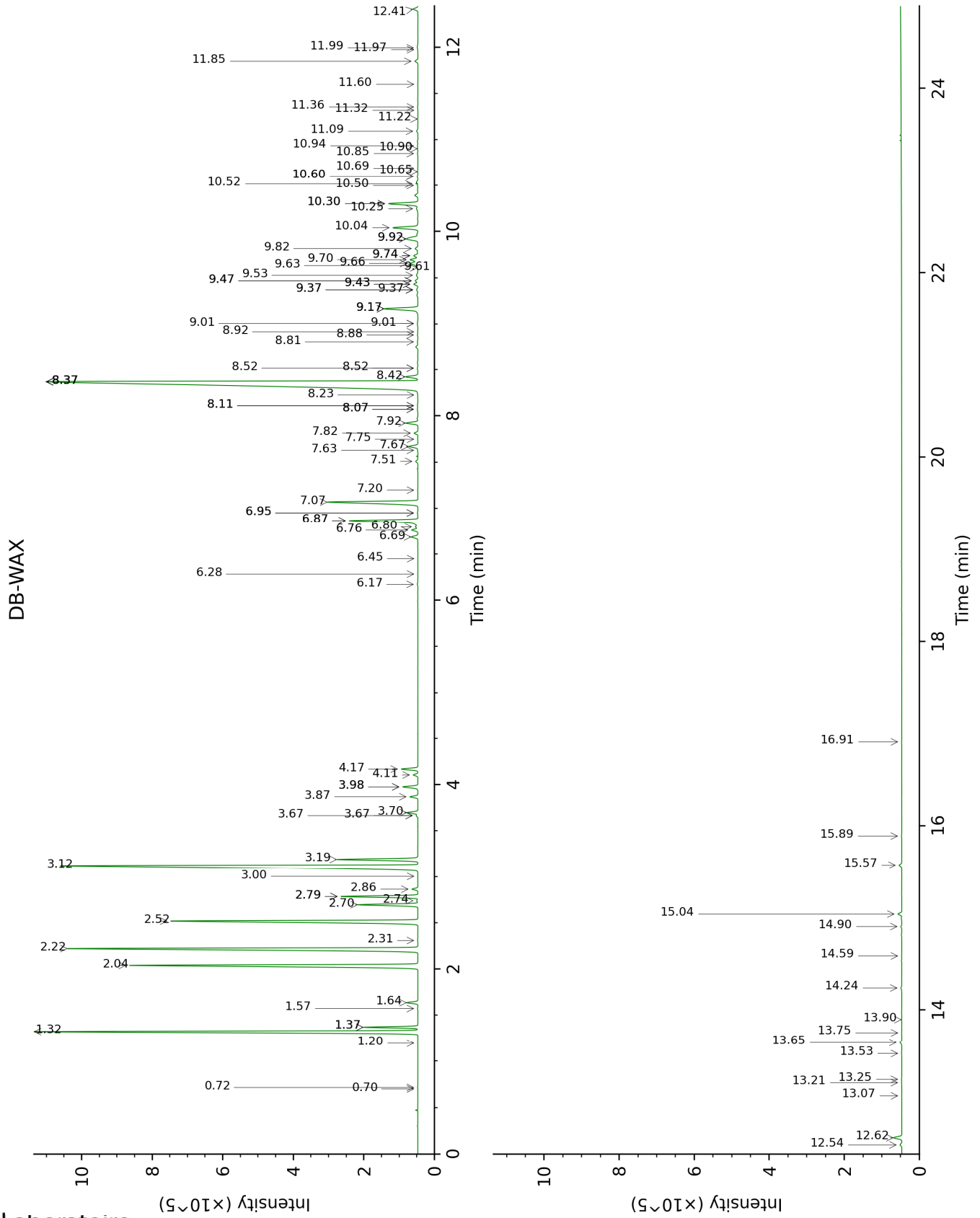
Note: no correction factor was applied

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	0.54	642	tr	0.72	887	tr
2-Methylbutyral	0.57	652	tr	0.70	881	tr
Toluene	1.04	758	tr	1.37*	1002	1.12
Tricyclene	2.68	917	0.01	1.20	974	tr
α -Thujene	2.80	925	1.11	1.37*	1002	[1.12]
α -Pinene	2.89	930	9.93	1.32	996	9.94
Camphene	3.05*	942	0.31	1.64	1029	0.29
α -Fenchene	3.05*	942	[0.31]	1.57	1022	0.01
3,7,7-Trimethylcyclohepta-1,3,5-triene	3.40	966	0.01	2.78*	1135	1.98
β -Pinene	3.49*	972	19.92	2.04	1069	8.98
Sabinene	3.49*	972	[19.92]	2.22	1088	10.98
Dehydro-1,8-cineole	3.72	987	0.01	3.00	1152	0.01
Myrcene	3.80	992	1.96	2.78*	1135	[1.98]
2-Carene	3.87	997	0.01	2.31	1096	0.01
Pseudolimonene	3.93*	1001	1.60	2.74	1131	0.03
α -Phellandrene	3.93*	1001	[1.60]	2.70	1128	1.59
Δ 3-Carene	4.03*	1008	7.74	2.52	1114	7.74
Unknown [m/z 91, 92 (53), 77 (19), 41 (14)... 134 (5)]	4.03*	1008	[7.74]			
α -Terpinene	4.12	1013	0.16	2.86	1141	0.16
meta-Cymene	4.22	1019	0.02	3.98*	1228	0.45
para-Cymene	4.25	1022	0.43	3.98*	1228	[0.45]
Limonene	4.35*	1028	16.31	3.12	1162	14.20
β -Phellandrene	4.35*	1028	[16.31]	3.19	1167	2.17
(Z)- β -Ocimene	4.53	1039	0.03	3.67*	1205	0.05
(E)- β -Ocimene	4.69	1049	0.21	3.87	1220	0.22
Unknown [m/z 93, 91 (54), 92 (31), 77 (29), 79 (17), 43 (13), 41 (10), 136 (9)]	4.76	1054	0.03	3.67*	1205	[0.05]
γ -Terpinene	4.80	1056	0.30	3.70	1208	0.31
cis-Sabinene hydrate	4.95	1066	0.18	6.76	1429	0.19
Isoterpinolene	5.22	1083	0.12	4.11	1238	0.14
Terpinolene	5.25	1084	0.47	4.17	1243	0.47
para-Cymenene	5.27	1086	0.02	6.17	1386	0.02
trans-Sabinene hydrate	5.44	1096	0.11	7.82	1507	0.11
Linalool	5.53	1102	0.37	7.92	1516	0.37
Verbenol analog?	5.60	1106	0.01	8.23	1539	0.01
trans-para-Mentha-2,8-dien-1-ol	5.79	1119	0.03	8.81	1584	0.02
cis-Limonene oxide	5.96	1130	0.02	6.28	1394	0.01
cis-para-Mentha-2,8-dien-1-ol	6.02	1134	0.02	9.37*	1629	0.05

<i>trans</i> -Limonene oxide	6.06	1136	0.01	6.45	1406	tr
<i>trans</i> -Verbenol	6.16	1142	0.02	9.37*	1629	[0.05]
meta-Mentha-4,6-dien-8-ol	6.20*	1145	0.02	9.17*	1612	1.17
1,4-Dimethyl-4-acetylcyclohexene	6.20*	1145	[0.02]	7.20	1461	0.01
Sabinaketon	6.32	1152	0.01	8.52*	1562	0.03
Pinocarvone	6.36	1155	0.01	7.75	1502	0.01
Borneol	6.42	1159	0.01	9.63	1650	0.06
<i>cis</i> -Sabinol	6.53	1166	0.01	10.69	1738	0.01
Terpinen-4-ol	6.64	1174	0.38	8.42	1554	0.39
meta-Cymen-8-ol	6.75	1180	0.02	11.32	1792	tr
para-Cymen-8-ol	6.79	1183	0.01	11.36	1795	0.01
α -Terpineol	6.88	1189	0.07	9.66	1652	0.29
Myrtenal	6.94	1192	0.01	8.52*	1562	[0.03]
<i>cis</i> - α -Phellandrene epoxide (iPr vs Me)	7.04	1199	0.01	10.85	1752	0.02
Unknown [m/z 109, 91 (100), 81 (88), 94 (75), 119 (74), 96 (73), 41 (63)... 150 (2)]	7.08	1201	0.01	10.66	1735	0.01
Verbenone	7.11	1204	0.01	9.47*	1637	0.09
Car-2-en-4-one?	7.27	1214	0.02	9.37*	1629	[0.05]
<i>trans</i> -Carveol	7.34	1219	0.01	11.22	1784	0.01
<i>cis</i> -Carveol	7.50*	1230	0.01	11.60	1816	0.01
Nerol	7.50*	1230	[0.01]	10.90	1756	0.01
Citronellol	7.58	1235	0.01	10.60*	1730	0.03
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.81	1250	0.02	10.94	1759	0.02
Methyl citronellate	7.99	1262	0.01	8.07*	1527	0.03
Bornyl acetate	8.27	1281	0.01	8.11*	1530	0.02
Unknown [m/z 43, 93 (66), 91 (44), 41 (38), 69 (35)... 152? (1)]	8.48	1295	0.01			
Myrtenyl acetate	8.82	1319	0.01	9.43*	1634	0.13
Methyl geranate	8.90	1324	0.01	9.61	1648	0.02
δ -Elemene	9.03*†	1333	2.32	6.87*	1437	2.35
Bicycloelemene	9.03*†	1333	[2.32]	6.95*	1443	0.02
α -Cubebene	9.19	1344	0.23	6.69	1423	0.22
Cyclosativene I	9.35	1356	0.11	6.80	1432	0.08
Cyclosativene II	9.39	1359	0.02	6.87*	1437	[2.35]
α -Ylangene	9.49	1366	0.02	6.95*	1443	[0.02]
α -Copaene	9.55	1370	3.21	7.07	1452	3.21
β -Cubebene	9.75	1384	0.30	7.67	1496	0.30
β -Elemene	9.79	1387	0.32	8.37*	1550	24.85
Isocaryophyllene	9.95	1398	0.02	8.07*	1527	[0.03]
α -Gurjunene	9.99	1401	0.08	7.51	1484	0.08
Unknown [m/z 105, 161 (84), 91 (80), 204]	10.04	1404	0.01	7.63	1493	0.01

(75), 119 (69), 189 (64)]						
<i>cis</i> - α -Bergamotene	10.17*	1414	24.38	8.11*	1530	[0.02]
β -Caryophyllene	10.17*	1414	[24.38]	8.37*	1550	[24.85]
β -Copaene	10.26	1421	0.20	8.37*	1550	[24.85]
γ -Elemene	10.38	1430	0.01	8.92	1593	0.01
<i>trans</i> - α -Bergamotene	10.41*	1432	0.06	8.37*	1550	[24.85]
α -Guaiene	10.41*	1432	[0.06]	8.37*	1550	[24.85]
Unknown [m/z 41, 97 (78), 69 (77), 43 (71), 125 (67), 55 (56)... 168 (39)]	10.46	1436	0.02	16.91	2338	0.01
Unknown [m/z 139, 69 (60), 41 (51), 43 (47), 119 (41)... 204 (1)]	10.54†	1442	1.16			
α -Humulene	10.58†	1444	[1.16]	9.17*	1612	[1.17]
Unknown [m/z 109, 110 (33), 43 (30), 95 (23), 71 (20), 41 (19)...]	10.63	1448	0.01			
allo-Aromadendrene	10.66	1451	0.02	8.88	1590	0.01
(<i>E</i>)- β -Farnesene	10.73*	1456	0.13	9.43*	1634	[0.13]
β -Santalene	10.73*	1456	[0.13]	9.01*	1600	0.03
γ -Gurjunene	10.83	1463	0.02	9.01*	1600	[0.03]
<i>trans</i> -Cadina-1(6),4-diene	10.87	1466	0.05	9.17*	1612	[1.17]
γ -Muurolene	10.92	1470	0.07	9.47*	1637	[0.09]
Germacrene D	10.95	1472	0.35	9.70	1656	0.27
β -Selinene	11.01	1476	0.13	9.74*	1659	0.14
ar-Curcumene	11.04	1478	0.01	10.50	1722	0.02
<i>trans</i> -Muurola-4(15),5-diene	11.08	1482	0.07	9.74*	1659	[0.14]
α -Selinene	11.16*	1487	0.47	9.82	1666	0.10
epi-Cubebol	11.16*	1487	[0.47]	11.85	1839	0.10
Viridiflorene	11.16*	1487	[0.47]	9.53	1642	0.06
Bicyclogermacrene	11.16*	1487	[0.47]	9.92*	1674	0.54
α -Muurolene	11.25	1494	0.53	9.92*	1674	[0.54]
β -Bisabolene	11.39	1505	0.83	10.04	1684	0.83
γ -Cadinene	11.41	1506	0.04	10.25	1701	0.07
Cubebol	11.44	1509	0.17	12.41	1889	0.16
7-epi- α -Selinene	11.47	1511	0.06	10.30*	1705	0.97
<i>trans</i> -Calamenene	11.54*	1517	1.03	11.09	1772	0.05
δ -Cadinene	11.54*	1517	[1.03]	10.30*	1705	[0.97]
<i>trans</i> -Cadina-1,4-diene	11.64	1524	0.05	10.52	1723	0.06
α -Calacorene	11.76	1534	0.01	11.97	1850	0.01
(<i>E</i>)- α -Bisabolene	11.83	1539	0.02	10.60*	1730	[0.03]
Isocaryophyllene epoxide B	11.86	1542	0.04	11.99	1852	0.03
α -Elemol	11.91	1545	0.03	13.90	2028	0.01

(E)-Nerolidol	12.13	1563	0.06	13.65	2004	0.06
Spathulenol	12.24*	1571	0.36	14.24	2062	0.04
Caryophyllene oxide	12.24*	1571	[0.36]	12.62	1908	0.27
Caryophyllene oxide isomer	12.24*	1571	[0.36]	12.54	1901	0.06
Globulol	12.32	1577	0.01	13.75	2014	0.01
Humulene epoxide I	12.45	1588	0.01	13.07	1950	0.01
Ledol	12.51	1592	0.01	13.25	1966	0.01
Humulene epoxide II	12.57	1597	0.02	13.21	1963	0.01
α-Corocalene	12.76	1613	0.01	13.53	1992	0.01
Alismol	12.88	1623	0.08	15.57	2196	0.09
Caryophylladienol II	12.94	1628	0.01	15.89	2228	0.01
Caryophylladienol I?	12.97	1630	0.01			
τ-Muurolol	13.03	1635	0.04	14.90	2127	0.03
α-Muurolol	13.08	1639	0.13	15.04	2141	0.12
Phytone	15.50	1848	0.01	14.59	2095	0.01
Total identified		98.29%			99.32%	
Total reported		99.55%			99.37%	

*: 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