

Date : March 07, 2018

CERTIFICATE OF ANALYSIS - GC PROFILING

SAMPLE IDENTIFICATION

Internal code : 18B21-CHS1-1-CC

Customer identification : Black pepper - Sri Lanka - HD/O/BP/180001

Type : Essential oil

Source : *Piper nigrum*

Customer : American College of Healthcare Sciences

ANALYSIS

Method: PC-PA-014-17J19 - 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 : March 01, 2018

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: 1.4815 ± 0.0003 (20 °C)

COMPLIANCE WITH ISO 3061:2008 (PIPER NIGRUM, SRI LANKA)

Compound	Min. Content	Max. Content	Observed Content	Complies?
α-Pinene	10	16	9.7	No
β-Pinene	9	12	11.6	Yes
Sabinene	10	17	9.4	No
Δ ³ -Carene	5	11	4.3	No
Limonene	13	16	15.7	Yes
δ-Elemene	0.5	3	1.3	Yes
α-Copaene	1.5	4	3.6	Yes
β-Caryophyllene	12	21	22.2	No
Germacrene D		1	0.4	Yes
α-Selinene	0.5	2	0.2	No
β-Selinene		2	0.4	Yes
Caryophyllene oxide		1	0.7	Yes
Refractive index	1.475	1.490	1.4815	Yes

CONCLUSION

No adulterant, contaminant or diluent has been detected using this method. The oil shows small non-compliances with the ISO standard, but nevertheless the oil corresponds to the expectations for the species.

ANALYSIS SUMMARY

Identification	DB-5 (%)	DB-WAX (%)	Classe
Isovaleral	tr	tr*	Aliphatic aldehyde
2-Methylbutyral	tr	[tr]*	Aliphatic aldehyde
Toluene	tr	tr	Simple phenolic
Tricyclene	tr	tr	Monoterpene
α -Thujene	1.17	1.18	Monoterpene
α -Pinene	9.74	9.77	Monoterpene
Camphene	0.26*	0.25	Monoterpene
α -Fenchene	[0.26]*	0.01	Monoterpene
meta-Cymene	0.01	1.75*	Monoterpene
Sabinene	20.93*	9.36	Monoterpene
β -Pinene	[20.93]*	11.63	Monoterpene
Myrcene	1.75	[1.75]*	Monoterpene
Pseudolimonene	0.85*	0.03	Monoterpene
α -Phellandrene	[0.85]*	0.83	Monoterpene
Δ^3 -Carene	4.27	4.27	Monoterpene
α -Terpinene	0.13	0.12	Monoterpene
ortho-Cymene	0.01	0.38*	Simple phenolic
para-Cymene	0.35	[0.38]*	Monoterpene
β -Phellandrene	17.12*	1.49	Monoterpene
Limonene	[17.12]*	15.67	Monoterpene
(Z)- β -Ocimene	0.03	0.03	Monoterpene
(E)- β -Ocimene	0.12	0.13	Monoterpene
Unknown	0.01	tr	Monoterpene
γ -Terpinene	0.21	0.22	Monoterpene
cis-Sabinene hydrate	0.10	0.14*	Monoterpenic alcohol
Isoterpinolene	0.05	0.06	Monoterpene
para-Cymenene	0.27*	0.01	Monoterpene
Terpinolene	[0.27]*	0.26	Monoterpene
trans-Sabinene hydrate	0.06	0.07*	Monoterpenic alcohol
Linalool	0.24	0.25	Monoterpenic alcohol
Verbenol analog?	0.01	0.01	Monoterpenic alcohol
trans-para-Mentha-2,8-dien-1-ol	0.03	0.03	Monoterpenic alcohol
cis-Limonene oxide	0.01	0.01	Monoterpenic ether
trans-Pinocarveol	0.03*	0.03	Monoterpenic alcohol
trans-Limonene oxide	[0.03]*	0.01	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	[0.03]*	0.03	Monoterpenic alcohol
trans-para-Menth-2-en-1-ol	0.01	0.02	Monoterpenic alcohol
trans-Verbenol	0.02	0.28*	Monoterpenic alcohol
Pinocarpone	0.02	[0.07]*	Monoterpenic ketone
cis-Sabinol	0.02*	0.01*	Monoterpenic alcohol
α -Phellandren-8-ol	[0.02]*	3.22*	Monoterpenic alcohol
Terpinen-4-ol	0.31	0.32	Monoterpenic alcohol
meta-Cymen-8-ol	0.02	0.01	Monoterpenic alcohol
Cryptone	0.01	0.02*	Normoterpenic ketone
para-Cymen-8-ol	0.02	0.02	Monoterpenic alcohol
α -Terpineol	0.07	0.61*	Monoterpenic alcohol
Myrtenol	0.02	[0.01]*	Monoterpenic alcohol
Unknown	0.01	0.01	Oxygenated monoterpene

Car-2-en-4-one?	0.01	[0.28]*	Monoterpenic ketone
<i>trans</i> -Carveol	0.01	0.01	Monoterpenic alcohol
<i>cis</i> -Carveol	0.01	0.01	Monoterpenic alcohol
Carvone	0.01	0.01	Monoterpenic ketone
Unknown	0.01	0.01	Unknown
Methyl citronellate	0.01	0.03*	Monoterpenic ester
Bornyl acetate	0.01	0.02*	Monoterpenic ester
Unknown	0.01	0.10	Unknown
Unknown	0.02	0.69*	Unknown
δ -Elemene isomer	0.04	[0.14]*	Sesquiterpene
δ -Elemene	1.30	1.38	Sesquiterpene
α -Cubebene	0.26	0.25	Sesquiterpene
Unknown	0.04		Sesquiterpene
Cyclosativene I	0.11	[0.14]*	Sesquiterpene
α -Copaene	3.58	3.56	Sesquiterpene
β -Cubebene	0.33	0.35	Sesquiterpene
β -Elemene	0.37	22.60*	Sesquiterpene
α -Gurjunene	0.11	0.11	Sesquiterpene
Isocaryophyllene	0.02	[0.03]*	Sesquiterpene
β -Caryophyllene	22.21*	[22.60]*	Sesquiterpene
<i>cis</i> - α -Bergamotene	[22.21]*	[0.02]*	Sesquiterpene
β -Copaene	0.19	[22.60]*	Sesquiterpene
<i>trans</i> - α -Bergamotene	0.01	[22.60]*	Sesquiterpene
α -Guaiene	0.05	[22.60]*	Sesquiterpene
Unknown	0.06		Unknown
α -Humulene	1.18	1.19	Sesquiterpene
(<i>E</i>)- β -Farnesene	0.26	[0.28]*	Sesquiterpene
<i>trans</i> -Cadina-1(6),4-diene	0.06	0.05	Sesquiterpene
γ -Gurjunene	0.03	[0.02]*	Sesquiterpene
γ -Murolene	0.09	0.12	Sesquiterpene
Germacrene D	0.35	[0.61]*	Sesquiterpene
β -Selinene	0.38	0.38	Sesquiterpene
<i>ar</i> -Curcumene	0.11	0.10	Sesquiterpene
α -Selinene	0.24	0.24	Sesquiterpene
Viridiflorene	0.45	0.10	Sesquiterpene
epi-Cubebol	0.12	0.18	Sesquiterpenic alcohol
α -Murolene	0.58	0.68	Sesquiterpene
β -Bisabolene	3.28	[3.22]*	Sesquiterpene
Cubebol	0.29	0.28	Sesquiterpenic alcohol
7-epi- α -Selinene	0.06	0.05	Sesquiterpene
δ -Cadinene	1.69*	1.53	Sesquiterpene
<i>trans</i> -Calamenene	[1.69]*	0.11	Sesquiterpene
(<i>E</i>)- γ -Bisabolene	0.09	0.10	Sesquiterpene
α -Calacorene	0.03	0.03	Sesquiterpene
(<i>E</i>)- α -Bisabolene	0.07	0.07	Sesquiterpene
Isocaryophyllene epoxide B	0.07	0.06	Sesquiterpenic ether
Unknown	0.01	0.01	Aliphatic alcohol
(<i>E</i>)-Nerolidol	0.13	0.18	Sesquiterpenic alcohol
Caryophyllene oxide isomer	0.93	0.13	Sesquiterpenic ether
Caryophyllene oxide	[0.93]	0.71	Sesquiterpenic ether
Unknown	0.03	0.03	Oxygenated sesquiterpene
Humulene epoxide II	0.07	0.06	Sesquiterpenic ether

α -Corocalene	0.07	0.05	Sesquiterpene
Guaia-6,10(14)-dien-4 β -ol	0.21	0.21	Sesquiterpenic alcohol
Caryophylladienol I	0.03	0.04	Sesquiterpenic alcohol
τ -Cadinol	0.18*	0.02	Sesquiterpenic alcohol
τ -Muurolol	[0.18]*	0.15	Sesquiterpenic alcohol
α -Muurolol	0.67	[0.69]*	Sesquiterpenic alcohol
<i>cis</i> -Calamene-10-ol	0.02	0.02	Sesquiterpenic alcohol
<i>trans</i> -Calamene-10-ol	0.01	0.01	Sesquiterpenic alcohol
(3 <i>Z</i>)-Caryophylla-3,8(13)-dien-5 β -ol	0.02	0.03	Sesquiterpenic alcohol
α -Bisabolol	0.01	0.02	Sesquiterpenic alcohol
Total identified	98.72%	98.50%	

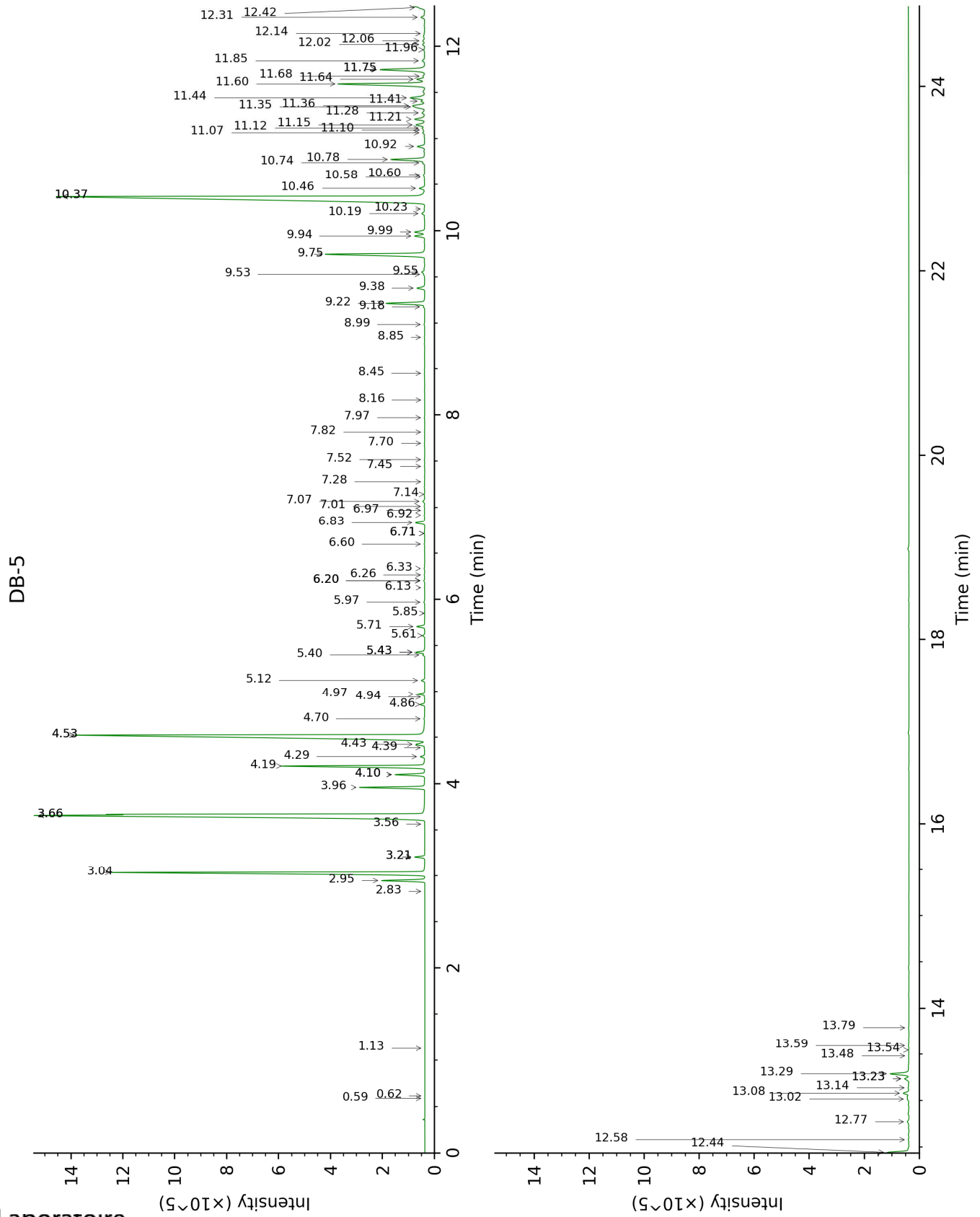
*: Two or more compounds are coeluting on this column

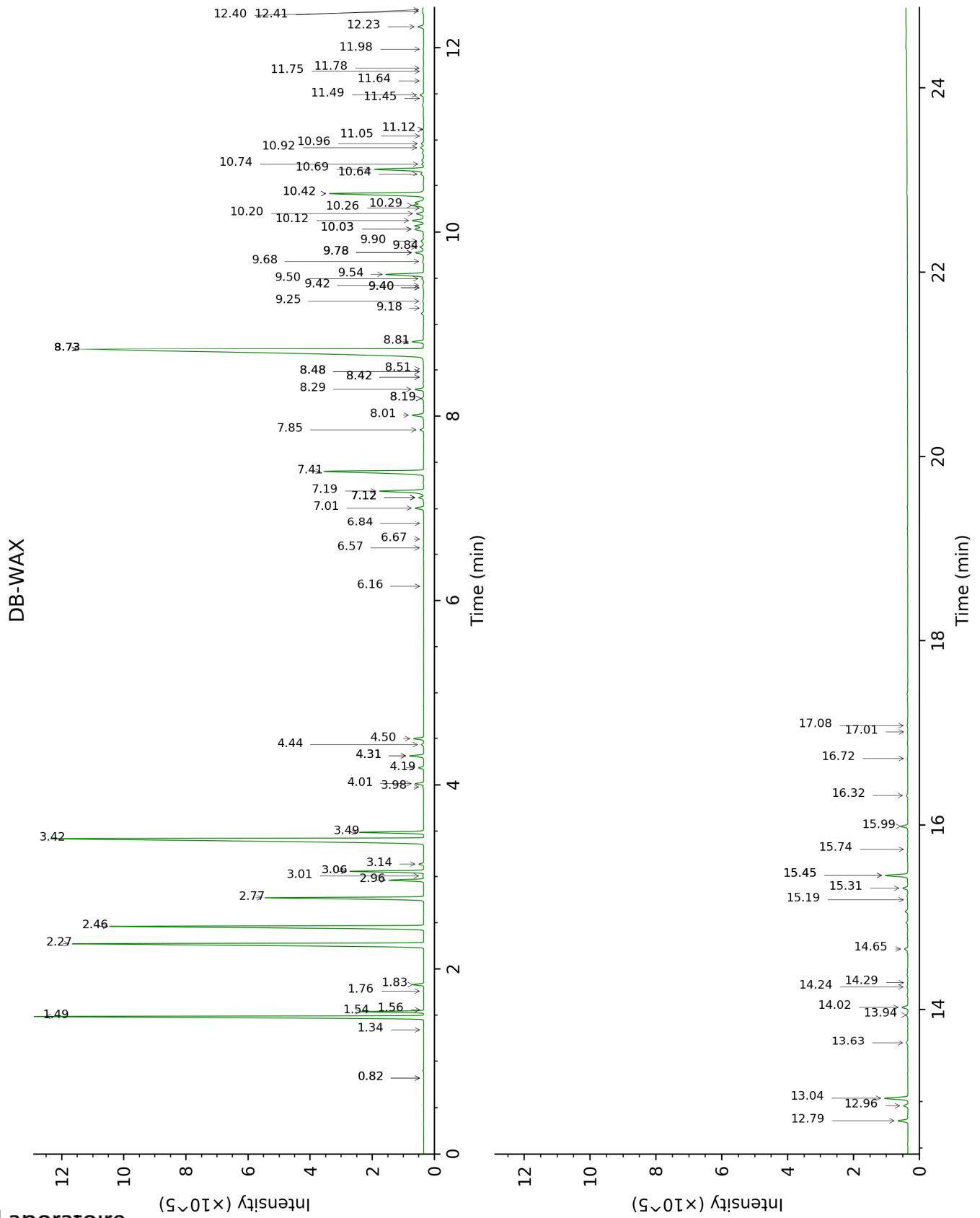
[xx]: Duplicate percentage due to coelutions, not taken account in the identified total

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

Note: no correction factor was applied

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FULL ANALYSIS DATA

Identification	Column DB-5			Column DB-WAX		
	R.T	R.I	%	R.T	R.I	%
Isovaleral	0.59	638	tr	0.82*	885	tr
2-Methylbutyral	0.62	648	tr	0.82*	885	[tr]
Toluene	1.13	753	tr	1.56	1004	tr
Tricyclene	2.83	913	tr	1.34	974	tr
α-Thujene	2.95	921	1.17	1.54	1002	1.18
α-Pinene	3.04	927	9.74	1.49	997	9.77
Camphene	3.21*	938	0.26	1.83	1030	0.25
α-Fenchene	3.21*	938	[0.26]	1.76	1023	0.01
meta-Cymene	3.56	962	0.01	3.06*	1135	1.75
Sabinene	3.66*†	968	20.93	2.46	1089	9.36
β-Pinene	3.66*†	968	[20.93]	2.28	1071	11.63
Myrcene	3.96	988	1.75	3.06*	1135	[1.75]
Pseudolimonene	4.10*	998	0.85	3.01	1131	0.03
α-Phellandrene	4.10*	998	[0.85]	2.96	1128	0.83
Δ ³ -Carene	4.19	1003	4.27	2.77	1113	4.27
α-Terpinene	4.29	1010	0.13	3.14	1141	0.12
ortho-Cymene	4.39	1016	0.01	4.31*	1228	0.38
para-Cymene	4.43	1018	0.35	4.31*	1228	[0.38]
β-Phellandrene	4.53*	1024	17.12	3.49	1168	1.49
Limonene	4.53*	1024	[17.12]	3.42	1162	15.67
(Z)-β-Ocimene	4.70	1036	0.03	3.98	1204	0.03
(E)-β-Ocimene	4.86	1045	0.12	4.19	1219	0.13
Unknown [m/z 41, 69 (97), 95 (33), 67 (31), 53 (18)...]	4.94	1050	0.01	6.16	1359	tr
γ-Terpinene	4.97	1052	0.21	4.01	1206	0.22
cis-Sabinene hydrate	5.12	1062	0.10	7.12*	1429	0.14
Isoterpinolene	5.40	1079	0.05	4.44	1236	0.06
para-Cymenene	5.43*	1081	0.27	6.57	1388	0.01
Terpinolene	5.43*	1081	[0.27]	4.50	1241	0.26
trans-Sabinene hydrate	5.61	1092	0.06	8.19*	1508	0.07
Linalool	5.71	1098	0.24	8.29	1516	0.25
Verbenol analog?	5.85	1108	0.01	8.51	1533	0.01
trans-para- Mentha-2,8-dien- 1-ol	5.97	1115	0.03	9.25	1590	0.03
cis-Limonene oxide	6.13	1125	0.01	6.67	1395	0.01
trans-Pinocarveol	6.20*	1130	0.03	9.42	1604	0.03
trans-Limonene oxide	6.20*	1130	[0.03]	6.84	1408	0.01
cis-para-Mentha- 2,8-dien-1-ol	6.20*	1130	[0.03]	9.68	1624	0.03
trans-para-Menth- 2-en-1-ol	6.26	1134	0.01	9.18	1584	0.02

<i>trans</i> -Verbenol	6.33	1138	0.02	9.78*	1632	0.28
Pinocarvone	6.60	1155	0.02	8.19*	1508	[0.07]
<i>cis</i> -Sabinol	6.71*	1163	0.02	11.12*	1742	0.01
α -Phellandren-8-ol	6.71*	1163	[0.02]	10.42*	1684	3.22
Terpinen-4-ol	6.83	1170	0.31	8.82	1556	0.32
meta-Cymen-8-ol	6.92	1176	0.02	11.75	1795	0.01
Cryptone	6.97	1179	0.01	9.40*	1602	0.02
para-Cymen-8-ol	7.01	1182	0.02	11.78	1798	0.02
α -Terpineol	7.07	1185	0.07	10.03*†	1652	0.61
Myrtenol	7.14	1190	0.02	11.12*	1742	[0.01]
Unknown [m/z 109, 91 (100), 81 (88), 94 (75), 119 (74), 96 (73), 41 (63)... 150 (2)]	7.28	1199	0.01	11.05	1736	0.01
Car-2-en-4-one?	7.45	1210	0.01	9.78*	1632	[0.28]
<i>trans</i> -Carveol	7.52	1215	0.01	11.64	1786	0.01
<i>cis</i> -Carveol	7.70	1226	0.01	11.98	1816	0.01
Carvone	7.82	1234	0.01	10.26	1671	0.01
Unknown [m/z 43, 97 (69), 107 (46), 41 (28), 55 (21), 109 (20)...]	7.97	1245	0.01	11.45	1770	0.01
Methyl citronellate	8.16	1258	0.01	8.42*	1526	0.03
Bornyl acetate	8.45	1277	0.01	8.48*	1531	0.02
Unknown [m/z 43, 111 (84), 109 (71), 126 (70)...]	8.85	1304	0.01	14.65	2061	0.10
Unknown [m/z 111, 126 (93), 43 (90), 71 (60)...]	8.99	1313	0.02	15.45*	2140	0.69
δ -Elemene isomer	9.18	1327	0.04	7.12*	1429	[0.14]
δ -Elemene	9.22	1330	1.30	7.19	1434	1.38
α -Cubebene	9.38	1341	0.26	7.01	1420	0.25
Unknown [m/z 95, 147 (61), 96 (39), 93 (37), 94 (37)... 204 (4)]	9.53	1352	0.04			
Cyclosativene I	9.55	1353	0.11	7.12*	1429	[0.14]
α -Copaene	9.75	1367	3.58	7.41	1450	3.56
β -Cubebene	9.94	1381	0.33	8.01	1495	0.35
β -Elemene	9.99	1384	0.37	8.73*	1550	22.60
α -Gurjunene	10.19	1398	0.11	7.85	1483	0.11
Isocaryophyllene	10.24	1401	0.02	8.42*	1526	[0.03]
β -Caryophyllene	10.37*	1411	22.21	8.73*	1550	[22.60]
<i>cis</i> - α -Bergamotene	10.37*	1411	[22.21]	8.48*	1531	[0.02]
β -Copaene	10.46	1418	0.19	8.73*	1550	[22.60]
<i>trans</i> - α -Bergamotene	10.58	1427	0.01	8.73*	1550	[22.60]

α-Guaiene	10.60	1429	0.05	8.73*	1550	[22.60]
Unknown [m/z 41, 97 (78), 69 (77), 43 (71), 125 (67), 55 (56)... 168 (39)]	10.74	1439	0.06			
α-Humulene	10.78	1442	1.18	9.54	1613	1.19
(E)-β-Farnesene	10.92	1452	0.26	9.78*	1632	[0.28]
trans-Cadina-1(6),4-diene	11.07	1463	0.06	9.50	1610	0.05
γ-Gurjunene	11.10	1465	0.03	9.40*	1602	[0.02]
γ-Murolene	11.12	1467	0.09	9.84	1637	0.12
Germacrene D	11.15	1469	0.35	10.03*†	1652	[0.61]
β-Selinene	11.21	1474	0.38	10.12	1660	0.38
ar-Curcumene	11.28	1479	0.11	10.92	1726	0.10
α-Selinene	11.35	1484	0.24	10.20	1666	0.24
Viridiflorene	11.36	1485	0.45	9.90	1642	0.10
epi-Cubebol	11.41	1488	0.12	12.23	1838	0.18
α-Murolene	11.44	1491	0.58	10.29	1673	0.68
β-Bisabolene	11.60	1502	3.28	10.42*	1684	[3.22]
Cubebol	11.64	1506	0.29	12.79	1888	0.28
7-epi-α-Selinene	11.68	1509	0.06	10.74	1711	0.05
δ-Cadinene	11.75*	1514	1.69	10.69	1706	1.53
trans-Calamenene	11.75*	1514	[1.69]	11.49	1774	0.11
(E)-γ-Bisabolene	11.84	1522	0.09	10.64	1702	0.10
α-Calacorene	11.96	1531	0.03	12.41	1854	0.03
(E)-α-Bisabolene	12.02	1536	0.07	10.96	1729	0.07
Isocaryophyllene epoxide B	12.06	1539	0.07	12.40	1853	0.06
Unknown [m/z 91, 41 (97), 107 (96), 93 (95), 133 (88), 69 (88), 149 (84)... 218 (19)]	12.14	1545	0.01	14.29	2026	0.01
(E)-Nerolidol	12.32	1559	0.13	14.02	2001	0.18
Caryophyllene oxide isomer	12.42†	1567	0.93	12.96	1902	0.13
Caryophyllene oxide	12.44†	1569	[0.93]	13.04	1910	0.71
Unknown [m/z 161, 105 (84), 43 (80), 119 (72), 93 (62), 121 (54)... 204 (38), 222 (2)]	12.58	1580	0.03	14.24	2022	0.03
Humulene epoxide II	12.77	1594	0.07	13.63	1964	0.06
α-Corocalene	13.02	1614	0.07	13.94	1993	0.05
Guaia-6,10(14)-dien-4β-ol	13.08	1620	0.21	15.99	2193	0.21
Caryophylladienol I	13.14	1624	0.03	16.32	2228	0.04
τ-Cadinol	13.23*	1632	0.18	15.19	2113	0.02

τ -Muurolol	13.23*	1632	[0.18]	15.31	2126	0.15
α -Muurolol	13.29	1637	0.67	15.45*	2140	[0.69]
<i>cis</i> -Calamenen-10-ol	13.48	1652	0.02	16.72	2269	0.02
<i>trans</i> -Calamenen-10-ol	13.54	1658	0.01	17.01	2300	0.01
(3 <i>Z</i>)-Caryophylla-3,8(13)-dien-5 β -ol	13.59	1662	0.02	17.08	2307	0.03
α -Bisabolol	13.79	1678	0.01	15.74	2168	0.02
Total identified		98.72%			98.50%	
Total reported		98.92%			98.66%	

*: Two or more compounds are coeluting on this column

[xx]: Duplicate percentage due to coelutions, not taken account in the identified 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