Date : March 24, 2020

Certificate of Analysis – GC Profiling

Sample identification

Internal code : 20C11-PSC02
Customer identification : Grapefruit White - Australia - NAP21376
Type : Essential oil
Source : *Citrus x paradisi* cv. White
Customer : Pacha Soap Co.

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 : Fanny Charlier, B. Sc.
Analysis date : March 23, 2020

Checked and approved by :

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

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PHYSICOCHEMICAL DATA

Physical aspect: Light yellow liquid
Refractive index: 1.4739 ± 0.0003 (20 °C)

ISO 3053:2005 - OIL OF GRAPEFRUIT, OBTAINED BY EXPRESSION

Compound	Min. %	Max. %	Observed %	Complies?
Nootkatone	0.01	0.80	0.15	Yes
β-Caryophyllene	0.2	0.5	0.2	Yes
Neral	0.02	0.04	0.05	No
Decanal	0.1	0.6	0.2	Yes
Nonanal	0.04	0.10	0.02	No
Octanal	0.2	0.8	0.3	Yes
Limonene	92	96	94	Yes
Myrcene	1.5	2.5	1.7	Yes
β-Pinene	0.05	0.20	0.04	No
Sabinene	0.1	0.6	0.4	Yes
α-Pinene	0.2	0.6	0.6	Yes
Refractive index	1.4740	1.4790	1.4739	No

CONCLUSION

The oil does not comply with the ISO standard for grapefruit oil.

This sample features Δ3-carene above 0.03%, which is considered as indicative of potential adulteration with sweet orange terpenes.

Δ3-carene is featured in sweet orange oil (and sweet orange terpenes, from which oxygenated compounds have been stripped) at a typical concentration in the range of ~0.10%[1,2]. Being the cheapest Citrus oil, sweet orange is a common adulterant of lemon and mandarin oils[2,3]. For these oils, Burfield observed that "its virtual absence (<0.01%) might indicate no orange oil or orange terpenes have been added to cheapen the oil" for "lemon oil, mandarin oil, etc."[4], an observation drawn from previous literature(1).

In his chapter on citrus oils adulterations, McHale further notes that "the level of Δ3-carene in sweet orange oil and its terpenes exceeded that in any of the other citrus oils", and additionally that "grapefruit oil contains virtually no Δ3-carene"[3]. This parallels the general trend from a literature review of Dugo et al,[5] covering 30 articles published between 1979 to 2009 from both industrial and laboratory oils. Of these, all but one paper report at most 0.03% of Δ3-carene (often only traces or undetectable amounts), with the remaining study reporting values of 0.04-0.27%, but for oil extricated from peel glands using a syringe and for various specific treatments (therefore not fully comparable to industrial extraction procedures). This sole article therefore stands out as a potential outlier and should not be given an inflated importance in view of the otherwise converging reports. The consensus in literature therefore appears to be that Δ3-carene should generally be found below 0.03% in grapefruit oil.

On this basis, PhytoChemia suggests that oils featuring Δ3-carene above 0.03% should be treated with caution, as it is for lemon and mandarin oils, to diminish risks of procuring material adulterated with sweet orange oil/terpenes.

References

(1) Dugo, B. G.; Lamonica, G.; Cotroneo, A.; Stagno, I.; Verzera, A.; Donato, M. G.; Dugo, P. High Resolution Gas Chromatography for Detection of Adulterations of Citrus Cold-Pressed Essential Oils. *Perfum. Flavorist* **1992**, *17*, 57–74.

(2) Tranchida, P. Q.; Bonaccorsi, I.; Dugo, P.; Mondello, L.; Dugo, G. Analysis of Citrus Essential Oils: State of the Art and Future Perspectives. A Review. *Flavour Fragr. J.* **2012**, *27* (2), 98–123. https://doi.org/10.1002/ffj.2089.

(3) McHale, D. Adulteration of Citrus Oils. In *Citrus: The Genus Citrus*; Dugo, G., Di Giacomo, A., Eds.; Taylor & Francis: London, 2002; pp 496–517. https://doi.org/10.1663/0013-0001(2004)058[0749:BREDFA]2.0.CO;2.

(4) Burfield, T. The Adulteration of Essential Oils - and the Consequences to Aromatherapy & Natural Perfumery Practice, 2003.

(5) Dugo, G.; Cotroneo, A.; Bonaccorsi, I.; Trozzi, A. Composition of the Volatile Fraction of Citrus Peel Oils. In *Citrus oils: Composition, advanced analytical techniques, contaminants, and biological activity*; Dugo, G., Mondello, L., Eds.; CRC Press: Boca Raton, FL, 2011; pp 1–162.

Analysis summary – Consolidated contents

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

Identification	%	Classe
α-Thujene	tr	Monoterpene
α-Pinene	0.55	Monoterpene
Sabinene	0.38	Monoterpene
β-Pinene	0.04	Monoterpene
Myrcene	1.74	Monoterpene
Octanal	0.31	Aliphatic aldehyde
α-Phellandrene	0.02	Monoterpene
Δ3-Carene	0.08	Monoterpene
Limonene	93.70	Monoterpene
1,8-Cineole	0.25	Monoterpenic ether
(*Z*)-β-Ocimene	0.01	Monoterpene
(*E*)-β-Ocimene	0.06	Monoterpene
γ-Terpinene	0.01	Monoterpene
cis-Sabinene hydrate	0.01	Monoterpenic alcohol
Octanol	0.03	Aliphatic alcohol
Terpinolene	0.01	Monoterpene
Linalool	0.07	Monoterpenic alcohol
Nonanal	0.02	Aliphatic aldehyde
trans-para-Mentha-2,8-dien-1-ol	0.03	Monoterpenic alcohol
cis-Limonene oxide	0.04	Monoterpenic ether
cis-para-Mentha-2,8-dien-1-ol	0.02	Monoterpenic alcohol
trans-Limonene oxide	0.02	Monoterpenic ether
Citronellal	0.02	Monoterpenic aldehyde
Terpinen-4-ol	tr	Monoterpenic alcohol
α-Terpineol	0.02	Monoterpenic alcohol
trans-Isopiperitenol	tr	Monoterpenic alcohol
Decanal	0.20	Aliphatic aldehyde
Octyl acetate	0.01	Aliphatic ester
trans-Carveol	0.02	Monoterpenic alcohol
cis-Carveol	0.01	Monoterpenic alcohol
Neral	0.05	Monoterpenic aldehyde
Geranial	0.01	Monoterpenic aldehyde
Neryl acetate	0.04	Monoterpenic ester
α-Copaene	0.06	Sesquiterpene
Geranyl acetate	0.09	Monoterpenic ester
β-Cubebene	0.06	Sesquiterpene
β-Elemene	0.02	Sesquiterpene
Capric acid	0.02	Aliphatic acid
Dodecanal	0.03	Aliphatic aldehyde
β-Caryophyllene	0.23	Sesquiterpene
α-Humulene	0.05	Sesquiterpene
(*E*)-β-Farnesene	0.01	Sesquiterpene
Germacrene D	0.05	Sesquiterpene
Bicyclogermacrene	0.02	Sesquiterpene
α-Muurolene	0.01	Sesquiterpene
Cubebol	0.01	Sesquiterpenic alcohol
δ-Cadinene	0.07	Sesquiterpene

α-Elemol	0.02	Sesquiterpenic alcohol
(*E*)-Nerolidol	0.01	Sesquiterpenic alcohol
Caryophyllene oxide	0.01	Sesquiterpenic ether
β-Sinensal	0.01	Sesquiterpenic aldehyde
Nootkatone	0.15	Sesquiterpenic ketone
Bergapten	0.09	Furanocoumarin
Osthole	0.01	Coumarin
Isoauraptene	0.02	Coumarin
Meranzin	0.05	Coumarin
Auraptenol	tr	Coumarin
Auraptene	0.20	Coumarin
Epoxyaurapten	0.06	Coumarin
Consolidated total	**99.12%**	

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.

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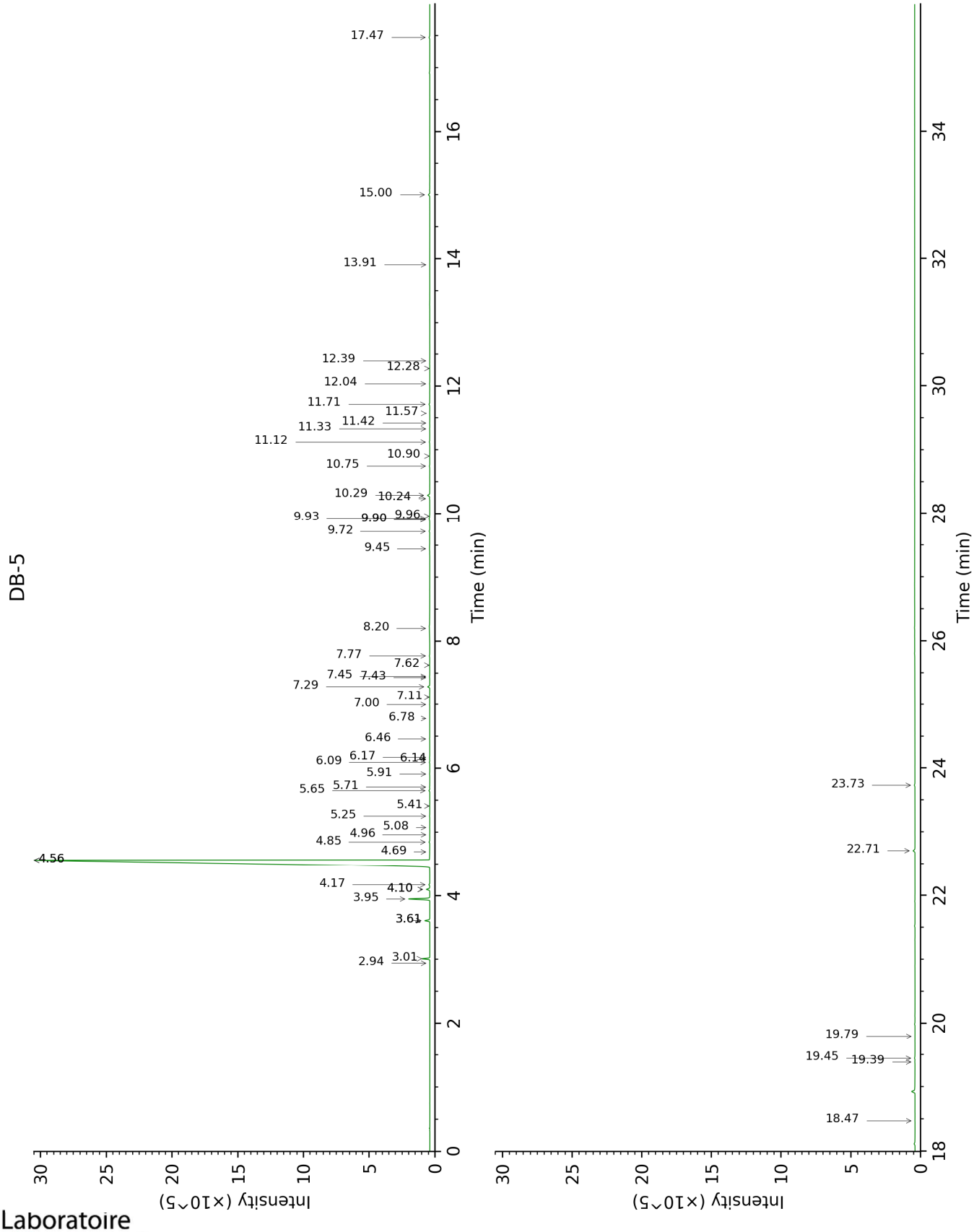
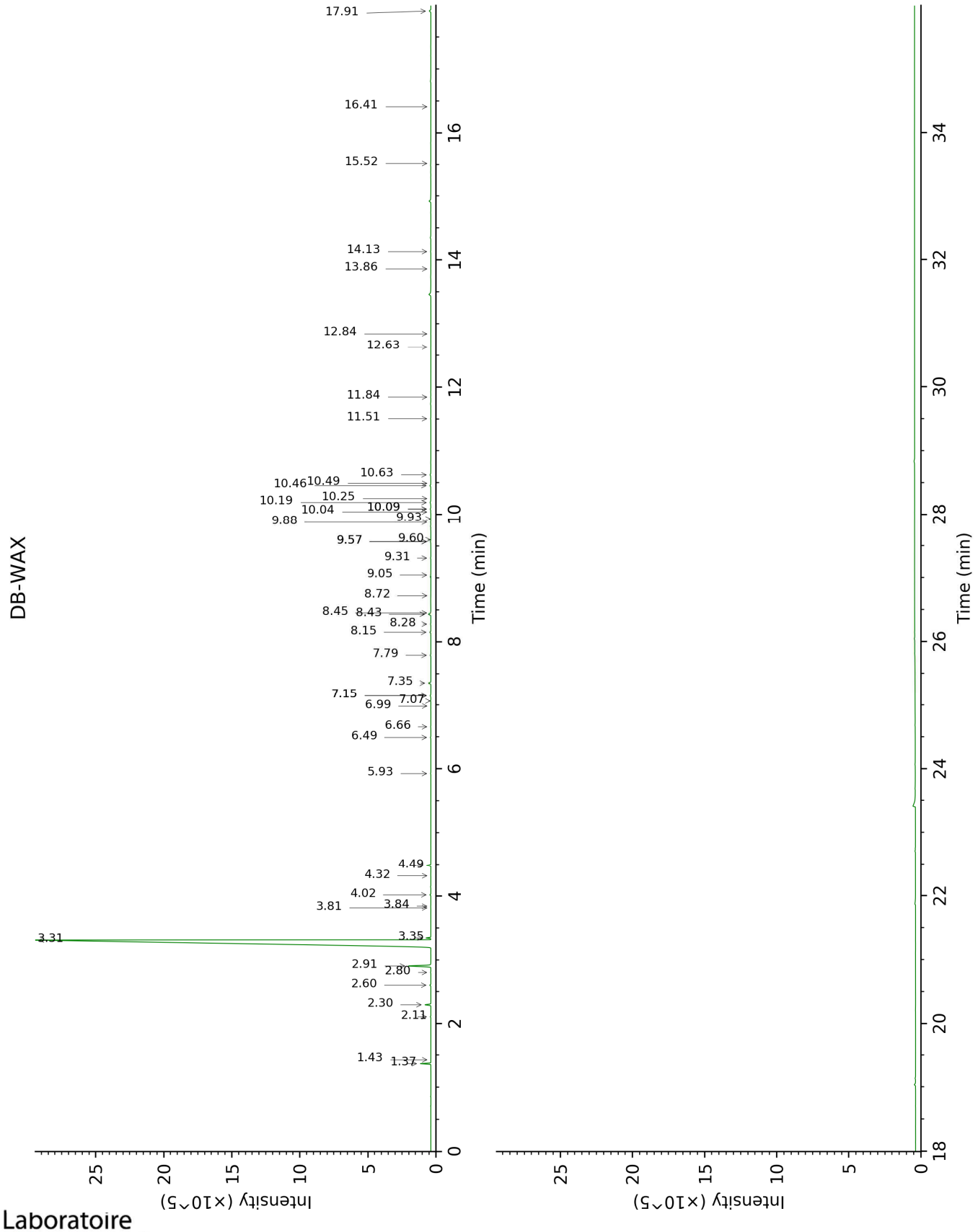

DB-5
17.47
15.00
13.91
12.39
12.28
12.04
11.71
11.57
11.33
11.42
11.12
10.90
10.75
10.29
10.24
9.93
9.90
9.96
9.72
9.45
8.20
7.77
7.62
7.45
7.43
7.29
7.11
7.00
6.78
6.46
6.09
6.17
6.14
5.91
5.65
5.71
5.41
5.25
5.08
4.85
4.96
4.69
4.56
4.17
4.10
3.95
3.61
2.94
3.01
23.73
22.71
19.79
19.45
19.39
18.47
Time (min)
Intensity (×10^5)


DB-WAX
17.91
16.41
15.52
14.13
13.86
12.84
12.63
11.84
11.51
10.63
10.46
10.49
10.19
10.25
10.04
10.09
9.88
9.93
9.57
9.60
9.31
9.05
8.72
8.45
8.43
8.28
8.15
7.79
7.35
7.15
7.07
6.99
6.66
6.49
5.93
4.49
4.32
4.02
3.81
3.84
3.31
3.35
2.91
2.80
2.60
2.30
2.11
1.43
1.37
Time (min)
Intensity (×10^5)

FULL ANALYSIS DATA

Identification	Column DB-5			Column DB-WAX		
	R.T	R.I	%	R.T	R.I	%
α-Thujene	2.94	925	tr	1.43	999	tr
α-Pinene	3.01	930	0.55	1.37	993	0.55
Sabinene	3.61*	969	0.42	2.30	1085	0.38
β-Pinene	3.61*	969	[0.42]	2.11	1067	0.04
Myrcene	3.95	992	1.74	2.91	1134	1.75
Octanal	4.10*	1002	0.32	4.49	1252	0.31
α-Phellandrene	4.10*	1002	[0.32]	2.80	1126	0.02
Δ3-Carene	4.17	1006	0.08	2.60	1111	0.08
Limonene	4.56*	1031	94.29	3.31	1166	93.70
1,8-Cineole	4.56*	1031	[94.29]	3.34	1168	0.25
(*Z*)-β-Ocimene	4.69	1039	0.01	3.84	1206	0.01
(*E*)-β-Ocimene	4.85	1049	0.06	4.02	1218	0.07
γ-Terpinene	4.96	1056	0.01	3.81	1204	tr
cis-Sabinene hydrate	5.08	1064	0.01	6.99	1432	0.01
Octanol	5.25	1075	0.03	8.28	1529	0.03
Terpinolene	5.41	1085	0.01	4.32	1240	0.02
Linalool	5.65	1100	0.07	8.15	1519	0.08
Nonanal	5.71	1104	0.02	5.93	1355	0.03
trans-para-Mentha-2,8-dien-1-ol	5.91	1117	0.03	9.05	1588	0.02
cis-Limonene oxide	6.09	1129	0.04	6.49	1396	0.04
cis-para-Mentha-2,8-dien-1-ol	6.14	1132	0.02	9.57*	1630	0.06
trans-Limonene oxide	6.17	1134	0.02	6.66	1408	0.02
Citronellal	6.46	1153	0.02	7.07	1438	0.02
Terpinen-4-ol	6.78	1174	tr	8.72	1563	0.01
α-Terpineol	7.00	1188	0.02	9.93	1659	0.02
trans-Isopiperitenol	7.11	1196	tr	10.49	1705	0.01
Decanal	7.29	1207	0.20	7.35	1459	0.19
Octyl acetate	7.43	1217	0.01	7.15*	1444	0.07
trans-Carveol	7.45	1218	0.02	11.50	1790	0.03
cis-Carveol	7.62	1231	0.01	11.84	1820	0.02
Neral	7.77	1241	0.05	9.57*	1630	[0.06]
Geranial	8.20	1271	0.01	10.25	1685	0.01
Neryl acetate	9.45	1361	0.04	10.19	1680	0.11
α-Copaene	9.72	1372	0.06	7.15*	1444	[0.07]
Geranyl acetate	9.90*†	1385	0.11	10.63	1716	0.09
β-Cubebene	9.90*†	1385	[0.11]	7.79	1491	0.06
β-Elemene	9.93†	1387	[0.11]	8.45	1542	0.02
Capric acid	9.96	1389	0.02	16.41	2256	0.02
Dodecanal	10.24	1409	0.03	10.09*	1672	0.06
β-Caryophyllene	10.29	1413	0.23	8.43	1541	0.21
α-Humulene	10.75	1447	0.05	9.31	1609	0.04

(*E*)-β-Farnesene	10.90	1458	0.01	9.60	1632	0.01
Germacrene D	11.12	1475	0.05	9.88	1655	0.04
Bicyclogermacrene	11.33	1490	0.02	10.09*	1672	[0.06]
α-Muurolene	11.42	1497	0.01	10.04	1668	0.02
Cubebol	11.57	1508	0.01	12.63	1890	0.01
δ-Cadinene	11.72	1520	0.07	10.46	1702	0.08
α-Elemol	12.04	1545	0.02	14.13	2029	0.03
(*E*)-Nerolidol	12.28	1564	0.01	13.86	2003	0.01
Caryophyllene oxide	12.40	1573	0.01	12.84	1909	0.01
β-Sinensal	13.91	1696	0.01	15.52	2165	0.01
Nootkatone	15.00	1791	0.15	17.91	2417	0.15
Bergapten	17.47	2022	0.09			
Osthole	18.47	2122	0.01			
Isoauraptene	19.39	2218	0.02			
Meranzin	19.44	2225	0.05			
Auraptenol	19.79	2262	tr			
Auraptene	22.71	2601	0.20			
Epoxyaurapten	23.73	2731	0.06			
Total identified		**99.40%**			**98.79%**	
Total reported		**99.40%**			**98.79%**	

*: 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