

Date : 2024-02-27

CERTIFICATE OF ANALYSIS - GC PROFILING

SAMPLE IDENTIFICATION

**Internal code :** 24B09-NPA06

**Customer Identification :** Lime - Italy - NPS00134 - Lot# NP0299

**Type :** Essential Oil

**Source :** *Citrus aurantifolia* ct. Distilled

**Customer :** Nature Packaged

Checked and approved by:



Sylvain Mercier, M. Sc., Chimiste 2014-005

*Notes: 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.*

This report is an update from the first version issued on 2024-02-22 to format it for online publication.



## GAS CHROMATOGRAPHIC ANALYSIS

**Method :** PC-MAT-014 - Analysis of the composition of an essential oil or other volatile liquide by FAST GC-FID



**Results :** See analysis summary (next page)

**Analyst :** Sylvain Mercier, M. Sc., Chimiste 2014-005

**Date :** 2024-02-19

## PHYSICOCHEMICAL DATA

**Refractive index :**  $1.4742 \pm 0.0003$  (20 °C)

**Method :** PC-MAT-016 - Measure of the refractive index of a liquid.

**Analyst :** Cindy Caron B. Sc.

**Date :** 2024-02-12

## CONCLUSION

The delta-3-carene content appears to be higher than expected based on literature for distilled lime essential oils,<sup>1</sup> and this constituent can be indicative of addition of sweet orange terpenes.

Overall, the oil shows several discrepancies with ISO 3519:2006 standard for distilled Mexican type lime oil, such as high limonene content and low bisabolene and alpha-farnesene contents.

The laboratory cannot confirm that this is a pure distilled lime (*Citrus aurantifolia*) fruit essential oil.

## REFERENCE

- (1) *Citrus Oils: Composition, Advanced Analytical Techniques, Contaminants, and Biological Activity*; Dugo, G., Mondello, L., Eds.; CRC Press: Boca Raton, FL, 2011.

## ANALYSIS SUMMARY - CONSOLIDATED CONTENTS

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

Identification	%	Class
Isoprene	tr	Alkene
4,5-Dihydrotoluene	tr	Alkene
Toluene	tr	Simple phenolic
1-Methylcyclohexa-1,3-diene	0.02	Alkene
3-Methylenecyclohexadiene	tr	Alkene
Octane	tr	Alkane
Nonane	0.01	Alkane
Bornylene	tr	Monoterpene
Tricyclene	0.01	Monoterpene
$\alpha$ -Thujene	0.02	Monoterpene
$\alpha$ -Pinene	0.80	Monoterpene
$\beta$ -Fenchene?	0.01	Monoterpene
Camphene	0.21	Monoterpene
$\alpha$ -Fenchene	0.08	Monoterpene
1,4-Dimethyl-4-vinylcyclohexene?	0.01	Monoterpene
Geranic oxide	0.01	Monoterpenic ether
$\beta$ -Pinene	1.07	Monoterpene
Sabinene	0.15	Monoterpene
3-Methyl-3-cyclohexenone	0.01	Aliphatic ketone
6-Methyl-5-hepten-2-one	0.01	Aliphatic ketone
Dehydro-1,8-cineole	0.01	Monoterpenic ether
trans-Dehydroxylinalool oxide	0.01	Monoterpenic ether
Myrcene	1.54	Monoterpene
Octanal	0.09	Aliphatic aldehyde
Pseudolimonene	0.03	Monoterpene
$\alpha$ -Phellandrene	0.17	Monoterpene
$\Delta^3$ -Carene	0.07	Monoterpene
1,4-Cineole	[2.00]	Monoterpenic ether
$\alpha$ -Terpinene	[2.00]	Monoterpene
para-Cymene	1.56	Monoterpene
Limonene	72.88	Monoterpene
1,8-Cineole	0.67	Monoterpenic ether
$\beta$ -Phellandrene	0.26	Monoterpene
(Z?) $\rightarrow$ Citroxide	0.03	Monoterpenic ether
(Z)- $\beta$ -Ocimene	0.09	Monoterpene
(E?) $\rightarrow$ Citroxide	0.10	Monoterpenic ether
(E)- $\beta$ -Ocimene	0.21	Monoterpene
$\gamma$ -Terpinene	6.12	Monoterpene
Unknown	0.02	Oxygenated monoterpene
Octanol	0.01	Aliphatic alcohol

Terpinolene isomer	0.08	Monoterpene
para-Cymenene	0.13	Monoterpene
Terpinolene	4.54	Monoterpene
Linalool	0.11	Monoterpenic alcohol
Nonanal	0.03	Aliphatic aldehyde
para-Menta-1,3,8-triene	0.02	Monoterpene
endo-Fenchol	0.14	Monoterpenic alcohol
trans-para-Mentha-2,8-dien-1-ol	0.02	Monoterpenic alcohol
Myrcenol	0.01	Monoterpenic alcohol
4-Hydroxy-4-methylcyclohex-2-enone	0.01	Aliphatic alcohol
cis-Limonene oxide	0.02	Monoterpenic ether
allo-Ocimene	0.01	Monoterpene
trans-Limonene oxide	0.02	Monoterpenic ether
1-Terpineol	0.12	Monoterpenic alcohol
Epoxyterpinolene	0.06	Monoterpenic ether
cis-β-Terpineol	0.14	Monoterpenic alcohol
Unknown	0.02	Unknown
Isoborneol	0.01	Monoterpenic alcohol
Citronellal	0.01	Monoterpenic aldehyde
(Z)-Ocimenol	0.01	Monoterpenic alcohol
Borneol	0.08	Monoterpenic alcohol
(E)-Ocimenol	0.03	Monoterpenic alcohol
Terpinen-4-ol	0.14	Monoterpenic alcohol
para-Cymen-8-ol	0.04	Monoterpenic alcohol
α-Terpineol	1.72	Monoterpenic alcohol
γ-Terpineol	0.21	Monoterpenic alcohol
trans-Piperitol	0.01	Monoterpenic alcohol
Decanal	0.02	Aliphatic aldehyde
trans-Carveol	0.02	Monoterpenic alcohol
2,3-Epoxyneral?	0.01	Monoterpenic aldehyde
cis-Carveol	0.01	Monoterpenic alcohol
Unknown	0.02	Oxygenated monoterpene
Neral	0.03	Monoterpenic aldehyde
Geraniol	0.01	Monoterpenic alcohol
Geranial	0.06	Monoterpenic aldehyde
Unknown	0.03	Oxygenated monoterpene
cis-Ascaridole glycol	0.01	Monoterpenic alcohol
Unknown	0.01	Unknown
δ-Elemene	0.03	Sesquiterpene
Neryl acetate	0.05	Monoterpenic ester
α-Copaene	0.01	Sesquiterpene
Geranyl acetate	0.04	Monoterpenic ester
β-Elemene	0.03	Sesquiterpene
Isocaryophyllene	0.01	Sesquiterpene
Dodecanal	0.01	Aliphatic aldehyde

<i>cis</i> - $\alpha$ -Bergamotene	0.01	Sesquiterpene
$\beta$ -Caryophyllene	0.17	Sesquiterpene
$\alpha$ -Santalene	0.02	Sesquiterpene
$\gamma$ -Elemene	0.01	Sesquiterpene
<i>trans</i> - $\alpha$ -Bergamotene	0.29	Sesquiterpene
$\alpha$ -Humulene	0.04	Sesquiterpene
$\beta$ -Santalene	0.03	Sesquiterpene
( <i>E</i> )- $\beta$ -Farnesene	0.03	Sesquiterpene
Selina-4,11-diene	0.05	Sesquiterpene
Germacrene D	0.02	Sesquiterpene
Unknown	0.03	Sesquiterpene
$\beta$ -Selinene	0.02	Sesquiterpene
<i>trans</i> - $\beta$ -Bergamotene	0.01	Sesquiterpene
Unknown	0.01	Sesquiterpene
Valencene	0.02	Sesquiterpene
$\delta$ -Selinene	0.06	Sesquiterpene
$\alpha$ -Selinene	0.04	Sesquiterpene
( <i>Z</i> )- $\alpha$ -Bisabolene	0.06	Sesquiterpene
$\beta$ -Bisabolene	0.52	Sesquiterpene
( <i>3E,6E</i> )- $\alpha$ -Farnesene	0.20	Sesquiterpene
$\delta$ -Cadinene	0.02	Sesquiterpene
Selina-4(15),7(11)-diene	0.02	Sesquiterpene
Selina-4,7(11)-diene?	0.07	Sesquiterpene
Selina-3,7(11)-diene	0.02	Sesquiterpene
( <i>E</i> )- $\alpha$ -Bisabolene	0.02	Sesquiterpene
Germacrene B	0.02	Sesquiterpene
Caryophyllenyl alcohol	0.02	Sesquiterpenic alcohol
Caryophyllene oxide	0.01	Sesquiterpenic ether
10-epi- $\gamma$ -Eudesmol	0.02	Sesquiterpenic alcohol
Clovan-2 $\beta$ -ol	0.01	Sesquiterpenic alcohol
$\gamma$ -Eudesmol	0.01	Sesquiterpenic alcohol
Unknown	0.01	Oxygenated sesquiterpene
Unknown	0.02	Oxygenated sesquiterpene
epi- $\alpha$ -Bisabolol	0.01	Sesquiterpenic alcohol
$\alpha$ -Bisabolol	0.01	Sesquiterpenic alcohol
<b>Consolidated total</b>	<b>98.40</b>	

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

Note: no correction factor was applied

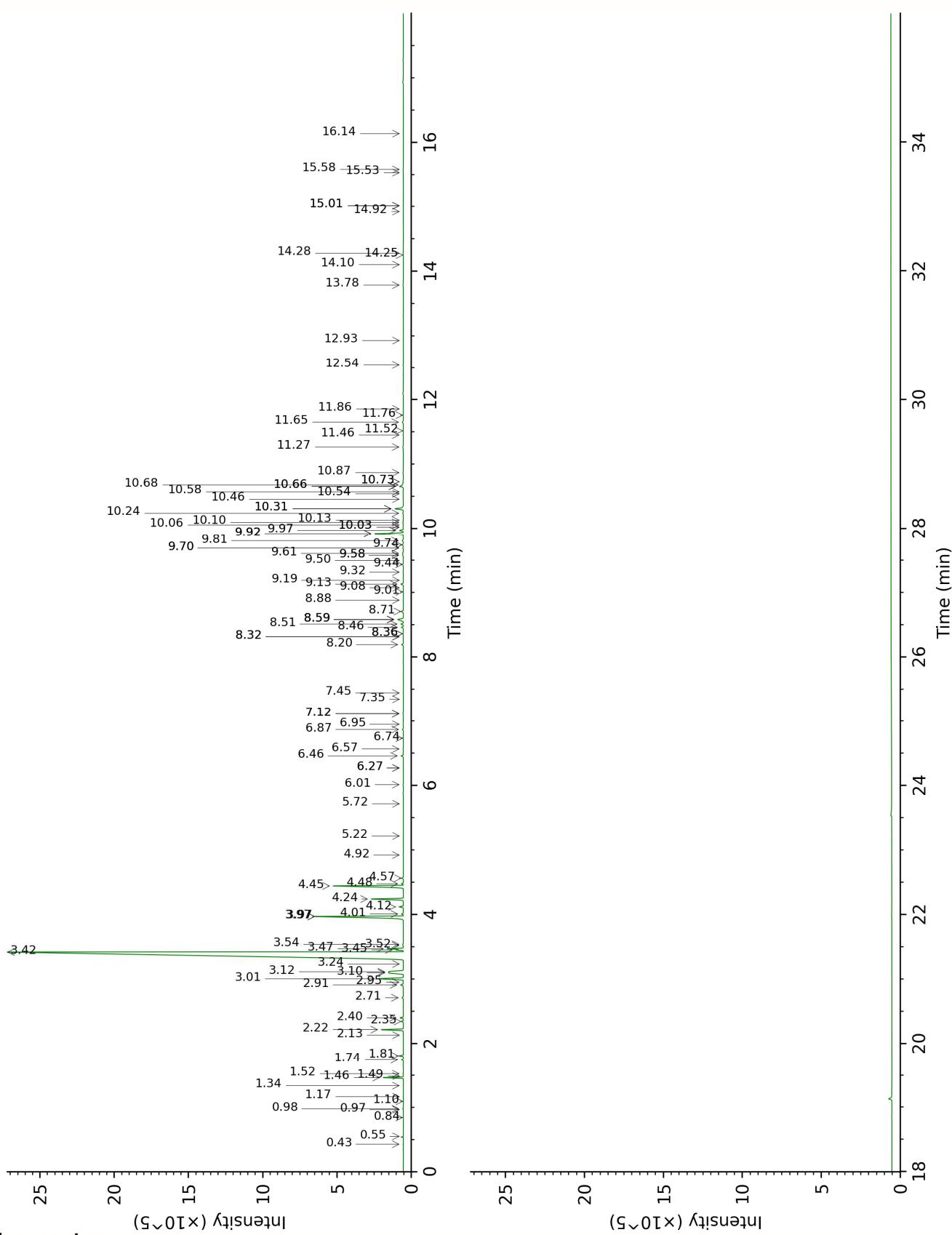
**About "consolidated" data:** The table above presents the breakdown of the sample volatile constituents after applying an algorithm to collapse data acquired from the multi-columns system of PhytoChemia into a single set of consolidated contents. In case of discrepancies between columns, the algorithm 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.

**Bracketed value ([xx]):** A bracketed percent value indicate that two or more compound percentage could not be solved due to coelution.

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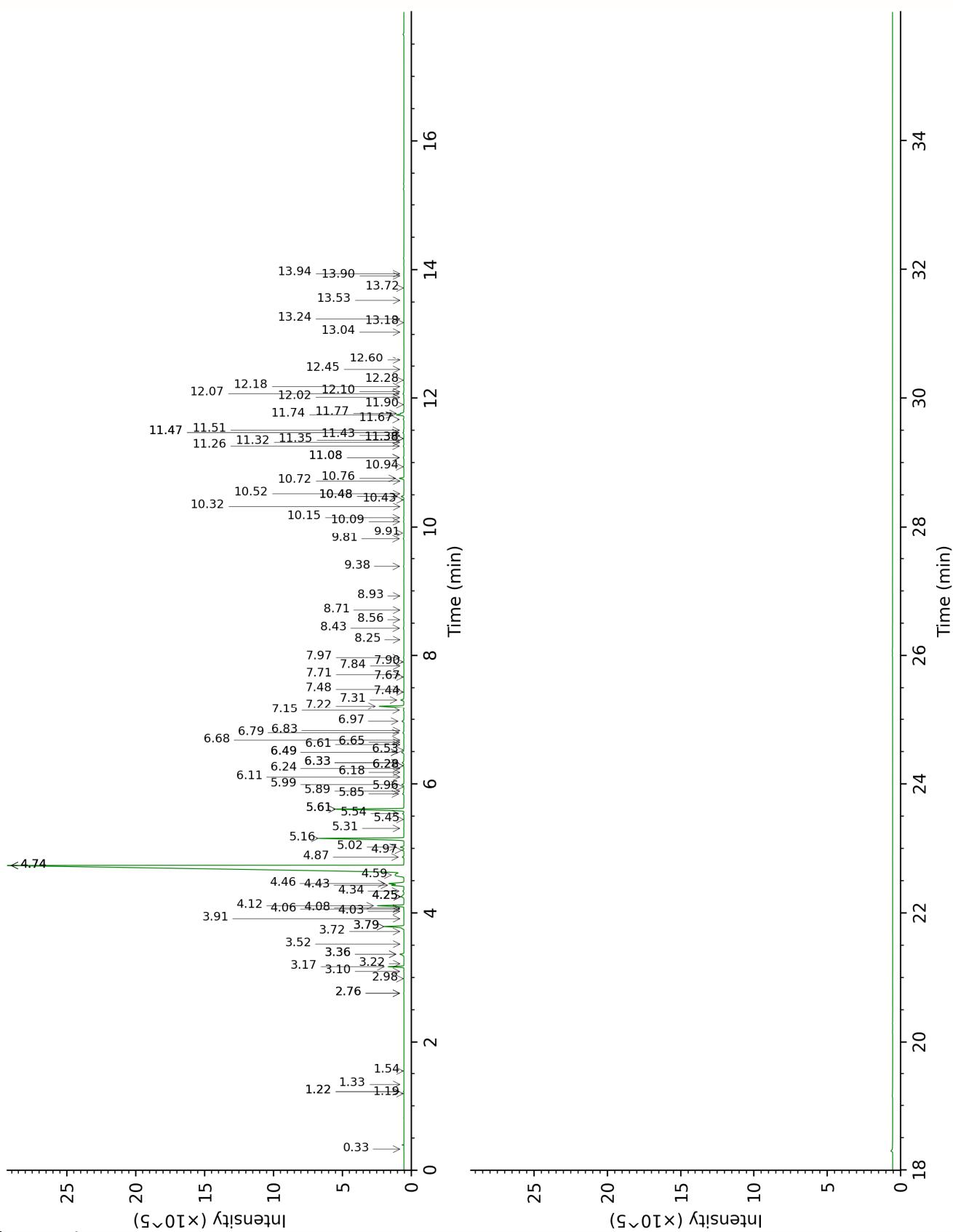
DB-WAX



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DB-5



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**PhytoChemia**

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FULL ANALYSIS DATA

Isoprene	Column DB-WAX			Column DB-5		
	0.43	677.9	0.01	0.33	500.7	tr
4,5-Dihydrotoluene	0.97	917.7	tr	1.19	754.9	tr
Toluene	1.49	996.0	tr	1.22*	759.0	[0.02]
1-Methylcyclohexa-1,3-diene	0.98	919.9	0.02	1.22*	759.0	[0.02]
3-Methylenecyclohexadiene	1.10	937.1	tr	1.33	774.4	tr
Octane	0.55	785.4	tr	1.54	803.2	tr
Nonane	0.84	893.3	0.01	2.76*	903.9	[0.02]
Bornylene	1.17	948.0	tr	2.76*	903.9	[0.02]
Tricyclene	1.34	973.6	0.01	2.98	919.1	0.01
$\alpha$ -Thujene	1.52	1001.5	0.01	3.10	926.5	0.02
$\alpha$ -Pinene	1.46	992.4	0.80	3.17	931.3	0.80
$\beta$ -Fenchene?				3.22	934.3	0.01
Camphene	1.81	1029.1	0.21	3.36*	944.0	[0.30]
$\alpha$ -Fenchene	1.74	1022.8	0.08	3.36*	944.0	[0.30]
1,4-Dimethyl-4-vinylcyclohexene?	2.13	1059.4	0.01	3.52	954.3	0.01
Geranic oxide	2.34	1079.5	0.10	3.72	967.2	0.01
$\beta$ -Pinene	2.22	1067.5	1.07	3.79*	972.3	[1.32]
Sabinene	2.40	1084.8	0.15	3.79*	972.3	[1.32]
3-Methyl-3-cyclohexenone	6.27*	1371.8	[0.02]	3.91	980.3	0.01
6-Methyl-5-hepten-2-one	5.22	1297.4	tr	4.03	987.8	0.01
Dehydro-1,8-cineole	3.24	1151.8	0.01	4.06	990.0	0.01
trans-Dehydroxylinalool oxide	3.52	1173.7	0.02	4.08	991.2	0.01
Myrcene	3.01	1134.3	1.56	4.12	993.5	1.54
Octanal	4.57	1250.3	0.09	4.26*	1002.7	[0.30]
Pseudolimonene	2.95	1130.3	0.03	4.26*	1002.7	[0.30]
$\alpha$ -Phellandrene	2.91	1127.1	0.17	4.26*	1002.7	[0.30]
$\Delta$ 3-Carene	2.71	1112.0	0.08	4.34	1008.3	0.07
1,4-Cineole	3.12*†	1142.6	[0.94]	4.43*†	1013.9	[0.86]
$\alpha$ -Terpinene	3.10*†	1141.5	[1.06]	4.46*†	1015.6	[1.14]
para-Cymene	4.24	1227.1	1.70	4.59	1023.8	1.56
Limonene	3.42	1165.9	72.88	4.74*	1033.1	[74.46]
1,8-Cineole	3.47	1169.8	0.67	4.74*	1033.1	[74.46]
$\beta$ -Phellandrene	3.45	1168.5	0.26	4.74*	1033.1	[74.46]
(Z?)-Citroxide	3.54	1175.2	0.03	4.74*	1033.1	[74.46]
(Z)- $\beta$ -Ocimene	3.97*	1207.7	[6.21]	4.87	1041.2	0.09
(E?)-Citroxide	4.01	1210.4	0.09	4.98	1047.9	0.10
(E)- $\beta$ -Ocimene	4.12	1218.4	0.21	5.02	1050.8	0.21
$\gamma$ -Terpinene	3.97*	1207.7	[6.21]	5.16	1059.4	6.12

Unknown PIMA I [m/z 79, 93 (60), 43 (40), 94 (35), 137 (33), 77 (26), 91 (20), 152 (18)]	4.92	1276.1	0.02	5.31	1069.0	0.02
Octanol	8.32*	1524.3	[0.02]	5.45	1077.7	0.01
Terpinolene isomer	4.48	1244.1	0.08	5.54	1083.3	0.08
<i>para</i> -Cymenene	6.46	1385.3	0.13	5.61*	1087.7	[4.69]
Terpinolene	4.44	1241.7	4.54	5.61*	1087.7	[4.69]
Linalool	8.20	1514.7	0.10	5.85	1102.4	0.11
Nonanal	6.02	1353.2	0.03	5.89	1105.1	0.03
<i>para</i> -Menta-1,3,8-triene	6.27*	1371.8	[0.02]	5.96	1109.3	0.02
endo-Fenchol	8.51	1539.4	0.14	5.99	1111.5	0.14
<i>trans-para</i> -Menta-2,8-dien-1-ol	9.08	1583.2	0.01	6.11	1119.1	0.02
Myrcenol	9.01	1578.2	0.01	6.18	1123.7	0.01
4-Hydroxy-4-methylcyclohex-2-enone	14.25	2032.2	0.01	6.24	1127.5	0.01
<i>cis</i> -Limonene oxide	6.57	1393.0	0.02	6.28*	1130.2	[0.02]
allo-Ocimene	5.72	1331.8	0.01	6.28*	1130.2	[0.02]
<i>trans</i> -Limonene oxide	6.74	1405.2	0.02	6.33*	1133.2	[0.13]
1-Terpineol	8.46	1535.5	0.12	6.33*	1133.2	[0.13]
Epoxyterpinolene	6.87	1415.3	0.06	6.49*	1143.4	[0.19]
<i>cis</i> - $\beta$ -Terpineol	9.13	1587.5	0.14	6.49*	1143.4	[0.19]
Unknown MEAL II [m/z 109, 124 (45), 119 (41), 43 (35), 91 (28), 95 (25)...]	6.95	1421.4	0.02	6.53	1145.7	0.02
Isoborneol	9.50	1616.6	0.01	6.61	1151.0	0.01
Citronellal	7.12*	1433.6	[0.03]	6.65	1153.4	0.01
(Z)-Ocimenol	9.58*	1623.3	[0.05]	6.68	1155.4	0.01
Borneol	9.92*	1651.1	[1.77]	6.80	1162.8	0.08
(E)-Ocimenol	9.81	1641.8	0.08	6.83	1165.0	0.03
Terpinen-4-ol	8.71	1554.6	0.13	6.97	1174.2	0.14
<i>para</i> -Cymen-8-ol	11.65	1795.9	0.08	7.15	1185.2	0.04
$\alpha$ -Terpineol	9.92*	1651.1	[1.77]	7.22	1189.6	1.72
$\gamma$ -Terpineol	9.98	1655.3	0.21	7.31	1195.8	0.21
<i>trans</i> -Piperitol	10.54	1701.7	0.02	7.44	1203.8	0.01
Decanal	7.45	1458.2	0.02	7.48	1206.4	0.02
<i>trans</i> -Carveol	11.52	1784.6	0.01	7.67	1219.3	0.02
2,3-Epoxyneral?				7.71	1221.6	0.01
<i>cis</i> -Carveol	11.86	1813.8	0.02	7.84	1230.7	0.01
Unknown CIAU II [m/z 137, 152 (28), 43 (25), 91 (24), 109 (23), 119 (19)]	11.46	1779.0	0.02	7.90	1234.9	0.02
Neral	9.61	1625.6	0.06	7.97	1239.3	0.03
Geraniol	11.76	1805.2	0.01	8.25	1257.9	0.01

Geranial	10.24	1677.0	0.04	8.43	1269.8	0.06
Unknown CIAU V [m/z 95, 67 (45), 41 (42), 110 (42), 43 (41), 59 (36)]	12.54	1874.4	0.02	8.56	1278.6	0.03
<i>cis</i> -Ascaridole glycol	14.92	2096.8	0.02	8.71	1288.6	0.01
Unknown CICA VI [m/z 112, 97 (93), 83 (60), 43 (46), 41 (20), 69 (19)...]	14.10	2017.9	0.01	8.93	1303.2	0.01
δ-Elemene	7.12*	1433.6	[0.03]	9.38	1335.4	0.03
Neryl acetate	10.31*	1682.7	[0.60]	9.81	1365.7	0.05
α-Copaene	7.35	1451.0	0.01	9.91	1372.2	0.01
Geranyl acetate	10.68	1713.2	0.07	10.09	1385.0	0.04
β-Elemene	8.58*	1544.9	[0.48]	10.15	1389.3	0.03
Isocaryophyllene	8.32*	1524.3	[0.02]	10.32	1401.4	0.01
Dodecanal	10.13	1668.3	0.02	10.43	1409.1	0.01
<i>cis</i> -α-Bergamotene	8.36*	1527.8	[0.03]	10.48*	1413.0	[0.19]
β-Caryophyllene	8.58*	1544.9	[0.48]	10.48*	1413.0	[0.19]
α-Santalene	8.36*	1527.8	[0.03]	10.52	1416.1	0.02
γ-Elemene	9.19	1592.1	0.01	10.72	1430.7	0.01
<i>trans</i> -α-Bergamotene	8.58*	1544.9	[0.48]	10.76	1433.9	0.29
α-Humulene	9.44	1611.7	0.05	10.94	1447.2	0.04
β-Santalene	9.32	1602.0	0.03	11.08*	1457.7	[0.05]
(E)-β-Farnesene	9.70*	1632.6	[0.04]	11.08*	1457.7	[0.05]
Selina-4,11-diene	9.58*	1623.3	[0.05]	11.26	1471.0	0.05
Germacrene D	9.92*	1651.1	[1.77]	11.32	1475.3	0.02
Unknown BOSE VII [m/z 91, 93 (92), 105 (71), 77 (69), 79 (68), 133 (63)... 204 (32)]	10.06	1662.2	0.02	11.35	1477.7	0.03
β-Selinene	10.02*	1659.4	[0.02]	11.38*	1479.7	[0.03]
<i>trans</i> -β-Bergamotene	9.70*	1632.6	[0.04]	11.38*	1479.7	[0.03]
Unknown CILI III [m/z 41, 69 (90), 79 (78), 93 (72), 91 (70)...204]	8.88	1568.2	0.02	11.42	1483.3	0.01
Valencene	10.02*	1659.4	[0.02]	11.47*	1486.6	[0.08]
δ-Selinene	9.74	1636.5	0.06	11.47*	1486.6	[0.08]
α-Selinene	10.10	1665.6	0.03	11.51	1489.3	0.04
(Z)-α-Bisabolene	10.46	1694.8	0.02	11.67	1501.7	0.06
β-Bisabolene	10.31*	1682.7	[0.60]	11.74	1507.1	0.52
(3E,6E)-α-Farnesene	10.66*	1711.4	[0.26]	11.76	1508.7	0.20
δ-Cadinene	10.58	1704.3	0.02	11.90	1519.5	0.02
Selina-4(15),7(11)-diene	10.73*	1717.5	[0.03]	12.02	1528.4	0.02
Selina-4,7(11)-diene?	10.66*	1711.4	[0.26]	12.07	1532.7	0.07
Selina-3,7(11)-diene	10.73*	1717.5	[0.03]	12.10	1535.2	0.02
(E)-α-Bisabolene	10.87	1729.4	0.03	12.18	1541.5	0.02

Germacrene B	11.27	1763.2	0.05	12.28	1549.2	0.02
Caryophyllenyl alcohol	13.78	1988.1	0.03	12.45	1562.3	0.02
Caryophyllene oxide	12.93	1908.9	0.01	12.60	1573.9	0.01
10-epi-γ-Eudesmol	14.28	2034.8	0.02	13.04	1608.5	0.02
Clovan-2β-ol				13.18	1620.7	0.01
γ-Eudesmol	15.01*	2105.6	[0.02]	13.24	1625.3	0.01
Unknown CILI I [m/z 94, 43 (89), 41 (67), 122 (46), 69 (41)...222]	15.01*	2105.6	[0.02]	13.53	1649.0	0.01
Unknown CILI II [m/z 69, 95 (100), 41 (89), 109 (68), 67 (61)...222]	16.14	2219.3	0.01	13.72	1664.7	0.02
epi-α-Bisabolol	15.58	2163.0	0.02	13.90	1680.2	0.01
α-Bisabolol	15.53	2158.0	0.01	13.94	1682.9	0.01
Total reported		98.63%			99.15%	

\*: Two or more compounds are coeluting on this column

[xx]: Duplicate percentage due to coelutions, only the first one is taken into account in the consolidated total

t: 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