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Comparison of Fast and Conventional GC Analysis for Citrus Essential Oils

LUIGI MONDELLO,*,† ALESSANDRO CASILLI,† PETER QUINTO TRANCHIDA,† LUCIA CICERO,† PAOLA DUGO,‡ AND GIOVANNI DUGO†

Dipartimento Farmaco-chimico, University of Messina, viale Annunziata, 98168 Messina, Italy and Dipartimento di Chimica organica e Biologica, Salita Sperone, University of Messina, 98166 Messina, Italy

This investigation concerns the application of fast GC in the analysis of essential oils. These are complex matrixes that usually undergo GC separation with conventional methods involving long columns, slow programmed temperature rates, and consequently, a high cost in terms of time. Fast GC techniques are based on the use of narrow bore capillary columns that allow the achievement of high-speed separations on complex samples while maintaining excellent resolution. This work saw the application of two methods on five different citrus essential oils and the comparison of all the results obtained.

KEYWORDS: GC analysis; narrow bore columns; citrus essential oils

INTRODUCTION

Conventional GC has an important role in the analysis of complex natural matrixes. Although this approach allows effective separations, these are often achieved at a high cost in time, and therefore there has always been a strong interest in the research of faster analytical methods. Various experimental routes proposed to minimize the analysis time in GC have been recently reviewed (*1*). Among them are the use of capillary columns with a reduced internal diameter and film thickness (*2*−*7*), multicapillary columns with more than 900 capillaries (i.d. = 40 μm) (*8*, *9*), or micro particle packed columns (*10*, *11*). The use of packed and multicapillary columns bring improvements in terms of velocity, but they also cause a loss in resolution. Therefore, their use is confined to simple samples.

The interest for fast GC has been enhanced by the introduction of narrow bore capillary columns and the development of the instrumental technology required by these types of columns. Narrow bore capillary columns have reduced inner diameters (0.1 mm or less), a thin stationary phase film, a high phase ratio (∼ 250), and more theoretical plates/m than conventional columns. Therefore, shorter columns can be used without sacrificing too much efficiency. Short, narrow bore columns will have a higher optimum linear carrier gas velocity, which is beneficial to attain faster analysis times. Theoretical and practical aspects of narrow-bore fast GC approach are well established (*1*, *12*). Fast GC requires instrumentation provided with high split ratio injection systems because of low sample column capacities, increased inlet pressures, rapid oven heating rates, and fast electronics for detection and data collection. The application of higher inlet pressures produces a gain in analysis time that can reach a factor of 10 or more. Hydrogen is used as carrier gas, because the optimum linear carrier gas velocity is higher than helium or nitrogen, and also, as shown by the ascending slope of the Van- Deemter HETP-u curve, the flatness of this curve for hydrogen allows opportunities to apply linear gas velocities higher than the optimum without significant loss of resolution.

This work compares fast and conventional GC in the analysis of citrus essential oils, very complex mixtures whose components can be divided into two fractions: a 90−95% volatile fraction (monoterpenes and sesquiterpenes hydrocarbons and their oxygenated derivatives along with aliphatic aldehydes, alcohols, and esters) and a 1−10% non volatile one (hydrocarbons, fatty acids, sterols, carotenoids, waxes, coumarines, psoralens, and flavonoids) (*13*). The determination of the volatile part has usually been achieved through conventional GC methods characterized by long capillary columns and slow temperature programs due to the presence of several compounds at very different concentrations. In this work, the same results are obtained with a fast GC technique in a greatly reduced time. The rapid and correct quantification of the volatile fraction of citrus essential oils is of great interest, especially for laboratories with a high sample through put and/or where quick results are required for the determination of quality and authenticity (*13*, *14*).

MATERIALS AND METHODS

Sample. Five cold-pressed citrus essential oils, bergamot, bitter orange, sweet orange, mandarin, and lemon, obtained from a local producer have been analyzed. The oils were diluted 1:10 (V/V) in hexane.

* Corresponding author. Tel.: +39-090-6766536. Fax: +39-090-6766532. E-mail: lmondello@pharma.unime.it.
† Dipartimento Farmaco-chimico.
‡ Dipartimento di Chimica organica e Biologica, Salita Sperone.

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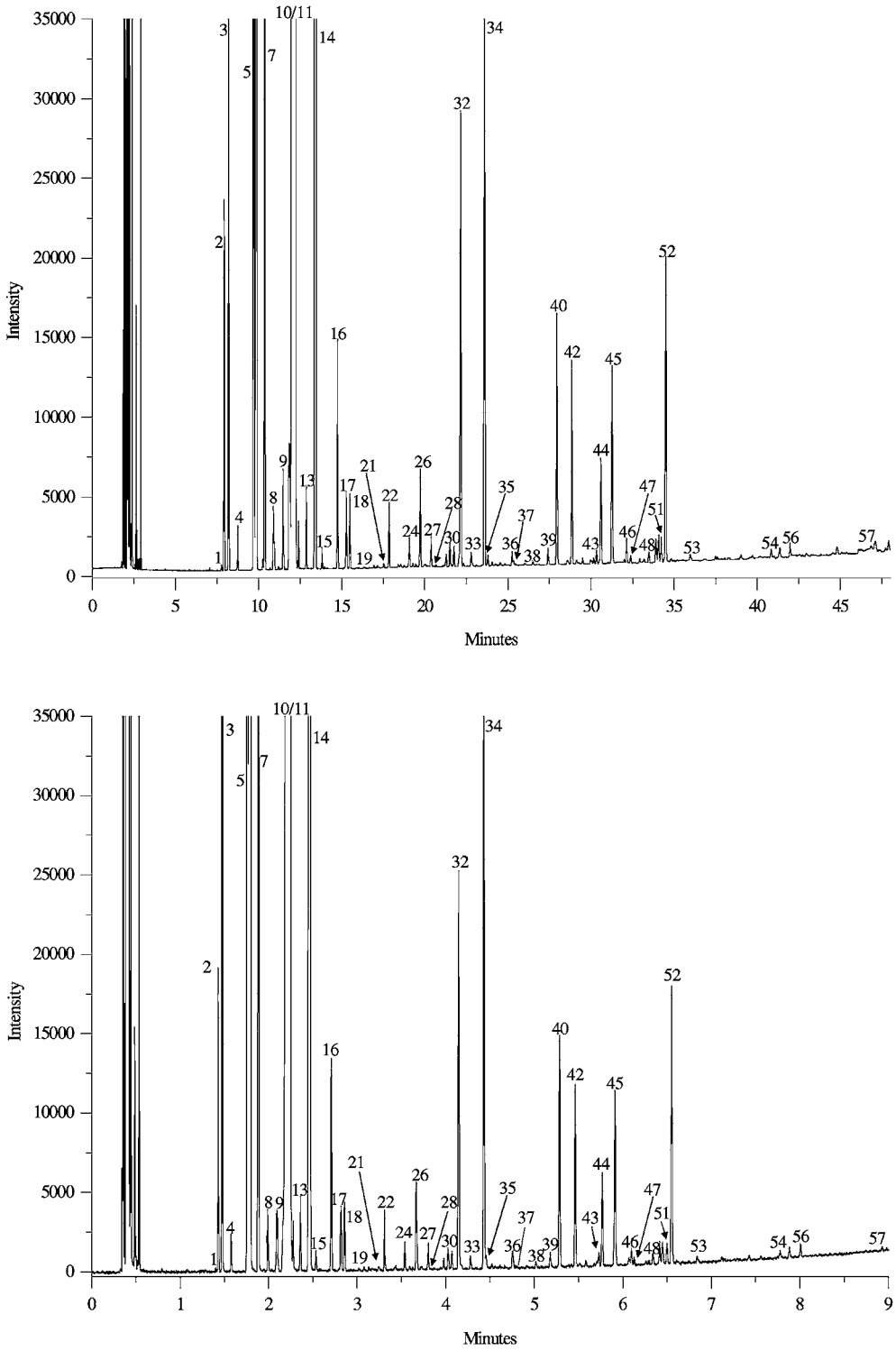

Figure 1. (A) Conventional GC chromatogram and (B) Fast GC chromatogram of a lemon essential oil. For peak identification see Table 1.

Instrumentation. *Conventional GC.* GC/FID analyses were performed on a Shimadzu system composed of GC-17A,ver.3 equipped with split/splitless injector, an autosampler AOC-20i, and an FID. Data acquisition was performed by the Class-VP 4.3 Software (Shimadzu, Milan, Italy).

Columns, RTX-5 MS 30-m × 0.25-mm i.d. × 0.25-μm film thickness. (Restek, Bellefonte, PA). Temperature program, 50−250 °C at 3 °C/min. Pressure program, 102 kPa at pressure constant. Carrier gas, He. u, 30 cm/sec. Injection volume, 1.0 μL. Split ratio, 1:100. Detector, FID H_2, 60 kPa. Air, 50kPa. Makeup, 80 kPa (He). Sampling frequency, 5 Hz.

Conventional GC/MS. GC/MS analyses were performed on a Shimadzu GC/MS instrument QP5050A equipped with Adams library (*15*) and a Flavor and Fragrance homemade library (16).

Columns, MDN-5S 30-m × 0.25-mm i.d. × 0.25-μm film thickness. (Supelco, Milan, Italy). Temperature program, 50−250 °C (10 min) at 3 °C/min. Pressure program, 35 kPa at pressure constant. Carrier Gas, He; u, 32 cm/sec. Injection volume, 1.0 μL. Split ratio, 1:26 (250 °C). Interface temperature, 230 °C. Ionization energy, 1.50 KV. Acquisition mass range, 40−400, Solvent cut, 4 min.

Fast GC. GC/FID analyses were performed on a Shimadzu system composed of GC-2010 equipped with split/splitless injector, an

Table 1. Peak Identification and Retention Time Values (min) Obtained for the Components of Lemon Essential Oil Analyzed by Conventional and Fast GC

	component	method of identification	conventional		fast	
			t_R	%CV	t_R	%CV
1	tricyclene	a,b	7.728	0.254	1.402	0.000
2	α-thujene	a,b,c	7.886	0.489	1.430	0.040
3	α-pinene	a,b,c	8.150	0.501	1.477	0.039
4	camphene	a,b,c	8.703	0.466	1.578	0.000
5	sabinene	a,b,c	9.661	0.441	1.760	0.057
6	β-pinene	a,b,c	9.835	0.420	1.793	0.064
7	myrcene	a,b,c	10.321	0.413	1.885	0.031
8	octanal + α-phellandrene	a,b,c	10.844	0.390	1.987	0.029
9	α-terpinene	a,b,c	11.437	0.352	2.092	0.055
10	p-cymene	a,b,c	11.804	0.362		
11	limonene	a,b,c	12.163	0.333	2.248	0.026
12	(Z)-β-ocimene	a,b	12.367	0.326	2.276	0.044
13	(E)-β-ocimene	a,b	12.827	0.314	2.358	0.024
14	γ-terpinene	a,b,c	13.389	0.288	2.468	0.041
15	cis-sabinene hydrate	a,b	13.772	0.282	2.532	0.023
16	terpinolene	a,b,c	14.705	0.250	2.707	0.043
17	linalool	a,b,c	15.241	0.239	2.816	0.054
18	nonanal	a,b,c	15.450	0.225	2.857	0.040
19	cis-limonene oxide	a,b,c	16.904	0.200	3.018	0.149
20	trans-limonene oxide	a,b,c	17.120	0.168	3.078	0.068
21	camphor	a,b,c	17.501	0.159	3.240	0.078
22	citronellal	a,b,c	17.809	0.167	3.308	0.046
23	borneol	a,b,c	18.729	0.159	3.433	0.034
24	terpinen-4-ol	a,b,c	19.036	0.143	3.536	0.043
25	p-cymen-8-ol	a,b	19.253	0.137	3.592	0.074
26	α-terpineol	a,b,c	19.691	0.129	3.667	0.027
27	decanal	a,b,c	20.351	0.125	3.801	0.040
28	octyl acetate	a,b,c	20.648	0.107	3.863	0.045
29	citronellol	a,b,c	21.254	0.105	3.977	0.025
30	nerol	a,b,c	21.494	0.104	4.025	0.025
31	carbonyl compound	a,b	21.733	0.106	4.070	0.028
32	neral	a,b,c	22.129	0.094	4.145	0.037
33	geraniol	a,b,c	22.767	0.089	4.278	0.023
34	geranial	a,b,c	23.566	0.079	4.430	0.034
35	perilla aldehyde	a,b,c	23.774	0.069	4.521	0.013
36	undecanal	a,b,c	25.237	0.056	4.756	0.021
37	nonyl acetate	a,b,c	25.320	0.069	4.793	0.032
38	methyl geranate	a,b	26.043	0.067	5.019	0.012
39	citronellyl acetate	a,b,c	27.402	0.047	5.180	0.011
40	neryl acetate	a,b,c	27.940	0.042	5.286	0.019
41	linalyl isobutyrate	a,b	28.593	0.020	5.388	0.037
42	geranyl acetate	a,b,c	28.831	0.037	5.462	0.021
43	sesquithujene	a,b	30.326	0.032	5.731	0.010
44	β-caryophyllene	a,b,c	30.586	0.032	5.767	0.017
45	trans-α-bergamotene	a,b	31.249	0.023	5.912	0.017
46	α-humulene	a,b,c	32.130	0.022	6.068	0.019
47	β-santalene	a,b	32.377	0.028	6.098	0.009
48	germacrene D	a,b	33.473	0.010	6.342	0.009
49	valencene	a,b,c	33.900	0.017	6.417	0.009
50	sesquiterpene	a	34.066	0.017	6.449	0.016
51	bicyclogermacrene	a,b	34.217	0.010	6.500	0.009
52	β-bisabolene	a,b	34.494	0.007	6.552	0.009
53	γ-bisabolene	a,b	35.970	0.007	6.840	0.015
54	2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	t	40.833	0.000	7.776	0.013
55	campherenol	t	40.833	0.000	7.883	0.007
56	α-bisabolol	a,b,c	41.012	0.020	8.009	0.012
57	nootkatone	a,b,c	46.848	0.009	8.930	0.000

[a] GC-MS spectrum. [b] Linear Retention Index. [c] Standard component; t = tentative.

autosampler AOC-20i, and an FID. Data acquisition was performed by the GC Solution Software (Shimadzu, Milan, Italy).

Columns, RTX-5 MS 10-m × 0.1-mm i.d. × 0.1-μm film thickness. (Restek, Bellefonte, PA). Temperature program 50−250 °C at 14 °C/min. Pressure program, 184.2 kPa at linear velocity constant. Carrier gas, H_2. u, 57 cm/sec. Injection volume, 0.4 μL. Split ratio, 1:250 (300 °C). Detector, FID (300 °C). H_2, 50 mL/min. Air, 400 mL/min. Makeup, 50 mL/min (N_2). Base period, 4 ms (250 Hz). Filter time constant, 20 ms (50 Hz). Sampling rate, 20 ms (50 Hz).

Table 2. Comparison of Chromatographic Parameters Retention Time (t_R), Peak Width (w_b), Selectivity Factor (α) and Resolution (R_s)obtained for Conventional and Fast GC Analysis

	component	conventional				fast			
		t_R	w_b	a	R_s	t_R	w_b	a	R_s
2	α-thujene	7.93	0.07			1.43	0.02		
3	α-pinene	8.20	0.07	1.04	3.76	1.48	0.02	1.04	2.35
17	linalool	15.28	0.09			2.82	0.02		
18	nonanal	15.49	0.09	1.01	2.29	2.86	0.02	1.02	1.95
51	bicyclogermacrene	34.22	0.09			6.50	0.02		
52	β-bisabolene	34.50	0.10	1.01	3.03	6.52	0.02	1.01	2.21

RESULTS

Conventional separation of the citrus essential oils was obtained using a standard column (RTX-5 MS 30-m × 0.25-mm × 0.25-μm).

Fast GC analysis was performed with an RTX-5 MS 10-m × 0.1-mm × 0.1-μm narrow bore column. Three repetitions on each sample for both methods were carried out, and the qualitative and quantitative results obtained were compared.

Figure 1 shows the chromatograms of conventional and fast GC analysis of a lemon essential oil. **Table 1** reports the qualitative results obtained with both columns, showing the average retention times resulting from the three repetitions, with their CV%. Peak identification was carried out by GC/MS analysis, with linear retention indices (*16*, *17*) and comparison with standard components where available. As can be seen from **Figure 1** and **Table 1**, 57 components were separated with both methods. The analysis with a conventional GC column takes about 46.8 min, while the fast analysis allows the separation of the same components in about 8.9 min with a very similar resolution. The fast GC technique performs the same separation with a speed gain of a factor of 5. CV% values resulted lower than 1% for both methods, but were significantly lower for the fast GC analysis than for the conventional one. These differences were larger for early eluting components and become small for late eluting components. This interesting result may indicate that fast GC using new instrumentation is more reliable than conventional GC.

Table 2 reports data regarding t_R, peak widths, selectivity factors, and resolution calculated for peaks regarding α-thujene and α-pinene, linalool and nonanal, and bicyclogermacrene and β-bisabolene in three different zones, both in the conventional and fast GC analyses.

As observed, t_R values and peak widths in the fast GC analysis are significantly lower than conventional ones, while selectivity factor values (α) are practically unchanged. Resolution values are slightly lower for fast GC peaks but are still high enough to allow baseline separation for all peaks present in the chromatogram. The negligable loss in resolution is greatly compensated by the reduction in analysis time.

The reproducibility of quantitative data in passing from one technique to the other is shown in **Table 3**. The information reported regards relative average areas and CV% for a lemon oil separation. These data show good agreement between fast and conventional results. Furthermore, accuracy was good, as CV% values are always lower than 1% for the three repetitions in both methods.

The fast method developed for lemon oil analysis has proven to be robust and reliable. Under the same experimental condi-

Table 3. Relative Area % and CV% for Conventional and Fast GC Analyses of Lemon Oil

	component	conventional		fast	
		rel area %	CV%	rel area %	CV%
1	tricyclene	0.01	0.42	0.01	0.54
2	α-thujene	0.42	0.15	0.43	0.25
3	α–pinene	1.89	0.10	1.89	0.15
4	camphene	0.06	0.39	0.06	0.86
5	sabinene	1.97	0.21	1.90	0.55
6	β-pinene	12.56	0.08	12.40	0.09
7	myrcene	1.47	0.14	1.51	0.18
8	octanal + α-phellandrene	0.12	0.28	0.12	0.80
9	α-terpinene	0.16	0.13	0.17	0.35
10	p-cymene	0.35	0.06	*	*
11	limonene	66.05	0.06	66.57	0.04
12	(Z)-β-ocimene	0.08	0.11	0.09	0.96
13	(E)-β-ocimene	0.12	0.10	0.13	0.45
14	γ-terpinene	9.16	0.08	8.94	0.59
15	cis-sabinene hydrate	0.03	0.59	0.03	0.87
16	terpinolene	0.35	0.09	0.36	0.64
17	linalool	0.12	0.03	0.13	0.57
18	nonanal	0.12	0.52	0.12	0.61
19	cis-limonene oxide	< 0.01	0.77	0.01	0.69
20	trans-limonene oxide	< 0.01	0.36	0.01	0.98
21	camphor	0.01	0.37	0.01	0.89
22	citronellal	0.10	0.85	0.11	0.82
23	borneol	0.01	0.52	0.01	0.63
24	terpinen-4-ol	0.05	0.20	0.06	0.89
25	p-cymen-8-ol	0.01	0.67	0.01	0.63
26	α-terpineol	0.17	0.22	0.17	0.45
27	decanal	0.05	0.71	0.05	0.89
28	octyl acetate	0.00	0.67	0.01	0.93
29	citronellol	0.02	0.33	0.04	0.65
30	nerol	0.04	0.83	0.03	0.57
31	carbonyl compound	0.04	0.52	0.04	0.23
32	neral	0.77	0.28	0.77	0.27
33	geraniol	0.03	0.50	0.03	0.79
34	geranial	1.29	0.34	1.30	0.13
35	perilla aldehyde	0.02	0.71	0.02	0.93
36	undecanal	0.03	0.34	0.04	0.71
37	nonyl acetate	0.01	0.63	0.01	0.58
38	methyl geranate	0.01	0.51	0.02	0.70
39	citronellyl acetate	0.03	0.86	0.04	0.94
40	neryl acetate	0.44	0.23	0.46	0.94
41	linalyl isobutyrate	0.01	1.00	0.01	0.75
42	geranyl acetate	0.35	0.38	0.36	0.98
43	sesquithujene	0.02	0.52	0.03	0.80
44	β-caryophyllene	0.21	0.86	0.20	0.60
45	trans-α-bergamotene	0.38	0.43	0.40	0.90
46	α-humulene	0.05	0.29	0.04	0.65
47	β-santelene	0.01	0.85	0.02	0.77
48	germacrene D	0.02	0.45	0.03	0.61
49	valencene	0.05	0.58	0.05	0.85
50	sesquiterpene	0.06	0.49	0.06	0.45
51	bicyclogermacrene	0.04	0.80	0.05	0.77
52	β-bisabolene	0.57	0.23	0.58	0.18
53	γ-bisabolene	0.02	0.27	0.02	0.81
54	2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	0.02	0.76	0.02	0.72
55	campherenol	0.02	0.74	0.03	0.75
56	α-bisabolol	0.03	0.97	0.03	0.87
57	nootkatone	0.01	0.57	0.01	0.96

tions used for lemon oil, other citrus essential oils were analyzed. **Table 4** presents the quantitative results obtained for conventional and fast analyses of bergamot, bitter orange, mandarin, and sweet orange oils, respectively.

For all citrus oils analyzed, the fast method allowed the separation of almost the same components as the conventional analysis. Quantitative data also showed good reproducibility.

The results presented here were to demonstrate the effectiveness of fast GC applications, through the use of narrow bore

Table 4. Relative Area % for Conventional and Fast GC Analyses of Bergamot, Sweet Orange, Bitter Orange, and Mandarin Oils

component	bergamot		sweet orange		bitter orange		mandarin	
	conv	fast	conv	fast	conv	fast	conv	fast
tricyclene	tr[a]	tr						
α-thujene	0.33	0.34	0.03	0.03	0.01	0.01	0.74	0.72
α-pinene	1.27	1.30	0.64	0.58	0.62	0.58	2.21	2.01
camphene	0.04	0.05	0.01	0.01	0.01	0.01	0.02	0.02
sabinene	1.16	1.17	0.52	0.47	0.29	0.27	0.24	0.24
β-pinene	7.04	7.06	0.91	0.81	1.01	0.93	1.47	1.41
6-methyl-5-hepten-2-one	0.01	tr						
myrcene	0.98	1.16	2.03	1.80	1.90	1.75	1.79	1.72
octanal + α-phellandrene	0.08	0.09	0.18	0.16	0.20	0.19	0.13	0.14
δ–3-carene	tr	tr	0.16	0.14	tr	tr	tr	tr
α-terpinene	0.15	0.16	0.02	0.02	tr	0.01	0.32	0.32
p-cymene + limonene	42.66	42.37	93.33	93.94	93.26	93.58	73.84	75.25
(Z)-β-ocimene	0.02	0.07	0.01	tr	0.01	tr	tr	tr
(E)-β-ocimene	0.21	0.28	0.62	0.60	0.62	0.52	0.02	0.02
γ-terpinene	7.84	7.91	0.59	0.52	0.08	0.08	17.06	16.01
cis-sabinene hydrate	0.04	0.04	0.04	0.04	tr	0.62	0.01	0.01
octanol	tr	0.02					0.01	
terpinolene	0.32	0.33	0.05	0.05	0.01	0.01	0.73	0.70
linalool	7.52	7.67	0.30	0.27	0.32	0.31	0.09	0.09
nonanal	0.03	0.04	0.04	0.03	0.03	0.03	0.03	0.02
cis-limonene oxide	tr	tr	0.01	0.01	0.01			
trans-limonene oxide	tr	0.01	0.02	0.02	tr	0.02		
E-myroxide							0.02	0.02
camphor	0.01							
citronellal	0.01	0.02	0.04	0.04	tr	tr	0.02	0.03
terpinen-4-ol	0.03	0.04	tr	0.01	tr	0.01	0.02	0.02
α-terpineol	0.09	0.10	0.06	0.05	0.03	0.04	0.08	0.09
decanal	0.06	0.07	0.17	0.16	0.13	0.13	0.08	0.08
octyl acetate	0.12	0.13	tr	tr	0.04	0.05		
nerol	0.05	0.05	0.02	0.02			0.01	0.02
neral	0.24	0.26	0.06	0.06	0.03	0.05	tr	tr
geraniol			0.01	0.02	0.01	0.02		
linalyl acetate	27.32	26.16			1.04	0.96		
geranial	0.36	0.38	0.13	0.11	0.05	0.07	0.04	0.06
perilla aldehyde			tr	0.02	0.02	0.01		
bornyl acetate	0.05	0.06	0.01	0.01				
undecanal	0.01	0.03	0.01	0.01	0.01	0.01	0.01	0.02
thymol							0.04	0.04
nonyl acetate	0.02	0.02			0.01	0.01		
δ-elemene					0.03	0.04		
α-terpinyl acetate	0.17	0.18			tr	0.01	0.03	0.04
citronellyl acetate	0.03	0.04	0.01	0.01	0.01	0.01	tr	0.01
neryl acetate	0.39	0.45	0.04	0.04	0.03	0.03	0.01	0.01
α-copaene acetate			0.02	0.02				
geranyl acetate	0.36	0.44	0.03	0.03	0.12	0.12	tr	tr
β-cubebene+β-elemene			0.03	0.03				
dodecanal	0.01		0.03	0.03	0.02	0.02		
decyl acetate	0.03	0.04	0.01	0.01				
N-methyl-methylantranilate						0.34	0.33	
sesquithujene	0.02	0.03						
β-caryophyllene	0.33	0.34	0.03	0.03	0.06	0.06	0.07	0.07
β-copaene			0.02	0.02				
trans-α-bergamotene	0.29	0.33	0.03	0.03	0.02	0.02		
cis-β-farnesene	0.03	0.03	0.02	0.01	0.02	0.01		
α-humulene			tr	tr			0.01	0.01
germacrene D	0.05	0.06	0.02	0.01	0.12	0.11		
sesquiterpene	0.02	0.02						
valencene								
α-selinene							0.03	0.04
α-farnesene + sesquiterpene	0.03		0.17	0.16			0.15	0.15
bicyclogermacrene	0.03	0.04			0.01	0.01		
β-bisabolene	0.43	0.46			0.01	0.01		
β-sesquiphellandrene	0.01	0.01						
δ-cadinene			0.02	0.02				
e-γ-bisabolol	0.01	0.02						
e-α-nerolidol	0.02	0.03			0.09	0.09		
trans-sesquisabinene hydrate	0.01	0.01						
tetradecanal			0.01	0.01	tr	0.01		
2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	0.01	0.02	0.01	0.01	tr	0.01		
campherenol	0.02	0.03						
α-bisabolol	0.03	0.03						
β-sinensal			0.02	0.02				
trans, cis-farnesol							tr	0.01
α-sinensal			0.01	0.02			0.29	0.29
nootkatone	0.05	0.06	0.02	0.02	0.01	0.01	0.01	

[a] tr = < 0.01.

capillary columns, in the separation of very complex matrixes. This technique did not affect analytical quality, and it proved its reliability for quick and correct identification. There is still much to be investigated, and therefore, there is room for development and application in all fields.

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