

## **The effect of time and temperature on shelf life of essential oil composition of *Teucrium polium* L.**

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### **Abstract**

*Teucrium polium* has been used to treat gastrointestinal disorders, inflammation, diabetes and rheumatism. This plant has beneficial therapeutic properties. In this research the changes in the composition of essential oils from the aerial parts of *Teucrium polium* were determined at different temperatures and times. The essential oil of air-dried samples was obtained by hydro distillation and analyzed by gas chromatography/mass spectrometry (GC/MS). Changes in essential oils compositions were detected during storage for 3 months in refrigerator, freezer, and at room temperature. The results indicated that at room temperature, the proportions of the compounds with lower boiling temperatures such as Cis-Verbenol (2.11-1.78%) and Germacrene D (1.56-0.94%) were decreased significantly. Furthermore, the essential oil composition showed the least alterations and *Teucrium polium* L. kept its primary quality when stored at low temperatures, particularly in refrigerator and freezer (Except:  $\alpha$ -Pinene,  $\beta$ -Pinene and Limonene).

**Keywords:** Felty germander; Storage conditions; Labiate; Limonene; Industrial compounds;  $\beta$ -Pinene.

### **Experimental**

#### **Collection of plants and identification**

Samples of *Teucrium polium* were prepared in May 2019 from plants growing in Behbahan city in the southeast of Khuzestan province of Iran. The plant materials were identified by Ahmad Hatami (faculty member at the herbarium of Fars Research Center for Agriculture and Natural Resources, Shiraz, Iran). A voucher specimen was sent to the herbarium of the Research Center for Agriculture and Natural Resources, Shiraz, Iran. (The plant specimen was specified as No: 14428). The plants were shade-dried at room temperature for 14 days (20-25°C). The essential oil content of all dried specimens (100 g) was isolated through hydro distillation for 3 h via a Clevenger-type apparatus, based on the technique proposed by the British Pharmacopoeia . Over anhydrous sodium sulfate, the distilled essential oils were dried and places in firmly closed dark vials for additional examinations.

#### **The circumstances for storing volatile oils**

To study the effects of various storage circumstances on compositions of the distilled essential oils, the prepared specimens were exposed to various storage temperatures including room temperature (25° C), freezer (-20° C), and refrigerator (4° C), for 3 consecutive months until examination. The essential oils were analyzed in all storage treatments on a monthly basis. Furthermore, the fresh extracted essential oil was examined directly followed by extraction to define the exact effects of storage circumstances on compositions of essential oils over the test course. The extracted EOs were yellow and had a sharp distinct odor (Rowshan et al. 2013 ; Najarian 2014).

GC analysis was carried out utilizing an Agilent gas chromatograph series 7890-A with anFID (flame ionization

detector). The temperatures of the detector were maintained at 250° C and 280° C. Nitrogen was utilized at a flow rate of 1 ml/min as the carrier gas. Oven temperature program was 60-210° C at the rate of 4° C /min and then was programmed to 240° C at the rate of 20° C /min. Eventually, it was kept at this temperature for 8.5 min. The split ratio was 1:50. GC-MS and the analysis were performed utilizing Agilent gas chromatograph armed with fused silica capillary HP-5MS column (0.25 mm × 30 mi.d.; film thickness 0.25 m) coupled with a 5975-C mass spectrometer. Helium was utilized as carrier gas with an ionizing voltage of 70 eV. Interface and ion source temperatures were 280° C and 230° C, respectively. The mass range was within 45-550 amu. Oven temperature program was like the one described for the GC (Rowshan et al. 2013 ; Najafian 2014, 2016).

### **Identifying the compounds**

The constituents of essential oils were determined by calculating their retention indices under temperature-programmed circumstances for n-alkanes (C8–C25) and the essential oil on an HP-5 column under similar chromatographic circumstances. The singular compounds were identified by comparing their mass spectra with reference standards or with the internal reference mass spectra library and their retention indices were compared with reference standards or with the literature (Adams, 2001; Najafian 2014, 2016). The percentages of the relative area obtained by FID were used for quantifying purpose, without using correction factors (Tables S1-S3).

### **Statistical analysis**

The tests were performed in triplicate. The findings were expressed as the average values plus standard deviations. Data analysis was conducted using ANOVA in SPSS software (v. 25.0). Duncan's Multiple Range test was employed to measure the important differences ( $P < 0.05$ ) between treatments.

### **References**

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N	Compound%	R <sup>a</sup>	After. D	After 1 month	After 2 month	After 3 month
1	$\alpha$ -Thujene	927	0.09	0.15	-	-
2	$\alpha$ -Pinene	932	6.97±0.008c	7.58±0.020b	6.56±0.005d	7.65±0.005a
3	Camphene	950	0.12	0.18	0.12	-
4	Thuja-2,4(10)-diene	955	0.12	0.13	-	-
5	Sabinene	974	0.33	0.35	0.32	0.36
6	$\beta$ -Pinene	978	12.97±0.008c	13.33±0.005b	12.01±0.012d	13.59±0.015a
7	6-methyl-5-Hepten-	987	0.18	0.24	0.21	-
8	Myrcene	991	2.19±0.005b	2.20±0.005b	2.14±0.005c	2.48±0.012a
9	n-Decane	997	0.12	0.10	-	-
10	$\alpha$ -Terpinene	1017	0.11	0.13	-	-
11	p-Cymene	1025	0.57±0.005d	0.68±0.003c	0.74±0.005b	0.93±0.011a
12	Limonene	1029	3.45±0.003c	3.53±0.005b	3.30±0.005d	3.65±0.005a
13	1,8-Cineole	1031	0.19	0.22	0.24	
14	(E)- $\beta$ -Ocimene	1046	0.35	0.31	0.23	
15	$\gamma$ -Terpinene	1058	0.43	0.34	0.23	
16	Terpinolene	1089	0.23	0.10	-	
17	Linalool	1098	0.29	0.31	0.34	
18	n-Nonanal	1104	0.20	0.25	0.18	
19	1-Octen-3-yl	1111	0.29	0.30	0.31	
20	$\alpha$ -Campholenal	1126	0.33	0.31	0.31	
21	trans-Pinocarveol	1138	0.16	0.26	0.32	
22	cis-Verbenol	1143	2.11±0.005a	1.70±0.008d	1.88±0.005b	1.78±0.005c
23	trans-Verbenol	1145	1.14±0.010c	1.14±0.010c	1.26±0.005b	1.30±0.005a
24	Pinocarvone	1163	0.25	0.75	0.15	-
25	p-Mentha-1,5-dien-	1167	0.82±0.008a	0.37±0.005d	0.74±0.005b	0.59±0.003c
26	Terpinen-4-ol	1177	0.35	0.34	0.37	
27	$\alpha$ -Terpineol	1190	0.38	0.24	0.24	
28	Myrtenal	1198	1.84	0.38	0.43	
29	Verbenone	1207	0.31±0.005d	1.89±0.003c	1.97±0.005b	2.00±0.003a
30	trans-Carveol	1222	0.23	0.31	0.24	
31	Nerol	1231	0.21	0.28	0.33	
32	Cumin aldehyde	1242	0.26	0.18	0.41	
33	Carvone	1244	3.85±0.005a	3.58±0.003c	3.81±0.005b	3.86±0.003a
34	Bornyl acetate	1287	1.10	1.06	1.05	-
35	Thymol	1290	1.23±0.008c	1.22±0.003c	1.32±0.005a	1.28±0.005b
36	Carvacrol	1299	0.12	0.15	0.17	
37	$\delta$ -Elemene	1337	0.20	0.23	0.14	
38	Piperitenone oxide	1368	0.41	0.37	0.41	
39	$\alpha$ -Copaene	1377	0.15	0.02	0.14	
40	$\beta$ -Bourbonene	1386	0.11	0.08	0.18	
41	$\beta$ -Elemene	1392	0.19	0.28	0.22	
42	n-Tetradecane	1399	0.08	0.06	-	
43	$\alpha$ -Gurjunene	1408	0.10	0.09	-	
44	(E)-Caryophyllene	1423	4.71±0.005a	4.39±0.005c	4.44±0.005b	4.30±0.005d
45	$\gamma$ -Elemene	1434	0.95	0.84	0.83	
46	$\alpha$ -Humulene	1452	0.08	0.07		
47	(E)- $\beta$ -Farnesene	1456	2.23±0.005a	2.12±0.005b	2.22±0.005a	2.24±0.005a
48	dehydro-	1471	2.86±0.005c	2.85±0.005c	3.04±0.010b	3.30±0.005a
49	Germacrene D	1484	1.56±0.005a	1.27±0.005b	1.12±0.011c	0.94±0.005d
50	Bicyclogermacrene	1499	1.00	0.57	0.20	
51	$\alpha$ -Muurolene	1502	0.42	0.34	0.36	
52	$\beta$ -Bisabolene	1508	0.99±0.005b	0.93±0.005c	1.00±0.006a	0.97±0.003b
53	$\gamma$ -Cadinene	1515	0.56	0.50	0.57	-
54	$\delta$ -Cadinene	1523	1.49±0.017a	1.36±0.005c	1.40±0.005b	1.34±0.005c
55	Unknown	1536	0.59	0.49	0.55	1.22
56	Elemol	1552	1.49±0.005a	1.42±0.005b	1.41±0.005b	1.48±0.005a
57	Germacrene B	1558	0.75	0.70	0.59	
58	(E)-Nerolidol	1565	0.37±0.005c	0.73±0.008a	0.37±0.005c	0.46±0.005b
59	Spathulenol	1578	0.44±0.005d	1.18±0.005c	1.54±0.005b	1.72±0.005a
60	Caryophyllene	1581	1.29±0.005c	2.08±0.005d	2.40±0.008b	2.58±0.005a
61	1,10-di-epi-	1621	0.64	0.65	0.71	0.68
62	Valerenol	1658	21.44±0.005b	20.81±0.008c	22.43±0.005a	22.47±0.09a
63	7-epi- $\alpha$ -Eudesmol	1663	0.45	0.53	0.60	0.60
64	epi- $\alpha$ -Bisabolol	1687	9.86±0.003d	9.96±0.003c	10.55±0.003b	10.89±0.003a
65	$\alpha$ -Eudesmol acetate	1792	0.68±0.003b	0.54±0.005d	0.66±0.003c	1.36±0.005a
	Total		100%	100%	100%	100%

Data are mean ± standard error of three replications. ND: Not detected.<sup>a</sup> R, retention indices. D:distillation (%) \*Means followed by the same letter within a row are not significantly different according to Duncan's multiple range test at P < 0.05.

No	Compound%	R <sup>a</sup>	After.D	After 1 month	After 2 month (%)	After 3 month
1	$\alpha$ -Thujene	927	0.09	0.10	-	-
2	$\alpha$ -Pinene	932	6.97 $\pm$ 0.025a	6.17 $\pm$ 0.021b	6.05 $\pm$ 0.015c	5.16 $\pm$ 0.061c
3	Camphepane	950	0.12	0.12	0.15	-
4	Thuja-2,4(10)-diene	955	0.12	0.15	0.23	-
5	Sabinene	974	0.33	0.29	0.36	-
6	$\beta$ -Pinene	978	12.97 $\pm$ 0.035a	11.51 $\pm$ 0.162b	11.81 $\pm$ 0.088b	10.80 $\pm$ 0.233c
7	6-methyl-5-Hepten-	987	0.18	0.17	0.20	-
8	Myrcene	991	2.19 $\pm$ 0.026a	2.03 $\pm$ 0.012b	2.17 $\pm$ 0.049ab	2.20 $\pm$ 0.018a
9	n-Decane	997	0.12	0.14	-	-
10	$\alpha$ -Terpinene	1017	0.11	0.12	0.07	-
11	p-Cymene	1025	0.57	0.54	0.59	0.55
12	Limonene	1029	3.45 $\pm$ 0.0158a	3.24 $\pm$ 0.069a	3.55 $\pm$ 0.125a	3.34 $\pm$ 0.059a
13	1,8-Cineole	1031	0.19	0.30	0.28	-
14	(E)- $\beta$ -Ocimene	1046	0.35	0.31	0.34	0.31
15	$\gamma$ -Terpinene	1058	0.43	0.43	0.47	0.44
16	Terpinolene	1089	0.23	0.14	0.18	-
17	Linalool	1098	0.29	0.28	0.28	-
18	n-Nonanal	1104	0.20	0.19	0.18	-
19	1-Octen-3-yl acetate	1111	0.29	0.28	0.33	-
20	$\alpha$ -Campholenal	1126	0.33	0.32	0.36	-
21	trans-Pinocarveol	1138	0.16	0.16	0.23	-
22	cis-Verbenol	1143	2.11 $\pm$ 0.035a	1.81 $\pm$ 0.012b	1.80 $\pm$ 0.047b	1.85 $\pm$ 0.020b
23	trans-Verbenol	1145	1.14 $\pm$ 0.041b	1.26 $\pm$ 0.029a	1.12 $\pm$ 0.037b	1.20 $\pm$ 0.060ab
24	Pinocarvone	1163	0.25 $\pm$ 0.0057a	0.70 $\pm$ 0.028b	0.70 $\pm$ 0.005b	0.76 $\pm$ 0.006a
25	p-Mentha-1,5-dien-8-	1167	0.82	0.44	0.46	0.46
26	Terpinen-4-ol	1177	0.35	0.38	0.39	0.39
27	$\alpha$ -Terpineol	1190	0.38	0.16	0.31	0.43
28	Myrtenal	1198	1.84 $\pm$ 0.017a	0.43 $\pm$ 0.014b	0.42 $\pm$ 0.005b	0.00
29	Verbenone	1207	0.31 $\pm$ 0.035c	1.89 $\pm$ 0.033c	1.99 $\pm$ 0.040ab	2.00 $\pm$ 0.005a
30	trans-Carveol	1222	0.23	0.22	0.31	-
31	Nerol	1231	0.21	0.26	0.27	-
32	Cumin aldehyde	1242	0.26	0.23	0.31	-
33	Carvone	1244	3.85 $\pm$ 0.26b	4.16 $\pm$ 0.06a	4.45 $\pm$ 0.13a	452 $\pm$ 0.17a
34	Bornyl acetate	1287	1.10 $\pm$ 0.04a	1.05 $\pm$ 0.16a	1.11 $\pm$ 0.00a	1.14 $\pm$ 0.04a
35	Thymol	1290	1.23 $\pm$ 0.03a	1.15 $\pm$ 0.45ab	1.23 $\pm$ 0.03a	1.27 $\pm$ 0.02a
36	Carvacrol	1299	0.12	0.10	0.13	-
37	$\delta$ -Elemene	1337	0.20	0.20	0.25	-
38	Piperitenone oxide	1368	0.41	0.47	0.44	0.56
39	$\alpha$ -Copaene	1377	0.15	0.06	0.20	-
40	$\beta$ -Bourbonene	1386	0.11	0.08	0.09	-
41	$\beta$ -Elemene	1392	0.19	0.23	0.22	-
42	n-Tetradecane	1399	0.08	0.07	0.06	-
43	$\alpha$ -Gurjunene	1408	0.10	0.11	-	-
44	(E)-Caryophyllene	1423	4.71 $\pm$ 0.088b	4.66 $\pm$ 0.029b	4.83 $\pm$ 0.014b	5.12 $\pm$ 0.043a
45	$\gamma$ -Elemene	1434	0.95 $\pm$ 0.015b	0.94 $\pm$ 0.003b	0.98 $\pm$ 0.005a	0.98 $\pm$ 0.005a
46	$\alpha$ -Humulene	1452	0.08	0.09	0.05	-
47	(E)- $\beta$ -Farnesene	1456	2.20 $\pm$ 0.008c	2.20 $\pm$ 0.008c	2.25 $\pm$ 0.005b	2.41 $\pm$ 0.005a
48	dehydro-	1471	2.86 $\pm$ 0.024c	2.80 $\pm$ 0.017c	2.91 $\pm$ 0.005b	3.07 $\pm$ 0.020a
49	Germacrene D	1484	1.56 $\pm$ 0.024ab	1.54 $\pm$ 0.023ab	1.52 $\pm$ 0.005b	1.61 $\pm$ 0.015a
50	Bicyclogermacrene	1499	1.00 $\pm$ 0.005b	1.21 $\pm$ 0.01a	0.80 $\pm$ 0.011c	1.02 $\pm$ 0.005b
51	$\alpha$ -Muurolene	1502	0.42	0.68	0.22	-
52	$\beta$ -Bisabolene	1508	0.99 $\pm$ 0.000a	0.96 $\pm$ 0.000a	1.00 $\pm$ 0.000a	0.000a
53	$\gamma$ -Cadinene	1515	0.56	0.52	0.41	-
54	$\delta$ -Cadinene	1523	1.49 $\pm$ 0.000a	1.45 $\pm$ 0.000a	1.43 $\pm$ 0.000a	1.51 $\pm$ 0.000a
55	Unknown	1536	0.59	0.57	0.39	0.39
56	Elemol	1552	1.49 $\pm$ 0.000a	1.71 $\pm$ 0.000a	1.57 $\pm$ 0.000a	1.74 $\pm$ 0.030a
57	Germacrene B	1558	0.75	0.87	0.68	-
58	(E)-Nerolidol	1565	0.37	0.34	0.43	0.79
59	Spathulenol	1578	0.44 $\pm$ 0.0030d	2.33 $\pm$ 0.012a	1.89 $\pm$ 0.050c	2.10 $\pm$ 0.050b
60	Caryophyllene oxide	1581	1.29 $\pm$ 0.005a	2.11 $\pm$ 0.008b	2.09 $\pm$ 0.020b	2.34 $\pm$ 0.23a
61	1,10-di-epi-Cubenol	1621	0.64	0.33	0.65	0.65
62	Valerenol	1658	21.44 $\pm$ 0.005b	21.19 $\pm$ 0.005c	20.54 $\pm$ 0.008d	22.47 $\pm$ 0.105aa
63	7-epi- $\alpha$ -Eudesmol	1663	0.45	0.38	0.56	0.56
64	epi- $\alpha$ -Bisabolol	1687	9.86 $\pm$ 0.005c	10.09 $\pm$ 0.005ba	9.84 $\pm$ 0.006c	10.95 $\pm$ 0.005a
65	$\alpha$ -Eudesmol acetate	1792	0.68 $\pm$ 0.005c	0.55 $\pm$ 0.005d	0.88 $\pm$ 0.005a	0.82 $\pm$ 0.005b
	Total		100%	100%	100%	100%

Data are mean  $\pm$  standard error of three replications. ND: Not detected. <sup>a</sup> R, retention indices. D:distillation (%)\*Means followed by the same letter within a row are not significantly different according to Duncan's multiple range test at P < 0.05.

No	Compound%	R <sup>a</sup>	a	After.D	After 1 month	After 2 month (%)	After 3 month
1	$\alpha$ -Thujene	927		0.09	0.01	-	-
2	$\alpha$ -Pinene	932		6.97 $\pm$ 0.008a	0.73 $\pm$ 0.014b	0.22 $\pm$ 0.008c	0.06 $\pm$ 0.005d
3	Camphepane	950		0.12	0.04	-	-
4	Thuja-2,4(10)-diene	955		0.12	0.08	-	-
5	Sabinene	974		0.33	0.07	-	-
6	$\beta$ -Pinene	978		12.97 $\pm$ 0.008a	3.04 $\pm$ 0.008b	1.30 $\pm$ 0.011c	0.61 $\pm$ 0.008d
7	6-methyl-5-Hepten-	987		0.18	0.17	0.14	-
8	Myrcene	991		2.19	0.75	0.29	-
9	n-Decane	997		0.12	0.08	-	-
10	$\alpha$ -Terpinene	1017		0.11	0.04	0.04	-
11	p-Cymene	1025		0.57	0.28	0.16	-
12	Limonene	1029		3.45 $\pm$ 0.048a	1.45 $\pm$ 0.015b	0.68 $\pm$ 0.005b	0.31 $\pm$ 0.008d
13	1,8-Cineole	1031		0.19	0.26	0.07	-
14	(E)- $\beta$ -Ocimene	1046		0.35	0.19	0.11	-
15	$\gamma$ -Terpinene	1058		0.43	0.23	0.12	-
16	Terpinolene	1089		0.23	0.14	0.10	-
17	Linalool	1098		0.29	0.34	0.33	0.35
18	n-Nonanal	1104		0.20	0.24	0.24	0.28
19	1-Octen-3-yl acetate	1111		0.29	0.30	0.33	0.25
20	$\alpha$ -Campholenal	1126		0.33	0.38	-	0.40
21	trans-Pinocarveol	1138		0.16	0.16	0.37	-
22	cis-Verbenol	1143		2.11 $\pm$ 0.000a	2.18 $\pm$ 0.000a	2.13 $\pm$ 0.000a	2.19 $\pm$ 0.000a
23	trans-Verbenol	1145		1.14 $\pm$ 0.000a	1.57 $\pm$ 0.000a	1.63 $\pm$ 0.000a	1.55 $\pm$ 0.000a
24	Pinocarvone	1163		0.25	0.41	0.23	0.61
25	p-Mentha-1,5-dien-8-	1167		0.82 $\pm$ 0.000a	0.98 $\pm$ 0.000a	0.67 $\pm$ 0.000a	0.49 $\pm$ 0.000a
26	Terpinen-4-ol	1177		0.35	0.45	0.42	0.46
27	$\alpha$ -Terpineol	1190		0.38	0.21	0.23	0.57
28	Myrtenal	1198		1.84	0.50	0.56	-
29	Verbenone	1207		0.31 $\pm$ 0.003d	2.08 $\pm$ 0.005c	2.26 $\pm$ 0.012a	2.20 $\pm$ 0.003b
30	trans-Carveol	1222		0.23	0.29	-	0.31
31	Nerol	1231		0.21	0.26	0.28	0.22
32	Cumin aldehyde	1242		0.26	0.27	0.21	-
33	Carvone	1244		3.85 $\pm$ 0.005d	4.71 $\pm$ 0.005b	4.42 $\pm$ 0.005c	4.96 $\pm$ 0.005a
34	Bornyl acetate	1287		1.10 $\pm$ 0.003c	1.30 $\pm$ 0.005b	1.30 $\pm$ 0.005b	1.47 $\pm$ 0.006a
35	Thymol	1290		1.23 $\pm$ 0.012a	1.51 $\pm$ 0.000b	1.55 $\pm$ 0.000c	1.80 $\pm$ 0.005d
36	Carvacrol	1299		0.12	0.18	0.20	-
37	$\delta$ -Elemene	1337		0.20	0.22	0.19	-
38	Piperitenone oxide	1368		0.41	0.52	0.52	-
39	$\alpha$ -Copaene	1377		0.15	0.09	0.06	0.56
40	$\beta$ -Bourbonene	1386		0.11	0.14	0.13	-
41	$\beta$ -Elemene	1392		0.19	0.33	0.38	0.32
42	n-Tetradecane	1399		0.08	0.04	-	-
43	$\alpha$ -Gurjunene	1408		0.10	0.19	-	-
44	(E)-Caryophyllene	1423		4.71 $\pm$ 0.010d	5.82 $\pm$ 0.020c	5.49 $\pm$ 0.005b	6.24 $\pm$ 0.005a
45	$\gamma$ -Elemene	1434		0.95	1.16	1.13	1.25
46	$\alpha$ -Humulene	1452		0.08	0.14	0.12	-
47	(E)- $\beta$ -Farnesene	1456		2.23 $\pm$ 0.008d	2.71 $\pm$ 0.005c	2.82 $\pm$ 0.005b	3.06 $\pm$ 0.010a
48	dehydro-	1471		2.86 $\pm$ 0.005d	3.51 $\pm$ 0.005c	3.57 $\pm$ 0.003b	4.10 $\pm$ 0.005a
49	Germacrene D	1484		1.56 $\pm$ 0.008c	1.76 $\pm$ 0.01b	1.67 $\pm$ 0.005a	1.77 $\pm$ 0.005a
50	Bicyclogermacrene	1499		1.00	1.25	0.98	0.23
51	$\alpha$ -Muurolene	1502		0.42	0.86	0.50	-
52	$\beta$ -Bisabolene	1508		0.99 $\pm$ 0.005d	1.22 $\pm$ 0.005c	1.24 $\pm$ 0.003b	1.37 $\pm$ 0.005a
53	$\gamma$ -Cadinene	1515		0.56	0.54	0.55	0.60
54	$\delta$ -Cadinene	1523		1.49 $\pm$ 0.005d	1.86 $\pm$ 0.005c	1.91 $\pm$ 0.005b	2.24 $\pm$ 0.01a
55	Unknown	1536		0.59	0.73	0.87	0.63
56	Elemol	1552		1.49 $\pm$ 0.005d	2.00 $\pm$ 0.005c	2.01 $\pm$ 0.003b	2.82 $\pm$ 0.008a
57	Germacrene B	1558		0.75 $\pm$ 0.005d	1.01 $\pm$ 0.003b	2.20 $\pm$ 0.005a	0.93 $\pm$ 0.005c
58	(E)-Nerolidol	1565		0.37 $\pm$ 0.011d	0.99 $\pm$ 0.003b	1.16 $\pm$ 0.017a	0.45 $\pm$ 0.015c
59	Spathulenol	1578		0.44 $\pm$ 0.005d	3.00 $\pm$ 0.005b	3.56 $\pm$ 0.005a	2.78 $\pm$ 0.005c
60	Caryophyllene oxide	1581		1.29 $\pm$ 0.005d	2.65 $\pm$ 0.005c	3.15 $\pm$ 0.05b	3.40 $\pm$ 0.005a
61	1,10-di-epi-Cubenol	1621		0.64	0.96	0.93	0.88
62	Valerenol	1658		21.44 $\pm$ 0.003d	26.77 $\pm$ 0.008c	29.55 $\pm$ 0.005b	30.46 $\pm$ 0.005a
63	7-epi- $\alpha$ -Eudesmol	1663		0.45	0.46	0.47	0.70
64	epi- $\alpha$ -Bisabolol	1687		9.86 $\pm$ 0.005d	12.45 $\pm$ 0.020c	12.72 $\pm$ 0.01b	14.55 $\pm$ 0.008a
65	$\alpha$ -Eudesmol acetate	1792		0.68 $\pm$ 0.005c	0.62 $\pm$ 0.005d	1.08 $\pm$ 0.005a	0.94 $\pm$ 0.003b
	Total			100%	100%	100%	100%

Data are mean  $\pm$  standard error of three replications. ND: Not detected. <sup>a</sup> R, retention indices. D: distillation (%)\*Means followed by the same letter within a row are not significantly different according to Duncan's multiple range test at P < 0.05.