

SUPPLEMENTARY MATERIAL

Essential Oil Composition of *Erica spiculifolia* Salisb. - First Report

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Abstract: The essential oil isolated from *Erica spiculifolia* Salisb. by hydrodistillation was analyzed by GC-MS. One hundred compounds representing 92.6% of total oil were identified. Among the detected compounds 38 were monoterpenoids (46.2%), 30 were sesquiterpenoids (31.7%), 2 diterpene (0.4%) and 30 compounds (14.3%) were with various non-terpenoid structures. Oxygenated monoterpenes were by far the main class (45.3%) with α -terpineol (7.5%) *endo*-borneol (7.2%), pinocarveol (5.9%) and thymol (3.7%) as the major compounds. The predominant components of sesquiterpenes were caryophyllene oxide (5.0%), caryophyllene (4.2%), τ -murolool (3.5%), spathulenol (2.9) and α -cadinol (2.3%).

Keywords: *Erica spiculifolia* Salisb. (*Bruckenthalia spiculifolia*); essential oil; chemical composition; GC-MS

Experimental

Plant material and sample preparation: Aerial parts of *E. spiculifolia* Salisb .were collected in July 2015 from the area of Pirin Mountain, Bansko, Bulgaria. A voucher specimen was deposited in the Herbarium Moesiacum Nis (HMN), Department of Biology and Ecology, University of Nis under the acquisition number 6924. The air-dried aerial parts of plant materials (500 g) were powdered and submitted for 2 h to water-distillation using a Clevenger-type apparatus. The oil was dried over anhydrous sodium sulphate and after filtration, stored at +4 °C prior to analysis. GC and GC/MS analysis

Gas Chromatography and Gas Chromatography-Mass Spectrometry Analysis: GC/MS analyses were performed on an Agilent 7890 gas chromatograph with 7000B GC/MS/MS triple quadrupole system, operating in MS1 scan mode, and equipped with a fused-silica capillary column Agilent HP-5 MS (30 m ×0.25 mm i.d.×0.25 µm film thickness). The chromatographic analyses were carried out in the following conditions: He as carrier gas at a flow rate of 1.0 mL/min, GC oven temperature was kept at 45°C for 2.25 min and programmed to 290°C at a rate of 4°C/min, split ratio was adjusted at 40:1, injection volume 1 µL. Post run: back flash for 1.89 min, at 280°C, with helium pressure of 50 psi. The injector temperature was set at 230°C. Ionization mode was electronic impact at 70 eV. Mass range was set from 40 to 440 Da. For GC/FID analysis, the same column and chromatographic conditions were applied as described for GC/MS. FID detector temperature was 300°C. The percentage amounts of the separated compounds were calculated from the GC peak areas using the normalization method without correction factors.

The data are reported as mean value of three sample injections.

Identification of Components:

Oil constituents were identified by comparison of their linear retention indices (relative to C₈-C₂₀ and C₂₁-C₄₄ alkanes (Van den Dool & Kratz 1963; Adams 2007) on the HP-5MS column) with literature values and their MS with those of authentic standards, as well as those from Wiley 6, NIST11, Agilent Mass Hunter Workstation B.06.00 software (Stein 1990) and a homemade MS library with the spectra corresponding to pure substances and components of known essential oils by the application of the AMDIS software (Automated Mass Spectral Deconvolution and Identification System, Ver. 2.1, DTRA-NIST, 2011).

Table S1. *Chemical composition of the *Erica spiculifolia* Salisb. essential oil volatiles achieved by GC and GC-MS

Peak	RI	RL	Compound	Composition %
1	861	867**	Heksanol	tr
2	930	939	α -Pinene	0.1
3	957	961	Benzaldehyde	0.2
4	969	968**	3,5,5-trimethyl-2-Hexene	0.2
5	975	979	1-Octen-3-ol	0.7
6	988	996	2-Pentylfuran	0.1
7	993	1012	(<i>E,E</i>)-2,4-Heptadienal	0.2
8	1008	1011	δ -3-Carene	0.7
9	1026	1031	Limonene	0.1
10	1041	1049**	Benzeneacetaldehyde	0.9
11	1055	1058**	(<i>Z</i>)-2-Octenal	0.2
12	1064	1067**	(<i>E</i>)-2-Octen-1-ol	0.2
13	1067	1068**	1-Octanol	tr
14	1067	1076**	(<i>E,E</i>)-3.5-Octadien-2-one	1.0
15	1070	1072**	<i>trans</i> -Linalooloxide (furanoid)	0.4
16	1089	1075**	<i>cis</i> -Linalooloxide (furanoid)	0.3
17	1097	1104	β -Linalool	2.6
18	1101	1102	Nonanal	1.2
19	1106	1102	4-Hydroxy-4-methylcyclohexanone	0.3
20	1138	1135**	Pinocarveol	5.9
21	1143	1141	Camphor	0.5
22	1147	1140	<i>trans</i> -Verbenol	0.2
23	1150	1153**	(<i>E,E</i>)-2,6-Nonadienal	0.1
24	1157	1162	(<i>E</i>)-2-Nonenal	0.1
25	1158	1166	<i>p</i> -Mentha-1,5-dien-8-ol	0.9
26	1161	1160	Benzenepropanal	0.7
27	1165	1166	<i>endo</i> -Borneol	7.2
28	1167	1168**	1-Nonanol	0.4
29	1176	1177	Terpinen-4-ol	1.0
30	1180	1185**	<i>p</i> -Cymen-8-ol	0.7
31	1186	1189	α -Terpineol	1.2
32	1189	1189	α -Terpineol	7.5
33	1195	1191	(-)-Myrtenol	1.8
34	1199	1206	Safranal	0.3
35	1202	1200	Decanal	0.5
36	1207	1207**	Verbenone	0.4
37	1217	1215	<i>trans</i> -Carveol	1.1
38	1219	1220**	β -Cyclocitral	0.5
39	1225	1225**	Grandlure II	0.3
40	1225	1229**	<i>cis</i> -Geraniol	0.3
41	1229	1229**	<i>cis</i> -Carveol	0.3

42	1232	1241	Thymol methyl ether	0.2
43	1236	1235	Isobornyl formate	0.1
44	1242	1268	<i>trans-p</i> -Menth-2-en-7-ol	0.9
45	1251	1254	Geraniol	1.0
46	1258	1265	(<i>E</i>)-2-Decenal	1.0
47	1268	1269**	Citral	0.5
48	1277	1284	<i>p</i> -Menth-4(8)-en-9-ol	0.3
49	1285	1289**	(-)-Bornyl acetate	0.7
50	1289	1290	Thymol	3.7
51	1299	1298	Carvacrol	0.8
52	1303	1305**	Undecanal	0.3
53	1313	1315	(<i>E,E</i>)-2,4-Decadienal	0.6
54	1315		4-Methyl-2-propylphenol	0.2
55	1348	1350**	α -Terpineol acetate	0.5
56	1356	1359**	Eugenol	0.5
57	1360	1364**	<i>E</i> -2-Undecenal	0.7
58	1373	1376**	Methyl 4-methoxybenzoate	0.1
59	1379	1382	Geranyl acetate	0.2
60	1392	1394	β -elemene	0.1
61	1401	1408	1,2,3-trimethoxy-5-methyl-benzene	0.7
62	1422	1418	Caryophyllene	4.2
63	1450	1454**	Geranylacetone	1.0
64	1456	1455	α -Humulene	0.8
65	1463	1464	9-epi-(<i>E</i>)-Caryophyllene	0.3
66	1463	1463	Dehydroaromadendrane	tr
67	1477	1465	<i>cis</i> -Muurolo-4(14),5-diene	0.2
68	1483	1480	Germacrene D	0.6
69	1485	1488	<i>trans</i> - β -Ionone	1.3
70	1489	1499**	Guaia-4,11-diene	0.2
71	1491	1492	10,11-epoxy-Calamenene	0.1
72	1498	1500	Bicyclogermacrene	1.4
73	1501	1499	α -Muurolole	tr
74	1508	1506	β - Bisabolene	0.5
75	1516	1511	γ -Cadinene	1.2
76	1524	1524	(+)- δ -Cadinene	1.7
77	1539	1544**	α -Cadinene	0.1
78	1545	1546	α -Calacorene	0.1
79	1570	1568	(<i>Z</i>)-3-Hexenyl benzoate	1.3
80	1574	1571	Caryolan-8-ol	0.2
81	1580	1582**	Spathulenol	2.9
82	1587	1581	Caryophyllene oxide	5.0
83	1595	1592	Viridiflorol	0.7
84	1608	1611**	Tetradecanal	0.6
85	1612	1615**	1,2-epoxide-Humulene	0.6
86	1617	1618	1,10-di-epi-Cubenol	0.4
87	1630	1627	1-epi-Cubenol	0.3

88	1636	1631	Caryophylla-4(12),8(13)-dien-5- α -ol	0.4
89	1640	1635	Caryophylla-4(12),8(13)-dien-5- β -ol	1.1
90	1644	1640	τ -Murolool	3.5
91	1648	1644	α -Muurolool	0.5
92	1657	1653	α -Cadinol	2.3
93	1673	1674	(Z)- α -Santalol	0.6
94	1694	1689	Shyobunol	1.7
95	1765	1763**	Benzyl benzoate	0.7
96	1820	1824**	1-methylethyl Tetradecanoate	0.2
97	1840	1847**	6,10,14-trimethyl-2-Pentadecanone	1.0
98	1870	1870**	Benzyl salicylate	0.1
99	1997	1989	Epimanoyl oxide (ent-8-epi-manoyl oxide)	0.2
100	2019	2007	18-Norabieta-8.11.13-triene	0.2
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Total				
			Monoterpenoids	46.2
			Hydrocarbons	0.9
			Oxygenated	45.3
			Sesquiterpenoids	31.7
			Hydrocarbons	11.4
			Oxygenated	20.3
			Diterpene	0.4
			Hydrocarbons	0.2
			Oxygenated	0.2
			Others	14.3

* Compounds are grouped under the classes and listed in order of elution from a HP-5MS column; RI: Experimental retention indices relative to C₈-C₃₂ *n*-alkanes; RL: Literature retention indices; (*): identified by NIST Chemistry Web Book Retention indices; tr: traces (<0.1%).

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