

# Composition of the Essential Oil of *Sideritis condensata* Boiss. et Heldr.

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Water distilled essential oils from six samples of *Sideritis condensata* Boiss. et Heldr., an endemic species in Turkey, were examined by GC and GC-MS. One-hundred-and-ninety-two components were characterized with  $\beta$ -caryophyllene (9.1-18.8%), germacrene-D (4.7% to 13.7%) and hexadecanoic acid (5.6% to 14.9%) as the major constituents.

KEY WORDS: *Sideritis condensata* Boiss. et Heldr.; Lamiaceae; essential oil;  $\beta$ -caryophyllene; germacrene-D; hexadecanoic acid

## INTRODUCTION

*Sideritis* (Lamiaceae) is represented by 41 species and 48 taxa in the *Flora of Turkey*.<sup>1,2</sup> Thirty-four of these are endemic in Turkey.

*Sideritis* species are widely used as herbal tea in Turkey due to their pleasant aroma. The tea is prepared by dipping a dried single inflorescence of the plant into a cup of hot water for half a minute. Then the plant part is removed and the tea is either sweetened with sugar or taken on its own.

In folk medicine, *Sideritis* species are attributed to have nervous system stimulant, sedative, antitussive, stomachic, carminative and anti-inflammatory activities.<sup>3,4</sup> Recent studies have shown that aqueous extracts of five *Sideritis* species of Turkey have nervous system stimulant or anti-stress activity in mice.<sup>3</sup>

*Sideritis condensata* Boiss. et Heldr. is an endemic plant of Turkey growing in Antalya province in southern Turkey and is locally known as 'Dağ Çayı' or 'Eşşek Çayı'. The present study covers the analysis of oils from six different samples of *S. condensata* collected in Antalya and neighbouring Isparta provinces.

## MATERIALS AND METHODS

### Plant Material

Plant materials were collected from the following localities:

	Oil yield (%)
A. Isparta. Eğirdir, Akpınar village in August 1992 (ESSE 7185).	tr
B. Antalya: Manavgat to Akseki, 31st km in August 1993 (ESSE 10427).	0.11
C. Antalya: Manavgat, Yukarı Işıklar village in August 1993 (ESSE 10123)	0.65
D. Antalya: Akseki, Aşağı Işıklar village in August 1993 (ESSE 9355).	tr
E. Antalya: Akseki, Taşlıca village in August 1993 (ESSE 9525).	tr
F. Antalya: sample purchased from market in August 1994 (ESSE 10748).	0.02

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Voucher specimens are kept at the herbarium of Faculty of Pharmacy Anadolu University in Eskişehir, Turkey (acronym: ESSE).

#### Essential Oil Distillation

Aerial parts of the air dried plants including inflorescence were subjected to water distillation for 3 h using a Clevenger apparatus to give oils in the above yields.

#### Gas Chromatography

The GC analysis was carried out using Shimadzu GC-9A with C-R4A integrator. A polar Thermo 600 T fused silica column (50 m × 0.25 mm i.d.) was used. The carrier gas was nitrogen. The oven temperature was kept at 70°C for 10 min and programmed to 180°C at a rate of 2°C/min, and then kept constant at 180°C for 30 min. The injector and detector (FID) temperatures were 250°C.

#### Gas Chromatography–Mass Spectrometry

The GC–MS analysis was carried out using Shimadzu GC–MS QP 2000A and Hewlett-Packard GCD systems.

In the Shimadzu GC–MS QP 2000A, a Thermo 600 T fused silica column (50 m × 0.25 mm i.d.) was used with helium as carrier gas, and MS were taken at 70 eV. The scanning speed was 2 scans/sec from 10 to 400 *m/z*. The same temperature programming as above was applied.

An Innovax FSC column (60 m × 0.25 mm i.d.) was also used with helium as the carrier gas in the Hewlett-Packard GCD system. GC oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min, kept constant at 220°C for 10 min, and then increased to 240°C at a rate of 1°C/min. Split flow was adjusted at 50 ml/min. The injector and detector temperatures were 250°C. MS were taken at 70 eV. Mass range was from 10 to 425 *m/z*.

Library search was carried out using LSS-30 Library Search Software from the NBS/NIH/EPA library, The Wiley/NBS Registry of Mass Spectral Data and TBAM Library of Essential Oil Constituents. The MS were also compared with reference compounds and confirmed with the aid of retention indices from published sources.<sup>5–8</sup>

## RESULTS AND DISCUSSION

*Sideritis* species generally give low yields of essential oil which is rich in sesquiterpenes.<sup>9</sup> *Sideritis* is taxonomically placed in the subfamily Nepetoideae according to Erdtman and known to possess hexacolpate pollen grains.<sup>10</sup> It was postulated that Lamiaceae genera possessing hexacolpate pollen grains were oil-rich.<sup>11,12</sup> However, *Sideritis* must be treated as an exception, with similar exceptions reported by Lawrence.<sup>10</sup> The sesquiterpene-rich oil of *Sideritis condensata* is reminiscent of those low oil yield species of Lamiaceae possessing tricolpate pollen grains, with β-caryophyllene and germacrene-D as the major constituents (Table 1).

Two sesquiterpene hydrocarbons, β-caryophyllene (9.1–18.8%) and germacrene-D (4.7–13.7%) were found as major constituents of the six oil samples analysed. Altogether 192 compounds were characterized. Sesquiterpene hydrocarbons were the predominant constituents in all the oils with ratios ranging from 26% to 45%. The ratio of oxygenated sesquiterpenes varied widely between 15% and 29%. On the other hand, cumulative percentage amounts of sesquiterpenoids showed a more consistent picture with values ranging from 47% to 61%.

Wide variations were observed with the percentage amounts of monoterpenoids. Cumulative values ranged from 7% to 17%. Except for B and E, the other oils possessed more hydrocarbons than their oxygenated derivatives. Non-terpenoid components constituted 15–32% of the oils.

These calculations are based on the characterized components. However, the remaining 7–13% unidentified components are not expected to change the picture in favour of monoterpenes, since most of these compounds are sesquiterpenoids. Recently, solvent-trapped volatiles of *S. condensata* were reported with β-caryophyllene (15.9%), β-pinene (12.1%), caryophyllene oxide (6.2%), germacrene-D (5.4%) and spathulenol (4.1%) as major constituents.<sup>13</sup> Dried *Sideritis condensata* inflorescences are sold in Antalya markets for use as herbal tea. The low oil content of this species does not deter people from using it to make a pleasant hot drink.

Table 1. Percentage composition of the essential oils of *Sideritis condensata* Boiss. et Heldr.

Compound	KI <sup>a</sup>	A	B	C	D	E	F
Tetrahydrofuran	877	0.01	0.01	–	–	–	–
α-Pinene	1032	0.3	0.1	0.1	0.3	0.02	1.9
α-Thujene	1035	–	–	–	0.02	–	0.1
1,3,5-Cycloheptatriene	1056	0.03	0.02	–	0.02	–	–
Camphene	1076	–	–	–	–	–	0.02
β-Pinene	1118	0.9	0.04	0.4	0.8	0.03	6.4
Sabinene	1131	0.1	0.03	0.04	0.1	0.02	0.2
δ-3-Carene	1159	–	–	0.04	0.02	–	0.02
Myrcene	1174	0.1	0.04	0.1	0.4	0.03	0.4
α-Phellandrene	1176	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
α-Terpinene	1188	0.02	0.01	–	0.1	–	0.1
Heptanal	1195	0.02	–	–	–	–	0.02
Dodecane	1200	–	0.01	–	–	–	–
Limonene	1203	0.5	0.3	0.5	0.9	0.2	0.7
1,8-Cineole	1213	0.2	0.5	0.6	0.2	0.4	–
β-Phellandrene	1213	0.02	0.01	0.04	0.1	< 0.01	0.1
Amyl furan	1244	–	–	–	–	–	0.1
(Z)-β-Ocimene	1246	0.01	< 0.01	0.04	0.1	–	0.6
γ-Terpinene	1255	0.1	0.04	0.1	0.1	0.1	0.3
(E)-β-Ocimene	1266	0.02	< 0.01	< 0.01	0.01	–	0.4
p-Cymene	1280	0.2	0.2	0.3	0.3	0.2	0.2
Terpinolene	1290	–	< 0.01	0.01	0.03	–	0.03
Octanal	1296	0.02	< 0.01	–	0.01	–	0.03
Tridecane	1300	0.02	0.01	–	0.01	0.01	–
6-Methylhept-5-en-2-one	1348	–	0.01	–	–	–	–
Hexanol	1360	–	0.01	–	0.01	–	–
6-Methylheptan-3-ol	1398	0.04	0.1	0.1	0.1	0.1	0.04
Nonanal	1400	0.3	0.2	0.2	0.2	0.2	0.3
Tetradecane	1400	–	0.01	–	–	–	–
α-Fenchone	1406	–	< 0.01	–	–	–	–
Perillene	1429	–	–	–	0.01	–	–
(E)-Oct-2-enal	1441	–	–	–	0.01	–	0.03
cis-Linalol oxide	1452	–	0.02	–	–	–	–
α-p-Dimethylstyrene	1452	–	–	–	0.01	–	–
β-Thujone	1457	–	–	–	0.01	–	–
Oct-1-en-3-ol	1457	0.5	0.03	0.3	0.3	0.2	0.2
α-Cubebene	1466	0.04	0.3	0.3	0.3	0.2	0.1
trans-Sabinene hydrate	1474	0.04	–	0.02	0.04	–	–
Menthone	1476	–	0.02	0.03	0.2	0.02	0.2
(Z) Hex-3-enyl 3-methylbutanoate	1479	0.01	–	0.02	0.03	0.01	0.04
Octyl acetate	1482	–	–	–	0.04	–	–
α-Ylangene	1492	–	–	0.01	–	–	0.04
Cyclosativene	1492	–	–	–	0.04	–	–
α-Copaene	1497	0.4	2.3	2.2	2.1	1.9	1.3
Decanal	1506	0.1	0.1	0.1	0.1	0.2	0.1
9-Hydroxytheaspiran-A*	1513	–	0.01	–	0.01	–	–
α-Bourbonene	1529	0.1	0.2	0.3	0.1	0.2	0.1
β-Bourbonene	1535	0.9	2.3	2.0	1.1	1.7	2.0
α-Gurjunene	1544	–	0.1	0.4	0.1	0.1	0.03
(E) Non-2-enal	1547	–	0.02	0.04	0.04	0.1	0.1
β-Cubebene	1547	–	–	–	–	–	–
Linalol	1523	0.7	8.4	2.8	2.2	1.5	0.9
Octan-1-ol	1562	0.1	0.1	0.1	0.1	0.1	0.3
Linalyl acetate	1565	–	0.03	0.1	0.01	0.1	–
trans-p-Menth-2-en-ol	1571	0.02	0.01	0.02	0.01	–	–
Pinocarvone	1586	0.1	0.04	0.1	0.1	0.1	0.1
trans-β-Bergamotene	1594	–	0.03	0.1	0.04	0.1	0.04
Hexadecane	1600	0.1	0.1	–	–	< 0.01	–
β-Elemene	1600	0.3	0.7	1.0	0.8	0.4	0.6
Terpinen-4-ol	1607	0.2	0.3	0.4	0.3	0.3	0.3

Table 1. (continued)

Compound	KI <sup>a</sup>	A	B	C	D	E	F
$\beta$ -Caryophyllene	1611	10.7	11.7	12.1	11.4	9.1	18.8
<i>allo</i> -Aromadendrene	1625	0.1	0.1	0.1	0.1	0.1	-
Hexyl tiglate	1631	0.1	-	0.1	-	0.1	0.1
<i>p</i> -Menth-1-en-9-al	1638	-	-	-	-	0.02	-
<i>cis-p</i> -Mentha-2,8-dien-1-ol	1638	-	-	0.03	-	-	-
$\beta$ -Cyclocitral	1638	-	0.03	-	-	0.03	0.1
Myrtenal	1648	0.2	0.03	0.2	0.1	0.1	0.1
( <i>E</i> )-Dec-2-enal	1655	-	0.1	0.3	0.3	0.3	0.2
Aromadendrene	1658	-	-	0.2	-	-	-
Pulegone	1661	-	0.3	-	0.3	0.2	0.2
Nonan-1-ol	1664	0.1	-	-	-	-	-
<i>cis</i> -Verbenol	1668	0.04	-	-	-	-	-
( <i>Z</i> )- $\beta$ -Farnescene	1668	-	-	0.2	-	-	-
<i>trans</i> -Pinocarveol	1671	0.3	0.1	0.2	0.1	0.1	0.1
( <i>E</i> )- $\beta$ -Farnesene	1671	1.4	0.5	1.3	0.8	0.7	1.9
( <i>Z</i> ) Hex-3-enyl tiglate	1681	0.1	0.1	0.3	0.1	0.1	0.2
Methyl chavicol	1684	-	-	-	-	-	0.6
$\alpha$ -Humulene	1684	0.8	1.3	0.9	0.9	0.7	0.7
Neral	1694	-	0.1	-	-	-	-
$\gamma$ -Muuroleone	1700	-	0.3	0.7	0.6	0.3	0.5
$\gamma$ -Curcumene	1704	0.6	-	-	-	-	-
$\alpha$ -Terpineol	1707	0.2	0.6	0.4	0.3	0.3	< 0.01
$\alpha$ -Terpinyl acetate	1707	-	-	0.2	-	-	-
( <i>E, E</i> ) Nona-2,4-dienal	1715	-	-	-	-	-	0.02
Dodecanal	1722	0.1	0.1	-	0.1	0.1	0.1
Germacrene-D	1726	6.8	7.9	12.1	9.7	4.7	13.7
$\alpha$ -Muuroleone	1737	0.3	0.7	0.6	0.5	0.4	0.3
$\alpha$ -Selinene	1744	-	-	0.2	0.1	-	-
Bicyclogermacrene	1751	2.0	1.5	0.9	2.8	1.6	1.1
( <i>E, E</i> )- $\beta$ -Farnesene	1755	0.7	0.3	0.2	0.7	0.5	0.5
Naphthalene	1762	-	-	-	-	-	2.4
Geranyl acetate	1765	-	0.2	-	0.4	-	-
( <i>E</i> )-Undec-2-enal	1765	-	-	0.2	-	0.4	-
$\delta$ -Cadinene	1772	0.7	2.4	2.3	2.7	2.0	1.2
$\gamma$ -Cadinene	1776	0.2	1.0	0.4	0.6	0.5	0.3
$\beta$ -Sesquiphellandrene	1783	-	-	0.03	-	-	-
<i>ur</i> -Curcumene	1786	1.2	0.3	0.6	0.2	0.1	0.2
Methyl salicylate	1797	-	0.1	0.02	0.1	0.1	0.1
Cadina-1,4-diene (= cubenene)	1800	0.2	0.1	0.4	0.2	0.04	0.1
Myrtenol	1808	0.4	0.1	0.3	0.2	0.1	0.03
Nerol	1808	-	0.1	-	0.04	-	-
Isobutyl benzoate	1808	-	-	-	0.02	-	-
Guaia-3,7-diene	1811	-	0.2	0.2	0.1	0.1	0.04
( <i>E, E</i> ) Deca-2,4-dienal	1827	0.2	0.2	0.1	0.1	0.3	0.2
Tridecanal	1830	-	-	-	0.1	-	-
2,6-Dimethyl octa-3( <i>E</i> ), 5( <i>E</i> ), 7-triene-2-ol	1830	-	-	0.1	-	-	-
Dihydro- $\alpha$ -ionone	1838	0.1	-	-	-	-	-
$\beta$ -Damascenone	1838	0.2	0.4	0.3	0.1	0.6	0.1
<i>trans</i> -Carveol	1845	0.1	0.04	0.1	0.1	0.04	-
Calamenene	1849	0.6	0.4	2.6	0.4	0.3	0.1
Geraniol	1856	-	0.7	-	-	-	-
<i>p</i> -Cymen-8-ol	1864	-	0.04	0.1	0.02	0.03	-
Hexanoic acid	1868	-	< 0.01	-	-	-	-
( <i>E</i> )-Geranyl acetone	1868	0.8	0.4	0.3	0.3	0.8	0.4
( <i>E</i> )-Dodec-2-enal	1875	-	0.01	-	0.02	0.02	-
1-Methylnaphthalene	1878	-	-	-	0.02	0.02	0.1
Aplotaxene*	1882	0.1	0.1	0.04	0.1	0.02	-
Epicubebol	1900	0.2	0.8	0.4	0.4	0.3	0.1
Nonadecane	1900	0.1	0.1	-	-	0.1	-
Benzyl isovalerate	1908	0.1	-	0.03	-	-	-

Table 1. (continued)

Compound	KI <sup>a</sup>	A	B	C	D	E	F
2-Methylbutyl benzoate	1929	0.04	0.1	–	0.01	0.1	0.03
Isoamyl benzoate	1933	–	0.1	–	–	0.1	–
Tetradecanal	1933	0.1	–	0.02	0.04	–	0.1
$\alpha$ -Calacorene-I	1941	0.1	0.1	0.1	0.1	0.2	0.04
Palustrol	1953	–	0.01	–	0.01	–	–
Cubebol	1957	0.3	0.7	0.4	0.4	0.5	0.1
$\beta$ -Ionone	1957	0.2	0.1	0.03	0.1	0.2	0.2
<i>cis</i> -Jasmone	1968	–	–	0.1	–	–	–
Dodecan-1-ol	1973	0.1	–	0.01	0.1	0.2	0.1
$\alpha$ -Calacorene-II	1984	0.1	0.2	0.1	0.1	0.1	0.1
Isocaryophyllene oxide	2000	0.5	0.5	0.3	0.2	0.4	0.8
Caryophyllene oxide	2008	11.3	4.3	4.4	4.9	6.5	7.2
Perillyl alcohol	2025	0.1	–	–	0.02	–	0.1
Methyl eugenol	2029	–	–	0.1	–	–	–
Pentadecanal	2041	–	0.2	–	0.1	–	–
11-Norbourbonan-1-one*	2045	0.3	–	0.1	–	0.3	–
( <i>E</i> )-Nerolidol	2053	0.1	0.1	0.3	0.3	0.6	0.4
Ledol	2057	0.1	0.2	0.3	0.2	0.2	–
Germacre-1,6-dien-5-ol	2069	–	0.7	–	0.4	–	–
Humulene epoxide II	2069	0.4	–	0.2	–	0.3	0.3
<i>p</i> -Mentha-1,4-dien-7-ol	2073	0.1	–	–	–	–	–
Octanoic acid	2084	–	0.2	0.3	0.2	0.5	0.1
Cubanol	2088	0.2	0.3	0.3	0.3	0.3	–
1-Epicubanol	2088	–	0.4	–	–	–	0.1
Globulol	2096	–	0.5	0.2	0.2	–	–
Hexyl benzoate	2096	0.2	–	–	–	0.3	0.1
Heneicosane	2100	0.2	–	–	–	–	–
Viridiflorol	2104	–	0.2	0.2	0.2	0.4	0.2
Hexahydrofarnesyl acetone	2131	1.4	0.9	1.0	1.6	2.1	0.4
Spathulenol	2144	7.3	2.7	1.8	2.9	3.5	0.9
( <i>Z</i> )-Hex-3-enyl benzoate	2148	0.7	0.4	0.04	0.2	0.2	0.1
$\beta$ -Bisabolol	2170	0.3	–	–	–	–	–
( <i>E</i> )-Hex-2-enyl benzoate	2170	0.5	–	0.5	–	0.6	–
Nonanoic acid	2192	0.1	0.5	0.9	0.9	0.6	0.1
Eugenol	2192	0.3	–	–	–	–	0.3
<i>T</i> -Cadinol	2192	–	0.8	0.4	0.2	0.2	–
Thymol	2205	< 0.01	0.2	–	–	< 0.01	< 0.01
<i>T</i> -Muurolol	2205	0.4	0.7	1.0	0.9	1.3	1.3
$\delta$ -Cadinol	2219	–	0.3	–	0.1	0.1	–
Dimyrcene-II-a	2223	–	–	–	0.3	–	–
Methyl hexadecanoate	2228	–	–	–	–	–	0.04
Carvacrol	2246	0.3	3.4	5.6	1.4	2.5	2.0
$\alpha$ -Cadinol	2255	0.2	1.9	0.6	0.9	1.0	0.3
Ethyl hexadecanoate	2260	–	–	–	–	–	0.04
Selin-11-en-4- $\alpha$ -ol	2264	0.1	0.1	–	–	–	–
Dimyrcene-II-b	2269	–	–	–	0.1	–	–
Decanoic acid	2296	0.2	0.3	0.5	0.8	0.5	0.2
Tricosane	2300	0.7	0.2	0.1	0.1	0.2	0.2
Caryophylladienol*	2320	0.7	0.9	–	–	–	0.6
Caryophylladienol*	2320	1.1	1.3	0.8	0.4	0.9	0.8
Kaur-15-ene	2337	–	–	–	0.04	–	–
Hexadecan-1-ol	2380	–	0.7	0.5	0.3	–	0.4
Farnesyl acetone	2384	0.3	< 0.01	–	0.4	–	–
Caryophyllenol II*	2396	2.1	–	1.0	1.1	1.2	0.9
8- $\alpha$ -13-Oxy-14-en-epilabdane	2396	–	2.0	–	–	–	–
Tetracosane	2400	–	< 0.01	–	< 0.01	–	–
Pentacosane	2500	2.0	0.9	–	–	1.5	–
Dodecanoic acid	2508	–	–	1.0	1.5	–	0.6
Isobutyl phthalate**	2556	0.8	0.3	0.2	0.5	1.3	3.1
Hexacos-9-ene	2585	0.1	0.4	–	0.04	0.1	0.1
Hexacosane	2600	0.3	0.1	0.04	0.1	0.1	–

Table 1. (continued)

Compound	KI <sup>a</sup>	A	B	C	D	E	F
Tridecanoic acid	2609	0.1	< 0.01	0.1	0.3	0.2	–
Phytol	2614	0.2	0.2	–	0.2	0.4	0.2
Benzyl benzoate	2651	0.3	0.2	0.1	0.1	0.2	0.1
Heptacosane	2700	2.7	1.2	0.6	1.3	2.2	0.6
Butyl phthalate**	2705	0.5	0.5	0.1	0.3	1.0	0.4
Tetradecanoic acid	2713	1.5	1.2	1.3	1.7	2.2	0.4
Octacosane	2800	0.4	0.2	0.04	0.1	0.3	–
Pentadecanoic acid	2819	0.3	0.3	0.2	0.4	0.6	0.03
Linoleic acid	2845	0.2	–	–	–	0.6	–
Nonacosane	2900	1.7	0.8	0.7	1.2	2.5	0.5
Hexadecanoic acid	2910	8.6	7.6	7.6	14.9	13.4	5.6
<b>Identified components (%)</b>		<b>87</b>	<b>90</b>	<b>87</b>	<b>90</b>	<b>88</b>	<b>93</b>
Monoterpene hydrocarbons		2	1	2	3	2	12
Oxygenated monoterpenes		5	16	12	7	7	5
<b>Monoterpenoids</b>		<b>7</b>	<b>17</b>	<b>14</b>	<b>10</b>	<b>9</b>	<b>17</b>
Sesquiterpene hydrocarbons		27	34	41	36	26	45
Oxygenated sesquiterpenes		29	19	15	16	21	16
<b>Sesquiterpenoids</b>		<b>56</b>	<b>53</b>	<b>56</b>	<b>52</b>	<b>47</b>	<b>61</b>
<b>Diterpenoids</b>		–	<b>2</b>	–	–	–	–
Others		24	18	17	28	32	15

<sup>a</sup>Tentative. <sup>a</sup>Retention index on Thermo 600T.

\*\*Impurity.

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