Progress in Essential Oils

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Artemisia annua Oil

The oil composition of *Artemisia annua* L. annual wormwood that was grown in Oregon (USA) was examined by Tellez et al. (1999). They found that most plants possessed capitate glandular trichomes (oil glands); however, a glandless biotype was found to arise spontaneously among the field-cultivated plants. As a result, the oil composition of both the glanded and glandless plants was the subject of analysis. As one might expect the glandless plants were oil-poor (0.06 percent yield). The components identified in this oil were:

4-methyl-3-penten-2-one (0.5 percent)
(E)-2-hexenal (0.4 percent)
(E)-3-hexenyl acetate (0.3 percent)
benzyl alcohol (0.1 percent)
phenylacetaldehyde (0.2 percent)
nonanal (0.3 percent)
indole (0.1 percent)
α-cubebene (0.1 percent)
α-copaene (1.9 percent)
β-caryophyllene (25.1 percent)
α-humulene (1.3 percent)
(E)-β-farnesene (5.4 percent)
β-cadinene[‡] (0.3 percent)
germacrene D (49.8 percent)
bicyclogermacrene (3.5 percent)
α-muurolene (0.4 percent)
germacrene A (0.1 percent)
(E,E)-α-farnesene (0.1 percent)
γ-cadinene (0.3 percent)
δ-cadinene (1.2 percent)
cadina-1,4-diene (0.2 percent)
caryophyllene oxide (0.3 percent)
T-muurolol (0.3 percent)
α-cadinol (0.3 percent)

[‡]incorrect identification based on GC elution order

In addition, trace amounts (< 0.1 percent) of 4-hydroxy-4-methyl-2-pentanone, (Z)-3-hexenol, hexanol, benzaldehyde, (E)-β-ocimene, acetophenone, octanol, isophorone, β-cubebene, β-elemene, tetradecane, α-cedrene, coumarin, γ-muurolene, α-cadinene, (E)-nerolidol, spathulenol, β-oplopenone, T-cadinol, cubenol and α-muurolol were also found in this unusual oil. In contrast, the composition of an oil of the typical glanded *A. annua* was found to be as follows:

4-methyl-3-penten-2-one (2.7 percent)
4-hydroxy-4-methyl-2-pentanone (2.2 percent)
(E)-2-hexenal (0.2 percent)
(Z)-3-hexenol (0.9 percent)
α-pinene (26.7 percent)
camphene (0.6 percent)
β-pinene (1.6 percent)
dehydro-1,8-cineole (0.2 percent)
yomogi alcohol (0.2 percent)
(E)-3-hexenyl acetate (1.4 percent)
α-terpinene (0.1 percent)
p-cymene (0.3 percent)
1,8-cineole (8.4 percent)
γ-terpinene (0.1 percent)
artemisia ketone (11.0 percent)
trans-sabinene hydrate (0.2 percent)
artemisia alcohol (0.4 percent)
cis-sabinene hydrate (0.2 percent)
isophorone (0.7 percent)
α-campholenal (0.5 percent)
trans-pinocarveol (9.0 percent)
pinacarvone (15.8 percent)
borneol (0.5 percent)
terpinen-4-ol (0.6 percent)
α-terpineol (0.1 percent)
myrtenal (0.7 percent)
myrtenol (0.4 percent)
trans-carveol (0.1 percent)
tridecene[*] (0.2 percent)
α-copaene (0.4 percent)
β-caryophyllene (2.6 percent)
coumarin (0.3 percent)
α-humulene (0.1 percent)
(E)-β-farnesene (0.7 percent)
germacrene D (6.1 percent)
bicyclogermacrene (0.3 percent)
(E,E)-α-farnesene (0.1 percent)

[*]correct isomer not identified

Trace amounts (< 0.1 percent) of furfural, hexanol, tricyclene, α-thujene, thuja-2,4(10)-diene, benzaldehyde, sabinene, santolina alcohol, phenylacetaldehyde, (E)-β-ocimene, acetophenone, o-guaiacol, terpinolene, nonanal, nopinone, pinocamphone, 4-methylacetophenone, p-cymen-8-ol, methyl salicylate, verbenone, *cis*-carveol, carvone, *trans*-myrtanol, indole, *trans*-pinocarvyl acetate, carvacrol, α-cubebene, eugenol, β-cubebene, β-elemene, (Z)-jasmone, tetradecane, α-cedrene, γ-muurolene, (E)-β-ionone, β-selinene, α-muurolene, γ-cadinene, δ-cadinene, *trans*-calamenene and (E)-nerolidol were also found in this oil.

Bouwmeester et al. (1999) determined that the sesquiterpene hydrocarbons found in an oil of *A. annua* were α-copaene, β-caryophyllene, (E)-β-farnesene, muurola-4,11-diene, selina-4,11-diene, germacrene D, β-selinene, bicyclogermacrene, germacrene A, γ-cadinene, α-humulene and amorpha-4,11-diene.

An oil produced from *A. annua* cultivated in the region of New Delhi (India) was analyzed by Ali and Siddiqui (1999). It revealed that the oil was rich in 1,8-cineole (12.8 percent). Thirty-three other components were supposedly characterized; however, as many were obviously identified in error, the data will not be reported. The analysis is only noted for completeness of a review on recently published material on this annual wormwood.

Lari-yazdi et al. (2002) screened a number of collections of *A. annua* from the wild in Iran. The oils, which were rich in monoterpenes, contained α-pinene (7.87-13.28 percent), 1,8-cineole (5.77-17.26 percent), artemisia ketone (1.44-6.97 percent), camphor (14.26-29.05 percent) and pinocarvone (3.64-6.97 percent). The major sesquiterpene hydrocarbons were β-caryophyllene (3.43-9.37 percent), germacrene D (3.07-6.26 percent) and β-selinene (0.46-10.36 percent). Numerous other components such as tricyclene, α-thujene, camphene, sabinene, β-pinene, myrcene, α-terpinene, p-cymene, *trans*-sabinene hydrate, artemisia alcohol, *cis*-sabinene hydrate, *trans*-pinocarveol, borneol, terpinen-4-ol, α-terpineol, myrtenol, verbenone, *cis*-carveol, *trans*-carveol, carvone, tridecane, benzyl butyrate, eugenol, α-copaene, β-cubebene, β-elemene, a jasmone isomer, β-caryophyllene, α-humulene, a β-farnesene isomer, an acoradiene, aromadendrene, germacrene D, β-selinene, bicyclogermacrene, γ-cadinene, δ-cadinene, eugenyl acetate, a bisabolene isomer, (E)-nerolidol, spathulenol, widdrol, a β-elemenone isomer, β-oplopanone, isocedrol, 5-cedranone, elemol, γ-eudesmol, T-cadinol, α-muurolol, α-eudesmol, α-cadinol, 5-isocedranol, tetradecanol, apiole, 8-cedran-13-ol, germacrone, (Z,E)-farnesol, 8-cedran-13-al, (Z,Z)-farnesol, (E,E)-farnesol, artemisinic acid, (Z)-α-santalyl acetate, (E,E)-farnesyl acetate, and 8,13-cedrane-diol.

Juteau et al. (2002) collected *A. annua* from a river bank near Marseilles (France). An oil produced from this plant was found to possess the following composition:

1,8-cineole (1.2 percent)
artemisia ketone (2.8 percent)
artemisia alcohol (0.2 percent)
trans-pinocarveol (10.9 percent)
camphor (43.5 percent)
pinocarvone (2.1 percent)
chrysanthenol* (0.3 percent)
borneol (0.2 percent)
terpinen-4-ol (0.3 percent)
myrtenal (0.1 percent)
α-terpineol (0.1 percent)
myrtenol (0.2 percent)
trans-pinocarvyl acetate (0.1 percent)
α-copaene (1.8 percent)
β-cubebene (0.1 percent)
β-caryophyllene (8.9 percent)
(E)-β-farnesene (0.1 percent)
germacrene D (15.6 percent)
β-selinene (9.4 percent)
bicyclogermacrene (0.3 percent)
germacrene A (0.7 percent)
γ-cadinene (0.1 percent)
T-cadinol (0.2 percent)

*correct isomer not identified

The composition of an oil of *A. annua* produced from plants collected in Gilan province (Iran) was the subject of analysis by Sefidkon et al. (2003). As a result of a combination of GC and GC/MS analysis, the oil was found to possess the following composition:

α-pinene (2.89 percent)
camphene (1.03 percent)
sabinene (0.65 percent)
limonene + 1,8-cineole (9.78 percent)
artemisia ketone (14.31 percent)
trans-sabinene hydrate (0.81 percent)
artemisia alcohol (0.78 percent)
terpinolene (3.81 percent)
camphor (8.11 percent)
trans-pinocarveol (7.75 percent)
trans-verbenol (0.47 percent)
cis-limonene oxide (2.69 percent)
pinocarvone (9.07 percent)
borneol (1.97 percent)
terpinen-4-ol (1.30 percent)
myrtenal (0.99 percent)
myrtenol (1.02 percent)
α-copaene (2.26 percent)
β-cubebene (0.60 percent)
β-caryophyllene (5.50 percent)
(Z)-β-farnesene (0.57 percent)
γ-muurolene (7.11 percent)
β-selinene (8.90 percent)
caryophyllene oxide (0.99 percent)

The main constituents of an oil of *A. annua* produced from plants collected in the Farahabad region (Iran) were determined by Rasooli et al. (2003) to be:

α-pinene (12.1 percent)
camphene (2.8 percent)
sabinene (2.5 percent)
β-pinene (1.4 percent)
myrcene (3.7 percent)
1,8-cineole (9.8 percent)
artemisia ketone (24.2 percent)
artemisia alcohol (0.6 percent)
linalool (0.5 percent)
camphor (8.4 percent)
borneol (6.0 percent)
terpinen-4-ol (0.5 percent)
α-terpineol (0.2 percent)
α-copaene (0.2 percent)
β-caryophyllene (3.5 percent)
germacrene D (2.5 percent)
α-selinene (7.5 percent)

Bagchi et al. (2003) compared the composition of the oils produced from different plant parts during different growth periods of *A. annua* grown in Srinigar (India). A summary of the results of this comparative study can be seen in Table I.

Vaze (2003) reported that production of *A. annua* oil in India was ca. 1 tonne. Production was centered in Jammu and some regions of Himachal Pradesh. He also determined that the oil composition of a commercially available Indian oil was as follows:

ethyl 2-methyl butyrate (0.27 percent)
(Z)-3-hexenol (0.02 percent)
santolinatriene (0.18 percent)
ethyl tiglate (0.03 percent)
tricyclene (0.06 percent)
α-thujene (0.04 percent)
α-pinene (3.83 percent)
propyl 2-methyl butyrate (0.10 percent)
camphene (1.02 percent)
sabinene (0.90 percent)
β-pinene (0.57 percent)
3-octanone (0.04 percent)
myrcene (0.14 percent)
dehydro-1,8-cineole (0.14 percent)
yomogi alcohol (1.25 percent)
p-cymene (0.50 percent)
limonene (0.80 percent)
1,8-cineole (12.07 percent)
santolina alcohol (0.36 percent)
artemisia ketone (38.51 percent)
trans-sabinene hydrate (0.45 percent)
(Z)-3-hexenyl isobutyrate (0.07 percent)
cis-linalool oxide (furanoid) (0.08 percent)
artemisia alcohol (4.85 percent)
trans-linalool oxide (furanoid) (0.12 percent)

Percentage composition of oils produced from *Artemisia annua* from different plant parts **T-1**

	Leaf oils				Flower oils		
Compound	**EV**	**LV**	**B**	**F**	**F**	**FI**	**FF**
α-pinene	2.3	2.2	1.2	0.4	0.1	0.3	-
α-thujene	0.9	1.2	2.2	0.2	1.8	t	-
camphene	0.3	0.6	0.8	10.7	28.4	15.1	4.5
β-pinene	0.2	0.4	-	-	-	-	-
sabinene	0.5	-	-	-	-	-	-
myrcene	0.1	0.3	0.4	0.4	0.2	1.2	0.1
1,8-cineole	2.0	20.1	8.2	1.8	0.4	2.8	0.6
β-ocimene*	0.3	0.5	1.8	0.5	t	-	-
γ-terpinene	1.4	1.2	0.8	0.3	0.1	-	-
p-cymene	-	0.3	-	-	0.1	-	-
artemisia ketone	0.1	0.1	0.2	0.2	0.1	0.2	0.2
6-methyl-5-hepten-2-one	0.2	0.5	0.4	0.2	0.1	0.2	0.3
3-octanol	0.1	1.1	0.8	0.5	1.0	0.3	1.5
trans-sabinene hydrate	0.4	0.4	0.2	0.1	0.1	t	-
artemisia alcohol	6.1	8.3	6.5	0.2	8.9	4.4	8.8
camphor	19.6	18.6	44.4	10.5	31.5	34.9	26.0
linalool	0.6	0.6	0.2	0.2	0.2	0.1	0.1
terpinen-4-ol	0.4	0.5	0.7	5.4	0.1	2.8	3.9
β-caryophyllene	0.3	0.4	0.4	2.8	0.1	2.8	3.8
α-terpineol	0.8	0.4	1.3	0.3	t	0.3	0.1
borneol	1.6	4.2	13.3	2.2	t	0.2	0.1
germacrene D	10.9	9.1	1.5	2.4	7.3	3.4	7.7
β-eudesmol	0.7	0.9	0.3	0.4	0.2	0.1	0.1

*correct isomer not identified; t = trace (< 0.1 percent); EV = early vegetative stage, LV = late vegetative stage, B = budding stage, F = flowering stage, FI = fruit initiation stage, FF = full fruiting stage

linalool (1.82 percent)
α-thujone (0.19 percent)
β-thujone (0.06 percent)
trans-pinocarveol (2.83 percent)
camphor (11.19 percent)
pinocarvone (0.22 percent)
borneol (1.25 percent)
terpinen-4-ol (0.68 percent)
p-cymen-8-ol (0.09 percent)
α-terpineol (0.54 percent)
myrtenal (0.44 percent)
myrtenol (0.28 percent)
verbenone (0.31 percent)
carveol* (0.25 percent)
bornyl formate (0.06 percent)
(Z)-3-hexenyl 2-methylbutyrate (0.10 percent)
carvone (0.08 percent)
linalyl acetate (2.59 percent)
myrtenyl acetate (0.23 percent)
isobornyl acetate (0.10 percent)
lavandulyl acetate (0.18 percent)
α-copaene (0.53 percent)
geranyl acetate (0.08 percent)
benzyl 2-methylbutyrate (0.35 percent)
β-elemene (0.14 percent)
β-caryophyllene (0.74 percent)
α-humulene (0.06 percent)
(Z)-β-farnesene (0.08 percent)
geranyl tiglate (0.12 percent)
β-selinene (1.17 percent)
caryophyllene oxide (2.06 percent)
humulene epoxide* (0.17 percent)
β-eudesmol (0.11 percent)

*correct isomer not identified

Scotch Spearmint Oil

Umemoto and Nagasawa (1980) analyzed an oil of Scotch spearmint that was produced experimentally from plants cultivated in Japan. This oil was found to contain the following constituents:

α-pinene (0.8 percent)
β-pinene (1.0 percent)
myrcene (0.7 percent)
limonene (16.5 percent)
1,8-cineole (2.6 percent)
3-octyl acetate (0.6 percent)
3-octanol (1.3 percent)
(Z)-3-hexenyl isovalerate (0.1 percent)
trans-sabinene hydrate (0.2 percent)
menthone (0.2 percent)
linalool (1.0 percent)
β-bourbonene (0.5 percent)
β-caryophyllene (0.4 percent)
menthol (0.2 percent)
dihydrocarvone* (5.0 percent)
dihydrocarvyl acetate (2.0 percent)
neodihydrocarveol (1.6 percent)
dihydrocarveol (1.2 percent)
carvone (60.5 percent)
trans-carvyl acetate (0.1 percent)
cis-carvyl acetate (0.2 percent)
δ-cadinene (0.1 percent)
γ-cadinene (0.1 percent)
trans-carveol (0.1 percent)
cis-carveol (0.2 percent)

*correct isomer not identified

Trace (< 0.1 percent) amounts of camphene, p-cymene, borneol and (Z)-jasmone were also found in the same oil.

An oil of Scotch spearmint was found (Shimizu et al. 1990) to contain the following major components:

α-pinene (1.2 percent)
β-pinene (1.1 percent)
sabinene (0.9 percent)
myrcene (1.5 percent)
limonene (19.7 percent)
1,8-cineole (2.8 percent)
3-octanol (3.1 percent)
carvone (54.6 percent)
trans-carveol (5.8 percent)
cis-carveol (1.7 percent)

In addition, *trans*-sabinene hydrate, linalool, *cis*-sabinene hydrate, terpinen-4-ol, borneol, neodihydrocarveol and dihydrocarveol were also found as minor constituents of this same oil. The authors also determined that the β-D-glucosides of 3-octanol, dihydrocarveol, neodihydrocarveol, *cis*-carveol and *trans*-carveol were constituents of the above-ground plants of Scotch spearmint.

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Odor description of some of the pyridines characterized in Scotch spearmint oil **T-2**

Pyridine	Odor description
2-acetyl-4-isopropenyl	slightly roasted, soft brown fermented odor, sweet terpenic undertone
4-isopropenyl	slightly sweet, green-bitter, nutty-beany odor
4-isopropenyl-2-methyl	slightly nutty, herbal, bitter character
2,4-diisopropenyl	hay-like, sweet, slightly brown, well-rounded odor
2-isopropyl-4-methyl	earthy-green, somewhat sour citrus
4-isopropyl-2-methyl	amine-like, ozone-green violet/perilla character
2-acetyl-4-isopropyl	sweet, spearmint-like rounded odor
4-acetyl-2-isopropenyl	weak herbal green fermented roasted notes
2-acetyl-4-isopropenyl	sweet-grassy, minty with amber-like undertones

Tsuneya et al. (1993) extracted 49 kg of Midwest Scotch spearmint oil three times with 7.5 kg 1 NHCl. From this acidic extract 2-8 g of basic components were produced (or 10.2 ppm of the original oil). Through the use of GC, GC/MS, IR and ^{1}H-NMR of subfractions of the basic fraction the basic compounds identified were 2-acetyl-4-isopropenylpyridine, 2-methylpyridine, 2-acetylpyridine, 3-[(E)-1-buten-1-yl]-4-propylpyridine, 3-[(E)-1-buten-1-yl]pyridine, 3-[(Z)-1-buten-1-yl]pyridine, 5-[(E)-1-buten-1-yl]-2-propylpyridine, 5-[(Z)-1-buten-1-yl]-2-propylpyridine, 2,4-diisopropenylpyridine, 2,6-dimethylpyridine, 3-ethylpyridine, 4-isopropyl-2-methylpyridine, 5-phenyl-2-propylpyridine, 3-phenylpyridine, pyridine, 2-acetyl-4-isopropylpyridine, 4-acetyl-2-isopropenylpyridine, 2-butylpyridine, 3-butylpyridine, 4-butylpyridine, 2-ethyl-4-isopropenylpyridine, 5-ethyl-2-methylpyridine, 2-ethylpyridine, 4-isopropenyl-2-methylpyridine, 2-isopropyl-4-methylpyridine, 2-pentylpyridine, 3-phenyl-4-propylpyridine, 3-propylpyridine, 4-propylpyridine, quinoline, 3-benzylpyridine, 4-isopropenylpyridine, 3-methylpyridine, 2-propylpyridine, 3-vinylpyridine, 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine and methyl anthranilate. The odor characters of a few of these heterocyclic nitrogen compounds can be seen in Table II.

Platin et al. (1994) determined that the key components of an oil of Scotch spearmint were as follows:

α-pinene (0.63 percent)
β-pinene (0.68 percent)
sabinene (0.42 percent)
myrcene (0.92 percent)
limonene (18.09 percent)
γ-terpinene (1.35 percent)
isomenthone (1.62 percent)
dihydrocarvone* (0.86 percent)
carvone (72.72 percent)
β-bourbonene (0.98 percent)
β-caryophyllene (1.24 percent)
β-cadinene† (0.49 percent)

*correct isomer not identified; †incorrect identification, probably germacrene D

Shi et al. (1996) analyzed a number of samples of Scotch spearmint oil, the origins of which were possibly Chinese although they were not disclosed. The oils were found to possess the following major constituents:

β-pinene (0.8-4.2 percent)
limonene (15.9-33.7 percent)
3-octanol (0.5-2.0 percent)
menthone (0-2.4 percent)
menthol (0.1-4.5 percent)
carvone (49.6-70.7 percent)

One of the oils was reported to contain α-terpinyl acetate (an unusual Scotch spearmint oil constituent) while another contained isovaleraldehyde (0.5 percent).

Tsuneya et al. (1998) determined the acids and phenols found in Scotch spearmint oil. Starting with 49 kg of Midwest oil they isolated a fraction (0.048 percent of the total oil) containing acids and another fraction (0.072 percent of the total oil) containing phenols. The compounds that were characterized in the acid fraction were:

acetic acid (0.07 percent)[a]
propionic acid (0.11 percent)
isobutyric acid (0.74 percent)
butyric acid (0.18 percent)
2-methylbutyric acid (4.50 percent)
isovaleric acid (3.80 percent)
valeric acid (0.21 percent)
hexanoic acid (5.38 percent)
tiglic acid (0.02 percent)
(Z)-3-hexenoic acid (0.55 percent)
heptanoic acid (0.37 percent)
(E)-2-hexenoic acid (4.98 percent)
(Z)-4-heptenoic acid (0.07 percent)
(Z)-5-heptenoic acid (0.03 percent)

octanoic acid (4.42 percent)
(Z)-5-octenoic acid (0.07 percent)
(Z)-3-octenoic acid (0.06 percent)
nonanoic acid (1.38 percent)
cis-2-pentylcyclopropane-1-carboxylic acid (2.98 percent)
citronellic acid (0.08 percent)
decanoic acid (0.05 percent)
benzoic acid (7.63 percent)
neric acid (0.14 percent)
geranic acid (0.23 percent)
phenylacetic acid (1.23 percent)
salicylic acid (32.07 percent)
3-isopropenylpentane-1,5-dioic acid (7.22 percent)
perillic acid (0.24 percent)
3-isopropenyl-6-oxoheptanoic acid (3.06 percent)
6-hydroxycarvone

[a]percent of the 0.048 percent of the acidic fraction

In addition, trace amounts of 2-ethylhexanoic acid, (Z)-3-heptenoic acid. (E)-2-octenoic acid, *trans*-2-pentylcyclopropane-1-carboxylic acid and (E)-2-nonenoic acid were also found in the same acidic fraction of the oil. The components that were characterized in the phenolic fraction were:

salicylaldehyde (0.20 percent)[a]
methyl salicylate (2.03 percent)
guaiacol (0.17 percent)
o-cresol (1.24 percent)
p-cresol (0.02 percent)
m-cresol (0.02 percent)
eugenol (72.97 percent)
carvacrol (0.13 percent)
thymol (0.15 percent)
4-vinylphenol (2.44 percent)
dehydrocarvacrol (2.41 percent)
vanillin (0.05 percent)

[a]percent of the 0.072 percent of the phenolic fraction

Lawrence (2000) analyzed a number of samples of Scotch spearmint oil produced both in the midwest and far west United States, and some oils produced in Canada. A summary of the variability of selected major components can be seen in Table III. Furthermore, Lawrence (2001) analyzed a number of commercial samples of Scotch spearmint oil their compositions are summarized as follows:

α-pinene (0.31-0.74 percent)
β-pinene (0.36-0.78 percent)
sabinene (0.29-0.51 percent)
myrcene (0.55-1.25 percent)
α-terpinene (< 0.01-0.10 percent)
limonene (11.85-18.50 percent)
1,8-cineole (0.97-1.57 percent)
γ-terpinene (<0.01-0.15 percent)
p-cymene (< 0.01 percent)
terpinolene (< 0.01 percent)
3-octyl acetate (0.24-0.27 percent)
3-octanol (1.57-2.35 percent)
trans-sabinene hydrate (< 0.01-0.20 percent)
menthone (1.07-1.36 percent)
β-bourbonene (0.74-1.14 percent)
β-caryophyllene (0.53-0.91 percent)
cis-dihydrocarvone (1.19-4.64 percent)
trans-dihydrocarvone (0.16-0.61 percent)
menthol (0.14-0.27 percent)
dihydrocarvyl acetate (0.60-0.80 percent)
α-terpineol (< 0.01-0.15 percent)
germacrene D (0.15-0.31 percent)
carvone (61.40-70.81 percent)
cis-carvyl acetate (0.18-0.24 percent)
trans-carveol (0.27-0.40 percent)
cis-carveol (0.29-0.38 percent)
(Z)-jasmone (< 0.01-0.11 percent)
viridiflorol (0-0.05 percent)

Coleman et al. (2002) used a non-equilibrated solid-phase microextraction (SPME) system to examine the volatiles of freshly distilled oils of Scotch spearmint. The compounds that were found in the headspace of these oils were as follows:

acetaldehyde (0.005 percent)
dimethyl sulfide (0.067-0.164 percent)
isobutanal (0.005-0.069 percent)

Comparative percentage composition of North American Scotch spearmint oil **T-3**

Compound	Midwest	Farwest	Canadian
myrcene	0.8-1.1	0.9-1.4	0.5-1.3
limonene	13.7-17.3	12.8-21.7	8.6-21.4
1,8-cineole	1.4-1.6	1.0-2.1	0.7-2.0
3-octyl acetate	0.1-0.2	0.1-0.3	0.1-0.2
3-octanol	2.1-2.7	1.7-4.3	1.7-3.4
trans-sabinene hydrate	1.0-1.4	0.1-1.4	0.1-0.3
β-bourbonene	0.9-1.2	0.5-1.4	0.6-1.4
carvone	65.8-71.6	59.9-74.2	64.4-78.6
trans-carveol	0.1-0.2	0.1-0.2	0.1-0.2
cis-carveol	0.3-0.5	0.2-0.4	0.2-0.5
No. of Samples	30	20	65

2-methylbutanal (0.142-0.433 percent)
2-ethylfuran (0.061-0.135 percent)
valeraldehyde (0.005 percent)
methyl 2-methylbutyrate (0.005-0.227 percent)
α-pinene (3.672-4.003 percent)
α-thujene (0.005-0.207 percent)
cis-2,6-diethyltetrahydrofuran (0.005-1.012 percent)
β-pinene (3.064-3.325 percent)
sabinene (1.775-2.242 percent)
myrcene (3.367-3.640 percent)
α-terpinene (0.107-0.228 percent)
limonene (42.196-48.943 percent)
1,8-cineole (4.850-6.272 percent)
(E)-2-hexenal (0.005-0.469 percent)
(Z)-β-ocimene (0.029-0.051 percent)
γ-terpinene (0.120-0.315 percent)
(E)-β-ocimene (0.005-0.121 percent)
p-cymene (0.005-0.057 percent)
3-octanol (3.515-4.436 percent)
menthone (1.208-1.424 percent)
trans-sabinene hydrate (0.005-0.195 percent)
menthofuran (0.005 percent)
pulegone (0.005 percent)
menthol (0.005 percent)
carvone (23.512-30.632 percent)

The authors noted that isomenthone (0.173-0.222 percent), menthyl acetate (0.005-0.048 percent), neomenthol (0.246-0.341 percent) and terpinen-4-ol (0.005-0.169 percent) were also found as constituents. However, it is unlikely that these four compounds were correctly characterized so they have been omitted from the list of constituents identified.

Kubeczka and Formacek (2002) examined an oil of Scotch spearmint that was produced in the United States using a combination of capillary GC and ^{13}C-NMR. The oil composition was found to be as follows:

2-methylbutanal (0.02 percent)
3-methylbutanal (0.05 percent)
α-pinene (0.65 percent)
α-thujene (0.04 percent)
trans-2,5-diethyltetrahydrofuran (0.10 percent)
β-pinene (0.63 percent)
sabinene (0.40 percent)
myrcene (0.81 percent)
α-terpinene (0.04 percent)
limonene (16.36 percent)
1,8-cineole (1.13 percent)
(Z)-β-ocimene (0.03 percent)
γ-terpinene (0.07 percent)
(E)-β-ocimene (0.04 percent)
p-cymene (0.02 percent)
terpinolene (0.04 percent)
3-octyl acetate (0.19 percent)
(Z)-3-hexenol (0.06 percent)
3-octanol (1.86 percent)
menthone (0.06 percent)
trans-sabinene hydrate (0.97 percent)
isomenthone (0.19 percent)
β-bourbonene (1.06 percent)
linalool (0.08 percent)
cis-sabinene hydrate (0.13 percent)
β-caryophyllene (0.86 percent)
terpinen-4-ol (1.12 percent)
cis-dihydrocarvone (1.42 percent)
trans-dihydrocarvone (0.19 percent)
γ-muurolene (0.35 percent)
(E)-β-farnesene (0.68 percent)
dihydrocarvyl acetate (0.34 percent)
α-terpineol (0.33 percent)
germacrene D (0.67 percent)
neodihydrocarveol (0.59 percent)
carvone (66.26 percent)
dihydrocarveol (0.25 percent)
cis-carvyl acetate (0.37 percent)
trans-carveol (0.43 percent)
cis-carveol (0.23 percent)
(Z)-jasmone (0.31 percent)
viridiflorol (0.24 percent)

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Laurel Leaf Oil and Extract

The main constituents of a laurel leaf oil produced from leaves collected in Poland were 1,8-cineole (60.1 percent) and α-terpinyl acetate (7.3 percent) (Gora et al. 1997).

An oil of laurel leaf that was used in an antifungal screening study against the bee chalkbrood fungus by Larran et al. (2001) was found to possess the following major components:

α-pinene (2.1 percent)
sabinene (4.2 percent)
β-pinene (2.0 percent)
limonene (0.9 percent)
1,8-cineole (29.3 percent)
linalool (31.3 percent)
β-caryophyllene (1.0 percent)

Caredda et al. (2002) compared the composition of an oil and a supercritical fluid (SFE) CO_2 extract of *Laurus nobilis* (laurel leaf). The authors showed that collection of samples at different extraction times allowed them to follow the changes in composition as extraction progressed. The leaves used in this study were collected from Porto Columbo (southern Sardinia, Italy). They were air-dried to a moisture content of 9.8 percent and ground to a particle size of 300-800 µm. Super-critical extraction was performed using a commercially available extraction system that used a 400 mL extraction vessel and two separators. Extraction was performed at 90 bar, 50°C with a CO_2 flow of 1.0 kg/h. During the extraction, the waxes were removed by trapping them in the first separators at 90 bar and -10°C. As expected, they comprised long chaim alkanes such as tricosane, pentacosane, octacosane, hentriacontane and triacontane. The composition of the SFE extract and an oil produced from the same batch of leaves by water distillation can be seen in Table IV. In addition, the authors also found trace amounts (< 0.2 percent) of 6-methyl-5-hepten-2-one, dehydro-1,8-cineole, ethyl hexanoate, α-phellandrene, (Z)-β-ocimene, *trans*-linalool oxide (furanoid), 2-nonanone, *cis*-p-menth-2-en-1-ol, endo-norbornyl acetate, *trans*-pinocarveol, borneol, p-cymen-8-ol, nerol, neryl acetate, geranyl acetate, β-cubebene, γ-muurolene, germacrene D, (Z)-nerolidol, α-calacorene and selin-11-en-4α-ol in both the extract and the oil. Trace amounts that were found exclusively in the extract were (E)-cinnamaldehyde, ascaridole, (E)-cinnamyl alcohol, aromadendrene and β-eudesmyl acetate, while p-mentha-1(7),8-diene was the only trace component found exclusively in the oil. Based on the data presented in Table IV, it would appear that as the oil and SFE were very similar, the increased cost in producing a supercritical fluid CO_2 extract of *L. nobilis* is not economically sound.

The headspace volatiles of bay leaves (*L. nobilis*) were determined by Diaz-Maroto et al. (2002) using solid-phase microextraction (SPME) coupled with GC/MS. The volatile components that were identified were:

α-pinene (0.8)[a]
sabinene (1.4)
β-pinene (0.3)
myrcene (0.2)
1,8-cineole (19.2)
linalool oxide[*] (0.2)
terpinolene (< 0.1)
linalool (10.8)
borneol (0.1)
terpinen-4-ol (0.6)
α-terpineol (1.1)
linalyl acetate (0.8)
bornyl acetate (0.1)
eugenol (0.4)
α-terpinyl acetate (12.3)
methyl eugenol (5.9)
β-elemene (1.4)
β-caryophyllene (1.4)
α-guaiene (0.7)
γ-cadinene (0.5)
elemicin (1.6)
diethyl phthalate[‡] (1.1)
β-eudesmol (0.6)

[a] = mg/kg of leaves; [*]correct isomer not identified; [‡]plasticizer contaminant

In a follow-up paper, Diaz-Maroto (2002) reported the results of an SPME-GC/MS headspace analysis of fresh bay leaves. In this study, the following components were identified:

α-pinene (0.85 percent)
sabinene (2.48 percent)
1,8-cineole (43.56 percent)
γ-terpinene (0.27 percent)
trans-sabinene hydrate (0.29 percent)
linalool (26.70 percent)
terpinen-4-ol (0.86 percent)
α-terpineol (4.95 percent)
nerol (0.09 percent)
linalyl acetate (0.80 percent)
bornyl acetate (2.27 percent)
eugenol (1.23 percent)
α-terpinyl acetate (11.75 percent)
methyl eugenol (3.12 percent)
spathulenol (0.41 percent)
β-eudesmol (0.29 percent)

Trace amounts (< 0.10 percent) of α-thujene, camphene, β-pinene, terpinolene, geraniol and elemicin were also found in the headspace.

Diaz-Maroto et al. also compared the volatile of bay leaves that were fresh, air-dried at room temperature, oven-dried at 45°C, frozen and freeze-dried using simultaneous-distillation and extraction (SDE) using methylene chloride as the solvent. As comparison of the results obtained from analyses of the SDE oils obtained from each drying process versus fresh leaves can be seen in Table V.

An oil of *L. nobilis* that was produced from leaves that were collected in Soukra (Tunisia) was analyzed by Bouzouita et al. (2003). The composition of this oil was found to be as follows:

α-thujene (0.5 percent)
α-pinene (7.8 percent)

Comparative percentage composition of a CO_2 extract and an oil of *Laurus nobilis* leaves **T-4**

Compound	SFE	Oil	Compound	SFE	Oil
α-thujene	0.43	0.53	eugenol	2.60	1.83
α-pinene	2.81	3.19	cyclosativene	0.79	t
camphene	0.39	0.51	hydrocinnamyl acetate	t	0.44
sabinene	4.30	4.23	α-copaene	0.31	t
β-pinene	2.57	2.72	β-elemene	1.18	0.66
myrcene	0.27	0.22	methyl eugenol	8.09	9.42
δ-3-carene	0.61	0.67	β-caryophyllene	1.36	0.92
α-terpinene	t	0.30	α-guaiene	0.82	t
o-cymene	t	0.23	(Z)-cinnamyl acetate	0.97	1.28
p-cymene	0.84	0.89	geranyl acetone	0.31	t
limonene	1.18	1.23	α-humulene	0.30	t
1,8-cineole	22.84	23.51	allo-aromadendrene	0.62	0.64
γ-terpinene	0.25	0.58	β-selinene	0.47	0.33
cis-linalool oxide (furanoid)	0.74	0.55	*cis*-β-guaiene	1.85	1.43
terpinolene	t	0.21	valencene	0.75	0.59
linalool	12.46	10.57	viridiflorene	0.63	0.43
trans-dihydro-α-terpineol	0.67	0.69	(E)-methyl isoeugenol	0.47	0.45
terpinen-4-ol	2.57	3.26	α-bulnesene	0.46	t
(Z)-3-hexenyl butyrate	0.29	-	γ-cadinene	0.42	0.37
α-terpineol	3.35	3.92	7-epi-α-selinene	1.01	0.79
linalyl acetate	1.02	0.38	elemicin	0.87	0.85
(Z)-cinnamyl alcohol	0.31	t	spathulenol	0.64	1.24
bornyl acetate	0.84	0.96	caryophyllene oxide	0.92	1.83
2-undecanone	t	0.32	β-oplopenone	t	0.33
neoiso(iso)pulegyl acetate	1.10	1.06	β-eudesmol	1.01	2.16
α-terpinyl acetate	11.36	10.79	bulnesol	0.62	0.93

t = trace (< 0.2 percent)

camphene (0.3 percent)
sabinene (5.4 percent)
β-pinene (5.9 percent)
α-phellandrene (0.7 percent)
δ-3-carene (0.1 percent)
α-terpinene (0.6 percent)
p-cymene (0.6 percent)
1,8-cineole (42.3 percent)
γ-terpinene (0.6 percent)
linalool (2.5 percent)
terpinen-4-ol (2.5 percent)
α-terpineol (2.1 percent)
bornyl acetate (0.4 percent)
α-terpinyl acetate (11.2 percent)
α-copaene (0.4 percent)
β-elemene (1.3 percent)
methyl eugenol (3.5 percent)
β-caryophyllene (1.3 percent)
allo-aromadendrene (0.3 percent)
α-humulene (0.2 percent)
germacrene D (0.8 percent)
δ-cadinene (0.8 percent)

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Spanish Oregano Oil

An oil of *Thymus capitatus* Hoffmanns et Link. (L.) [syn. *Coridothymus capitatus* (L.) Reichenb.] or Spanish oregano was produced from plants collected from their natural habitat on Mount Taygetos (Peloponnese, Greece) by Karpouhtsis et al. (1998). Analysis of this oil revealed that it contained the following constituents:

α-pinene (0.65 percent)
camphene (0.16 percent)

Comparative volatile composition of the oils of fresh and dried *Laurus nobilis*

T-5

Compound	Fresh leaf oil	Dried leaf oils 1	2	3	4
α-thujene	34.2[a]	37.2	36.0	25.6	22.5
α-pinene	338.2	355.3	350.0	193.4	165.7
camphene	83.4	87.0	78.7	16.5	14.9
sabinene	448.4	478.5	534.2	333.7	295.3
β-pinene	269.2	270.8	272.8	159.6	137.9
1,8-cineole	2515.8	2172.2	2349.4	1796.8	1621.2
γ-terpinene	35.7	35.1	32.7	33.0	27.8
trans-sabinene hydrate	32.4	35.0	38.2	28.2	26.0
terpinolene	11.7	10.8	11.2	10.9	9.4
linalool	1822.6	1708.3	1522.3	403.7	339.1
terpinen-4-ol	173.2	146.9	140.9	146.8	132.1
α-terpineol	306.7	278.6	324.3	96.1	86.3
nerol	21.9	23.3	22.8	15.4	13.0
geraniol	8.2	7.8	6.6	5.4	4.6
linalyl acetate	33.2	39.3	30.9	6.2	5.7
bornyl acetate	124.6	99.6	102.9	13.1	12.1
eugenol	222.5	451.0	445.0	431.7	445.5
α-terpinyl acetate	602.5	318.6	353.8	489.7	343.5
methyl eugenol	341.2	322.5	318.7	133.8	125.5
elemicin	32.0	30.6	30.8	75.3	64.2
spathulenol	41.8	35.3	37.7	117.9	105.5
β-eudesmol	27.3	23.2	23.6	70.2	54.5

[a] = mg/kg of leaves; 1 = air-dried (ambient), 2 = oven-dried (45°C), 3 = frozen, 4 = freeze-dried

β-pinene (0.07 percent)
sabinene (0.05 percent)
myrcene (0.94 percent)
α-phellandrene (0.06 percent)
α-terpinene (0.67 percent)
limonene (0.13 percent)
1,8-cineole (0.14 percent)
γ-terpinene (2.25 percent)
p-cymene (6.41 percent)
terpinolene (0.08 percent)
3-octanol (0.07 percent)
trans-sabinene hydrate (0.27 percent)
linalool (0.61 percent)
linalyl acetate (0.07 percent)
terpinen-4-ol (0.03 percent)
cis-sabinene hydrate‡ (0.13 percent)
β-caryophyllene (1.91 percent)
methyl carvacrol (0.03 percent)
trans-dihydrocarvone (0.06 percent)
isoborneol (0.61 percent)
α-terpineol (0.08 percent)
γ-elemene (0.06 percent)
β-bisabolene (0.05 percent)
γ-cadinene (0.04 percent)
p-cymen-8-ol (0.61 percent)
spathulenol (0.10 percent)
thymol (1.46 percent)
carvacrol (81.46 percent)

‡incorrect identification based on GC elution order

Arras and Usai (2001) examined the composition of *Th. capitatus* oil produced over three seasons. The range in composition found in the oil was as follows:

camphene (0-0.06 percent)
heptanol (0.08-1.66 percent)
β-pinene (0-0.15 percent)
3-octanone (0.65-0.68 percent)
myrcene (1.58-1.71 percent)
3-octanol (0.21-0.42 percent)
α-phellandrene (0.23-0.32 percent)
δ-2-carene (0-0.10 percent)
δ-3-carene (0.70-0.77 percent)
p-cymene (4.47-5.02 percent)
1,8-cineole (0.23-0.41 percent)
β-ocimene° (0-0.05 percent)
γ-terpinene (2.63-3.39 percent)
trans-sabinene hydrate (0.56-0.73 percent)
decahydronaphthalene‡ (0-0.08 percent)
linalool (1.19-1.29 percent)
borneol (0-0.16 percent)
terpinen-4-ol (0.55-1.27 percent)
α-terpineol (0-0.06 percent)
neral (0-0.21 percent)
carvone (0-0.08 percent)
thymol (0-0.16 percent)
carvacrol (80.98-83.50 percent)

β-caryophyllene (1.43-1.57 percent)
caryophyllene oxide (0-0.29 percent)

*correct isomer not identified; ‡not a naturally occurring constituent

An oil of *Th. capitatus* produced from plants collected in Greece was found be Daferera et al. (2002) to possess the following composition:

α-thujene (0.5 percent)
α-pinene (0.8 percent)
camphene (0.1 percent)
myrcene (1.1 percent)
α-terpinene (0.8 percent)
p-cymene (5.4 percent)
β-phellandrene (t)
γ-terpinene (2.6 percent)
terpinolene (0.2 percent)
linalool (0.4 percent)
borneol (0.2 percent)
terpinen-4-ol (0.5 percent)
α-terpineol (t)
thymol (0.2 percent)
carvacrol (81.5 percent)
β-caryophyllene (2.7 percent)

t = trace (< 0.1 percent)

Fleisher and Fleisher (2002) collected *Th. capitatus* along the Mediterranean coast in Israel, and oils produced from them were found to fall into three groups. An example of each of the three groups is shown in Table VI. As can be seen, the normally enountered carvacrol-rich oil is only one of the groups encountered. Because *Th. capitatus* oil is produced from plants collected in their wild state it can be seen that a reduction in the carvacrol content and an increase in the thymol content should not be unexpected in commercial Spanish oregano oils. Trace amounts (< 0.1 percent) of (Z)-3-hexenol, δ-3-carene, terpinolene, *trans*-dihydrocarvone, β-selinene, β-bisabolene, *cis*-calamenene, δ-cadinene, hexadecanoic acid and octadecanoic acid were found in all three example oils.

An oil of *Th. capitatus* that was produced from plants grown experimentally in Tunisia was analyzed by Hedhili et al. (2002). The components found in this oil were:

α-thujene (2.7 percent)
α-pinene (0.3 percent)
camphene (0.3 percent)
β-pinene (0.3 percent)
myrcene (1.7 percent)
α-phellandrene (0.3 percent)
α-terpinene (2.2 percent)
p-cymene (8.7 percent)
β-ocimene* (0.1 percent)
linalool (1.7 percent)
terpinen-4-ol (0.9 percent)
carvacryl acetate (0.1 percent)
β-caryophyllene (7.6 percent)
α-humulene (0.2 percent)
thymol (0.2 percent)
carvacrol (53.8 percent)
1-methyl-4-(5-methyl-1-methylene-4-hexenyl)cyclohexene‡ (0.3 percent)

Percentage composition of *Thymus capitatus* oils of Israeli origin **T-6**

Compound	Carvacrol-rich oil	Thymol-rich oil	Mixed oil	Compound	Carvacrol-rich oil	Thymol-rich oil	Mixed oil
α-thujene	0.2	0.2	0.3	carvone	t	-	t
α-pinene	0.3	0.5	0.2	methyl carvacrol	1.7	0.2	0.2
camphene	0.2	0.3	0.1	thymoquinone	0.3	1.3	1.0
sabinene	-	t	t	thymol	0.7	57.9	28.0
1-octen-3-ol	0.4	0.5	0.4	carvacrol	67.6	5.3	41.5
β-pinene	0.1	0.1	0.1	ascaridole	0.3	0.3	0.5
myrcene	0.4	1.1	0.8	eugenol	0.3	0.3	0.3
p-cymene	5.8	6.4	4.3	β-caryophyllene	1.8	1.2	2.3
limonene	0.2	0.4	0.3	aromadendrene	0.4	0.2	0.3
β-phellandrene	0.3	0.3	0.2	α-humulene	0.1	0.1	0.1
γ-terpinene	4.8	5.1	3.5	thymohydroquinone	0.4	1.2	1.4
trans-sabinene hydrate	0.3	0.5	0.3	spathulenol	0.5	0.6	0.4
linalool	0.5	0.6	0.6	caryophyllene oxide	0.2	2.4	1.8
borneol	0.9	1.4	0.8	humulene epoxide*	0.1	0.2	0.1
terpinen-4-ol	0.5	0.4	0.5	*cis*-coniferyl alcohol	0.7	0.4	0.3
α-terpineol	t	0.3	-	tetradecanoic acid	t	0.1	0.1
methyl thymol	0.3	0.4	0.3				

*correct isomer not identified; t = trace (< 0.1 percent)

cadina-1,4-diene (0.1 percent)
allo-aromadendrene (0.1 percent)
ledene (0.1 percent)

‡not a naturally occurring constituent; °correct isomer not identified

Trace amounts (< 0.1 percent) of δ-2-carene, limonene, bornyl acetate, a dihydrocarvone isomer, carvone, β-gurjunene and caryophyllene oxide were also found in the same oil. Furthermore, the authors examined the contents of four selected components in oils that were harvested monthly over a single year. They found variations such as γ-terpinene (2.8-16.4 percent), p-cymene (8.1-24.0 percent), β-caryophyllene (4.0-9.3 percent) and carvacrol (38.3-71.8 percent). The highest carvacrol level coincided with the highest oil content which was in August. As a result, this is the most cost effective month for commercial production of Spanish oregano oil in Tunisia.

An oil of *Th. capitatus* that was produced from plants collected from their natural habitat in Mornaguia (Tunisia) was the subject of analysis and antimicrobial screening by Bouzouita et al. (2003). The components identified in this oil were found to be:

α-thujene (0.7 percent)
α-pinene (0.3 percent)
camphene (0.1 percent)
1-octen-3-ol (0.1 percent)
β-pinene (0.1 percent)
myrcene (0.9 percent)
α-phellandrene (0.1 percent)
α-terpinene (1.0 percent)
p-cymene (4.0 percent)
β-phellandrene (0.2 percent)
γ-terpinene (5.3 percent)
trans-sabinene hydrate (0.1 percent)
terpinolene (0.1 percent)
linalool (1.0 percent)
borneol (0.4 percent)
terpinen-4-ol (0.7 percent)
thymol (0.2 percent)
carvacrol (81.4 percent)
carvacryl acetate (0.8 percent)
β-caryophyllene (2.2 percent)
(Z)-α-bisabolene (0.2 percent)
caryophyllene oxide (0.1 percent)

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