

### **Progress in Essential Oils**

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### Alaska Yellow Cedar Leaf Oil

On rare occasions, an oil of Alaskan cedar leaf (*Chamaecyparis nootkatensis* (D. Don) Spach.) is produced in North America and offered for sale commercially. A survey of the early literature reveals that the oil was thought to contain  $\alpha$ -pinene,  $\beta$ -pinene, sabinene, limonene and p-cymene (Gildemeister and Hoffmann, 1956).

Cheng and von Rudloff (1970) compared the composition of Alaskan cedar leaf oil produced from the leaves of young trees grown in a greenhouse with oils produced from a 50-55-year-old tree and that of a commercial oil. All oils were produced by steam distillation, although the distillation times were not given. The results of this comparative study are shown in **T-1**. Trace amounts (<0.1%) of  $\alpha$ -cubebene;  $\alpha$ -copaene; p-cymen-8-ol; isoprenyl senecoate; pentadecane; prenyl tiglate or angelate; nonadecane, tetradecanal, cedrol, heneicosane, abieta-7,13-diene, dehydroabietadiene, pentacosane, 8,13-diepi-manoyl oxide and docosanal were also characterized in one or all of these oils.

Andersen and Syrdal (1970) and Syrdal (1971) determined that a labdistilled needle oil of *C. nootkatensis* contained:

 $\begin{array}{l} \alpha \text{-pinene} \; (34.0\text{--}36.7\%) \\ \beta \text{-pinene} \; (2.0\text{--}2.3\%) \\ myrcene \; (4.0\%) \\ \delta \text{-}3\text{-carene} \; (42.5\text{--}44.4\%) \\ limonene \; (12.2\text{--}12.8\%) \end{array}$ 

In addition, the sesquiterpenes that were also characterized in this oil were  $\alpha$ -copaene,  $\alpha$ -ylangene, longifolene,  $\delta$ -cadinene,  $\gamma$ -cadinene, ar-curcumene,  $\beta$ -curcumene,  $\gamma$ -curcumene,  $\beta$ -bisabolene,  $\alpha$ -alaskene (syn. acora-4,7-(11)-diene,  $\beta$ -alaskene (syn. acora-3,7(11)-diene) and isomers of calamenene,  $\beta$ -farnesene and nerolidol. He also characterized 10aH-muurola-4,6-diene that was later (Andersen et al., 1973) given the trivial name 10-epi-zonarene.

Andersen and Syrdal (1970) reported the structure of  $\alpha$ - and  $\beta$ -alaskene and determined that the nerolidol found as a constituent of the needle oil of *C. nootkatensis* was (E)nerolidol.

Andersen et al. (1972) determined the absolute stereochemistry *cis*- and *trans*-calamenene and found that they were both components of Alaskan cedar needle oil.

The sesquiterpene fraction of the foliage oil of *C. nootkatensis* was determined (Banthorpe et al., 1977) to contain the following components:

A needle oil of yellow cedar was the subject of analysis by Lopes and Kolodziejczyk (2003). The oil was found to possess the following composition:

 $\begin{array}{l} \alpha \text{-pinene} \ (13.9\%) \\ \alpha \text{-fenchene} \ + \ camphene \ (1.3\%) \end{array}$ 

sabinene (0.1%)  $\beta$ -pinene (1.3%) myrcene (2.2%) δ-3-carene (30.8%)  $\alpha$ -terpinene (0.1%) p-cymene (0.2%) limonene +  $\beta$ -phellandrene (23.6%)  $\gamma$ -terpinene (0.2%) terpinolene (3.8%) nonanal (0.1%)camphor (0.6%) borneol (0.1%)terpinen-4-ol (0.5%)  $\alpha$ -terpineol (0.2%) citronellol (0.1%) piperitone (0.4%)(E)-anethole + U (0.1%) epi-cubebol + U (0.1%) (E)-nerolidol (0.2%) 1-epi-cubenol (0.1%)  $\alpha$ -cadinol (0.5%) abietatriene (1.1%)

U = unknown

Trace amounts (<0.05%) of tricyclene,  $\alpha$ -thujene,  $\alpha$ -phellandrene, 1,8-cineole, (E)- $\beta$ -ocimene, octanol,  $\alpha$ -fenchol, *trans*-p-menth-2-en-1-ol, p-cymen-8-ol, methyl thymol, methyl carvacrol,  $\gamma$ -muurolene,  $\alpha$ -muurolene and  $\alpha$ -muurolol were also found in this oil.

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#### **Ceylon Citronella Oil**

The so-called Ceylon citronella oil (known as *lenabata* in Sinhalese) is obtained by the steam distillation of *Cymbopogon nardus* (L.) Rendle (syn. *Andropogon nardus* L. var. *nardus*).

Mahalwal and Ali (2003) analyzed an oil of *C. nardus* produced from dried grass collected in the Nilgiris Hills near Ootakamond (Tamil Nadu, India). The constituents characterized in this oil were as follows:

 $\alpha$ -pinene (0.5%)  $\beta$ -pinene (1.5%) myrcene (2.9%) limonene (0.2%)cis-sabinene hydrate (3.8%) linalool (0.7%) citronellal (29.7%) borneol (2.5%)isoborneol (1.1%) lavandulol (0.7%)  $\alpha$ -terpineol (0.5%) γ-terpineol\* (9.2%) nerol (1.5%) geraniol (24.2%) geranial (0.3%) eugenol (0.5%)  $\beta$ -elemene (0.2%)  $\beta$ -caryophyllene (2.2%)  $\alpha$ -humulene (0.3%) spathulenol (0.2%)  $\beta$ -selinene (0.8%) valencene isomer\*\* (0.2%) valencene (0.5%)  $\alpha$ -selinene (1.0%) selinene \*\* (0.3%) 3,3,5-trimethyl-1,5-heptadiene\*\*\* (0.7%) germacrene D-4-ol (1.5%) (E)-nerolidol (4.8%) hexadecanol (0.5%) arachidic (C<sub>21</sub>) acid\*\*\* (0.1%)

behenic (C22) acid<sup>\*\*\*</sup> (0.1%)bis-(2-ethylhexyl)-phthalate<sup>\*\*\*\*</sup> (2.0%)phthalate<sup>\*\*\*\*</sup> (1.7%)

\*does not occur naturally

\*\*\*incorrect identification based on GC elution order

A sample of Ceylon citronella oil was reported (Anon., 2005) to possess the following composition:

-1

bornylene (0.05%)tricyclene (1.34%) $\alpha$ -pinene (2.45%)

### Comparative percentage composition of the leaf oils of *Chamaecyparis nootkatensis*

Compound	1	2	3
$\alpha$ -pinene	33.8	33.0	38.8
camphene	0.3	0.2	0.5
β-pinene	2.4	2.6	2.0
δ-3-carene	36.5	25.3	30.8
myrcene	2.5	4.2	2.3
limonene	12.1	23.5	9.2
β-phellandrene	1.3	2.8	2.9
p-cymene	t	0.1	0.3
terpinolene	3.8	2.5	2.0
isoprenyl isovalerate <sup>a</sup>	0.2	0.3	0.4
nonanal	0.1	0.2	0.2
prenyl isovalerate <sup>b</sup>	0.1	0.1	0.2
3-methyl-3-butenyl tiglate or angelate*	t	0.w3	0.2
bornyl acetate	0.2	0.2	0.2
terpinen-4-ol	0.1	0.7	0.2
thujopsene	t	t	0.1
$\alpha$ -terpineol	0.5	0.1	0.5
lpha-terpinyl acetate	0.9	0.3	1.2
piperitone	0.1	0.2	0.1
heptadecane	t	0.1	t
$\gamma$ -cadinene + $\delta$ -cadinene	0.1	0.1	0.1
citronellol	t	0.1	0.1
ar-curcumene	-	0.1	t
calamenene*	t	0.1	0.1
benzyl isovalerate	0.1	t	0.1
2-phenethyl isovalerate	t	-	0.1
nerolidol*	0.3	0.1	0.4
benzyl tiglate, or angelate	0.1	t	0.1
nonanoic acid	-	-	0.1
bisabolol*	0.3	0.3	0.7
α-cadinol	0.1	t	0.3
isopimara-8(9),15-diene	0.1	0.1	0.1
2-phenethyl tiglate, or angelate*	0.1	t	0.2
isohibaene	0.1	0.3	0.1
sandarapimaradiene + isophyllocladene	t	0.1	0.1
isopimaradiene	t	0.1	0.1
tricosane	t	0.1	t
manoyl oxide + 13-epi-manoyl oxide	0.6	0.5	0.3
phyllocladene	0.1	0.1	0.1
8-epi-manoyl oxide	0.5	0.5	1.1
phyllocladan-16-ol	0.1	0.3	0.1

1 = foliage oil from trees grown in greenhouse

2 = foliage oil from 50-55 year-old trees

3 = commercial sample of foliage oil

\*correct isomer not identified

t = trace (<0.1%)

<sup>a</sup>also known as 3-methyl-3-butenyl isovalerate

<sup>b</sup>also known as 3-methyl-2-butenyl tiglate or angelate

<sup>\*\*</sup>correct isomer not identified

<sup>\*\*\*\*</sup>plasticizer does not occur naturally

camphene (8.87%) sabinene (0.25%) β-pinene (0.25%) myrcene (1.21%) δ-2-carene (0.19%)  $\alpha$ -phellandrene (0.29%)  $\alpha$ -terpinene (0.18%) p-cymene (0.20%) limonene (9.68%)  $\beta$ -phellandrene (0.65%) 1,8-cineole (0.20%) (E)-β-ocimene (1.79%) 2,6-dimethyl-5-heptenal (0.09%) γ-terpinene (0.15%) octanol (0.05%) terpinolene (0.95%) camphenilone (0.09%) linalool (0.65%) (E,E)-allo-ocimene (0.12%) cis-p-menth-2-en-1-ol (0.06%)  $\alpha$ -campholenal (0.02%) trans-p-menth-2-en-1-ol (0.06%) citronellal (2.23%) isopulegol (0.07%) exo-methyl camphenilol (0.30%) isoborneol (0.07%) borneol (5.49%) terpinen-4-ol (0.54%)  $\alpha$ -terpineol (1.06%) myrtenol (0.09%) decanal (0.11%) cis-piperitol (0.02%) citronellol (1.89%) neral (0.38%) geraniol (27.85%) geranial (0.66%) bornyl acetate (1.07%) geranyl formate (0.05%) myrtenyl acetate (0.02%) citronellyl acetate (1.08%) α-terpinyl acetate (0.04%) neryl acetate (0.03%) geranyl acetate (11.59%)  $\beta$ -bourbonene (0.02%) β-elemene (0.28%) methyl eugenol (0.15%)  $\beta$ -caryophyllene (1.35%) cis-a-bergamotene (0.18%)  $\beta$ -sesquiphellandrene (0.01%) (Z)- $\beta$ -farmesene (0.04%) (Z)-methyl isoeugenol (0.03%)  $\alpha$ -cubebene (0.02%) α-humulene (0.18%)  $\beta$ -cubebene (0.02%) germacrene D (0.03%)  $\alpha$ -amorphene (0.18%) (Z,E)- $\alpha$ -farmesene (2.88%)(E)-methyl isoeugenol (0.94%) α-farnesene\* (0.41%) y-cadinene (0.38%)  $\delta$ -cadinene (0.78%) cadina-1,4-diene (0.01%)  $\alpha$ -cadinene (0.01%) elemicin (0.02%)geranyl butyrate (0.79%)

\*correct isomer not identified

## Ranade (2008) reported that the main constituents of Ceylon citronella oil range in composition as follows:

camphene (7–10%) limonene (7–11%) citronellal (3–6%) borneol (4–7%) citronellol (3–8.5%) geraniol (15–23%) methyl isoeugenol° (7–11%)

\*correct isomer not identified

Verma et al. (2009) compared the composition of oils produced in the laboratory by hydrodistillation from cultivars of *C. winterianus* over three seasonal times in the vicinity of Bageshwar (Uttarakhand, India). Examination of the data obtained revealed that the oil yield was highest (1.8%) during the summer harvest (May) compared to 1.3% for plants harvested in December (winter) and August (during monsoon season). A summary of the composite results of this study can be seen in **T-2**.

A commercial oil of citronella produced in Sri Lanka from *C. nardus* (L.) Rendle was the subject of analysis by Milchard et al. (2010). The components characterized in this oil were:

tricyclene (1.3%) α-pinene (2.3%) camphene (8.8%) myrcene (0.9%) limonene (9.3%) 1,8-cineole (0.5%) (Z)-β-ocimene (1.8%) (E)- $\beta$ -ocimene (1.1%) terpinolene (0.8%) linalool (0.7%) citronellal (4.5%) neoisopulegol (1.2%) borneol (5.9%)terpinen-4-ol (1.3%)  $\alpha$ -terpineol (1.3%) citronellol (3.5%)

neral (0.3%) geraniol (17.7%) geranial (0.4%) bornyl acetate (0.5%) citronellyl acetate (0.9%) geranyl acetate (3.1%) methyl eugenol (1.1%)  $\beta$ -elemene (1.0%)  $\beta$ -caryophyllene (1.9%) trans-α-bergamotene (0.9%) (E)-methyl isoeugenol (8.8%) germacrene D (1.6%)  $\alpha$ -farmesene\* (4.2%)  $\delta$ -cadinene (1.1%) elemol (1.6%) geranyl butyrate (1.1%)

\*correct isomer not identified

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#### **Spanish Oregano Oil**

The oil of Spanish oregano is obtained from Thymus capitatus (L.) Hoffm. et Link [syn. Coridothymus capitatus (L.) Reichenb. f.]. It has been valued for its richness in carvacrol, a compound whose antimicrobial activity has created a renewed interest in all carvacrol-rich oils. In addition to the fact that carvacrol is the main component of the desired chemotype of T. capitatus (oils rich in thymol are also known), it also contains minor amounts of other phenols. The other phenols were isolated by Kandil et al. (1994). In this study the author subfractioned an oil of T. capitatus of Egyptian origin. The phenols found were 2-methyl-5-isopropyl-phenol (carvacrol), 5-methyl-5-isopropylphenol, 3-methyl-2,4-di-isopropylphenol, 5-methyl-2,3-di-isopropyl-phenol, 3-methyl-2,3-di-isopropylphenol,

Comparative percentage composition of oils from five cultivars of *Cymbopogon winterianus* produced at three separate times of the year in Uttarakhand

Compound	'Manjusha' oil	'Mandakini' oil	'Jalpallavi' oil	'Bio-13' oil	'Medini' oil
myrcene	0.0–0.5	0.0–0.2	0.0–t	0.0–t	-
limonene	4.1–5.6	1.8–3.6	3.3–5.7	3.2-4.3	-
(Z)-β-ocimene	0.0–t	0.0–t	0.0–t	0.0–t	0.0–t
p-cymene	0.0–t	0.0–t	0.0–t	0.0–t	0.0–t
6-methyl-5-hepten-2-one	0.0–t	0.0–t	t–0.4	0.0–t	0.0–t
citronellal	28.3-37.3	27.7-34.8	34.9-40.0	35.4-42.5	11.0–18.9
linalool	0.6-0.8	0.6-0.8	0.6-0.8	0.6-0.8	0.7–0.8
linalyl acetate	0.1-0.2	t–0.3	0.1-0.3	t-0.2	t0.2
β-elemene	0.3-0.4	0.3–0.5	0.3–0.7	0.4-0.6	0.5–1.3
β-caryophyllene	0.2-0.6	0.4–1.3	t-0.2	0.1-0.3	0.0-0.1
citronellyl formate	0.0-0.1	-	-	-	0.0-0.2
citronellyl acetate	3.1-5.9	2.4-4.6	2.8-4.3	2.9-3.8	1.4–1.8
neral	0.0–t	0.0–t	0.0–t	0.0–t	5.8-8.1
α-terpineol	0.4-0.6	0.4–0.6	0.4–0.6	0.6-0.7	-
geranyl formate	0.6-1.5	0.8–1.8	0.5–1.0	0.5-1.0	0.2-0.4
borneol	t	0.0–t	t-0.2	t–0.1	0.0–t
geranial	0.4-0.8	0.5–1.0	0.5–0.8	t0.8	8.5–11.8
γ-cadinene	1.0-1.5	1.2-2.2	0.9–1.4	0.9–1.6	t0.2
geranyl acetate	4.4-9.3	4.1–5.3	5.3-5.9	4.6-6.4	4.9-6.0
citronellol	8.1-12.9	9.8-12.4	7.8–10.3	6.9–11.4	6.3–7.4
nerol	0.2-0.3	0.3–0.4	0.2-0.3	0.3-0.5	0.3–0.4
geraniol	20.9-23.5	18.8-23.2	18.6–23.4	18.4–24.7	41.6-45.7
caryophyllene oxide	0.0-2.2	0.0-2.0	0.0-1.9	0.0-1.8	0.0-0.5
elemol	5.5-9.6	5.6-8.1	3.7–7.1	3.3-7.6	1.8-5.2
$\alpha$ -cadinol	-	-	-	-	-
t = trace (<0.1%)					

5-methyl-2-isopropyl-phenol (thymol), 3-methyl-2,5-di-isopropylphenol, 2,5-di-isopropylphenol, 4-methyl-2,5di-isopropylphenol and 5-methyl-2,4di-isopropylphenol.

Hedhili et al. (2002) harvested plants of *T. capitatus* plants growing wild in the vicinity of Korba (Tunisia) monthly throughout the year. Oils isolated from each harvest time were examined for their main constituents. Over the year the oil yield ranged from a low of 0.13% in February to a high of 2.0% in August. The content of the four major constituents of the oil in February were found to be:

 $\begin{array}{l} \gamma \text{-terpinene} \ (7.1\%) \\ p \text{-cymene} \ (24.0\%) \\ carvacrol \ (38.3\%) \\ \beta \text{-caryophyllene} \ (9.0\%) \end{array}$ 

#### In contrast, the August oil contained:

 $\begin{array}{l} \gamma\text{-terpinene} \ (5.7\%) \\ p\text{-cymene} \ (8.1\%) \\ carvacrol \ (71.8\%) \\ \beta\text{-caryophyllene} \ (5.0\%) \end{array}$ 

In February, the plants are going through a dormancy period, whereas in August it is dry and hot and at the end of the flowering period.

The authors also analyzed an oil that was produced from plants harvested in June at the commencement of the flowering period. This oil, which as analyzed by GC-FID and GC/MS, was determined to contain the following constituents:

 $\alpha$ -pinene (0.3%) camphene (0.3%) sabinene (2.7%)  $\beta$ -pinene (0.3%) myrcene (1.7%) $\alpha\text{-phellandrene}\;(0.3\%)$  $\alpha$ -terpinene (2.2%) p-cymene (8.7%) γ-terpinene (16.4%)  $\beta$ -ocimene\* (0.1%) linalool (1.7%) terpinen-4-ol (0.9%) carvacryl acetate (0.1%) thymol (0.2%) carvaerol (53.8%) aromadendrene (0.1%)

ledene (0.1%) cadina-1(10),4-diene (0.1%) β-caryophyllene (7.6%) α-humulene (0.2%) β-bisabolene (0.3%)

\*correct isomer not identified

In addition, trace (<0.01%) amounts of  $\delta$ -3-carene, limonene, a dihydrocarvone isomer, carvone and caryophyllene oxide were also found in this same oil. The headspace volatiles obtained from *T. capitatus* collected from plants growing in their natural habitat in southern Italy was the subject of examination by D'Auria et al. (2005). Using SPME as their method of headspace trapping followed by GC/MS, the headspace was found to possess the following composition:

 $\begin{array}{l} \alpha \text{-thujene} \ (5.1\%) \\ \alpha \text{-pinene} \ (2.4\%) \\ \text{camphene} \ (0.5\%) \\ \text{sabinene} \ (1.1\%) \\ 3 \text{-octanone} \ (5.4\%) \\ \text{ethyl hexanoate} \ (1.3\%) \end{array}$ 

δ-3-carene (0.5%)  $\alpha$ -terpinene (5.5%) p-cymene (12.5%) 1,8-cineole (2.1%) (Z)-β-ocimene (0.1%) (E)-β-ocimene (0.5%) γ-terpinene (15.3%) trans-sabinene hydrate (2.8%) terpinolene (0.4%) linalool (2.6%) borneol (0.9%)  $\alpha$ -terpineol (0.8%) cis-dihydrocarvone (0.2%) cis-sabinene hydrate acetate\* (0.1%)neral\* (0.2%) geranial\* (0.3%) thymol (20.3%) carvacrol (0.1%) eugenol (0.1%) carvacryl acetate (0.1%) $\beta$ -caryophyllene (8.0%)  $\alpha$ -humulene (0.4%) allo-aromadendrene (17.0%)  $\gamma$ -elemene (0.1%) β-bisabolene (0.2%) (Z)- $\alpha$ -bisabolene (0.1%) caryophyllene oxide (0.2%)

\*incorrect identification based on GC elution order

Trace amounts (<0.05%) of 2-methylpentane, (E)-2-hexenal, thuja-2,4(10)-diene, methyl carvacrol, thymyl acetate,  $\alpha$ -gurjunene, (E)- $\beta$ -farnesene and  $\delta$ -cadinene were also found in the headspace.

Skoula and Grayer (2005) collected *T. capitatus* plants from Theriso (Crete, Greeece). Once dried they were hydrodistilled and the oil, which was produced in 4.6% yield, was analyzed by GC and GC/MS. The composition of this oil was found to be as follows:

 $\alpha$ -thujene (1.3%)  $\alpha$ -pinene (1.0%) camphene (0.5%) $\beta$ -pinene (0.2%) 3-octanone (0.1%) myrcene (2.1%)  $\alpha$ -phellandrene (0.1%) cis-dehydroxy linalool oxide<sup>f</sup> (0.1%) $\alpha$ -terpinene (1.8%) p-cymene (13.5%) limonene (0.4%)  $\beta$ -phellandrene (0.2%) γ-terpinene (7.4%) *cis*-sabinene hydrate (0.1%)terpinolene (0.2%) linalool (0.1%)trans-sabinene hydrate (0.4%) borneol (0.7%)terpinen-4-ol (3.5%)  $\alpha$ -terpineol (0.2%) thymoquinone (0.1%)

isobornyl acetate (0.1%)thymol (0.4%)carvacrol (62.6%) $\beta$ -caryophyllene (1.2%)<sup>f</sup>furanoid form

Skoula and Grayer (2005) also analyzed the oil produced from plants collected at a second site in Crete. This oil, which was produced in 2.79% yield, was found to be rich in p-cymene (19.6%),  $\gamma$ -terpinene (13.7%) and thymol (34.0%).

An oil produced from Tunisian *T. capitatus* collected at the end of the flowering stage in August was analyzed by Hedhili et al. (2005) using a combination of GC techniques. The components identified in this oil were:

 $\alpha$ -thujene (1.7%)  $\alpha$ -pinene (0.9%) camphene (0.4%) $\beta$ -pinene (0.2%) myrcene (1.9%)  $\alpha$ -phellandrene (0.4%)  $\alpha$ -terpinene (1.6%) p-cymene (10.0%) (E)-β-ocimene (1.2%)  $\gamma$ -terpinene (11.7%) linalool (2.6%) carvaerol (53.7%) isocaryophyllene (0.5%)  $\beta$ -caryophyllene (9.1%) aromadendrene (0.3%) germacrene D (0.4%)  $\beta$ -bisabolene (0.4%)  $\delta$ -cadinene (0.1%)  $\gamma$ -cadinene (0.3%) caryophyllene oxide (0.5%)

Bounatirou et al. (2006) collected aerial parts of T. capitatus plants during the season at vegetative, flowering and post-flowering development stages from three different locations, including Jendouba (interior north), Haouaria (littoral north) and Aintourine (littoral south) in Tunisia. Once the plant materials were air-dried they were hydrodistilled and the oils were analyzed by GC-FID and GC/MS. The authors found that oils produced from plants collected at different developmental stages showed chemical homogeneity regardless of the influence of extrinsic conditions such as collection site location, altitude, climate including temperature, humidity and rainfall. Examination of all of the data presented for the numerous

analyses the oil compositions can be seen summarized as follows:

α-thujene (t-1.8%) α-pinene (t-0.8%) camphene (t-1.0%) 1-octen-3-ol (t-0.2%) β-pinene (t-0.3%) myrcene (0.3-2.5%)  $\alpha$ -phellandrene (t-0.7%) δ-3-carene (t-0.2%)  $\alpha$ -terpinene (0.1–2.7%) p-cymene (3.0-17.0%) β-phellandrene (t–0.3%) limonene (0.1-0.5%) (Z)- $\beta$ -ocimene (t-0.1%) (E)-β-ocimene (t-0.1%) γ-terpinene (0.7-16.5%) trans-sabinene hydrate (t-0.6%) terpinolene (t-0.2%) cis-sabinene hydrate (t-0.2%) linalool (0.3-1.9%) borneol (0.2-1.7%) terpinen-4-ol (0.3-0.8%) thymol (0.3-12.7%) carvaerol (57.7-82.5%) eugenol (t-0.3%) carvacryl acetate (t-2.3%)  $\beta$ -caryophyllene (0.9–4.4%) aromadendrene (t-0.3%)  $\alpha$ -humulene (t-0.4%) viridiflorene (t-0.3%) β-bisabolene (t-0.4%) spathulenol (t-0.2%) caryophyllene oxide (0.1-1.2%)

Cultivated samples of *T. capitatus* were harvested from an experimental garden in Medenine (Tunisia) at two developmental stages by Zouari et al. (2007). Oils produced by hydrodistillation were analyzed by GC and GC/MS. The range in composition of the oils produced from all harvest times can be seen as follows:

α-thujene (0.4–1.9%) α-pinene (0.0–0.8%) myrcene (0.9–2.2%) α-terpinene (1.0–3.0%) p-cymene (8.3–14.9%) γ-terpinene (3.3–14.6%) linalool (0.9–1.9%) borneol (0.0–0.6%) terpinen-4-ol (0.7–1.0%) thymol (0.3–0.5%) carvacrol (47.4–66.3%) β-caryophyllene (1.8–4.7%) carvacryl acetate (0.0–2.4%) caryophyllene oxide (1.3–2.2%)

*Thymus capitatus* plants collected in the vicinity of Tetouan (northern Morocco) were hydrodistilled in the laboratory by El Ajjouri et al. (2008).

# Analysis of the oil by GC-FID and GC/MS revealed that it contained the following components:

α
-thujene (1.9%) α
-pinene (0.8%) camphene (0.1%) β
-pinene (2.5%) α
-phellandrene (0.3%) δ
-3
-carene (1.4%) p
-cymene (6.3%) γ
-terpinene (4.9%) terpinolene (0.1%) linalool (3.9%) borneol (0.9%) thymol (0.4%) carvacrol (70.9%) β
-caryophyllene (3.6%)

Galego et al. (2008) analyzed an oil produced from *T. capitata* [syn. *T. capitatus*, *Thymbra capitata* (L.) Griseb.] produced from plants obtained from an experimental garden in Algarve (Portugal). Using GC-FID and GC/MS as the method of analysis, the composition of the oils was found to be:

 $\alpha$ -thujene (1.5%)  $\alpha$ -pinene (0.6%) camphene (0.1%)  $\beta$ -pinene + sabinene (0.2%) myrcene (1.7%)  $\beta$ -terpinene\* (1.2%) limonene (2.6%) cis-sabinene\*\* (0.2%) (E)- $\beta$ -ocimene (4.8%) 1,8-cineole (0.3%)  $\gamma$ -terpinene (5.4%) terpinolene (0.1%)trans-sabinene hydrate (0.6%) linalool (0.7%)cis-sabinene hydrate (0.2%) terpinen-4-ol (0.6%) borneol (0.2%)

cis- $\alpha$ -terpineol<sup>\*\*</sup> (0.1%)  $\beta$ -caryophyllene (1.3%) thymol (0.2%) carvacrol (78.0%)

°does not exist naturally °° compound cannot exist

Marongiu et al. (2010) screened a supercritical  $CO_2$  extract of *T. capitatus* plants that were collected from their natural habitat in the vicinity of Sadali (Sardinia, Italy). The extract, which was analyzed using both GC-FID and GC/MS, was found to contain the following major components:

 $\begin{array}{l} p\text{-cymene} \ (2.2\%) \\ \gamma\text{-terpinene} \ (0.7\%) \\ linalool \ (1.0\%) \\ borneol \ (0.5\%) \\ carvacrol \ (87.8\%) \\ \beta\text{-caryophyllene} \ (2.3\%) \end{array}$ 

Trace amounts (<0.1%) of  $\alpha$ -pinene, camphene and  $\beta$ -pinene were also characterized in this extract.

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