Progress in Essential Oils

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Douglas Fir Needle Oil

There are seven species of *Pseudotsuga* found in their natural environment in eastern Asia and western North America (Airy Shaw, 1966). The taxonomic origin of Douglas fir is *Pseudotsuga menziesii* (Mirb.) Franco. This tree is highly valued in the timber industry of western North America because of its rapid growth habit.

According to Krüssman (1985), *P. menziesii* exists as a coastal form var. *menziesii* and a mountain form var. *glauca* (Beissn.) Franco. Numerous forms and cultivars of Douglas fir are known although no studies have been performed on the relationship between form or cultivar and essential oil composition.

A summary of the early literature reveals that the needle oil of *P. menziesii* was reported to contain camphene, limonene, α-pinene, β-pinene, borneol, bornyl acetate, geraniol, nerol, citral, furfurol, and a few acids, acetates and other esters (Gildemeister and Hoffmann, 1956).

Sakai et al. (1967) analyzed an oil produced from mature needles collected from trees found in the Pacific coast range west of Ukiah, California. Although 22 components were identified in the oil, no quantitative data were presented. Maarse and Kepner (1970) showed that there was some variation in essential oil components as the needles of Douglas fir growing in California matured. The components that ranged in composition were α-pinene, camphene, β-pinene, sabinene, δ-3-carene, myrcene, limonene, β-phellandrene, 1,8-cineole, an isomer of 2-hexenal, ethyl hexanoate, (Z)-β-ocimene, γ-terpinene, terpinolene, citronellal, linalool, α-fenchyl alcohol, bornyl acetate, terpinen-4-ol, β-caryophyllene, citronellyl acetate, α-terpineol, citronellol and geranyl acetate.

A comparison between the foliage oils of *P. menziesii* var. *menziesii* (coastal type) and *P. menziesii* var. *glauca* (mountain type) was studied

T-1 Comparative percentage composition of the oils of *Pseudotsuga menziesii* var. *menziesii* (PMM) and *P. menziesii* var. *glauca* (PMG)

Compound	PMM oil	PMG oil
2-hexenal*	0.2	0.1
santene	-	3.4
tricyclene	t	3.0
α-thujene + α-pinene[a]	13.1	15.7
camphene	0.4	25.5
β-pinene[a] + sabinene	46.3	8.5
myrcene	1.8	1.8
δ-3-carene	2.1	0.3
α-terpinene	2.0	0.5
limonene	1.5	6.4
β-phellandrene	1.4	0.4
(Z)-β-ocimene	0.1	-
γ-terpinene	4.3	1.2
terpinolene	11.4	3.1
linalool	0.2	0.1
α-fenchyl alcohol	0.1	t
camphene hydrate	t	0.1
borneol	t	0.1
terpinen-4-ol	6.7	3.1
α-terpineol	2.6	1.8
citronellol	0.9	0.1
bornyl acetate	0.2	24.3
citronellyl acetate	3.2	0.3
geranyl acetate	1.2	0.2
γ-cadinene + δ-cadinene	t	t
nerolidol*	t	t
α-cadinol + T-cadinol + α-muurolol + T-muurolol	0.1	0.1

*correct isomer not identified
t = trace (<0.1%)
[a]major component of mixed GC peak

by von Rudloff (1972). A summary of his findings are presented in **T-1**. Von Rudloff also found that the components of the needle oils of both varieties varied quantitatively from region to region and between trees. This variation was further examined by von Rudloff (1973a). He determined that the needle oils produced from the coastal Douglas fir and the Rocky mountain fir had intermediate types depending upon the tree origins. The oil compositions of the two types of each *Pseudostuga menziesii* varieties are shown in **T-2**. Von Rudloff (1973b) found that the compositions of needle oils of Douglas fir could be used to differentiate between populations of trees growing in British Columbia (Canada).

Ellison (1973) noted that longifolene, α-longipinene, β-elemene, *trans*-α-bergamotene, α-humulene, β-santalene, an α-bisabolene isomer, bisabola-3(15),7(14),10-triene, δ-cadinene and γ-cadinene were found in Douglas fir needle oil, according to Müller (unpublished). Ellison further examined a fraction of the oil of Douglas fir needles from which the monoterpene hydrocarbons had been removed. The components characterized in this fraction were *trans*-α-bergamotine, β-elemene, β-caryophyllene, terpinen-4-ol, α-humulene, α-terpineol, germacrene D, δ-cadinene, α-bisabolene isomer, p-cymen-8-ol, sibirene, citronellyl acetate and gernyl acetate; however, no quantitative data was given.

Kepner et al. (1975a) characterized the presence of germacrene D in Douglas fir needle oil. Kepner et al. (1975b) used a combination of analytical techniques to determine that an oil from mature needles of Douglas fir contained *trans*-α-bergamotene, an α-bisabolene isomer, bisabola-3(15),7(14),10-triene, γ-cadinene, δ-cadinene, β-caryophyllene, β-elemene, α-humulene, longifolene, α-longipinene, β-santalene and sibirene, whereas a young needle oil contained *trans*-α-bergamotene, an α-bisabolene isomer, δ-cadinene, β-caryophyllene, β-elemene, α-humulene and germacrene D in the sesquiterpene hydrocarbon fractions.

Von Rudloff and Rehfeldt (1980) analyzed the needle oils of 433 wild

T-2 **Percentage composition of the main components of two types of *Pseudotsuga menziesii* var. *menziesii* and *P. menziesii* var. *glauca***

Compound	*P. menziesii* var. *menziesii* oil A	*P. menziesii* var. *menziesii* oil B	*P. menziesii* var. *glauca* oil C	*P. menziesii* var. *glauca* oil D
santene	-	0.1–1.0	3–5	1–4
tricyclene	-	0.1–1.0	2.5–4.0	1–3
α-pinene	7–15	8–15	15–20	12–18
camphene	0.0–0.2	0.3–8.0	20–30	15–25
β-pinene	20–35	15–30	5–10	5–20
sabinene	2–15	2–12	0.1–0.5	0.5–5.0
α-terpinene	2–5	1–3	0.0–0.3	0.1–1.5
limonene	0.5–1.5	1–3	5–10	3–10
γ-terpinene	3–8	2–8	0.1–1.0	0.1–4.0
terpinolene	5–20	5–15	0.5–3.0	1–5
terpinen-4-ol	5–15	5–15	0.5–3.0	1–5
α-terpineol	1–3	1–3	0.2–1.0	0.5–2.0
citronellol	1–5	1–3	0.1–1.0	0.5–2.0
bornyl acetate	0.0–0.3	0.5–5.0	20–30	15–25
citronellyl acetate	2–4	2–6	0.1–2.0	1–3
geranyl acetate	1–3	2–5	0.1–1.0	0.5–2.0

A = Coastal type
B = Coastal intermediate type
C = Rocky Mountain type
D = Interior intermediate type

T-3 **Comparative percentage composition of the oils of *Pseudotsuga menziesii***

Compound	Commercial oil	Lab-distilled oil
santene	0.1	3.5
tricyclene	0.1	2.3
α-pinene	13.2	13.0
camphene	0.5	16.7
β-pinene	21.9	11.6
sabinene	19.4	7.4
δ-3-carene	8.9	2.0
myrcene	1.9	1.5
α-phellandrene	0.3	0.2
α-terpinene	2.2	2.0
limonene	1.6	3.4
β-phellandrene	1.4	0.8
γ-terpinene	3.4	3.2
p-cymene	0.7	0.1
terpinolene	13.6	9.1
p-cymenene	0.2	-
citronellal	-	0.8
bornyl acetate	-	10.0
terpinen-4-ol	3.8	4.6
citronellyl acetate	2.0	1.1
α-terpineol	0.4	0.7
α-terpinyl acetate + borneol	t	0.2
geranyl acetate	0.6	0.4
citronellol	1.0	2.1
geraniol	0.2	-

t = trace (<0.05%)

Percentage change in composition of Douglas fir needle oil as the needles matured **T-4**

Compound	1	2
tricyclene	2.8	3.9
α-pinene	17.5	15.8
camphene	13.9	28.5
β-pinene	30.6	14.4
myrcene	2.1	1.2
α-phellandrene	0.4	-
δ-3-carene	0.2	0.7
α-terpinene	0.6	-
p-cymene	1.1	0.1
limonene	16.5	12.2
(Z)-β-ocimene	1.8	1.4
γ-terpinene	0.2	-
(E)-β-ocimene	1.3	-
terpinolene	0.7	1.7
fenchone	0.2	0.3
linalool	0.2	2.1
camphor	0.2	0.3
borneol	0.6	0.5
terpinen-4-ol	0.3	0.3
citronellol	2.1	0.6
carvone	0.5	0.8
piperitone	0.6	0.2
β-caryophyllene	1.0	0.7
longifolene	0.6	2.1
methyl chavicol + α-terpineol	0.3	0.4

1 = oil produced from needles harvested June 6
2 = oil produced from needles harvested July 25

Douglas fir trees from 87 northwestern locations. They found that the components that varied the most were β-pinene, camphene, terpinolene/terpinen-4-ol and bornyl acetate in the oils of both Douglas fir varieties.

Kubeczka and Schultze (1987) analyzed a commercial sample of Douglas fir needle oil and compared it with that of a lab-distilled oil produced from twigs and needles collected from the Botanic Garden at the University of Würzburg (Germany). A summary of the analyses of these two oils is shown in **T-3**.

Changes in the composition of Douglas fir needle oils were studied by Wagner et al. (1989). They found that tricyclene, camphene, linalool and longifolene were major components of the oil that increased in amount as the needles matured, while many of the minor components decreased as reported in **T-4**.

Yskot and Coufalikova (1990) determined that a needle oil produced from *P. menziesii* grown in the former Czechoslovakia contained the following major components:

α-pinene (15.1–15.4%)
β-pinene (21.4–24.4%)
myrcene (2.8–2.9%)
phellandrene* (10.1–13.6%)
limonene (12.3–19.4%)
bornyl acetate (10.3–11.4%)

*correct isomer not identified

Buchbauer et al. (1994) analyzed an oil produced in the laboratory by distillation of the needles of Douglas fir harvested in Austria. The oil was analyzed using GCI-FID and GC-FTIR-MS. It was found to possess the following composition:

α-pinene (6.2%)
camphene (1.0%)
β-pinene (13.4%)
sabinene (15.4%)
δ-3-carene (4.3%)
myrcene (0.1%)
α-terpinene (4.1%)
α-phellandrene (0.1%)
limonene (3.2%)
β-phellandrene (0.3%)
(Z)-β-ocimene (0.1%)
γ-terpinene (4.2%)
(E)-β-ocimene (0.4%)
terpinolene (7.3%)
linalool (0.1%)
terpinen-4-ol (9.2%)
α-terpineol (1.4%)
citronellol (1.3%)
nerol (0.1%)
geraniol (0.3%)
borneol (0.3%)
(E)-anethole (0.8%)
carvone (0.2%)
carvacrol (0.1%)
bornyl acetate (3.1%)
citronellyl acetate (5.1%)
neryl acetate (0.9%)
geranyl acetate (3.6%)
farnesene* (0.2%)
β-caryophyllene (0.3%)
germacrene D (0.2%)
muurolene* (0.1%)
elemene* (0.2%)
α-humulene (0.2%)
cadinene* (0.8%)
elemol (1.0%)
3-hexenol* (0.2%)
isoeugenol* (0.2%)
methyl isoeugenol* (0.5%)
benzyl benzoate (1.4%)
decanol (1.0%)
isoamyl cinnamate (1.1%)

*correct isomer not identified

Trace amounts (<0.05%) of santene, tricyclene, α-thujene, 1,8-cineole, p-cymenene, α-fenchyl alcohol, citronellal, camphor, fenchone, piperitone, thymol, α-guaiene, longifolene, butanal, ethyl 2-methylbutyrate, a 2-hexenal isomer and eugenol. In addition, the authors noted that there were a number of uncharacterized acids and esters (4.2%) in the oil.

Gambliel and Cates (1995) examined the changes in needle oil composition during maturation of a single season's growth of the Rocky Mountain Douglas fir grown in the Barley Canyon, Cibola National Forest (New Mexico). They found that oils from bud break time through needle maturation accumulated α-pinene, tricyclene, camphene and bornyl acetate three- to tenfold as the needles matured. The components monitored by the authors were tricyclene, α-pinene, camphene, β-pinene,

Percentage composition of oils produced from Bulgarian *Pseudotsuga menziesii* T-5

Compound	1	2	3
(Z)-3-hexenol	0.1	-	0.1
hexanol	0.1	-	0.1
α-pinene	-	0.1	0.1
camphene	0.1	0.2	0.1
(Z)-3-hexenyl acetate	0.1	-	0.1
sabinene	21.0	22.2	29.7
β-pinene	39.5	24.4	31.2
myrcene	0.6	1.3	0.7
α-phellandrene	0.1	-	0.1
1,4-cineole	-	-	0.1
α-terpinene	0.9	1.8	0.6
p-cymene	0.1	0.6	0.1
1,8-cineole	0.1	-	-
limonene	0.4	1.0	0.4
(Z)-β-ocimene	7.1	5.6	4.9
β-phellandrene	0.5	1.0	0.5
(E)-β-ocimene	0.3	1.8	0.8
γ-terpinene	1.8	3.9	1.4
dihydromyrcenol	0.1	-	0.1
terpinolene	20.2	18.8	17.3
linalool	0.1	-	0.1
myrcenol	0.2	0.5	0.1
2-phenethanol	0.1	-	-
β-pinene oxide	0.1	-	0.1
α-fenchyl alcohol	0.2	-	0.1
isopulegol	-	-	0.1
camphor	0.1	-	0.1
trans-pinocarveol	0.1	-	-
citronellal	0.1	-	0.3
trans-verbenol	0.1	-	-
borneol	0.1	-	0.1
p-cymen-8-ol	0.1	-	0.1
α-terpineol	1.9	5.4	3.8
dihydrocarveol	0.1	-	0.1
octyl acetate	-	-	0.1
β-cyclocitral	0.1	-	0.1
carvone	0.1	-	0.2
geraniol	0.1	-	0.1
isopulegyl acetate	-	-	0.1
thymol	-	-	0.1
bornyl acetate	0.3	-	0.1
citronellyl acetate	0.5	5.9	2.2
α-longipinene	0.1	-	0.1
geranyl acetate	0.3	3.5	1.1
α-cubebene	0.1	-	-
α-copaene	-	-	0.1
decyl acetate	0.1	-	0.1
β-elemene	0.1	-	0.1
β-caryophyllene	0.1	-	0.1
(E)-β-farnesene	-	-	0.2
α-humalene	0.3	0.5	0.4
germacrene D	0.6	0.7	0.3
α-muurolene	0.1	-	0.1
δ-cadinene	0.2	-	0.2
caryophyllene oxide	0.1	0.6	0.4
α-muurolol	-	-	0.1

1 = fresh young needle oil
2 = fresh needles and twigs oil
3 = dried needles and twigs oil

myrcene, limonene, (E)-β-ocimene, terpinolene, citronellal, borneol, bornyl acetate, γ-cadinene and an isomer of α-bisabolene.

Jirovetz et al. (2000) sampled needles from Bulgarian grown *P. menziesii* from June to February and oils produced from these harvests were analyzed by GC-FID and GC/MS. The oil compositions were found to vary as follows:

(Z)-3-hexenol (0.3–2.7%)
hexanol (0.4–1.5%)
α-thujene (0.1–0.8%)
α-pinene (4.4–8.9%)
heptanol (0.1–0.5%)
camphene (0.3–1.4%)
(Z)-3-hexenyl acetate (0.1–1.3%)
sabinene (13.2–16.9%)
β-pinene (14.2–25.9%)
myrcene (0.9–2.3%)
α-phellandrene (0.8–2.1%)
1,4-cineole (t–0.3%)
α-terpinene (1.8–4.4%)
p-cymene (0.4–1.4%)
1,8-cineole (0.2–1.0%)
limonene (0.2–1.6%)
(Z)-β-ocimene (0.1–1.8%)
β-phellandrene (0.6–4.2%)
(E)-β-ocimene (0.2–1.5%)
γ-terpinene (2.3–5.7%)
dihydromyrcenol (0.0–0.1%)
terpinolene (6.4–16.2%)
linalool (0.5–1.6%)
myrcenol (0.0–0.2%)
2-phenethanol (0.0–0.6%)
β-pinene oxide (t–1.7%)
α-fenchyl alcohol (0.0–0.2%)
isopulegol (0.0–0.1%)
camphor (0.1–0.5%)
trans-pinocarveol (0.0–0.3%)
citronellal (0.4–3.0%)
trans-verbenol (0.0–0.2%)
borneol (0.0–0.2%)
p-cymen-8-ol (0.0–0.1%)
terpinen-4-ol (3.1–9.6%)
α-terpineol (0.9–5.1%)
dihydrocarveol (0.0–0.2%)
octyl acetate (0.0–0.1%)
β-cyclocitral (t–0.6%)
citronellol (1.3–3.9%)
carvone (0.0–0.1%)
geraniol (0.1–1.1%)
isopulegyl acetate (0.0–0.1%)
thymol (0.0–0.1%)
bornyl acetate (0.0–0.2%)
citronellyl acetate (1.5–4.8%)
α-longipinene (0.0–0.2%)
geranyl acetate (0.0–0.2%)
α-cubebene (0.0–0.2%)
α-copaene (0.0–0.2%)
decyl acetate (0.0–0.1%)
β-elemene (0.0–0.5%)
β-caryophyllene (0.1–0.9%)
(E)-β-farnesene (t–0.7%)
α-humulene (0.0–0.2%)
germacrene D (t–0.6%)
α-muurolene (0.0–0.2%)
δ-cadinene (0.0–0.8%)
caryophyllene oxide (t–0.2%)
α-muurolol (0.0–0.4%)

t = trace (<0.04%)

Stoyanova et al. (2001) analyzed the headspace volatiles of five essential oils produced from fresh needles and twigs that were distilled over different time periods. The components found by Jirovetz et al. (2000) were monitored in each of the oils. The main headspace volatiles were the same as those found in the oils.

Jirovetz et al. (2000) further examined the oils produced from fresh young needles, fresh needles and twigs, and air-dried needles and twigs of Bulgarian grown *P. menziesii.* The results of these analyses are listed in **T-5**. The authors also examined an oil produced from the bark of Douglas fir. The reason for examining the composition of the bark oil was not given other than to include it in a microbiological activity screening. The major components of the bark oil were:

sabinene (14.9%)
β-pinene (15.1%)
(Z)-β-ocimene (19.3%)
(E)-β-ocimene (15.4%)
terpinolene (11.3%)

Lopes and Kolodziejczyk (2003) analyzed an oil produced from Douglas fir needles of Western Canadian origin. They found that the oil contained the following components:

(E)-2-hexenal (0.3%)
tricyclene (0.1%)
α-thujene (1.8%)
α-pinene (9.4%)
α-fenchene + camphene (1.5%)
sabinene (19.2%)
β-pinene (21.7%)
myrcene (3.1%)
α-phellandrene (0.1%)
δ-3-carene (7.4%)
α-terpinene (0.9%)
p-cymene (1.0%)
limonene + β-phellandrene (4.2%)
(Z)-β-ocimene (0.2%)
(E)-β-ocimene (0.2%)
γ-terpinene (2.2%)
cis-sabinene hydrate (0.2%)
terpinolene (11.4%)
cis-p-menth-2-en-1-ol (0.2%)
citronellal (0.4%)
borneol (0.1%)
terpinen-4-ol (2.9%)
p-cymen-8-ol (0.3%)
α-terpineol (0.2%)
citronellol (0.8%)
(E)-anethole + U (0.7%)
citronellyl acetate + U (1.2%)
geranyl acetate (0.4%)
β-caryophyllene (0.2%)
α-humulene (0.3%)
β-selinene (0.1%)
α-muurolene (0.1%)
γ-cadinene + U (0.3%)
δ-cadinene (0.5%)
α-cadinol (0.1%)
manool (0.1%)

U = unknown

Trace amounts (<0.05%) of hexanol, α-copaene and α-cadinene were also found in this oil.

Rohloff and Langleite (2005) examined the monoterpene patterns of a number of oils of conifers, among which was *P. menziesii.* They found that this oil, which was produced in a commercial steam distillery in central Norway was found to contain the following components:

α-pinene (12.2%)
β-pinene (26.6%)
sabinene (11.7%)
δ-3-carene (4.5%)
myrcene (2.0%)

α-terpinene (2.9%)
limonene (1.7%)
β-phellandrene (1.6%)
γ-terpinene (4.7%)
terpinolene (12.5%)
terpinen-4-ol (7.8%)

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