Progress in Essential Oils

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Thyme Oil

The main thyme oil of commerce that is produced from *Thymus vulgaris* L. is obtained solely from the thymol chemotype.

An oil of *T. vulgaris* obtained commercially in Italy was determined by Giamperi et al. (2002) to contain the following components:

α-thujene (0.1%)
α-pinene (0.5%)
camphene (1.0%)
sabinene (0.5%)
β-pinene (0.5%)
myrcene (0.1%)
p-cymene (15.0%)
limonene (0.6%)
1,8-cineole (0.3%)
(E)-β-ocimene (0.1%)
γ-terpinene (12.0%)
linalool (0.1%)
camphor (1.0%)
menthone (13.0%)
isomenthone (3.0%)
borneol (0.8%)
α-terpineol (0.6%)
methyl thymol (0.5%)
geraniol (2.0%)
thymol (47.0%)
carvacrol (0.1%)
geranyl acetate (0.2%)
β-caryophyllene (0.1%)
α-humulene (0.1%)
γ-muurolene (0.2%)

The occurrence of menthone and isomenthone is an anomaly. Neither compound exists in *T. vulgaris* oil; consequently, assuming that their characterizations by retention indices and mass spectra are correct, the only conclusion that can be drawn is that the oil was either contaminated or adulterated.

Kreck et al. (2002) compared the enantiomeric purity of selected components of thyme oil and living thyme plants (ex *T. vulgaris*) using stir bar sorptive extraction coupled to GC/MS. The results of this study are shown in **T-1**.

Baranauskiene et al. (2003) analyzed an oil of *T. vulgaris* of its second year of cultivation in Lithuania. The composition of the oil obtained from fresh herbage was found to be as follows:

α-thujene (1.2–1.4%)
α-pinene (0.8–0.9%)
camphene (0.4–0.7%)
sabinene (0.3–0.4%)
β-pinene (0.4–0.6%)
1-octen-3-ol (t–0.2%)
myrcene (1.7–1.8%)
α-phellandrene (0.1–0.2%)
δ-3-carene (0.1%)
α-terpinene (1.3–1.6%)
p-cymene (9.7–13.7%)
1,8-cineole (1.1–1.6%)
(Z)-β-ocimene (0–t)
(E)-β-ocimene (t–0.1%)
γ-terpinene (12.6–17.8%)
trans-sabinene hydrate (0.8–1.0%)
terpinolene (t–0.1%)
cis-sabinene hydrate (0–t)
linalool (1.5–2.5%)
camphor (t–0.2%)
isoborneol (0.1–0.3%)
borneol (0.7–1.1%)
terpinen-4-ol (0.3–0.4%)
α-terpineol (0.1–0.2%)
p-cymen-8-ol (0–t)
methyl thymol (0.1–0.8%)
methyl carvacrol (0.2–0.5%)
linalyl acetate (0–0.1%)
geraniol (0–0.1%)
geranial (0–t)
bornyl acetate (0–0.1%)
thymol (49.1–56.1%)
carvacrol (2.5–3.1%)
thymyl acetate (0–0.2%)
α-copaene (0.1–0.3%)
β-bourbonene (t–0.1%)
β-elemene (0–t)
β-caryophyllene (1.3–2.0%)
β-gurjunene (0–t)
α-humulene (0.1–0.2%)
allo-aromadendrene (0–t)
γ-gurjunene (0–t)
γ-muurolene (0–0.1%)
germacrene D (0–0.3%)
α-muurolene (t–0.1%)
β-bisabolene (0–0.1%)
γ-cadinene (0–0.1%)
δ-cadinene (0.1–0.2%)
spathulenol (t–0.1%)
caryophyllene oxide (0.1–0.2%)
1,10-di-epi-cubenol (0–t)
γ-eudesmol (t–0.1%)
T-cadinol (0–0.2%)
α-cadinol (0–0.2%)
farnesol* (0–t)

*correct isomer not identified; t = trace (< 0.05%)

In addition, trace amounts of tricyclene and β-cubebene were also found in this oil.

Lucchesi et al. (2004) analyzed a sample of thyme oil produced from *T. vulgaris* plants grown in Reunion by either microwave distillation or direct hydrodistillation. The results of this study are reported in **T-2**.

Atti-Santos et al. (2004) studied the changes that took place when the oils were produced from *T. vulgaris* over a season in southern Brazil. The range in composition found across the season was as follows:

α-pinene (0.5–1.2%)
α-thujene (0.8–1.7%)
camphene (0.3–1.4%)
sabinene (0.1–0.4%)
myrcene (1.1–1.9%)
α-terpinene (0.7–1.8%)
limonene (0.3–0.6%)
1,8-cineole (0.6–1.5%)
γ-terpinene (6.7–14.5%)
p-cymene (17.1–34.4%)
1-octen-3-ol (0.7–1.6%)
trans-sabinene hydrate (0.7–1.2%)
camphor (0.3–1.0%)
linalool (2.7–4.5%)
methyl thymol (0.6–1.5%)
methyl carvacrol (0.5–1.2%)
α-terpineol (0.2–0.9%)
germacrene D (0.2–0.3%)
borneol (1.2–3.3%)
caryophyllene oxide (0.4–1.1%)
thymol (31.5–52.4%)
carvacrol (2.3–3.1%)

A commercial sample of thyme oil purchased in Germany was analyzed by Sipailiene et al. (2006). This oil was found to possess the following composition:

α-pinene (5.4%)
camphene (0.1%)
sabinene (0.1%)
β-pinene (1.3%)
myrcene (0.3%)
α-phellandrene (0.1%)
δ-3-carene (4.1%)
α-terpinene (0.1%)
p-cymene (25.2%)
limonene (6.3%)
1,8-cineole (9.4%)
γ-terpinene (0.1%)
linalool (1.2%)
cis-β-terpineol (0.1%)
isoborneol (0.3%)
trans-β-terpineol (0.6%)
borneol (2.2%)
α-terpineol (2.5%)
methyl thymol (0.6%)
linalyl acetate (0.8%)
thymol (31.4%)
carvacrol (3.8%)
α-copaene (0.1%)
β-caryophyllene (2.6%)
α-humulene (0.3%)
caryophyllene oxide (0.2%)
Trace amounts (< 0.1%) of terpinolene, β-fenchyl alcohol, terpinen-4-ol, α-cubebene, longifolene and δ-cadinene were also characterized in this oil.

Dried thyme leaves (ex *T. vulgaris*) were either treated with γ-irradiation or e-beam ionization prior to oil isolation, and this oil was compared with an oil produced from untreated

T-1 **Enantiomeric purity (%) of selected components of thyme oil and living thyme plants**

Compound	Thyme oil	Thyme plants
(1R,5R)-(+)-α-pinene	89–93	90–94
(1R,5R)-(+)-β-pinene	65–71	57–62
(1R,5R)-(+)-sabinene	82–85	82–89
(4S)-(-)-α-phellandrene	94–98	98–99
(4R)-(+)-limonene	55–59	55–58
(3R)-(-)-linalool	96–98	> 99
(1S,4S)-(+)-terpinen-4-ol	64–69	64–69
(1R,4R)-(+)-α-terpineol	72–76	72–77
(1S,4S)-(-)-borneol	> 99	> 99

T-2 **Comparative percentage composition of oils of *Thymus vulgaris* produced by different isolation procedures**

Compound	Microwave distilled oil	Hydrodistilled oil
α-thujene	0.6	1.7
α-pinene	0.3	0.8
camphene	0.1	0.4
sabinene	–	0.2
1-octen-3-ol	2.9	2.5
3-octanone	0.2	–
myrcene	1.8	2.8
3-octanol	0.2	0.2
α-phellandrene	0.2	0.3
α-terpinene	1.7	2.7
p-cymene	7.5	11.1
limonene	0.6	0.9
1,8-cineole	0.5	0.7
γ-terpinene	17.1	22.8
cis-sabinene hydrate	2.8	0.9
terpinolene	–	0.2
linalool	4.6	4.0
borneol	1.1	1.2
terpinen-4-ol	0.5	1.4
α-terpineol	0.2	0.2
methyl thymol	–	0.2
methyl carvacrol	1.0	1.0
thymol	51.0	40.5
eugenol	1.5	0.3
β-caryophyllene	2.2	1.8
geranyl propionate	0.3	0.2
γ-muurolene	0.8	1.0
T-cadinol	0.3	–

thyme leaves. Haddad et al. (2007) showed that there were no changes in the oil composition either quantitatively or qualitatively irrespective of radiation treatment.

A wild-growing thyme (*T. vulgaris*) that was collected in Ramtha (Jordan) was lab-distilled to produce an oil that was analyzed by Hudaib and Aburjai (2007). This oil, which was rich in thymol, contained the following components:

tricyclene (0.6%)
α-thujene (0.5%)
α-pinene (0.3%)
sabinene (0.3%)
β-pinene (0.4%)
α-phellandrene (0.1%)
α-terpinene (1.4%)
p-cymene (8.2%)
limonene (0.2%)
β-phellandrene (0.1%)
1,8-cineole (2.1%)
γ-terpinene (7.7%)
cis-sabinene hydrate (0.4%)
trans-sabinene hydrate (0.1%)
α-thujone (1.2%)
β-thujone (0.7%)
camphor (1.1%)
borneol (0.2%)
terpinen-4-ol (0.3%)
α-terpineol (0.1%)
dihydrocarveol (0.1%)
trans-carvone oxide (0.3%)
thymol (63.8%)
carvacrol (6.9%)
thymyl acetate (0.1%)
β-caryophyllene (0.2%)
aromadendrene (0.1%)
caryophyllene oxide (0.5%)
γ-eudesmol (0.9%)

Martínez-Pérez et al. (2007) analyzed an oil produced from *T. vulgaris* grown in Cuba that was harvested when the plant was in full flower. The components characterized in this oil were:

α-thujene (0.2%)
α-pinene (0.7%)
camphene (0.5%)
β-pinene (0.2%)
1-octen-3-ol (1.7%)
3-octanone (0.1%)
3-octanol (0.1%)
p-cymene (26.1%)
1,8-cineole (1.5%)
γ-terpinene (0.9%)
trans-linalool oxide[f] (0.2%)
cis-linalool oxide[f] (0.1%)
terpinolene (0.1%)
p-cymenene (0.2%)
linalool (5.8%)
isoprenyl isovalerate (0.2%)
cis-p-menth-2-en-1-ol (0.1%)
trans-pinocarveol (0.1%)
camphor (0.5%)
camphene hydrate (0.1%)
isoborneol (0.1%)
borneol (4.1%)
terpinen-4-ol (3.1%)
α-terpineol (0.6%)
methyl thymol (2.1%)
methyl carvacrol (1.0%)
thymol (37.4%)
carvacrol (4.4%)
eugenol (0.5%)
α-ylangene (0.1%)
α-copaene (0.1%)
isobornyl propionate (0.2%)
β-bourbonene (0.1%)
methyl eugenol (0.2%)
β-caryophyllene (1.3%)
α-humulene (0.1%)
geranyl propionate (0.2%)
γ-muurolene (0.1%)
ar-curcumene (0.1%)
β-selinene (0.1%)
valencene (0.1%)
α-muurolene (0.1%)
β-bisabolene (0.1%)
γ-cadinene (0.4%)
δ-cadinene (0.5%)
α-cadinene (0.1%)
geranyl butyrate (0.1%)
caryophyllene oxide (1.8%)
humulene epoxide II (0.1%)
1,10-di-epi-cubenol (0.1%)
γ-eudesmol (0.2%)
(E)-sesquilavandulol (0.1%)
caryophylla-4(14),8,15-dien-5β-ol (0.1%)
T-muurolol (0.3%)
α-cadinol (0.1%)
cadalene (0.2%)

[f] = furanoid form

In addition, trace amounts (< 0.1%) of valeraldehyde, hexanal, 2-furfural, (E)-2-hexenal, (Z)-3-hexenol, hexanol, 2-heptanone, (E,E)-2,4-hexadienal, tricyclene, benzaldehyde, α-terpinene, limonene, salicylaldehyde, (E)-β-ocimene, 2,6-dimethyl-5-heptenal[‡], methyl benzoate, hotrienol, 2-phenethanol, α-fenchyl alcohol, β-thujone, *trans*-rose oxide, terpinen-1-ol, nerol oxide, pinocarvone, methyl salicylate, cuminaldehyde, geraniol, geranial, β-copaene, *trans*-α-bergamotene, aromadendrene, α-terpinyl isobutyrate, 2-phenethyl isovalerate, neryl isobutyrate, geranyl isobutyrate, α-calacorene, (E)-nerolidol, humulene epoxide I, geranyl isovalerate, α-muurolol, β-eudesmol and phytol were found in the same oil.

[‡] = also known as bergamal or melonal (tentative identification)

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Spanish Thyme Oil

A commercial sample of red thyme oil (ex *Thymus zygis* L.) was analyzed by Kubeczka and Formacek (2002). Using a combination of GC and ^{13}C-NMR, the authors determined that the oil contained the following components:

α-pinene (1.10%)
α-thujene (0.61%)
camphene (0.79%)
β-pinene (0.21%)
δ-3-carene (0.08%)
myrcene (1.38%)
α-phellandrene (0.09%)
α-terpinene (1.24%)
limonene (0.55%)
β-phellandrene (0.21%)
1,8-cineole (0.31%)
γ-terpinene (3.63%)
p-cymene (22.20%)
terpinolene (0.13%)
trans-sabinene hydrate (0.58%)
camphor (0.32%)
linalool (5.47%)
cis-sabinene hydrate (0.24%)
bornyl acetate (0.10%)
β-caryophyllene (1.07%)
methyl carvacrol (0.53%)
terpinen-4-ol (1.10%)
aromadendrene (0.19%)
α-terpineol (0.27%)
borneol (1.72%)
δ-cadinene (0.16%)
geraniol (0.29%)
caryophyllene oxide (0.16%)
thymol (49.70%)
carvacrol (2.64%)

White thyme oil of Spanish origin (ex *Thymus zygis*) was reported by Milchard et al. (2004) to possess the following major constituents:

α-pinene (1.0%)
myrcene (2.1%)
α-terpinene (1.4%)
p-cymene (17.5%)
γ-terpinene (8.1%)
linalool (6.1%)
borneol (1.4%)
terpinen-4-ol (1.4%)
thymol (48.6%)

carvacrol (2.7%)
β-caryophyllene (1.5%)

Currently 1500 tonnes of *T. zygis* leaves are exported from Spain each year. In addition, ca. 15–20 tonnes of thyme oil are produced; however, the demand particularly for leaves is not met with *T. zygis* subsp. *gracilis* (known as Red Thyme). Consequently, to satisfy the market needs wild plants from southeastern Spain are collected for both oil and leaf production.

Sotomayor et al. (2004) evaluated the effect of watering on both biomass production and oil composition. They found that the highest thymol levels in the oil were obtained at 30% and 40% of the local potential evapotranspiration. The composition of the oil at the lowest watering level was determined to be as follows:

α-thujene (1.00%)
α-pinene (0.57%)
camphene (0.25%)
sabinene (0.01%)
β-pinene (0.14%)
myrcene (1.30%)
α-phellandrene (0.14%)
δ-3-carene (0.08%)
α-terpinene (1.22%)
p-cymene (14.21%)
limonene (0.45%)
(E)-β-ocimene (0.01%)
γ-terpinene (2.99%)
terpinolene (0.15%)
β-caryophyllene (0.62%)
aromadendrene (0.22%)
α-humulene (0.16%)
allo-aromadendrene (0.02%)
γ-cadinene (0.06%)
δ-cadinene (0.11%)
(Z)-α-bisabolene (0.01%)
(Z)-3-hexenol (0.05%)
hexanol (0.02%)
1-octen-3-ol (0.19%)
3-octanol (0.17%)
trans-sabinene hydrate (0.49%)
cis-sabinene hydrate (0.09%)
linalool (2.48%)
cis-verbenol (0.01%)
trans-verbenol (0.01%)
isoborneol (0.01%)
borneol (0.60%)
terpinen-4-ol (0.62%)
α-terpineol (0.06%)
nerol + citronellol (0.05%)
spathulenol (0.07%)
nonanal (0.01%)
decanal (0.02%)
3-octanone (0.13%)
pinocarvone (0.02%)
dihydrocarvone (0.06%)
verbenone (0.01%)
(Z)-jasmone (0.01%)
β-ionone* (0.01%)
methyl acetate (0.01%)
benzyl acetate (0.02%)
bornyl acetate (0.04%)
citronellyl acetate (0.01%)
methyl thymol (0.02%)
methyl carvacrol (0.51%)
thymol (64.16%)
carvacrol (3.33%)
eugenol (0.08%)
isoeugenol* (0.02%)
cis-linalool oxide[f] (0.01%)
trans-linalool oxide[f] (0.01%)
caryophyllene oxide (0.19%)
1,8-cineole (0.03%)

*correct isomer not identified; [f] = furanoid form

Trace amounts (< 0.01%) of tricyclene, verbenene, (Z)-β-ocimene, isoamyl alcohol, 3-methyl-2-butenol, geraniol, neral, geranial, perillaldehyde, 2-butanone, 3-hexanone, 3-heptanone, β-thujone, pinocarvone, thymoquinone, an α-ionone isomer, octyl acetate, isobornyl acetate, α-terpinyl acetate, neryl acetate, butyl octanoate, (E)-anethole, methyl eugenol and *cis*-limonene oxide were also found in this oil.

An oil produced in the laboratory by hydrodistillation of *T. zygis* leaves was screened for its antimicrobial properties by Dorman and Deans (2004). Analysis of the oil revealed that it was found to contain the following constituents:

α-thujene (0.1%)
α-pinene (2.1%)
camphene (0.2%)
β-pinene (0.1%)
myrcene (1.2%)
α-terpinene (0.4%)
p-cymene (25.5%)
1,8-cineole (1.3%)
limonene (1.1%)
γ-terpinene (4.4%)
terpinolene (0.5%)
linalool (4.6%)
camphor (0.1%)
borneol (0.9%)
terpinen-4-ol (0.2%)
α-terpineol (0.9%)
β-terpineol* (0.3%)
bornyl acetate (1.9%)
thymol (48.0%)
carvacrol (3.0%)
β-caryophyllene (1.0%)

*correct isomer not identified

A lab-distilled oil of thyme leaves acquired at a Spanish market was produced by simultaneous distillation and extraction using dichloromethane as the solvent (Diaz-Maroto et al. 2005). The same batch of leaves was subjected to supercritical fluid CO_2 extraction (SFE) and the volatiles were compared using GC/MS as shown in **T-3**. It should be noted that the taxonomic origin of the thyme was not determined even though the authors reported that it was *T. vulgaris* and Spanish thyme originates from *T. zygis*. Also, the fact that the SFE volatile concentrate was rich is thymol is not unexpected as the method of oil isolation was hydrodistillation from a minute sample.

An oil produced from *T. zygis* subsp. *gracilis* was reported by Martinéz et al. (2006) to possess the following composition:

α-thujene (0.1%)
α-pinene (0.5%)
camphene (0.2%)
β-pinene (0.1%)
1-octen-3-ol (0.3%)
3-octanone (0.1%)
myrcene (1.0%)
α-phellandrene (0.1%)
α-terpinene (0.9%)
p-cymene (17.0%)
limonene (0.4%)
γ-terpinene (3.1%)
trans-sabinene hydrate (0.5%)
terpinolene (0.2%)
linalool (3.0%)
borneol (0.4%)
terpinen-4-ol (0.6%)
p-cymen-8-ol (0.1%)
α-terpineol (0.2%)
methyl carvacrol (0.8%)
geranial (0.1%)

Comparative percentage composition of an oil and a supercritical fluid CO_2 (SFE) extract of *Thymus zygis* **T-3**

Compound	Oil	SFE
α-thujene	0.5	t
α-pinene	2.1	0.2
camphene	1.5	0.1
verbenene	0.1	9.1
1-octen-3-ol	0.2	0.1
1-octen-3-one	0.1	–
sabinene	0.1	–
β-pinene	0.2	–
myrcene	0.5	t
α-phellandrene	0.1	–
δ-3-carene	t	–
α-terpinene	0.8	0.2
p-cymene	33.0	8.5
limonene + 1,8-cineole	2.8	2.3
(E)-β-ocimene	t	–
γ-terpinene	4.8	1.6
trans-sabinene hydrate	1.0	0.6
cis-linalool oxide[f]	0.1	t
trans-linalool oxide[f] + p-cymenene	0.2	t
terpinolene	0.1	t
linalool	3.6	3.3
camphor	0.7	1.4
trans-verbenol	0.5	0.2
borneol	3.6	4.0
p-cymen-8-ol	0.3	0.2
terpinen-4-ol	1.0	1.1
α-terpineol	0.9	1.0
verbenone	1.7	1.4
methyl thymol	0.3	0.5
methyl carvacrol	0.6	0.4
linalyl acetate	0.2	1.0
thymol	31.1	60.9
carvacrol	5.2	8.9
β-caryophyllene	0.5	0.9
β-gurjunene	t	t
γ-cadinene	t	t
δ-cadinene	t	0.1
caryophyllene oxide	0.6	0.5
α-cadinol	0.1	0.1

[f] = furanoid form; t = trace (< 0.1%)

bornyl acetate (0.1%)
thymol (62.1%)
carvacrol (3.1%)
β-caryophyllene (0.7%)
α-humulene (0.1%)
caryophyllene oxide (0.4%)

In addition, trace amounts (< 0.1%) of sabinene, 1,8-cineole, *cis*-sabinene hydrate, *trans*-pinocarveol, *cis*-verbenol, camphor, a dihydrocarvone isomer, verbenone, valencene, γ-cadinene and spathulenol were also found in this oil.

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