Progress in Essential Oils

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Native Spearmint Oil

Singh et al. (1990) determined that the effect of drying and storage of spearmint leaves prior to distillation for a period of greater than 20 days in India resulted in a reduction of the carvone content of oil produced from such leaves from 72.6% to 66.3%.

Adamovic (1993) reported that the carvone content of commercially available spearmint oil produced in Serbia during the years 1981–1990 ranged from 53–78%.

Tsuneya et al. (1993) determined that the major pyridines and pyrazines found in Native spearmint oil were as follows:

pyridine (0.46)[a]
2,5-dimethylpyrazine (0.58)
2-acetylpyridine (0.13)
2-isopropyl-4-methylpyridine (0.13)
4-isopropyl-2-methylpyridine (0.14)
4-isopropenyl-2-methylpyridine (0.02)
3-[(Z)-1-buten-1-yl]pyridine (0.23)
3-[(E)-1-buten-1-yl]pyridine (0.59)
2-ethyl-4-isopropenylpyridine (0.14)
quinoline (0.11)
2-acetyl-4-isopropylpyridine (0.04)
2,4-diisopropenylpyridine (0.40)
2-acetyl-4-isopropenylpyridine (3.54)
4-acetyl-2-isopropenylpyridine (0.03)
5-[(Z)-1-buten-1-yl]-2-propylpyridine (0.02)
5-[(E)-1-buten-1-yl]-2-propylpyridine (0.14)
3-[(Z)-1-buten-1-yl]-4-propylpyridine (0.49)
3-[(E)-1-buten-1-yl]-4-propylpyridine (0.05)
3-phenylpyridine (0.57)
3-phenyl-4-propylpyridine (0.07)
5-phenyl-2-propylpyridine (0.22)

[a] = ppm in Native spearmint oil

A commercial sample of *M. spicata* oil that originated from Guangzhou (China) was analyzed by Zhu et al. (1995) using GC/MS. The components identified in this oil were:

α-pinene (0.06%)
β-pinene (0.08%)
limonene (6.79%)
linalool (0.12%)
menthone (1.86%)
lavandulol (1.05%)
menthol (1.46%)
terpinen-4-ol (0.21%)
α-terpineol (0.26%)
cis-dihydrocarvone (1.82%)
trans-dihydrocarvone (0.17%)
carveol* (0.82%)
carvone (76.54%)
piperitone (0.14%)
geranyl acetate (0.18%)
menthyl acetate (0.29%)
dihydrocarvyl acetate (0.30%)
carvyl acetate* (0.14%)
β-bourbonene (1.50%)
β-elemene (0.31%)
β-caryophyllene (1.58%)
β-cubebene† (0.17%)
α-humulene (0.09%)
calamenene* (0.16%)

*correct isomer not identified; †incorrect identification based on GC elution order

Using a two-dimensional GC technique, Dimandja et al. (2000) analyzed a commercial sample of Native spearmint oil. The components characterized in this oil were as follows:

α-pinene (0.6%)
sabinene (0.3%)
β-pinene (0.7%)
3-octanol (0.6%)
myrcene (0.3%)
α-terpinene (t)
p-cymene (t)
1,8-cineole (0.3%)
limonene (15.0%)
terpinolene (t)
isopulegol (0.4%)
menthone (9.7%)
isomenthone (4.4%)
neomenthol (2.4%)
lavandulol (0.4%)
menthol + neoiso(iso)pulegol (16.2%)
dihydrocarvone* (1.1%)
α-terpineol (0.4%)
neodihydrocarveol (t)
trans-carveol + pulegone (0.4%)
carvone (37.7%)
menthyl acetate (1.4%)
dihydrocarvyl acetate (0.5%)
β-bourbonene (0.6%)
β-elemene (0.2%)
β-caryophyllene (0.9%)

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

Based on the fact that the spearmint oil analyzed by Dimandja et al. contained high levels of menthone, isomenthone, menthol, menthyl acetate and unusual constituents such as isopulegol, neomenthol, lavandulol and neoiso(iso)pulegol, it is obvious that the oil was not pure but was adulterated. It is a pity that the authors didn't pay more attention to the oil authenticity when describing an analysis using an advanced GC technique.

The main component composition of Native spearmint oil produced either in the Midwest, the Far West or a second cutting in the Far West can be seen in **T-1** (Lawrence 2000).

A commercial sample of Native spearmint oil produced in the United States was analyzed by Kubeczka and Formacek (2002) using GC and ^{13}C-NMR. The oil composition was

found to be as follows:

2-methylbutanal (0.03%)
isovaleraldehyde (0.07%)
α-pinene (0.79%)
α-thujene (0.08%)
trans-2,5-diethyltetrahydrofuran (0.06%)
β-pinene (0.73%)
sabinene (0.51%)
myrcene (2.74%)
α-terpinene (0.19%)
limonene (10.62%)
1,8-cineole (2.15%)
(Z)-β-ocimene (0.20%)
γ-terpinene (0.38%)
(E)-β-ocimene (0.09%)
p-cymene (0.36%)
terpinolene (0.13%)
3-octyl acetate (0.35%)
(Z)-3-hexenol (0.05%)
3-octanol (0.95%)
menthone (0.09%)
trans-sabinene hydrate (1.21%)
isomenthone (0.06%)
β-bourbonene (1.52%)
linalool (0.08%)
cis-sabinene hydrate (0.13%)
β-caryophyllene (0.86%)
terpinen-4-ol (1.12%)
cis-dihydrocarvone (1.42%)
trans-dihydrocarvone (0.19%)
γ-muurolene (0.35%)
(E)-β-farnesene (0.68%)
dihydrocarvyl acetate (0.34%)
α-terpineol (0.33%)
germacrene D (0.67%)
neodihydrocarveol (0.59%)
carvone (66.26%)
dihydrocarveol (0.25%)
cis-carvyl acetate (0.37%)
trans-carveol (0.43%)
cis-carveol (0.23%)
(Z)-jasmone (0.31%)
viridiflorol (0.24%)

Through the use of a non-equilibrated solid-phase microextraction technique combined with GC/MS, the headspace volatiles of more than 20 samples of freshly distilled Native spearmint oil were examined by Coleman et al. (2002). The results of this study can be seen summarized as follows:

acetaldehyde (0.005%)
dimethyl sulphide (0.054–0.100%)
isobutanal (0.041–0.087%)
2-methylbutanal (0.340–0.517%)
2-ethylfuran (0.096–0.195%)
valeraldehyde (0.005%)
methyl 2-methylbutyrate (0.108–0.167%)
α-pinene (3.746–4.035%)
α-thujene (0.376–0.451%)
trans-2,6-diethyltetrahydrofuran (0.224–0.600%)
β-pinene (2.961–3.238%)
sabinene (2.017–2.397%)
myrcene (8.517–9.572%)
α-terpinene (1.097–1.296%)
limonene (31.627–35.089%)
1,8-cineole (7.293–8.868%)
(E)-2-hexenal (0.219–0.456%)
(Z)-β-ocimene (0.401–0.578%)
γ-terpinene (1.499–1.702%)
(E)-β-ocimene (0.230–0.327%)
p-cymene (0.250–0.333%)
3-octanol (1.498–1.769%)
menthone (0.005%)
trans-sabinene hydrate (1.001–1.862%)
menthofuran (0.005%)
isomenthone (0.005%)
menthyl acetate (0.005%)
pulegone (0.005%)
menthol (0.005%)
carvone (26.664–33.500%)

In the original report the authors noted that neomenthol (0.422–0.886%) and terpinen-4-ol (0.724–0.887%) were found as constituents. However, it is unlikely that these two compounds were correctly characterized, as a result they have been omitted from the above list of constituents identified.

Mestri (2005) reported that the main constituents found in Native spearmint oil (origin unknown) were:

limonene (9.0%)
3-octanol (1.0%)
trans-sabinene hydrate (1.5%)
menthone (< 0.1%)
dihydrocarvone* (< 2.5%)
carvone (56.0%)
dihydrocarvyl acetate (0.6%)
cis-carvyl acetate (0.6%)

*correct isomer not identified

In addition, the oil was reported to also contain α- and β-pinene, α-phellandrene and 1,8-cineole.

An oil of *M. spicata* of Indian origin was reported by Ranade (2005) to contain the following constituents:

α-pinene (0.70%)
β-pinene (3.10%)
limonene (8.65%)
1,8-cineole (3.65%)
3-octanol (1.05%)
3-octyl acetate (0.35%)
sabinene hydrate* + sabinyl acetate† (1.15%)
linalool (5.20%)
menthofuran† (0.10%)
menthone (0.30%)
isomenthone (0.10%)
carvomenthone† (1.00%)
dihydrocarvone* (4.45%)
pulegone (0.50%)
dihydrocarvyl acetate (2.30%)
carvone (63.60%)
trans-carvyl acetate (0.90%)
cis-carvyl acetate (1.55%)
trans-carveol (0.20%)
cis-carveol (0.50%)

*correct isomer not identified; †incorrect identification based on GC elution order

Comparative main component composition (%) of Native spearmint oils produced in the United States **T-1**

Compound	Midwest oil (15)[a]	Far West oil (50)	2nd Cutting oil (20)
myrcene	2.51–2.85	2.30–4.76	2.72–3.36
limonene	9.06–10.90	8.32–13.36	10.39–11.93
1,8-cineole	1.77–1.81	1.49–2.11	1.63–2.00
3-octyl acetate	0.20–0.29	0.20–0.54	0.24–0.47
3-octanol	0.83–0.95	0.82–1.23	0.79–1.02
trans-sabinene hydrate	0.84–0.93	0.75–1.68	0.95–1.50
β-bourbonene	2.07–2.22	1.78–2.66	1.50–1.88
carvone	65.40–66.59	58.47–66.59	65.42–69.44
trans-carveol	0.80–1.15	0.32–1.15	0.30–0.58
cis-carveol	0.28–0.41	0.26–0.45	0.28–0.47

[a] = number of samples analyzed

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Chirality

The technique of "heart cutting" from a DB-1701 to a heptakis (3-O-acetyl-2,6-di-O-pentyl)-b-cyclodextrin chiral column known as multidimensional gas chromatography was used by Mosandl et al. (1991) to determine the enantiomeric distribution of three monoterpene hydrocarbons in a commercial sample of Native spearmint oil. They found the chiral distribution of these compounds to be as follows:

(1R,5R)-(+)-α-pinene (57%):(1S,5S)-(-)-α-pinene (43%)
(1R,5R)-(+)-β-pinene (40%):(1S,5S)-(-)-β-pinene (60%)
(4R)-(+)-limonene (19%):(4S)-(-)-limonene (81%)

Ravid et al. (1992) determined that the enantiomeric distribution of carvone in a range of spearmint oils was as follows: (4S)-(+)-carvone (< 1.0%):(4R)-(-)-carvone (> 99%).

Nakamoto et al. (2006) reported that the enantiomeric distribution of cis- and trans-carveol in Native spearmint oil was as follows:

(2R,4S)-(+)-trans-carveol (15%):(2S,4R)-(-)-trans-carveol (85%)
(2S,4S)-(+)-cis-carveol (4%):(2R,4R)-(-)-cis-carveol (96%)

Coleman and Lawrence (2001) used chiral GC to examine the enantiomeric distribution of four monoterpene hydrocarbons in Native spearmint oil. Their results are as follows:

(1R,5R)-(+)-α-pinene (40.3%):(1S,5S)-(-)-α-pinene (59.7%)
(1R)-(+)-camphene (99.9%):(1S)-(-)-camphene (0.1%)
(1R,5R)-(+)-β-pinene (48.7%):(1S,5S)-(-)-β-pinene (51.3%)
(4R)-(+)-limonene (1.9%):(4S)-(-)-limonene (98.1%)

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Oils of *Mentha spicata* Rich in Carvone

As part of a survey of the aromatic plants found in the Nainital district (Uttarakhand, India), Misra et al. (1989) collected 42 genotypes of *M. spicata* from which they produced oils for analysis. The composition of an example of a carvone-rich oil can be seen as follows:

α-pinene (0.4%)
sabinene (0.5%)
α-terpinene (0.5%)
α-phellandrene (1.2%)
limonene (22.5%)
β-ocimene (0.4%)
1,8-cineole (0.9%)
3-octanol (1.1%)
trans-sabinene hydrate (0.2%)
menthone (1.9%)
pulegone (1.7%)
carvone (61.5%)
trans-carveol (2.0%)
cis-carveol (0.2%)
piperitenone oxide (2.0%)

Terpinolene, 3-octyl acetate, linalool, isomenthone and a carvyl acetate isomer were also found as trace constituents (< 0.1%) of this same oil.

Although it has been known for a long time that (-)-carvone was the major enantiomer found in a sample of carvone-rich spearmint oil, König et al. (1990) determined that its enantiomeric distribution was as follows: (4S)-(+)-carvone (0.67%):(4R)-(-)-carvone (99.33%).

Tucker et al. (1991) analyzed the composition of three oils of *M. spicata* that were found to be rich in carvone. The components identified in these oils were as follows:

α-pinene (0.01–0.38%)
camphene (0–0.01%)
β-pinene (0.07–0.46%)
sabinene (0.08–0.42%)
myrcene (0–4.13%)
α-terpinene (0.01%)
limonene (4.51–7.86%)
1,8-cineole (0.55–1.30%)
(Z)-β-ocimene (0.15–0.49%)
γ-terpinene + (E)-β-ocimene + 3-octanone (0.07–0.12%)
p-cymene (0.01%)
terpinolene (0.05–0.09%)
3-octyl acetate (0.02–0.17%)
3-octanol (0.03–0.13%)
1-octen-3-ol (0.03–0.05%)
trans-sabinene hydrate (0.03–1.32%)
menthone (0–0.94%)
menthofuran (0.08–0.11%)
isomenthone (0–0.24%)
β-bourbonene (0.38–0.46%)
linalool (0.03–0.20%)
terpinen-4-ol (0.14–0.20%)
β-caryophyllene + *cis*-dihydrocarvone (3.64–9.96%)
trans-dihydrocarvone (0–0.15%)
menthol (0–0.11%)
pulegone (0–0.52%)
isomenthol (0–0.28%)
dihydrocarvyl acetate (0–0.95%)
α-humulene (0.37–0.89%)
α-terpineol (0.63–0.78%)
germacrene D (0–0.12%)
neodihydrocarveol (0–6.67%)
carvone (64.24–77.35%)
dihydrocarveol (0.63–2.08%)
cis-carvyl acetate (0.07–0.20%)
trans-carveol (0.07–0.16%)
cis-carveol (0.04–0.10%)
eugenol (0.01–0.03%)

Mentha spicata oil rich in carvone that was produced in Moldova was the subject of analysis by Shikimaka et al. (1993). They found that the oil contained the following main components:

α-pinene (0.3%)
β-pinene (0.6%)
myrcene (0.2%)
limonene (6.2%)
1,8-cineole (0.1%)
dihydrocarvone* (9.7%)
dihydrocarvyl acetate (3.8%)
dihydrocarveol (16.5%)
carvone (59.8%)
carvyl acetate* (0.2%)
carveol* (0.1%)

*correct isomer not identified

Koyalta et al. (1993) reported that *M. spicata* cultivated in Senegal contained the following main constituents:

β-pinene (0.3%)
sabinene (0.2%)
myrcene (0.4%)

α-terpinene (0.2%)
limonene (8.0%)
1,8-cineole (16.0%)
β-caryophyllene (4.0%)
terpinen-4-ol (2.0%)
carvone (68.0%)
piperitenone oxide (0.2%)
germacrene D (0.2%)

Avato et al. (1995) analyzed a carvone-rich oil of spearmint that was cultivated in Italy and found that it contained:

α-pinene (0.1%)
sabinene (0.3%)
myrcene (0.4%)
limonene (2.0%)
1,8-cineole (3.6%)
p-menth-2-en-1-ol* (0.2%)
β-bourbonene (1.8%)
α-gurjunene (0.1%)
linalool (1.6%)
germacrene A (0.2%)
β-caryophyllene (3.7%)
trans-dihydrocarvone (6.1%)
β-sesquiphellandrene (0.7%)
dihydrocarvyl acetate (6.1%)
germacrene D (0.8%)
isodihydrocarveol (1.4%)
trans-carvyl acetate (2.6%)
carvone (58.7%)
δ-cadinene (0.9%)
cis-carvyl acetate (6.4%)
trans-carveol (0.5%)
calamenene* (0.1%)
cis-carveol (0.6%)
8-hydroxy-p-menth-4-en-3-one (0.7%)
piperitenone (0.3%)
(Z)-jasmone (0.1%)
piperitenone oxide (0.2%)
caryophyllene oxide (0.4%)
germacrene D-4-ol (0.1%)
cadin-4-en-1-ol (0.1%)
viridiflorol (0.3%)
α-cadinol (0.3%)

*correct isomer not identified

Trace amounts of β-pinene, phenylacetaldehyde, germacrene B and α-terpineol were also found in the same oil.

Oil produced from a single strain of *M. spicata* was analyzed by Kokkini et al. (1995) and found to possess the following composition:

α-pinene (0.2%)
β-pinene (0.6%)
sabinene (0.4%)
myrcene (0.6%)
limonene (14.8%)
1,8-cineole (4.5%)

Comparative main component composition (%) of Native spearmint oils produced in the United States **T-2**

Compound	Fresh leaf oil	Shade-dried leaf oil	Sun-dried leaf oil
α-pinene	0.77	0.76	0.01
β-pinene	1.04	0.86	0.03
myrcene	2.84	1.53	0.30
limonene	26.82	26.96	3.78
β-ocimene*	0.52	0.28	0.27
3-octanol	0.43	0.16	0.31
1-octen-3-ol	0.35	0.20	0.34
β-bourbonene	0.40	0.50	1.13
linalool	0.58	0.64	1.20
trans-dihydrocarvone	0.75	0.60	1.84
germacrene D	0.14	0.31	0.49
dihydrocarveol	0.12	0.97	1.06
carvone	59.59	62.78	82.90
trans-carvyl acetate	4.34	0.91	0.68
cis-carvyl acetate	0.15	0.13	0.32
eucarvone†	0.14	0.15	0.19
(Z)-jasmone	0.12	0.15	0.19
cubenol†	0.20	0.17	0.42
α-cubenol†	0.30	0.16	0.36

*correct isomer not identified; †identity not corroborated with previously published data

Comparative percentage composition of an oil and a supercritical fluid CO_2 extract of spearmint **T-3**

Compound	Oil	SFE
α-pinene	0.47	0.02
β-phellandrene‡	0.18	0.03
β-pinene	0.57	0.05
1,8-cineole	7.98	3.65
trans-sabinene hydrate	0.64	0.69
linalool	0.29	0.29
menthone	0.08	0.13
borneol	0.80	0.84
terpinen-4-ol	0.82	0.56
dihydrocarvone*	1.76	1.91
neodihydrocarveol	3.38	3.01
cis-carveol	5.33	4.56
carvone	74.49	81.85
eugenol	0.01	0.16
trans-carvyl acetate	0.14	0.22
(Z)-jasmone	0.32	0.52
α-cubebene	0.04	0.11
β-bourbonene	1.06	2.31
β-elemene	0.22	0.44
β-caryophyllene	0.80	1.39
β-selinene	0.07	0.16
1-epi-bicyclosesquiphellandrene	0.08	0.26
spathulenol	0.46	0.60

SPE = supercritical fluid CO_2 extract; ‡incorrect identification based on elution order; *correct isomer not identified

β-ocimene (t)
γ-terpinene (t)
p-cymene (t)
terpinolene (t)
3-octanol (0.4%)
1-octen-3-ol (t)
trans-sabinene hydrate (0.2%)
β-caryophyllene (0.3%)
trans-dihydrocarvone (7.2%)
terpinen-4-ol (0.1%)
cis-dihydrocarvone (0.1%)
dihydrocarvyl acetate (0.2%)
α-terpineol (0.1%)
germacrene D (0.9%)
neoisodihydrocarvyl acetate (0.6%)
carvone (68.4%)
dihydrocarveol (0.1%)
trans-carveol (0.1%)
cis-carveol (0.1%)

t = trace (< 0.1%)

An oil of *M. spicata* produced in the laboratory from plants collected along the Sithonia Peninsula (northern Greece) was subjected to GC and GC/MS analysis by Adam et al. (1998). The composition of the oil was found to be:

α-pinene (0.91%)
camphene (0.04%)
β-pinene (0.25%)
sabinene (0.08%)
δ-3-carene (0.04%)
myrcene (0.45%)
limonene (5.07%)
1,8-cineole (5.42%)
β-phellandrene (0.07%)
(Z)-β-ocimene (0.14%)
γ-terpinene (0.04%)
(E)-β-ocimene (0.03%)
1-octen-3-ol (0.08%)
trans-sabinene hydrate (0.53%)
cis-sabinene hydrate (0.38%)
linalool (0.06%)
terpinen-4-ol (0.60%)
β-caryophyllene (0.90%)
trans-dihydrocarvone (0.12%)
cis-dihydrocarvone (4.90%)
α-terpineol + borneol (0.83%)
dihydrocarvyl acetate (0.19%)
germacrene D (1.76%)
neodihydrocarveol (0.90%)
carvone (59.12%)
dihydrocarveol (6.27%)
neoisodihydrocarveol (2.11%)
trans-carvyl acetate (0.24%)
trans-carveol (0.68%)
cis-carveol (3.90%)
(Z)-jasmone (0.16%)

Karousou et al. (1998) determined that the main p-menthane

Comparative percentage composition of the oil and headspace volatiles of spearmint oil of Cameroonian origin **T-4**

Compound	Oil	Headspace
3-methylbutanal	0.25	0.37
2-methylbutanal	0.10	0.22
valeraldehyde	–	0.02
2-butanol	0.01	0.01
α-pinene	0.17	0.45
camphene	0.41	0.92
β-pinene	0.31	0.22
sabinene	0.14	0.05
myrcene	0.88	0.36
α-terpinene	0.15	0.09
α-phellandrene	0.44	0.24
δ-3-carene	0.02	0.03
limonene	6.55	8.31
1,8-cineole	4.19	7.12
γ-terpinene	0.12	0.18
(Z)-β-ocimene	0.21	0.06
p-cymene	0.52	0.23
(Z)-3-hexenol	0.04	0.06
(E)-β-ocimene	0.56	0.38
terpinolene	0.05	0.44
(E)-1-hexen-3-ol‡	0.66	1.72
hexanol	0.12	0.09
3-octanol	0.17	0.14
trans-sabinene hydrate	0.02	t
α-copaene	0.77	0.45
linalool	0.48	0.87
β-bourbonene	0.19	0.12
linalyl acetate	0.11	0.08
β-elemene	0.45	0.37
trans-dihydrocarvone	0.34	0.31
terpinen-4-ol	0.08	0.07
cis-dihydrocarvone	0.27	0.21
β-caryophyllene	0.42	0.16
menthol	0.21	0.56
α-terpineol	0.53	0.36
α-humulene	0.18	0.18
pulegone	0.24	0.13
(E)-β-farnesene	0.07	0.06
dihydrocarvyl acetate	0.22	0.23
borneol	0.26	0.18
germacrene D	1.78	0.92
trans-dihydrocarveol†	0.19	0.17
bicyclogermacrene	0.06	0.03
carvone	69.34	66.77
cis-dihydrocarveol††	0.31	0.19
trans-carvyl acetate	1.23	0.99
trans-carveol	0.66	0.57
cis-carvyl acetate	0.18	0.14
germacrene A	0.23	0.19
cis-carveol	2.14	1.86
geraniol	0.87	0.74
(Z)-jasmone	0.80	0.66

t = trace (< 0.01%); ‡1-hexen-3-ol cannot exist in (E)- or (Z)-forms; †probably neodihydrocarveol; ††probably dihydrocarveol

Comparative percentage composition of oils of *Mentha spicata* produced from plants grown in Egypt **T-5**

Compound	Siwa Oasis oil	Owainat oil	Kharga oil	Samallot oil	Arish oil	Cairo oil
α-pinene	0.9	1.0	0.9	0.9	0.9	0.7
sabinene	0.7	0.5	0.6	0.6	0.6	0.6
β-pinene	1.2	1.2	1.2	1.2	1.2	1.0
myrcene	0.8	0.8	0.8	0.7	0.7	0.7
p-cymene	0.2	0.3	0.1	0.1	0.1	0.1
limonene	33.2	26.7	34.8	26.2	30.7	5.0
1,8-cineole	4.9	4.9	4.5	5.7	6.1	5.1
(Z)-β-ocimene	0.1	0.1	0.1	0.1	0.2	0.1
(E)-β-ocimene	0.4	0.5	0.1	0.2	0.1	0.1
γ-terpinene	0.7	1.4	1.3	1.2	1.4	0.4
menthol	1.1	1.2	0.5	0.6	0.4	0.8
α-terpineol	0.1	0.2	0.1	0.1	0.2	0.1
dihydrocarveol*	0.2	0.2	0.2	0.4	7.1	0.5
cis-dihydrocarvone	0.1	t	t	t	–	t
trans-dihydrocarvone	0.1	t	t	0.1	–	0.3
trans-carveol	0.1	0.1	0.1	0.1	0.1	0.1
cis-carveol	0.2	0.2	0.1	0.3	0.1	0.9
pulegone	1.0	0.7	1.4	1.1	3.1	0.8
carvone	51.6	57.6	49.5	55.4	42.2	73.2
cis-carvyl acetate	0.3	0.2	0.8	0.9	0.5	0.8
β-caryophyllene	0.7	0.6	1.2	1.4	0.8	1.6

*correct isomer not determined; t = trace (< 0.1%)

Volatile concentration of the oils obtained from fresh and dried spearmint leaves **T-6**

Compound	1	2	3	4
α-thujene	10a	15	15	12
α-pinene	307	407	423	380
camphene	38	48	53	43
1-octen-3-ol	12	8	11	6
sabinene	296	364	349	333
β-pinene	464	584	609	545
myrcene	325	361	360	320
limonene + 1,8-cineole	6488	8319	7909	7492
trans-sabinene hydrate	465	595	526	482
borneol	99	100	107	81
terpinen-4-ol	58	59	73	58
dihydrocarvone*	736	565	672	622
dihydrocarveol*	1733	1561	1434	1109
cis-carveol	115	58	60	70
carvone	14399	15324	14702	14229
bornyl acetate	85	74	98	65
dihydroedulan	42	38	50	40
dihydrocarvyl acetate*	430	525	443	286
cis-p-mentha-6,8-dien-2-yl acetate	87	77	83	51
trans-carvyl acetate	80	79	103	63
(Z)-jasmone	100	87	105	65
β-bourbonene	303	225	295	271
β-elemene	79	64	85	87
β-caryophyllene	534	406	481	425
β-selinene	31	21	28	19
1-epi-bicyclosesquiphellandrene	425	296	377	336

*correct isomer not identified; [a] = mg/g dry leaf weight; 1 = fresh leaf volatiles; 2 = air-dried leaf volatiles; 3 = oven-dried leaf volatiles; 4 = freeze-dried (-18°C) leaf volatiles

compounds in a carvone-rich oil of *M. spicata* of Greek origin were as follows:

limonene (7.04–11.04%)
trans-dihydrocarvone (0.30–0.78%)
cis-dihydrocarvone (4.01–11.11%)
neodihydrocarveol (0.02–0.20%)
neoisodihydrocarvyl acetate (0.04–0.40%)
carvone (56.21–62.95%)
dihydrocarveol (0–0.33%)
neoisodihydrocarveol (0.21–0.45%)
trans-carvyl acetate (0.01–0.23%)
trans-carveol (0.36–0.46%)
cis-carveol (0–0.08%)

Abou-Mandour and Binder (1998) produced an oil from *M. spicata* grown in Egypt, which on analysis comprised the following components:

α-thujene (0.02%)
α-pinene (0.15%)
β-pinene (1.01%)
sabinene (0.09%)
myrcene (0.09%)
limonene (0.70%)
1,8-cineole (0.85%)
(Z)-β-ocimene (0.05%)
γ-terpinene (0.01%)
(E)-β-ocimene (0.01%)
terpinolene (0.01%)
3-octanol (0.10%)
menthone (0.31%)
menthofuran (0.05%)

isomenthone (0.07%)
β-bourbonene (0.12%)
linalool (0.39%)
terpinen-4-ol (0.06%)
β-caryophyllene (2.28%)
trans-dihydrocarvone (2.46%)
cis-dihydrocarvone (0.08%)
α-terpineol (0.09%)
dihydrocarveol 1* (0.02%)
dihydrocarveol 2* (0.02%)
pulegone (0.05%)
α-humulene (0.23%)
germacrene D (3.10%)
carvone (75.51%)
neoisodihydrocarvyl acetate (0.09%)
trans-carvyl acetate (0.02%)
isodihydrocarvyl acetate (0.02%)
δ-cadinene (0.09%)
cis-carvyl acetate (0.06%)

*correct isomer note identified

Mentha spicata oils produced from plants grown in Hungary from Hungarian and German clones were found by Hethelyi et al. (1998) to possess the following constituents:

α-pinene (< 0.1–0.4%)
β-pinene (0.7–1.7%)
limonene (8.9–19.3%)
dihydrocarvone* (8.0–19.1%)
carvone (63.0–63.5%)
β-caryophyllene (1.6–5.4%)
β-cubebene† (1.0–1.1%)

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

Özgüven and Kirici (1999) compared the composition of *M. spicata* grown in two locations in Turkey over two seasons and two harvesting times. They found that the major components varied as follows:

α-pinene (0–2.2%)
β-pinene (0.7–7.9%)
limonene (0–0.5%)
1,8-cineole (9.6–22.7%)
pulegone (0.1–11.3%)
menthyl acetate (0–5.9%)
menthone (0–1.8%)
menthofuran (0.2–4.7%)
menthol (0–1.1%)
carvone (39.4–69.4%)
β-caryophyllene (2.4–4.3%)

It should be noted that the amounts of 1,8-cineole, menthofuran, pulegone and menthyl acetate found were quite unusual. These could be examples of a mixed chemotype or more than one chemotype used to produce a composite oil.

Hadjiakhoondi et al. (2000) analyzed an oil of *M. spicata* produced from plants grown in an experimental garden in Iran. The oil was found to possess the following composition:

α-pinene (0.5%)
camphene (0.1%)
β-pinene (0.9%)
3-octanol (1.0%)
limonene (10.8%)
linalool (11.3%)
α-thujone† (0.7%)
menthofuran† (1.6%)
trans-dihydrocarvone (4.0%)
cis-dihydrocarvone (7.1%)
carvone (22.4%)
piperitone (1.8%)
isopulegyl acetate (8.4%)
p-mentha-1,3-dien-7-al† (0.3%)
menthyl acetate (0.4%)
carvacrol (0.6%)
trans-carvyl acetate (2.7%)
cis-carvyl acetate (0.9%)
piperitone oxide* (1.6%)
β-bourbonene (1.2%)
β-caryophyllen (5.8%)
α-humulene (0.8%)
germacrene D (0.9%)
calamenene* (0.5%)
globulol (0.4%)
T-cadinol (0.7%)
α-cadinol (0.5%)

*correct isomer not identified; †unusual constituent for spearmint oil, corroboration required before acceptance as a constituent can be given

Comparative percentage composition of oils of *Mentha spicata* produced by different methods **T-7**

Compound	Microwave distilled oil	Hydrodistilled oil
α-pinene	0.1	0.8
sabinene	0.3	1.0
β-pinene	0.4	1.4
myrcene	0.5	1.3
3-octanol	1.0	1.8
limonene	9.7	20.2
1,8-cineole	1.5	–
γ-terpinene	0.2	0.8
cis-sabinene hydrate	2.5	1.2
terpinolene	–	0.1
linalool	0.4	0.4
borneol	–	1.2
terpinen-4-ol	0.4	2.6
α-terpineol	0.5	2.1
carvone	64.9	52.3
thymol	5.2	1.9
eugenol	1.2	0.2
β-bourbonene	1.9	2.1
β-elemene	1.7	1.7
β-caryophyllene	3.5	3.4
α-humulene	0.3	0.2
γ-muurolene	2.4	2.1
bicyclogermacrene	0.6	0.5
cis-calamenene	0.3	–

Mohan Rao (2000) compared the lab-distilled composition of fresh and dried leaves of *M. spicata* grown in India. The results obtained have been summarized in **T-2**. The oil yield of fresh leaf oil produced by hydrodistillation was 1.48% compared with oils produced in 1.28% and 0.66% from shade-dried and sun-dried leaves, respectively.

Diaz-Maroto et al. (2002) compared the composition of a simultaneous distillation-extraction produced spearmint oil with that of a supercritical CO_2 extract of the same batch of commercially available spearmint leaves. The comparative compositions can be seen in **T-3**. The conditions used for extraction were 40°C, 120 bar and a CO_2 density of 0.72 g/mL.

An oil of spearmint, which was

produced from *M. spicata* plants collected in the vicinity of Ngaoundere (Cameroon) was the subject of analysis by Jirovetz et al. (2002). The authors compared the composition of the oil with that of its headspace volatiles as determined by SPME-GC/MS. The results of these analyses can be seen in **T-4**.

In addition, the authors examined the chirality of some of the constituents of the Cameroonian spearmint oil. They found that the oil contained (-)-limonene, (+)-menthol, (+)-pulegone, (+) and (-)-dihydrocarvyl acetate, (-)-carvone, (+) and (-)-*trans*-carveol and *cis*-carveol and (-)-*cis*-carvyl acetate. Unfortunately, they did not present any enantiomeric distribution data.

Edris et al. (2003) transplanted stolons of a carvone-rich *M. spicata* clone that was found in Siwa Oasis (Egypt) to a variety of Egyptian locations such as Owainate, Kharga, Samallot and Arish. An oil produced from plants collected in the original location was compared both with oils produced from plants grown in the various locations and one produced from plants obtained from a local market in Cairo. The comparative compositions are presented in **T-5**. As can be seen, the *M. spicata* obtained from the local market possessed an oil that was richest in carvone.

Fresh leaves of *M. spicata* that were collected from Ciudad Real province (Spain) were subjected to different drying conditions such as oven-drying at 45°C, freeze-drying at -198°C, freeze-drying at -18°C and air-drying at 28°C by Diaz-Maroto et al. (2003) to reach an average moisture content of < 10%. Each batch of dried leaves was subjected to micro-simultaneous distillation-extraction using methylene chloride as the solvent. The results of the analyses of the fresh leaf oil and three dried leaf oils are shown in **T-6**. The authors found that air-drying at ambient temperatures and oven-drying at 45°C retained the most volatiles and maintained the structural integrity of the leaves better than freeze-drying.

Oils produced from a carvone-rich *M. spicata* (as *M. crispa*) grown in Reunion by different isolation methods were the subject of analysis by Lucchesi et al. (2004). The results of these analyses are shown in **T-7**.

Benyoussef et al. (2004) compared the composition of oils of *M. spicata* produced from plants collected in the vicinity of Algiers (northern Algeria) and Ouargla (southeastern Algeria). The results of this study are summarized in **T-8**. This oil produced from plants collected in Ouargla and its fractions were found to cause high mortality rates in *Rhyzopertha dominica*, a stored grain pest that causes considerable damage to stored wheat in Algeria (Khalfi et al. 2006).

An oil produced in the laboratory from *M. spicata* plants grown from underground runners collected along the Blue Nile riverbank (Khartoum, Sudan) was analyzed by GC and GC/MS (Younis and Beshir 2004). It was found to contain the following composition:

α-pinene (0.1%)
β-pinene (0.4%)
sabinene (0.3%)
myrcene (0.2%)
limonene (8.8%)

Comparative percentage composition of Algerian oils of *Mentha spicata* **T-8**

Compound	Northern oil	Southern oil
thuja-4(10)-diene	0.3	–
sabinene	–	0.2
β-pinene	0.6	0.3
3-octanol	1.6	–
myrcene	–	0.6
phenylacetaldehyde†	0.3	–
1,8-cineole	8.2	4.3
limonene	0.5	4.0
β-ocimene*	0.5	t
α-terpinene†	0.4	–
trans-sabinene hydrate	2.8	t
octanol	–	t
α-pinene oxide	t	–
terpinolene	0.1	–
cis-sabinene hydrate	0.2	t
nonanal	t	–
linalool	0.1	–
1-octen-3-yl acetate	t	–
3-octyl acetate	t	–
borneol	1.0	1.9
terpinen-4-ol	1.5	–
cis-dihydrocarvone	0.7	3.4
α-terpineol	0.5	1.3
trans-dihydrocarvone	–	0.3
dihydrocarveol	7.1	t
carvone	56.4	79.9
(Z)-3-hexenyl isovalerate	0.3	–
hexyl isovalerate	0.2	–

*correct isomer not identified; †incorrect identification based on GC elution order; t = trace (< 0.1%)

1,8-cineole (2.6%)
(Z)-β-ocimene (0.l%)
p-cymene (0.1%)
3-octanol (0.1%)
menthone (1.6%)
isomenthone (0.6%)
cis-sabinene hydrate (0.4%)
linalool (3.2%)
β-caryophyllene (0.1%)
terpinen-4-ol (0.1%)
α-terpineol (0.1%)
germacrene D (0.2%)
carvone (78.9%)
dihydrocarveol (0.2%)
bicyclogermacrene (0.1%)
trans-carveol (0.3%)
thymol (0.1%)

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