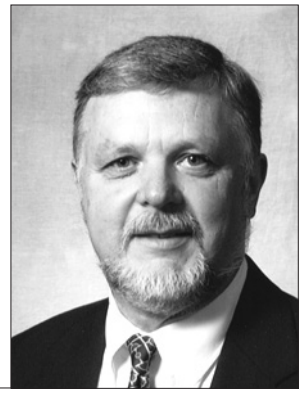

Progress in Essential Oils

By Brian M. Lawrence, R.J. Reynolds Tobacco Company

Bergamot Oil

A number of years ago, Frey (1988) used the selective ion monitoring function of GC/MS to detect the addition of synthetic linalool and linalyl acetate by determining their impurities (dihydrolinalool and dihydrolinalyl acetate). He found that the addition of as little as 2% synthetic linalool and linalyl acetate to genuine bergamot oil could be detected.

Inoma et al. (1989) examined the composition of a commercial sample of bergamot oil available in Japan. The compounds identified in this oil were:

α-thujene (0.15%)
α-pinene (0.70%)
camphene (t)
sabinene (0.72%)
β-pinene (5.11%)
myrcene (0.36%)
p-cymene (3.61%)
limonene (26.71%)
(E)-β-ocimene (0.13%)
γ-terpinene (1.15%)
terpinolene (t)
linalool (18.60%)
limonene oxide* (t)
terpinen-4-ol (t)
α-terpineol (0.12%)
decanal (t)
trans-carveol (t)
cis-carveol (t)
carvone (0.13%)
neral (0.13%)
perillaldehyde (0.11%)
geranial (0.16%)
linalyl acetate (38.13%)
undecanal (t)
neryl acetate (0.47%)
geranyl acetate (0.64%)
dodecanal (0.16%)
β-caryophyllene (0.18%)
α-bergamotene* (0.20%)
β-bisabolene (0.31%)
bergapten (0.11%)

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

Zani et al. analyzed an Italian sample of bergamot oil (1991); it was found to contain the following major constituents:

α-thujene (0.29%)
α-pinene (1.11%)
camphene (0.93%)
β-pinene (6.12%)
myrcene (0.89%)
limonene (39.9%)
γ-terpinene (6.38%)
linalool (10.56%)
linalyl acetate (28.85%)

Mondello et al. (1995 and 1996) and Dugo et al. (1999) determined that Italian bergamot oil contained 60.58% monoterpene hydrocarbons and 1.31% sesquiterpene hydrocarbons in the non-polar hydrocarbon fraction. The monoterpene hydrocarbons identified were:

tricyclene (0.01%)
α-thujene (0.56%)
α-pinene (2.24%)
δ-3-carene (<0.01%)
α-terpinene (0.12%)
p-cymene (0.68%)
camphene (0.10%)
sabinene (1.95%)
β-pinene (11.93%)
myrcene (1.64%)
α-phellandrene (0.05%)
limonene (65.71%)
(Z)-β-ocimene (0.03%)
(E)-β-ocimene (0.42%)
γ-terpinene (14.13%)
terpinolene (0.43%)

In the same oil, the authors also identified the following sesquiterpene hydrocarbons:

cis-α-bergamotene (2.5%)
β-caryophyllene (24.0%)
trans-α-bergamotene (21.9%)
α-humulene (2.0%)
(Z)-β-farnesene (4.1%)
(Z)-β-santalene (1.2%)
germacrene D (4.9%)
β-bisabolene (32.7%)
(Z)-γ-bisabolene (0.1%)
(E)-β-farnesene (0.1%)
β-sesquiphellandrene (0.1%)

In 1996, Verzera et al. examined numerous samples of bergamot oil produced in Italy, the results of which have been previously reported (Lawrence 1999). In addition to the analyses, the authors compared the composition of more than 1,000 genuine oils with two adulterated commercial oils. They found that through the use of component ratios they were able to differentiate between authentic genuine oils with oils that had been adulterated. The results of this component ratio study can be found in Table I.

Using a heptakis-(2,3-di-O-acetyl-6-O-t-butyl-trimethylsilyl)-β-cyclodextrin chiral GC column, Mosandl and Juchelka (1997) determined that the enantiomeric distribution of three components of bergamot oil was as follows:

(3R)-(-)-linalyl acetate (>99%) : (3S)-(+)-linalyl acetate (<1%)
(lS,5S)-(-)-β-pinene (91%) : (lR,5R)-(+)-β-pinene (9%)
(4R)-(+)-limonene (97%) : (4S)-(-)-limonene (3%)

DiGiacomo and DiGiacomo (1999) described the analytical methods used to determine the furanocoumarin content of bergamot oil. They found that, based on the HPLC method (the method of choice), bergamot oil contained the following components:

bergamottin (1.14-2.73%)
citropten (0.1-0.30%)
bergapten (01.6-0.40%)

Furthermore, they stated that the average bergapten content of Calabrian bergamot oil was 0.25%, which is higher than the IFRA regulation recommendation of 0.35%.

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Table I. Component ratio of genuine and two adulterated bergamot oils

Ratios	Genuine Oil	2 Adulterated Commercial Oils
citronallal/terpinen-4-ol	0.167-1.875	0.119-0.136
octyl acetate/α-terpineol	0.842-4.742	0.500-0.561
γ-terpinene/sabinene + β-pinene	0.661-12.79	0.670-0.733
trans-sabinene hydrate acetate/α-terpineol	0.704-3.323	0.303-0.354

Table II. Comparative percentage composition of SFE produced from fresh and dried bergamot peel using different processing conditions

	80 bar, 40°C		90 bar, 50°C		100 bar, 60°C	
Compound	fresh (4.26)[b]	dry[a] (4.31)	fresh (4.37)	dry (5.01)	fresh (3.82)	dry (5.43)
α-thujene	0.2	0.3	0.2	0.3	0.3	0.3
α-pinene	0.7	1.2	1.0	1.3	1.2	1.2
camphene	t	t	t	t	0.1	t
sabinene	0.9	1.0	0.9	0.9	1.0	1.1
β-pinene	6.0	7.6	5.9	7.4	6.9	7.1
myrcene	0.8	1.0	0.9	1.0	1.0	1.0
octanal + α-phellandrene	0.1	0.1	0.1	0.1	0.1	0.1
α-terpinene	0.2	0.2	0.2	0.2	0.2	0.2
p-cymene	1.1	0.1	0.3	0.2	0.4	0.1
limonene	40.0	40.2	40.9	39.7	41.0	41.2
(Z)-β-ocimene	t	t	t	t	t	t
(E)-β-ocimene	0.2	0.3	0.3	0.3	0.3	0.3
γ-terpinene	8.9	8.6	8.3	8.5	9.3	8.5
trans-sabinene hydrate	0.1	t	0.1	t	0.5	0.4
linalool	7.8	4.0	7.8	5.0	8.1	4.6
citronellal	t	t	t	t	0.1	t
terpinen-4-ol	0.1	t	0.1	t	0.3	t
α-terpineol	0.1	t	0.1	t	0.2	t
decanal	0.1	0.1	0.1	0.1	0.1	0.1
octyl acetate	0.1	0.1	0.1	0.1	0.1	0.1
neral	0.2	0.2	0.3	0.1	0.3	0.2
linalyl acetate	28.8	31.2	29.0	31.9	25.6	30.4
geranial	0.3	0.2	0.4	0.2	0.4	0.2
bornyl acetate	0.1	t	t	t	0.1	t
nonyl acetate	t	t	t	t	t	t
linalyl propionate	0.1	t	0.1	0.1	0.1	0.1
α-terpinyl acetate + citronellyl acetate	0.3	0.3	0.3	0.2	0.3	0.2
neryl acetate	0.3	0.3	0.3	0.3	0.3	0.3
geranyl acetate	0.5	0.5	0.4	0.3	0.4	0.4
decyl acetate + dodecanal	0.1	0.1	0.1	t	0.1	t
β-caryophyllene	0.3	0.4	0.3	0.3	0.3	0.3
trans-α-bergamotene	0.4	0.4	0.3	0.3	0.3	0.3

Table II. Continued

Compound	80 bar, 40°C fresh (4.26)[b]	80 bar, 40°C dry[a] (4.31)	90 bar, 50°C fresh (4.37)	90 bar, 50°C dry (5.01)	100 bar, 60°C fresh (3.82)	100 bar, 60°C dry (5.43)
α-humulene + β-farnesene*	0.1	0.1	0.1	0.1	0.1	0.1
germacrene D	0.1	0.1	0.1	t	0.1	0.1
β-bisabolene	0.6	0.6	0.3	0.3	0.4	0.4
nerolidol*	t	0.1	t	t	t	t
tetradecanal	t	t	0.1	0.1	t	t
nootkatone	0.2	0.1	0.1	0.1	0.1	0.1

[a]peel state; [b]kg of CO_2 consumed

Poiana et al. (1999) compared the composition of supercritical fluid CO_2 extracts (SFE) produced separately from fresh and dried bergamot peel using different SFE parameters. The results of this study are summarized in Table II. In addition, the authors also showed that the bergapten content of extracts of fresh peels was higher than those produced from dry peels. They also showed that the bergapten content increased with increased extraction time. Finally, they recommended that the best processing conditions for dried peels was 90 bar at 50°C.

Sawamura et al. (1999) compared the composition of C.P. oils of bergamot produced in Italy and Japan. The results of these analyses are shown in Table III. Sawamura (2000) reported these same results in a summary report covering the composition of many different Citrus species, cultivars and hybrids.

G. Dugo et al. (1999) examined the coumarin and psoralen content of bergamot oil. They found that genuine cold-pressed bergamot oil contained:

bergamottin (1000-2750)[a]
5-geranyloxy-7-methoxycoumarin (80-270)
citropten (120-350)
bergapten (100-320)

[a]mg/100 g of oil

Furthermore, they found that if bergamot oil was treated with NaOH, the lactonic ring of bergapten and citropten would be hydrolyzed while the 5-geranyloxy-7-methoxycoumarin and bergamottin (5-geranyloxypsoralen) were not hydrolyzed because the geranyloxy group sterically protected the lactonic function. As a result, the NaOH treated oil contained the following psoralens and coumarins:

bergamottin (1170-1620)[a]
5-geranyloxy-7-methoxycoumarin (150-200)
citropten (0-50)
bergapten (0-90)

[a]mg/100g of oil

Furthermore, they determined that if the oil was redistilled, the psoralens and coumarins were mostly left behind in the residue, and their level was generally less than 350 mg/100 g of oil for bergamottin and 5-geranyloxy-7-methoxycoumarin, while the content of citropten and bergapten rarely exceeded 50 mg/100 g of oil.

These same authors examined the coumarin and psoralen content of some commercial samples of bergamot oil. They found that their oxygen heterocyclic contents ranged as follows:

bergamottin (680-1160)[a]
5-geranyloxy-7-methoxycoumarin (180-370)
5-geranyloxy-8-methoxypsoralen (+)
5-isopentenyloxy-7-methoxycoumarin (+)
5-isopentenyloxy-8-methoxypsoralen (+)
citropten (100-130)
8-geranyloxypsoralen (+)
herniarin (+)
bergapten (40-100)
isopimpinellin (+)
oxypeucedanin (+)

[a]mg/100g of oil; + presence although actual amount was low (<50 mg/100 g of oil)

As can be seen, the citropten and bergapten levels were lower than those found in genuine oils. Also, the authors pointed out that the presence of 5-geranyloxy-8-methoxypsoralen, 5-isopentenyloxy-7-methoxycoumarin, 5-isopentenyloxy-8-methyoxypsoralen, herniarin and isopimpinellin indicate that the oils have been adulterated with lime oil.

In addition to the previously listed coumarins and psoralens, P. Dugo et al. (1999) also characterized and confirmed the presents of tetra-O-methylscutellarein (4l,5,6,7-tetramethoxyflavone) and sinensetin (3l,4l,5,6,7-pentamethoxyflavone) in genuine bergamot oil using liquid chromatography-atmospheric pressure chemical ionization mass spectrometry.

Kawaii et al. (1999) reported that the major furanocoumarins found in bergamot oil were:

bergamottin (96.7)[a]
bergapten (152.5)
citropten(21.7)

[a]µg/100mg of dried peel

Table III. Comparative percentage composition of Italian and Japanese bergamot oils

Compound	Fantasicoa	Consorziob	Balotinc	Compound	Fantasicoa	Consorziob	Balotinc
α-pinene	1.52	1.45	1.29	(E)-2-decenal	t	t	-
camphene	0.03	0.03	0.03	β-santalene	t	t	t
β-pinene	10.09	8.93	7.47	α-humulene	t	t	t
sabinene	1.47	1.16	1.25	(E)-β-farnesene	0.06	0.07	0.11
myrcene	0.87	0.83	0.66	neral	0.25	0.16	0.27
α-phellandrene	0.03	0.03	0.02	δ-muurolene‡	t	t	-
α-terpinene	0.16	0.10	0.13	decyl acetate	t	t	t
limonene	35.66	35.62	21.31	α-terpineol	0.19	0.16	0.10
β-phellandrene	0.23	0.19	0.19	α-terpinyl acetate	-	-	t
(E)-β-ocimene	0.03	0.03	0.02	germacrene D	0.03	0.03	0.03
γ-terpinene	7.32	6.53	5.30	dodecanal	t	t	-
p-cymene	0.33	0.97	0.02	neryl acetate	-	-	t
terpinolene	0.28	0.23	0.21	β-bisabolene	0.45	0.45	0.76
2,7-dimethyl-1, 6-octadiene	-	-	t	geranial	0.39	0.47	-
heptyl acetate	t	t	t	geranyl acetate	0.42	0.47	0.24
cis-limonene oxide	t	t	-	decanol	t	-	-
trans-limonene oxide	t	t	-	citronellol	t	t	t
trans-linalool oxide†	t	-	-	nerol	0.02	0.07	0.07
octyl acetate	0.19	0.12	0.16	germacrene B	t	t	-
decanal	0.11	-	0.08	*trans*-carveol	t	-	-
solanone	0.12	0.10	0.08	geraniol	0.09	0.11	0.02
linalool	4.81	8.67	20.02	α-sinensal	t	-	-
linalyl acetate	30.76	29.12	36.37	perillyl alcohol	t	-	-
cis-sabinene hydrate	0.07	0.04	0.03	caryophyllene oxide	t	-	-
nonyl acetate	t	t	t	(Z)-nerolidol	t	t	-
α-bergamotene*	0.25	0.30	0.25	(E)-nerolidol	t	t	t
β-elemene	t	t	-	verbenol‡	t	t	t
β-caryophyllene	0.15	0.21	0.22	hydroxylinalool‡	t	t	t
terpinen-4-ol	0.03	0.03	0.05	nootkatone	0.18	0.07	0.07
undecanal	-	-	t	elemol	-	-	t

[a]Italian cultivar oil; [b]oil produced by Consortium of Bergamot; [c]Japanese cultivar oil; *correct isomer not identified; †furanoid form; ‡incorrect identification based on elution order; t = trace (<0.01%)

In 1999, Bonaccorsi et al. developed the technique known as fast HPLC to analyze the non-volatile oxygen heterocyclic components of Italian bergamot oil. Although the authors did not present any quantitative data, they showed that they were able to reduce the analysis time from 20-45 min to only 7 min with good separation of the following components:

citropten
bergapten
bergamottin
5-geranyloxy-7-methoxycoumarin

A combination of this technique along with GC/MS and chiral GC analysis will go a long way to determine the authenticity of bergamot oil.

Dugo et al. (1999 and 2001) also determined that the enantiomeric distribution of seven components in oils produced between December and March in Italy was as follows:

(1R,5R)-(+)-α-pinene (6.8-9.5%) : (1S,5S)-(-)-α-pinene (90.5-93.2%)
(1R,5R)-(+)-sabinene (14.1-18.2%) : (1S,5S)-(-)-sabinene (81.2-85.9%)
(4R)-(+)-limonene (97.3-98.1%) : (4S)-(-)-limonene (1.9-2.7%)
(3S)-(+)-linalool (0.3-0.6%) : (3R)-(-)-linalool (99.4-99.7%)
(3S)-(+)-linalyl acetate (0.1-0.3%) : (3R)-(-)-linalyl acetate (99.7-99.9%)

(4S)-(+)-terpinen-4-ol (9.7-26.3%) :
(4R)-(-)-terpinen-4-ol (73.7-90.3%)
(4R)-(+)-α-terpineol (30.6-82.5%) :
(4S)-(-)-α-terpineol (17.5-69.4%)

An Italian bergamot oil was heated on an aroma lamp to reduce its weight by 50%. The headspace composition of this heated oil was compared with that of the headspace composition of the original oil and the actual oil composition (Oberhofer et al 1999). The results of this comparative study are shown in Table IV. As can be seen, after heating the oil, the linalyl acetate is thermally degraded to linalool while the other terpene esters increased.

Bergamot oil and bergamot extract was produced by either water distillation or a pentane/diethyl ether (1:1) solvent extraction of either the zest or the whole fruit by Kiwanuka et al. (2000). The extract was further water distilled separately at pH 2.5 and pH 5.3 to determine the effect of pH on the oil composition. A summary of the results of this study can be seen in Table V. The authors further examined the enantiomeric distribution of six oil constituents using multidimensional GC with the chiral GC column of choice being heptakis-(2,3,6-tri-O-methyl)-β-cyclodextrin. The enantiomeric distribution of limonene, linalool, linalyl acetate, α-and β-pinene and α-terpineol can be seen in Table VI. Examination of this data revealed that racemization of (3R)-(-)-linalyl acetate by various processing conditions did not occur, whereas it did show that pH had a marked effect on the racemization of α-terpineol and linalool.

Verzera et al. (2000) analyzed bergamot oils produced from different cultivars grown in Sicily and compared them against the range of oil composition of Calabrian bergamot oil. These analyses can be seen summarized in Table VII. The authors also determined that the enantiomeric distribution of linalool and linalyl acetate for the cultivar oils were as follows:

(3R)-(-)-linalool (99.4-99.5%) : (3S)-(+)-linalool (0.5-0.6%)
(3R)-(-)-linalyl acetate (99.7-99.8%) : (3S)-(+)-linalyl acetate (0.2-0.3%)

Kirbaslar et al. (2000) analyzed a bergamot oil produced from fruit grown in Antalya (Turkey) and found that it contained:

α-thujene (0.2%)
α-pinene (0.5%)
camphene (t)
sabinene (0.5%)
β-pinene (3.0%)
myrcene (2.0%)
hexyl acetate (0.2%)
δ-3-carene (0.9%)
α-terpinene (0.2%)
p-cymene (0.1%)
m-cymene (0.3%)
limonene + β-phellandrene (23.7%)
(Z)-β-ocimene (t)
γ-terpinene (4.7%)
terpinolene (0.4%)
linalool (14.7%)
2-phenethyl alcohol (0.5%)
nonanal (t)
citronellal (t)
terpinen-4-ol (t)
α-terpineol (0.1%)
decanal (0.1%)
octyl acetate (0.2%)
nerol + citronellol (0.3%)
neral (0.4%)
linalyl acetate (38.7%)
geranial + perillaldehyde (t)
bornyl acetate (t)
nonyl acetate (t)
octadienyl formate* (0.1%)
citronellyl acetate (0.1%)
p-menth-1-en-8-yl acetate (t)
neryl acetate (1.6%)
geranyl acetate (1.6%)
dodecanal (t)
decyl acetate (0.1%)
cis-α-bergamotene (t)
β-caryophyllene (0.7%)
trans-α-bergamotene (0.9%)
α-humulene (0.1%)
1-hydroxy-linalool† (0.1%)
(Z)-β-farnesene (0.1%)
germacrene D (0.1%)
β-bisabolene (1.2%)
(E)-nerolidol (0.1%)
α-bisabolol (0.1%)
nootkatone (0.5%)
dimethoxycoumarin* (0.1%)
bergapten (0.2%)

*correct isomer not identified; † identity in question; t = trace (<0.1%)

These same authors (Kirbaslar et al 2001) compared the composition of Turkish bergamot oil produced from fruit

Table IV. Percentage composition of bergamot oil and the headspace of a original oil and a heated oil

Compound	Oil	Headspace Original Oil	Headspace Heated Oil
α-thujene	t	t	t
α-pinene	0.46	0.72	t
sabinene	t	t	t
β-pinene	2.97	4.28	t
myrcene	1.78	1.87	3.05
p-cymene	0.67	1.24	t
limonene	24.50	33.34	0.65
(Z)-β-ocimene	0.46	0.35	t
(E)-β-ocimene	0.82	0.62	3.50
γ-terpinene	2.54	2.54	t
terpinolene	t	t	t
linalool	22.49	18.69	78.84
decanal	t	t	0.91
geranial	0.26	0.24	0.70
linalyl acetate	40.61	33.56	4.07
neryl acetate	0.21	t	1.82
geranyl acetate	0.38	0.23	0.87
β-caryophyllene	0.11	t	t
trans-β-bergamotene	0.91	t	t
β-bisabolene	0.13	t	t

t = trace (<0.01%)

Table V. Average percentage composition of an extract and oils produced in different ways from bergamot zest and whole fruit

Compound	Zest Extract	Zest Oil	Whole Fruit Oil	Oil from Extract at pH 2.5
α-pinene	0.81	0.78	1.18	0.68
β-pinene	4.94	4.96	5.29	3.71
sabinene	1.04	0.98	0.24	0.54
myrcene	1.53	2.13	2.49	1.79
α-terpinene	0.13	0.17	0.46	0.27
limonene	40.20	41.65	44.43	42.19
β-ocimene*	0.12	0.46	0.50	0.73
β-phellandrene	0.31	0.35	0.31	0.38
γ-terpinene	7.27	7.73	10.05	7.82
terpinolene	0.29	0.43	1.16	0.76
linalool	5.22	10.68	13.59	12.79
linalyl acetate	30.50	21.41	5.38	15.86
decanal	0.10	0.14	0.20	0.20
terpinen-4-ol	0.02	0.15	2.11	0.55
α-terpineol	0.04	2.04	4.77	5.15
neryl acetate	0.25	0.77	0.65	0.67
geranyl acetate	0.24	1.27	1.15	0.96
geranial	0.22	0.22	0.11	0.19
nerol	0.06	0.48	1.24	0.73
geraniol	0.10	1.24	1.97	2.07
nootkatone	0.25	0.11	0.06	0.13

*correct isomer not identified

Table VI. Enantiomeric distribution of selected components of an extract and oils produced in different ways from bergamot zest and whole fruit

Compound	Zest Extract	Whole Fruit Oil	Oil Produced from Extract pH 5.3	pH 2.5
(4R)-(+)-limonene	98.1	98.0	98.2	98.5
(4S)-(-)-limonene	1.9	2.0	1.8	1.5
(3R)-(-)-linalool	>99.0	56.3	78.1	61.9
(3S)-(+)-linalool	<1.0	43.7	21.9	38.1
(3R)-(-)-linalyl acetate	>99.0	>99.0	>99.0	<99.0
(3S)-(+)-linalyl acetate	<1.0	<1.0	<1.0	<1.0
(1S,5S)-(-)-α-pinene	67.8	67.5	65.7	65.8
(1R,5R)-(+)-α-pinene	32.2	32.5	34.3	34.2
(1S,5S)-(-)-β-pinene	92.9	92.3	92.7	92.0
(1R,5R)-(+)-β-pinene	7.1	7.7	7.3	8.0
(4R)-(+)-α-terpineol	-	51.3	71.8	54.5
(4S)-(-)-α-terpineol	-	48.7	38.2	45.5

Table VII. Percentage composition of Sicilian (cultivars) and Calabrian bergamot oils

Compound	Sicilian Oils				Calabrian oils
	Castagnaro	Fantastico	Feminello	PCF	(range)
tricyclene	t	t	t	t	t
α-thujene	0.30	0.29	0.19	0.22	0.19-0.46
α-pinene	1.18	1.14	0.75	0.90	0.72-1.76
camphene	0.03	0.03	0.02	0.02	0.02-0.05
sabinene + β-pinene*	7.09	6.63	3.70	5.90	4.81-12.69
6-methyl-5-hepten-2-one	t	t	t	t	t-0.01
myrcene	1.20	1.07	0.96	0.96	0.63-1.23
octanal	0.03	0.03	0.02	0.02	0.03-0.08
α-phellandrene	0.02	0.04	0.03	0.02	0.02-0.04
hexyl acetate	t	t	t	t	t
δ-3-carene	t	t	t	t	t
α-terpinene	0.17	0.16	0.10	0.12	0.10-0.24
p-cymene	0.04	0.04	0.02	0.03	0.01-0.72
limonene + β-phellandrene	51.32	43.80	40.48	40.89	25.38-45.41
1,8-cineole	t	t	t	t	0.01-0.02
(Z)-β-ocimene	0.01	0.01	0.01	0.01	0.01-0.05
(E)-β-ocimene	0.13	0.15	0.13	0.16	0.17-0.32
γ-terpinene	7.63	7.07	4.68	6.03	5.27-11.15
cis-sabinene hydrate	0.04	0.05	0.03	0.04	0.01-0.05
octanol	t	t	t	t	t-0.01
terpinolene	0.32	0.31	0.21	0.26	0.22-0.45
linalool	1.32	4.93	9.27	4.23	3.63-22.68
nonanal	0.04	0.02	0.01	0.03	0.02-0.06
heptyl acetate	t	t	t	t	t-0.02
cis-limonene oxide	t	t	t	t	t
trans-limonene oxide	t	t	t	t	t
isopulegol	t	t	t	t	t
camphor	t	t	t	t	t-0.08
citronellal	0.02	0.02	0.02	0.02	t-0.02
terpinen-4-ol	0.02	0.02	0.01	0.02	0.01-0.03
α-terpineol	0.03	0.05	0.05	0.03	0.04-0.13
decanal	0.07	0.04	0.03	0.06	0.05-0.10
octyl acetate	0.16	0.07	0.04	0.16	0.07-0.20
nerol + citronellol	0.01	0.02	0.03	0.02	0.02-0.08
neral	0.13	0.26	0.29	0.20	0.10-0.28
trans-sabinene hydrate acetate	0.08	0.08	0.05	0.07	0.05-0.10
linalyl acetate	26.01	30.91	36.43	36.67	21.84-41.36
geraniol	t	t	t	t	t
geranial* + perillaldehyde	0.19	0.38	0.43	0.29	0.20-0.44
bornyl acetate	0.02	0.02	0.01	0.02	0.01-0.02
undecanal	0.01	0.01	t	0.01	t-0.02
nonyl acetate	0.04	0.03	0.01	0.04	0.01-0.03

Table VII. Continued

Compound	Sicilian Oils				Calabrian oils
	Castagnaro	Fantastico	Feminello	PCF	(range)
methyl geranate	0.01	0.01	0.01	t	t-0.01
δ-elemene	t	t	t	t	t
linalyl propionate	0.04	0.04	0.04	0.04	0.02-0.06
α-terpinyl acetate	0.16	0.15	0.10	0.16	0.07-0.22
citronellyl acetate	0.03	0.02	0.03	0.03	0.01-0.03
neryl acetate	0.25	0.31	0.30	0.39	0.13-0.64
geranyl acetate	0.34	0.20	0.16	0.17	0.11-0.51
dodecanal	0.05	0.03	0.02	0.04	0.02-0.05
decyl acetate	0.02	0.02	0.02	0.02	0.01-0.03
cis-α-bergamotene	t	t	t	t	0.02-0.05
β-caryophyllene	0.28	0.23	0.23	0.27	0.15-0.43
trans-α-bergamotene	0.30	0.27	0.26	0.28	0.16-0.36
α-humulene	0.02	0.06	0.01	0.01	0.01-0.04
β-santalene	0.01	t	t	t	t-0.02
(Z)-β-farnesene	0.05	0.02	t	t	0.03-0.09
dodecanol	t	t	t	t	0-t
germacrene D	0.05	0.05	0.04	0.08	0.03-0.11
bicyclogermacrene	0.03	0.02	0.03	0.04	0.01-0.04
(E,E)-α-farnesene	0.01	0.02	t	t	t
β-bisabolene	0.41	0.39	0.37	0.41	0.21-0.51
(Z)-γ-bisabolene	t	t	t	t	t-0.01
germacrene B	0.01	0.01	0.01	0.01	t-0.01
(E)-nerolidol	0.01	t	0.01	0.01	t-0.01
tetradecanal	0.01	0.01	t	0.01	0-t
2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	0.01	0.01	0.01	0.01	0.01-0.02
campherenol	0.02	0.20	0.02	0.02	0.01-0.02
α-bisabolol	0.02	0.02	0.02	0.02	0.01-0.02
nootkatone	0.06	0.07	0.06	0.08	0.01-0.09

t = trace (<0.01%); * major component; PFC is a new clone of the Feminello cultivar

harvested at three different times. The tree harvesting dates were December 25 (completely green fruit), January 23 (50-75% green fruit) and February 20 (yellow-fully ripe fruit). The results of this study are presented in Table VIII. As can be seen, the linalyl content reached its maximum only when the fruit were mature. Kirbaslar et al. also water distilled some fresh, mature bergamot fruit and found that the oil composition was as follows:

α-thujene (0.2%)
α-pinene (0.9%)
camphene (t)
sabinene (0.5%)
β-pinene (3.8%)
myrcene (2.3%)
α-terpineol (3.0%)
nerol (0.8%)
linalyl acetate (17.3%)
geranial (0.8%)
geraniol (1.7%)
bornyl acetate (0.1%)
α-phellandrene (0.1%)
hexyl acetate (0.1%)
α-terpinene (0.7%)
p-cymene (t)
m-cymene (t)
limonene (33.2%)
(E)-β-ocimene (0.3%)
γ-terpinene (6.5%)
1,8-cineole (t)
linalool (20.1%)
1,3-vinylbenzene† (0.2%)
camphor (0.1%)
citronellol (t)
terpinene-4-ol (0.5%)
nonyl acetate (t)
citronellyl acetate (t)
neryl acetate (2.1%)
geranyl acetate (2.7%)
α-terpinyl isobutyrate (0.2%)
dodecanal (t)
decyl acetate (t)
cis-α-bergamotene (0.4%)
β-caryophyllene (0.3%)
trans-α-bergamotene (t)
(Z)-β-farnesene (t)
cis-β-santalene (0.1%)
β-bisabolene (0.8%)

t = trace (<0.1%); † identity required confirmation

Table VIII. Percentage composition of cold-pressed Turkish bergamot oils during fruit maturation

Compound	Green Fruit Oil	Green/Yellow Fruit Oil	Yellow Fruit Oil
α-thujene	0.1	0.2	0.2
α-pinene	0.5	0.6	0.6
camphene	t	t	t
sabinene	0.3	0.4	0.4
β-pinene	2.7	3.5	3.9
myrcene	1.2	1.3	1.3
hexyl acetate	0.1	0.1	0.1
α-terpinene	0.2	0.2	0.2
p-cymene	0.1	0.1	0.1
limonene	36.4	36.9	37.2
(Z)-β-ocimene	t	t	t
(E)-β-ocimene	0.3	0.3	0.4
γ-terpinene	5.1	5.7	5.9
1,8-cineole	t	t	t
terpinolene	0.3	0.3	0.3
linalool	18.7	13.8	7.9
nonanal	t	t	t
citronellal	t	t	t
terpinen-4-ol	t	t	t
α-terpineol	0.1	0.1	0.1
decanal	t	t	t
octyl acetate	0.1	0.1	0.2
neral	0.4	0.4	0.4
nerol	0.2	0.2	0.2
linalyl acetate	29.5	31.9	36.3
geranial	t	t	t
bornyl acetate	t	t	t
nonyl acetate	t	t	t
citronellyl acetate	0.1	0.1	0.1
3,7-dimethyl,3-hydorxy, 1,6-octadienyl formate†	0.1	0.1	0.1
p-menth-1-en-8-yl-acetate	0.1	0.1	0.2
neryl acetate	0.7	0.8	1.1
geranyl acetate	0.5	0.6	0.7
dodecanal	t	t	t
decyl acetate	0.1	0.1	0.1
cis-α-bergamotene	t	t	t
β-caryophyllene	0.3	0.3	0.3
trans-α-bergamotene	0.4	0.4	0.4
(Z)-β-farnesene	0.1	0.1	0.1
α-humulene	0.1	0.1	0.1
1-hydroxy linalool†	0.1	0.1	0.1
germacrene D	t	t	t
cis-β-santalene	t	t	t
β-bisabolene	0.6	0.6	0.6
(E)-nerolidol	0.1	0.1	0.1
α-bisabolol	t	t	t
nootkatone	0.1	0.1	0.1

† identity requires confirmation

Table IX. Comparative percentage composition of bergamot oil and an oil partially de-terpenified with solid CO_2

Compound	Original Oil	Partially Deterpenified Oil
α-thujene	0.3	0.1
α-pinene	1.2	0.3
sabinene	1.2	0.5
β-pinene	7.3	2.8
myrcene	5.1	4.5
p-cymene	1.0	0.8
limonene	41.3	30.2
(Z)-β-ocimene	1.5	1.5
(E)-β-ocimene	2.7	2.5
γ-terpinene	7.9	6.9
terpinolene	0.6	0.6
linalool	6.9	9.3
linalyl acetate	18.8	35.2
neryl acetate	1.2	1.4
geranyl acetate	2.4	2.6
β-bisabolene	0.6	0.8

Ogawa et al. (2000) screened a large number of Citrus species, hybrids and cultivars for their auraptene content. They found that bergamot peel and fruit was completely devoid of auraptene. As a result, if auraptene is ever found in bergamot oil, it is an indicator of adulteration.

Russo et al. (2001) used solid CO_2 to deterpenify bergamot oil. The oil and dry ice were mixed in a cylindrical vessel and during stirring the hydrocarbon fraction became entrained in the CO_2 gas stream. A comparison between the original oil (100 mL) and the partially deterpenified oil which used 1,500 g dry ice to achieve the compositional change, which can be seen in Table IX.

This same year, Feger et al. (2001) examined the sesquiterpene hydrocarbon fraction of a range of Citrus oils to see if the germacrene content differed amongst the oils. In addition to finding that the ratio of germacrenes in a citrus oil was somewhat specific to the oil type, they found that bergamot oil contained the following germacrenes:

germacrene A (0.01%)
germacrene B (0.01%)
germacrene C (0.02%)
germacrene D (0.5-0.6%)
bicyclogermacrene (0.1%)

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Lemongrass Oil

In 1992, Lamaty et al. analyzed a number of oils produced from plants harvested in tropical parts of Africa. Using GC/MS as their method of analysis, the authors analyzed lemongrass oils produced from plants harvested in the Congo, Benin and Cameroon. These oils, which were produced from

C. citratus possessed the same three major constituents in approximately the same amounts as shown below:

geranial (46%)
neral (34%)
myrcene (13%)

The following year, Zhu et al. (1993) analyzed an oil of *C. citratus* produced in the Kunming region of Yunnan (China). This oil was reported to contain:

6-methyl-5-hepten-2-one (0.10%)	neral (35.24%)
myrcene (14.61%)	geranial (36.69%)
(Z)-β-ocimene (0.61%)	2-undecanone (0.15%)
(E)-β-ocimene (0.48%)	geranyl acetate (0.52%)
linalool (1.98%)	β-caryophyllene (0.18%)
citronellal (0.65%)	α-bergamotene* (0.21%)
limonene oxide* (6.43%)	2-tridecanone (0.19%)
cis-carveol (0.27%)	

*correct isomer not identified

A sample of lemongrass oil produced in New Zealand was found by Lis-Balchin et al. (1996) to possess the following major constituents:

limonene (4.6%)
neral (26.1%)
geranial (42.5%)

Changonda and Chalchat (1997) found that the main constituents of an oil of *C. citratus* produced from plants grown in Zimbabwe were neral (30%) and geranial (41%)

with two other less major constituents being myrcene and geraniol.

Chalchat et al. (1997) analyzed an oil of lemongrass produced from *C. citratus* plants of Cameroonian origin. They found that the oil contained the following major constituents:

6-methyl-5-hepten-2-one (0.8%)	neral (33.5%)
myrcene (12.8%)	geraniol (2.5%)
limonene (0.3%)	geranial (45.9%)
linalool (1.2%)	geranyl acetate (0.2%)
citronellol (0.1%)	

Also in 1997, Bhattacharya et al. reported that growing in India there were four *C. flexuosus* cultivars (OD-19, Pragati, Cauvery, SKK-7) rich in neral/geranial, one rich in geraniol (GR-1) and one hybrid (CKP-25) [*C. flexusus x C. khasianus* (Hack) Stapf ex. Bor.] rich in neral/geranial that have been released for commercial cultivation. A comparison of their chemical compositions can be seen in Table X.

In addition to the above named lemongrass cultivars, Kulkarni (2000) listed "SD-68" and "Krishna" as additional cultivars grown in India. He also noted that the oil yield per hectare of the Krisha cultivar was more than double that of any other cultivars.

Baratta et al. (1998) screened a commercial oil of lemongrass for its antimicrobial and antioxidant properties. The composition of this oil was follows:

6-methyl-5-hepten-2-one (2.7%)	allo-ocimene* (t)
myrcene (3.9%)	citronellal (1.7%)
1,8-cineole (0.1%)	geranial (45.7%)
limonene (0.1%)	citronellyl acetate (0.1%)
(Z)-β-ocimene (0.9%)	geranyl acetate (0.5%)
(E)-β-ocimene (0.7%)	β-caryophyllene (0.2%)
α-thujone (1.7%)	*trans*-α-bergamotene (0.1%)

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

This oil would appear to be either poorly analyzed, adulterated or lab-made, because neral was not identified as a major constituent.

Chisowa et al. (1998) analyzed an oil of *C. citratus* produced from plants grown in Zambia. This oil was found to contain the following constituents:

α-pinene (t)	linalool (1.3%)
myrcene (18.0%)	2-undecanone (0.5%)
limonene (0.2%)	neral (29.4%)
1,8-cineole (1.0%)	α-terpineol (0.3%)
(Z)-β-ocimene (0.4%)	geranial (39.0%)
(E)-β-ocimene (0.3%)	citronellol (0.3%)
6-methyl-5-hepten-2-one (0.8%)	2-tridecanone (0.2%)
verbenol* (t)	geraniol (1.7%)

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

Numerous oils of *C. citratus* produced from plants grown in Mali were subjected to analysis by Chalchat et al. (1998). They found that the oils ranged in composition in the following way:

myrcene (6.2-9.1%)	*cis*-α-bergamotene (0.1-0.3%)
limonene (0.1-0.2%)	neral (20.1-32.5%)
(Z)-β-ocimene (0.2-0.4%)	geranial (45.3-56.7%)
(E)-β-ocimene (0.1-0.3%)	γ-cadinene (0.1-0.3%)
6-methyl-5-hepten-2-one (1.2-2.3%)	δ-cadinene (t-0.1%)
citronellal (t-0.1%)	geranyl acetate (0.2-0.4%)
cis-verbenol (0.3-0.6%)	citronellol (0.1-0.2%)
linalool (0.6-0.9%)	nerol (0.2-0.4%)
trans-verbenol (0.7-1.9%)	geraniol (3.0-5.6%)
bornyl acetate (t-0.2%)	

t = trace (<0.1%)

Chalchat et al. also reported that oils produced from *C. citratus* from plants collected in the Ivory Coast were found to possess the following composition:

sabinene (0.2%)	neral (23.6-26.3%)
myrcene (18.1-22.0%)	geranial (30.5-34.0%)
limonene (t)	γ-cadinene (0.4-0.6%)
1,8-cineole (t-0.1%)	geranyl acetate (2.1-3.5%)
(Z)-β-ocimene (0.3-0.4%)	citronellol (0.1-0.2%)
(E)-β-ocimene (0.2-0.3%)	nerol (0.1%)
geijerene (0.4%)	geraniol (6.2-7.7%)
6-methyl-5-hepten-2-one (2.4-2.5%)	elemol (0.2-0.7%)
citronellol (t-0.1%)	germacrene D-4-ol (0.1-0.2%)
cis-verbenol (t-0.6%)	α-muurolol (0.1%)
linalool (1.2-1.3%)	T-muurolol (0.1-0.2%)
trans-verbenol (1.1-1.2%)	α-eudesmol (0.1%)
bornyl acetate (t-0.1%)	β-eudesmol (0.1-0.2%)
cis-α-bergamotene (0.6-0.8%)	α-cadinol (0.3-0.4%)

t = trace (<0.1%)

These same analyses were also repeated in a second publication (Sidibé et al. 2001).

Bhattacharya et al. (1998) studied the effect of a prolonged storage (1 year) under Indian ambient conditions of lemongrass oil ex. *C. flexusus*. The results of this study, which can be seen in Table XI, revealed that Indian ambient conditions of storage are deleterious to the composition of lemongrass oil; particularly the neral and geranial levels. The user of Indian lemongrass oil should note that high neral/geranial levels can be maintained if an antioxidant is used. As a result, it is advisable to check these oils to ensure that no antioxidant is present.

Viturro and Bucu (1998) used a combination of chromatographic techniques including GC/MS to analyze lemongrass oil produced from *C. citratus* plants grown in Jujuy (Argentina). The composition of this oil was found to be as follows:

myrcene (10.36%)	geraniol (2.42%)
(Z)-β-ocimene (0.42%)	geranyl acetate (0.20%)
(E)-β-ocimene (0.26%)	β-caryophyllene (0.09%)
α-thujone† (0.13%)	α-humulene (0.03%)
linalool (0.76%)	germacrene D (0.04%)
6-methyl-5-hepten-2-one (0.56%)	2-undecanone (0.37%)

Table X. Comparative percentage composition of the oils of *Cymbopogon flexuosus* cultivars and one hybrid

Compound	OD-19	Pragati	Cauvery	SHK-7	GRL-1	CKP-25
(Z)-3-hexenol	-	-	-	-	0.08	0.08
α-pinene	-	-	-	-	-	0.06
6-methyl-5-hepten-2-one	0.48	0.54	0.34	0.45	0.07	0.87
myrcene	0.16	0.03	0.04	0.06	3.97	1.40
α-phellandrene	-	0.03	-	-	0.07	0.27
p-cymene	0.05	-	-	-	-	0.22
limonene	0.13	0.29	0.10	0.84	0.28	3.79
(Z)-β-ocimene	-	0.03	-	0.10	0.96	0.07
(E)-β-ocimene	0.06	0.03	0.06	0.07	1.37	0.13
γ-terpinene	-	-	-	-	0.54	0.04
terpinolene	-	-	0.05	-	-	0.09
linalool	0.42	0.47	0.59	0.49	1.42	0.79
camphor	0.14	0.17	0.23	0.16	-	0.53
β-terpineol*	-	-	-	-	-	0.17
citronellal	0.07	0.10	0.06	0.07	0.22	0.38
isopulegol	0.27	0.47	0.27	0.25	-	1.23
borneol	-	0.02	0.03	-	-	-
terpinen-4-ol	0.57	0.04	0.55	0.58	-	0.15
p-cymen-8-ol	-	0.14	-	-	-	-
α-terpineol	0.13	0.10	-	-	0.08	0.06
decanal	-	-	-	-	0.18	0.26
citronellol	0.05	0.04	-	-	0.60	-
neral	33.31	36.06	33.31	33.32	-	34.93
piperitone	-	-	-	-	0.07	0.06
geraniol	0.31	0.23	0.60	0.34	80.21	1.03
geranial	53.38	53.63	52.11	54.52	-	45.07
bornyl acetate	-	-	-	-	-	0.31
citronellyl acetate	-	0.87	1.04	-	0.10	-
neryl acetate	-	-	0.06	-	-	-
geranyl acetate	0.11	0.10	0.25	0.38	4.60	3.97
α-copaene	-	-	0.04	-	-	-
β-elemene	-	0.30	0.09	0.08	0.15	0.14
β-caryophyllene	0.14	0.68	0.27	0.19	0.18	0.09
α-humulene	0.14	0.11	0.12	0.08	0.18	0.06
β-bisabolene	0.09	0.05	0.06	0.05	0.08	-
γ-cadinene	0.26	0.32	0.60	0.38	-	-
δ-cadinene	-	0.04	0.03	-	-	-
elemol	0.66	0.09	0.07	0.06	-	-
caryophyllene oxide	1.55	1.21	1.38	1.46	0.09	-
α-cadinol	-	-	-	-	0.44	0.31

*correct isomer not identified

camphor (0.02%)
cis-linalool oxide* (0.01%)
citronellal (0.20%)
trans-linalool oxide* (0.14%)
neral + geranial (77.94%)
β-pinene oxide† (1.66%)
borneol (0.04%)
aromadendrene (0.06%)
nerol (0.55%)
viridiflorol (0.05%)

*correct isomer not identified; †probable misidentification

This same year, Liu et al. (1998) determined the composition of an oil of *C. citratus* produced from plants grown in China. They reported that the oils contained:

2-pentene† (3.59%)
valancene (1.86%)
myrcene (4.93%)
aromadendrene (0.19%)
(E)-β-ocimene (1.29%)
γ-gurjunene (0.40%)
neral (29.45%)
T-muurolol (0.39%)
geranial (39.12%)
selin-7(11)-4α-ol (0.31%)
α-farnesene* (0.31%)
geranic acid (2.98%)
δ-cadinene (0.21%)
α-cadinol (0.15%)
citronellol (0.39%)
farnesol* (0.27%)
nerol (0.49%)
(E,E)-farnesol (0.27%)
geraniol (4.03%)
dibutyl phthalate‡ (0.20%)
caryophyllene oxide (0.15%)
palmitic acid (0.20%)

*correct isomer not identified; †probably misidentification; ‡artifact, does not occur naturally but is obtained from plastic (it is a plasticizer)

A commercial sample of lemongrass oil of unknown geographic origin was determined by Reichling et al. (1999) to possess the following composition:

α-pinene (0.29%)
geranial (43.44 %)
camphene (0.63%)
citronellol (2.80%)
limonene (3.60%)
β-cubebene (5.01%)
methyl eugenol (0.28%)
β-caryophyllene (2.38%)
linalool (0.70%)
α-humulene (0.51%)
citronellal (0.57%)
caryophyllene oxide (0.50%)
borneol (0.85%)
α-terpineol (0.25%)
neral (30.19%)

A commercial sample of lemongrass oil purchased in Austria was subjected to GC/MS analysis by Oberhofer et al. (1999) and its composition can be seen in Table XII. Furthermore, the authors compared the headspace composition of the neat oil with that of an oil that had been reduced in weight by 50% by heating with an aroma lamp. As can be seen, the headspace composition changed drastically between the neat oil and the heated oil.

Lemongrass oil produced from plants collected in Burkina Faso were analyzed by Menut et al. (2000) and found to contain:

6-methyl-5-hepten-2-one (0.7%)
citronellol (0.4%)
myrcene (10.7%)
nerol (1.5%)
1,8-cineole (0.2%)
neral (33.0%)
(Z)-β-ocimene (0.1%)
geraniol (2.3%)
(E)-β-ocimene (0.1%)
geranial (44.6%)
linalool (0.8%)
2-undecanone (0.1%)
nonanal (0.1%)
neric acid (0.2%)
camphor (0.1%)
heliotropin (0.3%)
citronellal (1.1%)
geranic acid (0.4%)
isoborneol (1.0%)
eugenol (0.7%)
terpinen-4-ol (0.3%)
geranyl acetate (0.2%)
α-terpineol (0.2%)
2-tridecanone† (0.1%)

† tentative identification

Lemongrass oil was produced from *C. citratus* in Zimbabwe for four consecutive years; Changonda et al. (2000) subjected the oils to analysis. The oil compositions were found to range as follows:

α-pinene (t-0.4%)
linalool (0.2-1.5%)
camphene (0-0.1%)
trans-verbenol (0.5-1.2%)
β-pinene (t-0.2%)

Table XI. Comparative percentage composition of fresh and aged Indian lemongrass oil

Compound	Fresh Oil	Aged Oil
α-pinene	0.10	0.18
6-methyl-5-hepten-2-one	0.50	3.79
myrcene	0.15	0.86
α-phellandrene	0.06	0.49
p-cymene	0.18	0.57
limonene	0.29	1.26
(Z)-β-ocimene	0.03	0.31
(E)-β-ocimene	0.07	0.32
γ-terpinene	0.10	1.19
terpinolene	0.05	0.16
linalool	0.45	1.47
camphor	0.15	0.71
citronellal	0.11	4.71
isopulegol	0.25	1.43
borneol	0.12	1.15
terpinen-4-ol	0.51	0.48
α-terpineol	0.10	0.31
citronellol	0.06	0.52
neral	33.21	25.63
geraniol	0.51	3.49
geranial	53.11	38.42
geranyl acetate	0.22	1.68
β-caryophyllene	0.68	1.51
γ-cadinene	0.30	0.96
caryophyllene oxide	1.30	0.11

Table XII. Comparative percentage composition of the oils and headspace of lemongrass

Compound	Commercial Oil	Headspace Neat Oil	Headspace Heated Oil
α-pinene	t	1.33	t
camphene	t	t	t
sabinene	0.27	4.26	t
6-methyl-5-hepten-2-one	2.39	12.52	0.48
myrcene	0.99	6.21	0.32
1,8-cineole	0.44	2.50	0.29
limonene	2.05	24.25	0.48
(Z)-β-ocimene	t	0.74	0.18
(E)-β-ocimene	t	0.51	0.12
linalool	3.47	4.37	0.84
isopulegol	1.68	2.47	0.62
citronellal	2.82	0.68	2.23
δ-terpineol	0.79	0.50	2.02
isopulegone*	1.68	2.47	0.62
terpinen-4-ol	1.44	1.25	0.15
neral	34.78	18.68	30.94
geranial	46.89	16.37	56.78
terpinen-4-yl acetate	0.49	t	0.15
geranyl acetate	0.29	t	0.62
β-cubebene	0.30	t	0.89
β-caryophyllene	0.24	t	0.66

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

neral (29.9-33.4%)
geranial (37.7-41.3%)
geranyl acetate (0.4-1.9%)
citronellol (0.2-0.5%)
nerol (0.2-0.4%)
geraniol (2.5-6.7%)
isocaryophyllene oxide (t-0.1%)
α-muurolol (t-1.1%)
sabinene (t)
myrcene (5.6-18.6%)
limonene (0.1-0.8%)
1,8-cineole (0.1-2.9%)
(Z)-β-ocimene (t-0.5%)
(E)-β-ocimene (t-0.6%)
6-methyl-5-hepten-2-one (0.8-2.6%)

t = trace (<0.1%)

Pino and Rosado (2000) analyzed, by GC/MS, an oil of *C. citratus* produced from plants grown in Cuba. It was found to possess the following composition:

α-pinene (0.1%)
6-methyl-5-hepten-2-one (1.8%)
myrcene (1.7%)
isophorone (t)
cis-linalool oxide† (t)
trans-linalool oxide† (t)
linalool (1.5%)
2,2-dimethyl-3,4-octadienal‡ (0.3%)
geraniol (0.1%)
piperitone (t)
geranial (49.5%)
2-undecanone (0.5%)
geranyl formate (0.4%)
α-cyclogeraniol‡ (0.3%)
neryl acetate (0.7%)
β-caryophyllene (0.1%)
trans-α-bergamotene (0.1%)
2-tridecanone (0.4%)
α-thujone (0.2%)
β-thujone (0.2%)
citronellol (t)
neral (38.2%)
caryophyllene oxide (0.1%)
β-bisabolenol‡ (0.5%)
(Z,E)-farnesyl acetate (0.2%)
(E,E)-farnesyl acetate (0.3%)

† furanoid form; ‡ tentative identification; t = trace (<0.1%)

An oil of *C. citratus* produced from plants grown in Nigeria was found by Kasali et al. (2001) to contain the following components:

2,6-dimethyloctane‡ (0.1%)
myrcene (25.3%)
(Z)-β-ocimene (1.0%)
(E)-β-ocimene (0.7%)
p-cymene (0.5%)
(E,E)-allo-ocimene (0.1%)
tetrahydrolinalol† (0.3%)
fenchone (0.2%)
citronellal (0.3%)
β-patchoulene‡ (0.2%)
linalool (0.6%)
camphor (0.1%)
neomenthol‡ (3.3%)
terpinen-1-ol (0.4%)
linalyl acetate (2.3%)
myrcen-8-ol (0.2%)
β-elemene (0.1%)
β-caryophyllene (0.3%)
neral (26.5%)
sabinol‡ (0.1%)
geranial (33.7%)
nerol (0.8%)
geraniol (0.2%)

† does not occur naturally; ‡ probably misidentification

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Cassie Extract

In 1989 Saleem and Ahmad examined the range of *Acacia farnesiana* growth in Pakistan. They determined that *A. farnesiana* could be found growing in Multan in Lahore district, in the Changa Manga range in Kohistan district and in Azak Kashmir. These authors reported that a Pakistani extract of *A. farnesiana* (cassie) contained:

anisaldehyde
cresol
benzaldehyde
methyl salicylate
benzyl alcohol
geranyl acetate
geraniol
β-ionone
linalool

An absolute of cassie produced from *A. farnesiana* in Egypt was analyzed by Ehret et al. (1991). The strong, herbaceous, floral odored portion of the absolute with balsamic undertones was fractionated into neutral (11.5%), carboxylic acid (32.8%), phenolic (3.0%) and nitrogen heterocyclic (0.16%) fractions. They found that the major contributors to the odor of cassie were as follows:

methyl salicylate (1.1%)
benzyl alcohol (1.0%)
hexahydrofarnesylacetone (0.6%)
methyl o-anisate (0.6%)
ethyl linolenate (0.5%)
ethyl palmitate (0.5%)
p-anisaldehyde (0.4%)
geraniol (0.3%)
anisyl alcohol (0.2%)
nonadecane (0.2%)
palmitic acid (9.0%)
linolenic acid (8.9%)
linoleic acid (5.2%)
oleic acid (1.9%)
(Z)-3-methyl-3-decenoic acid (0.6%)

In addition, Ehret et al. also identified a number of new constituents in the cassie absolute, although these were not qualified. The new compounds were as follows:

hexadecane
octadecane
nonadecane
p-menth-1-ene
1-hepten-3-ol
(E,Z)-2,6-nonadienol
lauric aldehyde
undecanol
p-cymene
terpinolene
allo-ocimene*
β-caryophyllene
16-kaurene
1,8-cineole
rose oxide*
cis-linalool oxide (furanoid)
trans-linalool oxide (furanoid)
2-amylfuran
methyl eugenol
methyl chavicol
1,1-diethoxypentane (valeraldehyde diethyl acetal)
1,1-diethoxy-3-methylbutane (isovaleraldehyde diethyl acetal)
1,1-diethoxyhexane (hexanal diethyl acetal)
1,1-diethoxyheptane (heptanal diethyl acetal)
1,1-diethoxynonane (nonanal diethyl acetal)
1,1-diethoxydecane (decanal diethyl acetal)
valeraldehyde
(E)-2-hexenal
(E)-2-heptenal
(E,Z)-2,4-heptadienal
decanal
(E)-2-dodecenal
myristic aldehyde
neral
geranial
cinnamaldehyde
3-phenylpropanal
2-octanone
2-nonanone
4-nonanone
2-nonen-3-one
1,2-epoxy-β-ionone
geranylacetone
acetophenone
vanillylacetone
amyl alcohol
isoamyl alcohol
hexanol
3-hexanol
(Z)-3-hexenol
(E)-2-hexenol
heptanol
2-nonanol
1-undecen-10-ol
citronellol
nerol
phytol
isophytol
phenol
guaiacol
chavicol
isoeugenol*
carvacrol
thymol
methyl 4-hydroxy-3-methoxyphenyl acetate
methyl β-orcinecarboxylate
hexanoic acid
heptanoic acid
octanoic acid
nonanoic acid
decanoic acid
dodecanoic acid
pentadecanoic acid
heptadecanoic acid
octadecanoic acid
nonadecanoic acid
eicosanoic acid
heneicosanoic acid
dieicosanoic acid
2-phenethyl alcohol
ethyl hexanoate
ethyl benzoate
hexyl acetate
octyl acetate
(Z)-3-hexenyl acetate
(Z)-3-hexenyl valerate
(Z)-3-hexenyl benzoate
benzyl formate
benzyl tiglate
benzyl palmitate
2-phenethyl acetate
2-phenethyl tiglate
geranyl formate
geranyl benzoate
methyl palmitate
methyl linolenate
3-methoxypyridine
2-phenylpyridine
isoquinoline
2,6-dimethylquinoline
2-aminobenzaldehyde

*correct isomer not identified

The authors further reported that the major components in the headspace of cassie absolute were:

benzyl alcohol (22.0%)
methyl salicylate (21.0%)
benzaldehyde (14.0%)
hexanal (2.5%)
benzyl acetate (2.1%)

Compounds present in 1-2% amounts:

(E)-2-hexenal
tiglate aldehyde
1,1-diethoxyethane (acetaldehyde diethyl acetal)
hexanol
(Z)-3-hexenol
(E)-2-hexenol
2-phenethyl alcohol
ethyl hexanoate
butyl acetate

Compounds present in 0.1-1% amounts:

nonane
isobutyl (E)-3-hexenoate
sabinene
1,1-diethoxypentane
α-pinene
1,1-diethoxyheptane
δ-3-carene
1,1-diethoxynonane
α-p-dimethylstyrene
butanal
2-methyl-6-heptanone
(E)-2-heptenal
amyl acetate
(E,Z)-2,4-heptadienal
(Z)-3-hexenyl acetate
1-octen-3-ol
ethyl butyrate
guaiacol
ethyl (E)-3-hexenoate

Trace compounds present in less than 0.1%:

(E)-2-octenal
6-methyl-3,5-heptadien-2-one
salicylaldehyde
2-heptanone
2-amylfuran
2-octanone
cis-linalool oxide (pyranoid)
1-penten-3-ol
trans-linalool oxide (pyranoid)
6-methyl-5-hepten-2-ol
methyl chavicol

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Port Orford Cedarwood Oil

Port-Orford-Cedar (*Chamaecyparis lawsoniana* (A. Murray) Parl.) is a native North American tree that can be found in a narrow zone of the Pacific Northwest from southwest Oregon south to northwest California. A limited quantity of oil is produced from cedarwood shavings.

In 1986, Karawya et al. analyzed the leaf oil of *C. lawsoniana* by GC/MS and found that it contained the following constituents:

(Z)-3-hexenol (1.42%)
terpinen-4-ol (0.84%)

δ-3-carene (14.09%)
sabinene (10.13%)
limonene (15.84%)
γ-terpinene (3.31%)
isopulegol (5.23%)
bornyl acetate (4.10%)
α-terpinene† (11.16%)
p-menth-1-ene† (t)
(E)-β-farnesene (0.48%)

† incorrect identification based on elution order; t = trace (<0.1%)

In 1989, McDaniel examined the composition of a hexane extract of the heart wood shavings of Port-Orford-Cedar and found that it contained:

γ-muurolene (3.28%)
α-terpineol (1.38%)
β-selinene (1.59%)
α-muurolene (4.48%)
γ-cadinene (14.49%)
T-cadinol (11.01%)
T-muurolol (16.33%)
α-muurolol (5.91%)
α-cadinol (30.05%)

In addition, the authors also identified borneol and bornyl acetate in an acetone extract of the same heartwood shavings.

Two years later, De Pooter et al. (1991) examined a concrete of the leaves of *C. lawsoniana* using GC and GC/MS and found that it contained the following components:

tricyclene (t)
α-thujene (t)
carvone (0.6%)
α-terpinyl acetate (1.3%)
α-pinene (0.3%)
sabinene (0.3%)
myrcene (0.8%)
p-cymene (0.3%)
limonene (58.6%)
terpinen-4-ol (0.5%)
α-terpineol (0.1%)
carveol* (0.1%)
β-caryophyllene (0.4%)
α-humulene (0.1%)
calamenene* (t)
δ-cadinene (2.4%)
oplopanone (0.7%)
oplopanyl acetate (8.7%)
hybaene (3.1%)

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

More recently, Tucker et al. (2000) used GC/MS to examine the composition of three commercial samples of Port Orford cedarwood oil. A summary of their results can be seen as follows:

tricyclene (0.02%)
α-pinene (6.53%)
α-fenchene (0.42%)
camphene (1.29%)
myrcene (0.25%)
1,4-cineole (1.30%)
limonene (2.69%)
1,8-cineole (0.69%)
γ-terpinene (0.24%)
p-mentha-3,8-diene (0.02%)
p-cymene (1.27%)
terpinolene (1.55%)
6-methyl-5-hepten-2-one (0.09%)
cis-rose oxide (0.09%)
fenchone (4.67%)
α-p-dimethylstyrene (0.96%)
α-fenchyl acetate (0.13%)
citronellal (0.04%)
camphor (5.94%)
isopulegol (1.41%)
α-fenchol (5.51%)
bornyl acetate (0.54%)
β-elemene (1.01%)
terpinen-4-ol (0.73%)
β-terpineol* (3.31%)
myrtenal (0.24%)
citronellyl acetate (0.63%)
isoborneol (0.15%)
α-humulene (0.02%)
α-terpineol (14.33%)
α-amorphene (1.87%)
borneol (1.31%)
zonarene (0.08%)
α-muurolene (4.23%)
citronellol (2.28%)
δ-cadinene (8.17%)
myrtenol (1.04%)
cis-calamenene (0.38%)
p-cymen-8-ol (0.25%)
α-calacorene (0.35%)
T-cadinol (3.42%)
T-muurolol (2.71%)
α-cadinol (5.30%)
hexadecanoic acid (0.05%)

*correct isomer not identified

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