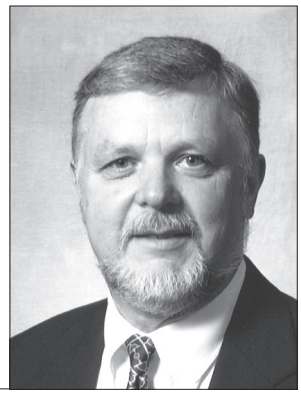
Progress in Essential Oils



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Lemon Oil

In 1982, Kekelidze et al. analyzed the peel oil of the Novogruzinski cultivar of lemon produced in Georgia (at that time part of USSR). They found that this local oil contained the following constituents:

α-pinene (1.47%)
β-pinene (13.75%)
sabinene (2.32%)
myrcene (1.24%)
limonene (61.89%)
ocimene* (1.48%)
γ-terpinene (5.39%)
p-cymene (3.20%)
terpinolene (0.50%)
hexanal (0.22%)
citronellal (0.18%)
linalool (0.67%)
terpinen-4-ol (1.34%)
α-terpineol (2.00%)
β-caryophyllene (0.10%)
bergamotene* (0.20%)
β-bisabolene (0.60%)
citronellol (0.15%)
neryl acetate (0.20%)
neral (0.64%)
geranial (1.21%)
geranyl acetate (0.18%)
nerol (0.14%)
geraniol (0.10%)

*correct isomer not identified

Koepsel and Surburg (1988) characterized two new naturally occurring ketones in orange oil. They were identified as (exo)-*cis*-4,7-dimethylbicyclo[3.2.1]oct-3-en-6-one. The authors noted in this report that both ketones were also found in lemon oil.

Dellacassa et al. (1991) compared the composition of lemon oil produced by the FMC process throughout the season in Uruguay. The range of composition of the oils is shown in Table I. While examining the less volatile portion of lemon oil, Ziegler and Spiteller (1992) identified the following unusual compounds:

3-(3',4',5'-trimethoxyphenyl)-propyl acetate
3-(3',4',5'-trimethoxyphenyl)-propyl propionate
3-(3',4',5'-trimethoxyphenyl)-2-propenyl acetate
3-(3',4',5'-trimethoxyphenyl)-2-propenyl propionate

Also in 1992, Dugo et al. used component ratios of some oxygenated constituents of lemon oil to determine whether an oil had been adulterated with distilled lemon oil. The component ratios found to be discriminatory were:

terpinen-4-ol/*cis*-sabinene hydrate (0.21-1.18)[a]
terpinen-4-ol/*trans*-sabinene hydrate (0.22-1.69)
terpinen-4-ol/citronellal (0.02-0.53)
terpinen-4-ol/decanal (0.17-1.28)

[a] component ratio range at 95% confidence limits

The authors found that oils that had been adulterated with 8% distilled lemon oil would be differentiated from authentic lemon oil by the above component ratios.

Table I. Percentage composition of lemon oil produced in Uruguay by the FMC process

Compound	Percentage Range
α-thujene	0.41-0.66
α-pinene	1.65-1.98
camphene	0.05-0.07
sabinene + β-pinene	11.13-12.24
myrcene	1.51-1.78
octanal + α-phellandrene	0.16-0.21
δ-3-carene	0-t
α-terpinene	0.19-0.28
p-cymene + limonene	69.05-70.17
(Z)-β-ocimene	t-0.10
(E)-β-ocimene	0.09-0.17
γ-terpinene	8.36-9.18
cis-sabinene hydrate	0.04-0.12
terpinolene	0.25-0.41
trans-sabinene hydrate	t
linalool	0.10-0.13
nonanal	0.08-0.12
citronellal	0.08-0.12
terpinen-4-ol	0.02-0.05
α-terpineol	0.11-0.14
decanal	0.04-0.06
nerol + citronellol	0.03-0.04
neral	0.67-0.73
geraniol	0.03-0.05
geranial	1.09-1.27
undecanal	0.01-0.03
citronellyl acetate	0.02-0.04
neryl acetate	0.32-0.37
geranyl acetate	0.30-0.32
β-caryophyllene	0.24-0.31
bergamotene*	0.36-0.40
α-humulene	0.02-0.08
valencene	t-0.02
β-bisabolene	0.53-0.62

*correct isomer not identified
t = trace (<0.01%)

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Table II. Average percentage composition of lemon oils from the north and south of Uruguay (1992/1993) compared with Italian oils (1991)

Compound	North Uruguay Oils	South Uruguay Oils	Italian Oils
α-thujene	0.410	0.418	0.436
α-pinene	1.875	1.878	1.947
camphene	0.057	0.058	0.059
sabinene + β-pinene	13.509	14.354	14.313
myrcene	1.532	1.480	1.478
α-phellandrene + octanal	0.117	0.111	0.118
δ-3-carene	0.005	0.005	0.005
α-terpinene	0.198	0.204	0.181
limonene	67.747	66.046	65.866
(Z)-β-ocimene	0.058	0.068	0.066
(E)-β-ocimene	0.106	0.129	0.120
γ-terpinene	8.857	9.334	9.393
trans-sabinene hydrate	0.031	0.043	0.041
octanol	t	t	0.003
terpinolene	0.380	0.392	0.375
cis-sabinene hydrate	0.028	0.038	0.034
linalool	0.102	0.121	0.109
nonanal	0.108	0.121	0.107
citronellal	0.071	0.083	0.099
terpinen-4-ol	0.039	0.038	0.037
α-terpineol	0.157	0.196	0.166
decanal	0.040	0.038	0.041
citronellol + nerol	0.031	0.029	0.035
neral	0.867	0.935	0.866
geraniol	0.022	0.061	0.021
geranial	1.455	1.554	1.496
undecanal	0.023	0.024	0.022
citronellyl acetate	0.020	0.023	0.029
neryl acetate	0.353	0.346	0.431
geranyl acetate	0.274	0.350	0.399
β-caryophyllene	0.251	0.252	0.233
trans-α-bergamotene	0.337	0.326	0.336
α-humulene	0.016	0.016	0.016
β-santalene + (Z)-farnesene	0.048	0.047	0.044
valencene	0.021	0.028	0.028
β-bisabolene	0.520	0.509	0.495
2,3-dimethyl-3(4-methyl-3-pentenyl) -2-norbornanol	0.017	0.017	0.018
campherenol	0.020	0.020	0.018
α-bisabolol	0.027	0.024	0.022

t = trace (<0.01%)

In 1993, Braunsdorf et al. demonstrated that the use of gas chromatography-isotope mass spectrometry using a suitable internal isotopic standard such as neryl acetate or limonene not only eliminated the influence of CO_2 fixation during photosynthesis, but allowed the analyst to determine whether a sample of lemon oil had been adulterated or not.

Two years later, Dellacassa et al. (1995) reported that lemon oils produced in North (Salto, Paysandu, Rio Negro and Rivera) and South Uruguay (Canelones, Maldonado, Montevideo, San José and Colonia) had a very similar average composition. The compositions of oils from each area were compared also with Italian lemon oils, and the results of these comparisons can be seen in Table II.

In 1996, Usai et al. used a combination of GC and GC/MS to analyze the peel oils produced from 12 cultivars of lemon grown in Sardinia. They found that the oil compositions ranged as follows:

α-thujene (0.07-0.28%)
α-pinene (0.44-1.26%)
camphene (0-0.07%)
sabinene (0.85-1.51%)
β-pinene (4.93-8.64%)
myrcene (1.14-1.58%)
octanal (0.02-0.11%)
α-phellandrene (0-0.01%)
α-terpinene (0.06-0.13%)
p-cymene (0.06-0.08%)
limonene (70.08-76.50%)
(E)-β-ocimene (0.11-0.19%)
γ-terpinene (6.00-9.51%)
octanol (0.06-0.15%)
terpinolene (0.38-0.59%)
linalool (0.21-0.63%)
nonanal (0.02-0.32%)
p-menth-4(8)-en-9-ol (0-0.05%)
pulegone (0-0.24%)
citronellal (0.06-0.16%)
borneol (0.01%)
terpinen-4-ol (0.04-0.06%)
α-terpineol (0.21-0.63%)
decanal (0.01-0.10%)
nerol (0.04-0.07%)
neral (0.57-1.98%)
geraniol (0.02-0.10%)
geranial (0.81-3.56%)
perillaldehyde (0.02-0.03%)
undecanal (0.02-0.04%)
citronellyl acetate (0-0.40%)
neryl acetate (0.57-2.54%)
geranyl acetate (0.31-3.18%)
α-copaene (0-0.08%)
β-caryophyllene (0.12-0.93%)

trans-α-bergamotene (0.11-1.76%)
α-humulene (0-0.34%)
β-bisabolene (0.20-2.50%)
α-bisabolol (0.04-0.05%)

The cultivar that was richest in carbonyl compounds was Santa Teresa 146.

Also in 1996, Ayeddoun et al. examined the peel oil composition of lemons grown in Benin using GC and GC/MS. The composition of this oil was found to be:

α-thujene (0.3%)
α-pinene (1.8%)
camphene (0.1%)
sabinene (t)
β-pinene (4.2%)
octanal (0.1%)
myrcene (1.8%)
α-phellandrene (0.1%)
α-terpinene (0.3%)
p-cymene (0.4%)
1,8-cineole (3.3%)
limonene (70.4%)
(Z)-β-ocimene (0.2%)
(E)-β-ocimene (0.3%)
γ-terpinene (11.8%)
terpinolene (0.8%)
linalool (0.1%)
α-fenchyl alcohol (t)
cis-β-terpineol (0.1%)
borneol (t)
terpinen-4-ol (0.7%)
α-terpineol (1.4%)
decanal (t)
citronellol (t)
neral (t)
β-caryophyllene (0.3%)
trans-α-bergamotene (0.3%)
α-humulene (t)
germacrene D (0.2%)
(E,E)-α-farnesene (0.2%)
β-bisabolene (0.5%)
δ-cadinene (0.1%)

t = trace (<0.1%)

The sesquiterpene hydrocarbon fraction of Sicilian lemon oils was found by Mondello et al. (1996) to comprise the following components:

cis-α-bergamotene (2.7%)
β-caryophyllene (13.8%)
trans-α-bergamotene (24.5%)
α-humulene (1.2%)
(Z)-β-farnesene (1.6%)
(Z)-β-santalene (4.1%)
γ-curcumene (0.7%)
valencene (1.7%)
β-bisabolene (37.1%)
(Z)-γ-bisabolene (0.5%)
(E)-β-farnesene (0.4%)
(E)-γ-bisabolene (0.4%)

A year later, Verzera et al. (1997) analyzed Italian lemon peel oil wax, a mixture, which was ca. 2.0% of lemon oil. Initially, the wax was separated from the oil and freeze dried. It was then examined using TLC, GC and GC/MS and the components characterized were:

1. fatty acids from C_{12}-C_{25} with palmitic (28.1%) and linoleic acids (12.7%) being major
2. linear alcohols from C_{21}-C_{28} with C_{24} (34%) being major
3. linear, iso and anteiso hydrocarbons from C_{20}-C_{33} with C_{29} and C_{31} (27.9% and 19.0%, respectively) being major
4. sterols and triterpene alcohols with C_{24} (33.7%) being major

The authors determined that the essential oil contained the following components:

tricyclene (0.01%)
α-thujene (0.37%)
α-pinene (1.70%)
camphene (0.05%)
sabinene + β-pinene (13.67%)
6-methyl-5-hepten-2-one (t)
myrcene (1.47%)
octanal (0.05%)
α-phellandrene (0.07%)
δ-3-carene (t)
α-terpinene (0.18%)
p-cymene + limonene (66.30%)
(Z)-β-ocimene (0.08%)
(E)-β-ocimene (0.13%)

Table III. Average percentage composition of lemon oils from the north and south of Uruguay (1995) compared with Italian oils

Compound	North Uruguay Oils	South Uruguay Oils	Italian Oils
tricyclene	0.01	0.01	0.01
α-thujene	0.40	0.04	0.43
α-pinene	1.92	1.89	1.95
camphene	0.06	0.06	0.06
sabinene + β-pinene	13.88	14.31	15.03
6-methyl-5-hepten-2-one	t	t	t
myrcene	1.58	1.49	1.44
α-phellandrene	0.07	0.07	0.05
octanol	0.04	0.04	0.06
δ-3-carene	0.01	0.01	0.01
α-terpinene	0.18	0.19	0.19
p-cymene	0.15	0.15	0.13
limonene	67.00	67.02	65.23
(Z)-β-ocimene	0.05	0.07	0.07
(E)-β-ocimene	0.10	0.12	0.13
γ-terpinene	8.88	8.81	9.54
cis-sabinene hydrate	0.04	0.05	0.04
octanol	0.01	0.01	t
terpinolene	0.36	0.35	0.38
trans-sabinene hydrate	0.03	0.03	0.03
linalool	0.12	0.12	0.11
nonanal	0.10	0.12	0.11
cis-limonene oxide	0.01	0.01	0.01
trans-limonene oxide	0.01	0.01	0.01
camphor	0.01	0.01	0.01
citronellal	0.09	0.08	0.09
borneol	0.02	0.02	0.01
terpinen-4-ol	0.04	0.06	0.04
α-terpineol	0.16	0.19	0.17
decanal	0.05	0.04	0.04
octyl acetate	t	0.01	t
citronellol + nerol	0.03	0.02	0.04
neral	0.76	0.81	0.83
piperitone	t	t	t
geraniol	0.03	0.04	0.02
geranial + perillaldehyde	1.32	1.38	1.39
bornyl acetate	0.01	0.01	t
undecanal	0.03	0.02	0.02
nonyl acetate	0.01	0.01	0.01
methyl geranate	0.01	t	t
citronellyl acetate	0.03	0.02	0.03

γ-terpinene (9.47%)
cis-sabinene hydrate (0.04%)
octanol (0.01%)
terpinolene (0.39%)
trans-sabinene hydrate (0.05%)
linalool (0.14%)
nonanal (0.13%)
cis-limonene oxide (0.01%)
trans-limonene oxide (0.01%)
camphor (t)
citronellal (0.10%)
borneol (0.02%)
terpinen-4-ol (0.07%)
α-terpineol (0.19%)
decanal (0.06%)
octyl acetate (t)
nerol + citronellol (0.10%)
neral (0.79%)
piperitone (t)
geraniol (0.04%)
geranial + perillaldehyde (1.34%)
bornyl acetate (0.02%)
undecanal (0.03%)
nonyl acetate (t)
methyl geranate (t)
citronellyl acetate (0.04%)
neryl acetate (0.47%)
geranyl acetate (0.47%)
dodecanal (0.01%)
decyl acetate (0.04%)
β-caryophyllene (0.22%)
trans-α-bergamotene (0.39%)
α-humulene (0.02%)
(Z)-β-santalene + (E)-β-farnesene (0.05%)
γ-muurolene (0.02%)
germacrene D (0.01%)
valencene (0.04%)
bicyclogermacrene (0.05%)
(Z)-α-bisabolene (0.04%)
β-bisabolene (0.57%)
γ-elemene† (0.02%)
tetradecanal (t)
2,3-dimethyl-3(4-methy-3-pentenyl)-2-norbornanol (0.03%)
campherenol (0.03%)
α-bisabolol (0.03%)
nootkatone (0.01%)

†incorrect identification based on elution order
t = trace (<0.01%)

A sample of Sicilian lemon oil was found by Chamblee et al. (1997) to contain bicyclogermacrene and (Z)-α-bisabolene as minor constituents.

Dellacassa et al. (1997) analyzed the 1995 season North Uruguay and South Uruguay lemon oils and compared them with Italian lemon oils produced by the FMC process. They found that the 1995 season oils were similar to the 1992/1993

season oils, although this time they identified more components as can be seen in Table III.

Also in 1997, Sawada and Yamada compared the compositions of Californian and Sicilian lemon oils using GC and GC/MS. From their results, which are presented in Table IV, one could conclude from this study that Sicilian oils were slightly richer in nonanal, citronellal, α-terpineol, neryl acetate and geranyl acetate. Also, the differences in the Sicilian oils produced by different processes were quite small.

Asano (1997) analyzed two oils of lemon, one from the United States and the other from Spain and found them as expected to be very similar. The components identified in these two oils were as follows:

α-pinene (1.73-2.11%)
camphene (0.06%)
β-pinene (10.15-11.24%)
sabinene (1.86-1.91%)
myrcene (1.23-1.65%)
α-terpinene (0.16-0.20%)
limonene (66.64-67.11%)
1,8-cineole (0.26-0.67%)
γ-terpinene (7.84-8.39%)
p-cymene (0.09-1.01%)
decanal (0-0.07%)
trans-sabinene hydrate (0-0.09%)
linalool (0.12-0.13%)
terpinen-4-ol (0.43-0.49%)
undecanal (0.26-0.29%)
neral (1.25-1.31%)
α-terpineol (0.04-0.29%)
geranial (1.88-1.93%)
neryl acetate (0.71-1.50%)
β-bisabolene (0.42-0.74%)
geranyl acetate + α-farnesene* (0.05-0.48%)
nerol (0.07-0.10%)
geraniol (0.05-0.07%)

*correct isomer not identified

The author pointed out that the US oil was richer in the important carbonyl compounds than the Spanish oil.

In 1998, Caccioni et al. analyzed three lemon oils of Italian origin using both GC and GC/MS. The components identified in these oils were as follows:

α-thujene (0.27-0.43%)
α-pinene (1.27-2.27%)
camphene (0.05-0.10%)
sabinene (0.93-1.52%)
β-pinene (8.34-9.42%)
octanal (0.06-0.07%)
myrcene (1.39-1.52%)
α-phellandrene (0.04-0.06%)
δ-3-carene (t)
α-terpinene (0.12-0.30%)
β-phellandrene + p-cymene (0.17-0.42%)
limonene (60.20-71.06%)
(Z)-β-ocimene (0-0.04%)
(E)-β-ocimene (0.07-0.15%)
γ-terpinene (8.06-9.45%)
octanol (0.01-0.06%)
terpinolene (0.39-0.52%)
nonanal (0.10-0.13%)
linalool (0.18-0.46%)
citronellal (0.04-0.08%)
terpinen-4-ol (0.34-0.67%)
α-terpineol (0.41-0.86%)
decanal (0.03-0.05%)
nerol (0.17-0.86%)
neral (0.38-0.90%)
geraniol (0.18-1.05%)
geranial (0.56-1.23%)
citronellyl acetate (0.02-0.05%)
neryl acetate (0.21-0.44%)
geranyl acetate (0.23-0.45%)
β-caryophyllene (0.13-0.19%)
trans-α-bergamotene (0.17-0.28%)
α-humulene (0.01-0.03%)
β-bisabolene (0.23-0.47%)

An oil of lemon of commercial origin was analyzed by Baratta et al. (1998) using both GC (for quantitative data and retention indices) and GC/MS (for component identity confirmation). The components identified in the oil were:

α-thujene (0.3%)
α-pinene (1.8%)
sabinene (1.2%)
β-pinene (12.8%)
myrcene (1.3%)
α-terpinene (0.1%)
p-cymene (0.7%)
limonene (68.8%)
(Z)-β-ocimene (t)
(E)-β-ocimene (0.1%)
γ-terpinene (8.7%)
terpinolene (0.2%)
linalool (0.1%)
α-terpineol (0.1%)
geranial (1.3%)
geranyl acetate (0.2%)
β-caryophyllene (1.2%)
trans-α-bergamotene (0.3%)
β-bisabolene (0.5%)

Table III (cont.). Average percentage composition of lemon oils from the north and south of Uruguay (1995) compared with Italian oils

Compound	North Uruguay Oils	South Uruguay Oils	Italian Oils
neryl acetate	0.42	0.33	0.40
geranyl acetate	0.31	0.31	0.42
dodecanal	0.01	0.01	t
decyl acetate	0.01	0.01	t
β-caryophyllene	0.26	0.24	0.23
trans-α-bergamotene	0.34	0.30	0.34
α-humulene	0.02	0.02	0.02
γ-muurolene	t	t	0.01
(E)-β-farnesene + β-santalene	0.05	0.05	0.04
germacrene D	0.02	0.01	0.01
valencene	0.04	0.02	0.03
bicyclogermacrene	0.06	0.07	0.07
(Z)-α-bisabolene	0.04	0.04	0.04
β-bisabolene	0.51	0.44	0.51
germacrene B	0.01	0.01	0.01
tetradecanal	0.01	0.01	0.01
2,3-dimethyl-3-(4-methyl-3-pentyl)-2-nrobornanol	0.02	0.02	0.02
campherenol	0.02	0.02	0.02
β-bisabolol	0.02	0.02	0.02
nootkatone	0.01	t	t

t = trace (<0.01%)

Table IV. Comparative percentage composition of Californian and Sicilian lemon oils produced with different processes

	Californian	Sicilian		
Compound	FMC	FMC	Sfumatrice	Pelatrice
α-pinene	2.22	2.37	2.26	2.32
camphene	0.06	0.06	0.06	0.05-0.06
β-pinene	11.54	12.93	11.54	12.54-12.55
sabinene	1.61	2.08	1.84	1.98-2.00
6-methyl-5-hepten-2-one	t	t	t	t
myrcene	1.47	1.49	1.53	1.47
octanal	t	t	t	t
α-phellandrene	0.03	0.04	0.03	0.03
α-terpineol	0.15	0.18	0.20	0.20-0.21
p-cymene	0.97	0.35	0.26	0.31
limonene	68.80	64.84	67.10	65.21-65.30
β-phellandrene	0.28	0.37	0.32	0.36
β-ocimene*	0.04	0.06	0.08	0.08
γ-terpinene	7.98	9.38	9.14	9.51-9.53
cis-sabinene hydrate	0.05	0.04	0.03	t-0.05
terpinolene	0.36	0.45	0.47	0.47-0.48
trans-sabinene hydrate	0.04	t	t	t
linalool	0.12	0.12	0.10	0.09-0.11
nonanal	0.05	0.10	0.09	0.09-0.10
citronellal	0.06	0.09	0.12	0.08-0.12
α-terpineol	0.09	0.23	0.15	0.19-0.20
decanal	0.04	0.04	0.05	0.05
nerol	t	t	t	0.03
neral	0.73	0.91	0.87	0.86-0.88
geraniol	t	t	t	t
geraniol	1.45	1.49	1.40	1.42-1.43
neryl acetate	0.35	0.48	0.48	0.46
geranyl acetate	0.27	0.44	0.46	0.49
trans-α-bergamotene				
β-caryophyllene	0.22	0.22	0.20	0.18-0.20
(E)-β-farnesene	t	t	t	t
β-santalene	t	t	t	t
β-sesquiphellandrene	t	t	0.04	0.04
β-bisabolene	0.56	0.71	0.76	0.76-0.77

*correct isomer not identified
t = trace (0.01%)

Jazet Dongmo et al. (1998) used GC and GC/MS to analyze the peel oils of two lemon cultivars grown in the Cameroon. The constituents identified in these oils were as follows:

α-thujene (0.20-0.28%)
α-pinene (1.20-1.30%)
camphene (0-0.05%)
6-methyl-5-hepten-2-one (0.07-0.20%)
sabinene (1.23-1.30%)
β-pinene (8.78-9.74%)
myrcene (1.34-1.40%)
α-phellandrene (0-0.03%)
δ-3-carene (0-0.04%)
α-terpinene (0-t)
p-cymene (1.02-2.93%)
limonene (56.99-60.71%)
(E)-β-ocimene (0.11-0.19%)
γ-terpinene (4.79-7.97%)
terpinolene (0.30-0.39%)
linalool (0.57-1.29%)
α-pinene oxide (0-0.33%)
cis-limonene oxide (0-0.11%)
trans-limonene oxide (0.05-0.12%)
terpinen-1-ol (0.05-0.08%)
isopulegol (0.09-0.14%)

Table V. Comparative composition of lemon oil produced from seven cultivars grown in Uruguay

Compound	1	2	3	4	5	6	7
α-thujene	0.494	0.333	0.394	0.394	0.364	0.412	0.382
α-pinene	2.057	1.164	1.763	1.760	1.584	1.814	1.678
camphene	0.094	0.086	0.053	0.054	0.044	0.055	0.049
sabinene + β-pinene	14.400	12.781	12.612	12.813	10.172	13.096	11.715
myrcene	1.509	1.544	1.546	1.516	1.597	1.518	1.540
octanal + α-phellandrene	0.142	0.092	0.119	0.136	0.097	0.117	0.100
α-terpinene	0.247	0.164	0.194	0.196	0.178	0.203	0.183
limonene	64.073	69.248	67.129	66.547	70.774	65.935	69.082
(Z)-β-ocimene	0.039	0.071	0.060	0.060	0.090	0.056	0.087
(E)-β-ocimene	0.065	0.121	0.096	0.091	0.152	0.109	0.133
γ-terpinene	10.904	7.329	8.567	8.775	8.146	9.157	8.682
cis-sabinene hydrate	0.085	0.070	0.073	0.074	0.061	0.073	0.067
terpinolene	0.474	0.321	0.376	0.381	0.352	0.395	0.369
trans-sabinene hydrate	0.101	0.074	0.087	0.089	0.067	0.081	0.074
linalool	0.116	0.133	0.138	0.135	0.137	0.145	0.128
citronellal	0.074	0.066	0.109	0.107	0.060	0.067	0.067
terpinen-4-ol	0.030	0.034	0.023	0.024	0.024	0.025	0.023
α-terpineol	0.301	0.254	0.250	0.274	0.233	0.267	0.251
decanal	0.047	0.026	0.054	0.041	0.035	0.038	0.032
nerol + citronellol	0.027	0.041	0.068	0.058	0.031	0.052	0.028
neral	0.914	1.192	1.519	1.515	1.277	1.492	1.163
geraniol	0.024	0.018	0.032	0.034	0.028	0.032	0.027
geranial	1.489	1.899	2.428	2.427	2.071	2.410	1.896
undecanal	0.017	0.028	0.020	0.020	0.023	0.025	0.022
citronellyl acetate	0.018	0.017	0.019	0.022	0.015	0.019	0.018
neryl acetate	0.261	0.284	0.319	0.358	0.297	0.397	0.244
geranyl acetate	0.348	0.216	0.271	0.402	0.343	0.325	0.376
β-caryophyllene	0.310	0.297	0.227	0.279	0.283	0.273	0.239
trans-α-bergamotene	0.360	0.400	0.377	0.355	0.306	0.296	0.330
α-humulene	0.020	0.019	0.014	0.017	0.020	0.018	0.016
(Z)-β-santalene	0.048	0.057	0.052	0.051	0.046	0.046	0.046
β-bisabolene	0.545	0.603	0.557	0.549	0.464	0.456	0.504
2,3-dimethyl-3-(4-methyl- 3-pentenyl-2-norbornanol	0.017	0.019	0.018	0.017	0.015	0.017	0.016
campherenol	0.022	0.026	0.023	0.022	0.019	0.021	0.020
β-bisabolol	0.026	0.030	0.028	0.025	0.023	0.022	0.024

1 = Verna, 2 = Eureka, 3 = Messero, 4 = Lisbon, 5 = Ferres I, 6 = Ferres II, 7 = Vila

menthone (0-0.03%)
β-terpineol* (0.09-0.21%)
terpinen-4-ol (1.17-1.88%)
myrtenal (0-0.04%)
α-terpineol (2.19-2.92%)
p-cymen-8-ol (0-0.11%)
verbenone (0.04-0.05%)
carveol* (0.03-0.14%)
neral (0.27-1.06%)
nerol (1.82-4.26%)
geraniol (0.26-0.58%)
geranial (2.39-5.43%)
methylnaphthalene* (0.03-0.10%)
methylnaphthalene* (0-0.05%)
thymol (0-0.04%)
bornyl acetate (0-0.07%)
δ-elemene (0-0.06%)
α-terpinyl acetate (0.26-0.56%)
geranyl acetate (0.20-0.26%)
α-copaene (0-0.41%)
β-elemene (0-0.07%)
β-caryophyllene (0.23-0.47%)
trans-α-bergamotene (0.17-0.82%)
α-humulene (0-1.07%)
allo-aromadendrene (0-0.05%)
dodecanol (0-0.17%)
germacrene D (0-0.16%)
valencene (0-0.24%)
bicyclogermacrene (0-0.12%)
β-bisabolene (0-0.29%)
γ-cadinene (0-0.42%)

Table VI. Percentage composition of lemon oil produced from two hybrids at three distinct times of the year in Italy

Compound	Feminello commune			Feminello precorce Brolo		
	Winter	Spring	Summer	Winter	Spring	Summer
α-thujene	0.29	0.25	0.31	0.21	0.20	0.35
α-pinene	1.23	1.16	1.61	0.94	1.04	1.79
sabinene	1.46	1.71	1.76	1.11	2.03	1.30
β-pinene	8.32	11.15	14.25	6.84	13.66	15.41
myrcene	1.53	1.27	1.37	1.46	1.08	1.40
α-phellandrene	-	0.14	0.14	-	0.14	0.12
α-terpinene	0.13	0.19	0.15	0.10	0.20	0.13
p-cymene	2.00	0.43	0.47	2.54	0.69	0.48
limonene	70.05	64.07	63.54	72.23	60.07	61.94
(Z)-β-ocimene	0.12	0.13	0.14	0.10	0.13	0.09
(E)-β-ocimene	0.10	0.16	0.10	0.11	0.21	0.15
γ-terpinene	8.48	10.54	9.09	7.67	10.96	6.74
terpinolene	0.38	0.45	0.35	0.34	0.50	0.40
linalool	0.26	0.25	0.37	0.23	0.18	0.29
nonanal	0.10	0.18	-	0.10	0.13	-
citronellal	0.12	0.22	0.10	0.15	0.19	0.31
terpinen-4-ol	0.32	0.18	0.35	0.36	0.10	0.34
α-terpineol	0.10	0.10	0.10	0.10	0.10	0.13
nerol	0.18	0.14	0.26	0.14	0.10	0.16
neral	0.60	1.49	1.30	0.90	1.09	1.23
geraniol	0.19	0.15	0.17	0.10	0.11	0.14
geranial	1.22	2.45	1.63	1.60	2.82	1.52
citronellyl acetate	0.10	0.12	0.10	0.10	0.10	0.16
neryl acetate	0.38	0.61	0.39	0.49	0.61	0.70
geranyl acetate	0.24	0.86	0.79	0.49	1.51	0.81
β-caryophyllene	0.18	0.25	0.18	0.23	0.36	0.25
trans-α-bergamotene	0.45	0.54	0.37	0.55	0.69	0.38
β-bisabolene	0.63	0.81	0.62	0.81	1.00	0.57

δ-cadinene (0-0.42%)
cadina-1,4-diene (1.34%)
globulol (0-0.03%)
spathulenol (0-0.09%)
caryophyllene oxide (0-0.18%)
β-eudesmol (0.04-0.06%)
(Z,E)-farnesol (0-0.03%)
β-sinensal (0.04-0.09%)
(Z,Z)-farnesol (0-0.08%)
(E,E)-farnesol (0-0.05%)

*correct isomer not identified

Campisi et al. (1998) compared the composition of lemon oil produced from various cultivars in Uruguay between March and October 1993. The results of this study can be found in Table V. Also in 1998, Geraci et al. compared the composition of lemon oil produced from two Italian cultivars. In particular, they compared the composition of oils produced in winter, spring and summer as can be seen in Table VI. From these data, it could be concluded that oils produced in the spring were richest in the odor-characteristic neral and geranial.

In 1999, Sawamura et al. compared the oil compositions of two Italian lemon oils (Feminello and Monachello) and one Japanese lemon oil (Lisbon). The results of this comparative study are shown in Table VII. From these data, it can be concluded that the Lisbon oil from Japan had more intensity of lemon odor based on the fact that its neral/geranial content of 6.29% was far greater than that of either Italian cultivar oil.

The composition of an authentic lemon oil was compared with that of an Austrian commercial lemon oil by Oberhofer et al. (1999). In addition, the author also compared the headspace volatiles of the neat Italian oil with that of an oil that had evaporated to 50% of its original weight as might happen when the oil was heated with an aroma lamp. The results of the oil and headspace analyses can be seen in Table VIII. From an examination of the

Table VII. Comparative percentage composition of two Italian and one Japanese oil of lemon

Compound	Feminello Italy	Monachello Italy	Lisbon Japan
α-pinene	2.29	2.04	2.64
camphene	0.06	0.05	0.06
β-pinene	17.10	14.46	14.23
sabinene	2.62	2.18	2.42
myrcene	1.30	1.38	1.61
α-phellandrene	0.04	0.04	0.04
α-terpinene	0.20	0.21	0.22
limonene	56.60	59.34	59.76
β-phellandrene	0.55	0.39	0.37
(E)-β-ocimene	0.10	0.08	0.04
γ-terpinene	9.08	10.24	9.29
p-cymene	0.20	0.18	0.11
terpinolene	0.33	0.40	0.38
octanal	0.13	0.06	-
tridecane	t	t	-
heptyl acetate	t	-	-
nonanal	0.17	0.18	-
tetradecane	t	t	t
1-tetradecene	t	t	-
cis-limonene oxide	t	t	-
trans-limonene oxide	t	t	-
trans-sabinene hydrate	0.14	0.12	0.13
citronellal	0.07	0.06	0.04
decanal	0.08	0.04	-
camphor	t	t	t
pentadecane	t	t	-
solanone	-	t	-
linalool	0.23	0.27	0.28
p-menth-1-en-9-ol	t	t	-
octanol	t	-	-
cis-sabinene hydrate	0.03	0.04	0.02
α-santalene	0.01	t	-
bornyl acetate	t	t	-
nonyl acetate	-	t	-
trans-α-bergamotene	0.39	0.46	0.27
β-caryophyllene	0.13	0.23	0.16
terpinen-4-ol	0.02	0.02	-
undecanal	t	t	-
γ-elemene	t	t	-
(E)-2-decenal	t	-	-

quantitative data, it is not unexpected that the heated oil was found to have a musty, dirty geraniol-like odor as compared to the characteristic lemon-like odor of the pure authentic Italian oil.

Dugo et al. (1999) reported that the average compo–sition for Sicilian lemon oil was as follows:

tricylene (0.008%)
α-thujene (0.427%)
α-pinene (1.921%)
camphene (0.060%)
sabinene (1.902%)
β-pinene (13.128%)
6-methyl-5-hepten-2-one (t)
myrcene (1.466%)
octanal (0.061%)
α-phellandrene (0.039%)
δ-3-carene (t)
α-terpinene (0.204%)
p-cymene + β-phellandrene + 1,8-cineole + limonene (65.227%)
(Z)-β-ocimene (0.094%)
(E)-β-ocimene (0.149%)
γ-terpinene (9.758%)
cis-sabinene hydrate (0.038%)
octanol (t)
terpinolene (0.392%)
trans-sabinene hydrate (0.024%)
linalool (0.108%)
nonanal (0.129%)
cis-limonene oxide (t)
allo-ocimene* (t)
trans-limonene oxide (t)
camphor (t)
citronellal (0.079%)
borneol (t)
terpinen-4-ol (0.050%)
α-terpineol (0.166%)
decanal (0.054%)
octyl acetate + nerol (t)
citronellol (0.050%)
neral (0.701%)
piperitone (t)
geraniol (0.032%)
perillaldehyde + geranial (1.176%)
bornyl acetate (t)
undecanal (0.039%)
nonyl acetate (t)
methyl geranate (t)
citronellyl acetate (0.023%)
neryl acetate (0.352%)
geranyl acetate (0.412%)
β-caryophyllene (0.241%)
cis-α-bergamotene (t)
decyl acetate (t)
trans-α-bergamotene (0.341%)
α-humulene (0.016%)
(Z)-β-santalene + (Z)-β-farnesene (0.038%)
(E)-β-farnesene (0.013%)
valencene (0.025%)
bicyclogermacrene (0.041%)
(Z)-α-bisabolene (0.041%)
β-bisabolene (0.499%)
(Z)-γ-bisabolene (t)
(E)-α-bisabolene (0.021%)
germacrene D-4-ol (0.020%)
selin-11-en-4α-ol (0.009%)
2,3-dimethyl-3-(4-methyl-3-pentyl)-norbornanol (0.021%)
campherenol (0-0.02%)
β-bisabolol (0.008%)
α-bisabolol (0.031%)
nootkatone (0.021%)

t = trace <0.001%

Verzera et al. (1999) compared the average compo–sition of Sicilian lemon oil produced by different cold pressing methods such as Sfumatrice, Pelatrice, FMC and Torchi. A summary of their results can be seen in Table IX.

In 2000, Corleone et al. reported their results of multiple analyses of lemon oil produced from three cultivars over the months of January-April (winter season) in Italy. The monthly results showing the changes in composition for oils produced each month from the Feminello cultivar are shown in Table X. Although the authors reported similar data for the Lo

Porto and Monachello cultivars, the data presented in Table XI shows a summary of the seasonal range data.

Sawamura (2000) compared the composition of the cold-pressed oils of four lemon cultivars (Bagheria, Eureka, Feminello and Lisbon) grown in Japan. The oils were analyzed using a combination of GC and GC/MS. A summary of the results can be found in Table XII.

More recently, Verzera et al. (2001) compared the composition of the cold-pressed peel oils of nine cultivars of lemon grown in Sicily. The cultivars examined were Feminello siracusano, Feminello continella, Feminello Santa Teresa, Feminello fior d'aracio, Feminello dosaco, Feminello incappuciato, Monachello, Interdonato and fino. The oil compositions of these nine cultivars can be seen summarized in Table XIII. As the carbonyl compounds neral and geranial were highest in the oils of cultivars Feminello incappucciato and Feminello Santa Teresa, these must be considered to be the most desired of the lemon cultivar oils studied.

This same year, Feger et al. (2001) examined the germacrene content of citrus oils. Within this study, the authors found that lemon oil contained only bicyclogermacrene (0.04-0.12%) and a trace of germacrene A.

Table VII (cont.). Comparative percentage composition of two Italian and one Japanese oil of lemon

Compound	Feminello Italy	Monachello Italy	Lisbon Japan
β-santalene	t	t	t
α-humulene	0.02	0.02	-
citronellyl acetate	0.03	0.02	-
(E)-β-farnesene	0.04	0.06	-
neral	0.77	0.59	2.00
δ-muurolene	t	t	t
decyl acetate	t	-	-
α-terpineol	0.31	0.26	0.27
2,7-dimethyl-2,6-octadien-1-ol	-	t	t
β-selinene	t	t	-
bicyclogermacrene	t	t	-
geranial	2.57	2.48	4.29
geranyl acetate	0.68	0.86	0.30
citronellol	t	-	t
perillaldehyde	0.07	0.09	0.09
geranyl propionate	-	t	t
nerol	0.03	0.02	0.02
p-menth-1(7)-en-9-ol	t	-	-
geranyl 2-methylbutyrate	-	-	t
trans-carveol	t	t	-
geraniol	0.01	0.02	0.02
α-sinensal	t	t	-
p-mentha-1,8-dien-9-ol	t	-	t
(Z)-nerolidol	-	t	-
ledol	-	-	t
α-muurolol	t	-	-
verbenol*	t	t	t
hexadecanol	t	-	-
β-bisabolol	t	t	-
neryl acetone	t	t	t
α-bisabolol	0.03	0.03	0.02

*correct isomer not identified
t = trace (<0.01%)

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Table VIII. Percentage composition of two lemon oils and the headspace of one neat oil and one heated oil

Compound	Austrian Oil	Italian Oil	Headspace Neat Oil	Headspace Heated Oil
α-thujene	t	t	0.32	t
α-pinene	0.62	1.11	1.42	0.07
camphene	t	t	t	t
sabinene	0.41	0.27	0.11	t
β-pinene	15.16	12.66	6.86	0.46
myrcene	1.61	2.43	1.84	1.10
δ-3-carene	0.11	t	t	t
α-terpinene	0.24	t	t	t
p-cymene	0.46	0.22	0.99	5.73
limonene	65.95	69.07	73.13	58.24
(Z)-β-ocimene	t	-	-	-
(E)-β-ocimene	t	t	t	t
γ-terpinene	1.51	1.96	9.05	5.03
terpinolene	0.09	0.24	0.58	0.11
linalool	t	0.53	0.66	0.89
citronellal	t	0.15	0.27	0.04
decanal	0.16	0.62	0.57	2.09
neral	0.42	0.57	0.08	0.02
geranial	0.66	1.12	1.00	3.98
geraniol	t	-	-	-
linalyl acetate	1.07	0.65	0.39	3.85
neryl acetate	0.34	1.85	1.23	3.99
geranyl acetate	0.46	t	0.22	1.34
β-caryophyllene	0.23	0.16	t	0.82
trans-β-bergamotene	0.35	1.02	0.77	3.96
β-bisabolene	0.54	0.61	0.08	4.44
α-bisabolol	t	-	-	-

t = trace (<0.01%)

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Table IX. Comparative percentage composition of Sicilian lemon oil produced by four different processes

Compound	Sfumatrice (100)[a]	Pelatrice (59)	FMC (124)	Torchi (81)
tricyclene	0.01	0.01	0.01	0.01
α-thujene	0.48	0.47	0.47	0.46
α-pinene	2.21	2.15	2.23	2.11
camphene	0.07	0.07	0.07	0.06
sabinene + β-pinene	16.56	16.74	18.15	16.04
6-methyl-5-hepten-2-one	0.01	0.01	0.01	0.01
myrcene	1.62	1.57	1.52	1.60
octanal	0.11	0.11	0.08	0.08
α-phellandrene	0.04	0.04	0.08	0.04
δ-3-carene	t	t	t	t
α-terpinene	0.19	0.17	0.16	0.17
p-cymene	0.12	0.16	0.16	0.12
limonene	61.84	61.13	60.18	63.31
(Z)-β-ocimene	0.07	0.08	0.07	0.07
(E)-β-ocimene	0.13	0.15	0.13	0.13
γ-terpinene	10.26	10.42	10.10	10.25
cis-sabinene hydrate	0.03	0.06	0.05	0.01
octanol	t	t	t	t
terpinolene	0.40	0.39	0.36	0.39
trans-sabinene hydrate	0.02	0.04	0.04	0.01
linalool	0.12	0.12	0.13	0.07
nonanal	0.15	0.16	0.16	0.14
cis-limonene oxide	t	t	t	t
trans-limonene oxide	t	t	0.01	t
camphor	t	t	t	t
citronellal	0.09	0.10	0.10	0.08
borneol	0.01	0.01	0.01	t
terpinen-4-ol	0.07	0.04	0.05	0.04
α-terpineol	0.18	0.21	0.23	0.10
decanal	0.05	0.05	0.06	0.05
octyl acetate	t	t	t	t
nerol	0.03	0.04	0.07	0.02
neral	0.95	1.00	0.86	0.68
piperitone	t	t	t	t
geraniol	0.02	0.04	0.02	0.02
geranial + perillaldehyde	1.64	1.74	1.44	1.22
bornyl acetate	t	t	t	t
undecanal	0.03	0.03	0.04	0.03
nonyl acetate	t	t	0.01	t
methyl geranate	t	t	t	t

Table IX (cont.). Comparative percentage composition of Sicilian lemon oil produced by four different processes

Compound	Sfumatrice (100)[a]	Pelatrice (59)	FMC (124)	Torchi (81)
citronellyl acetate	0.03	0.03	0.03	0.03
neryl acetate	0.40	0.42	0.49	0.43
geranyl acetate	0.39	0.45	0.50	0.41
dodecanal	0.01	0.01	0.01	0.01
decyl acetate	t	0.01	0.01	0.01
β-caryophyllene	0.23	0.23	0.24	0.24
trans-α-bergamotene	0.36	0.38	0.39	0.38
α-humulene	0.02	0.02	0.02	0.02
(Z)-β-farnesene	0.03	0.03	0.03	0.03
(Z)-β-santalene	0.01	0.01	0.01	0.01
γ-muurolene	0.01	0.01	0.01	0.04
germacrene D	t	0.01	0.01	0.01
valencene	0.02	0.02	0.02	0.03
bicyclogermacrene	0.07	0.06	0.06	0.07
(Z)-α-bisabolene*	0.04	0.04	0.04	0.04
β-bisabolene	0.54	0.56	0.58	0.56
tetradecanal	t	0.01	t	0.01
2,3-dimethyl-3-(4-methyl-3-pentyl) -2-norbornanol	0.02	0.02	0.02	0.02
campherenol	0.02	0.02	0.02	0.02
α-bisabolol	0.02	0.02	0.02	0.02
nootkatone	t	t	t	t

[a] number of samples analyzed
t = trace (<0.01%)

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Enantiomeric Distribution

The enantiomeric distribution of some of the chiral constituents of lemon oil has been a more important subject of study for the past few years. For example, although α-terpineol is only a minor constituent of lemon oil (ca. 0.6%), Ravid et al. (1995) determined that its enantiomeric distribution in Israeli lemon oil was as follows:

(4R)-(+)-α-terpineol (33%) : (4S)-(-)-α-terpineol (67%)

The following year, Casabianca et al. (1996) used chiral GC to determine that the enantiomeric distribution of citronellal and citronellol in lemon oil were as follows:

(3R)-(+)-citronellal (8.5-10.0%) : (3S)-(-)-citronellal (90.0-91.5%)
(3R)-(+)-citronellol (8.5%) : (3S)-(-)-citronellol (91.5%)

In 1998, Mondello et al. determined that the enantiomeric distribution of six chiral components of lemon oils was as follows:

Table X. Comparative percentage composition of lemon oil produced from Feminello cultivar over winter (January-April) harvesting season

Compound	January	February	March	April
heptanal	0.0069	0.0045	0.0032	0.0046
tricyclene	0.0062	0.0062	0.0065	0.0074
α-thujene	0.3789	0.3984	0.4124	0.4478
α-pinene	1.8023	1.8911	1.9152	2.0671
camphene	0.0635	0.0617	0.0637	0.0712
sabinene	2.2011	2.2561	2.2436	2.4157
β-pinene	13.0830	14.3652	14.2940	15.3908
myrcene	1.5353	1.4460	1.4171	1.4319
octanal	0.0868	0.1012	0.0980	0.1203
α-phellandrene	0.0422	0.0411	0.0419	0.0437
δ-3-carene	0.0045	0.0042	0.0044	0.0048
α-terpinene	0.2114	0.2175	0.2219	0.2311
p-cymene	t	t	t	t
limonene	62.9809	61.4988	61.7350	60.0468
(Z)-β-ocimene	0.0709	0.1346	0.1297	0.1476
(E)-β-ocimene	0.1362	0.2457	0.2241	0.2573
γ-terpinene	9.9271	10.6521	10.6038	10.5499
cis-sabinene hydrate	0.0931	0.0850	0.0813	0.0928
octanol	t	0.0023	-	0.0109
terpinolene	0.4358	0.4386	0.4426	0.4487
trans-sabinene hydrate	0.0913	0.0725	0.0511	0.0556
linalool	0.1958	0.1919	0.1801	0.1824
nonanal	0.1806	0.1902	0.1907	0.2003
trans-limonene oxide	0.0066	0.0051	0.0052	0.0063
camphor	0.0195	0.0156	0.0121	0.0116
citronellal	0.1292	0.1149	0.1234	0.1470
borneol	0.0264	0.0183	0.0182	0.0153
terpinen-4-ol	0.0292	0.0269	0.0280	0.0288
α-terpineol	0.3232	0.2910	0.2492	0.2610
decanal	0.0475	0.0560	0.0580	0.0669
octyl acetate	-	0.0030	0.0036	0.0056
nerol + citronellol	0.0453	0.0289	0.0292	0.0273
neral	1.0989	0.8548	0.7467	0.8200
piperitone	0.0050	0.0048	0.0047	0.0052
geraniol	0.0150	0.0133	0.0120	0.0121
carvone	0.0015	0.0016	-	0.0015
geranial + perillaldehyde	1.8238	1.4683	1.2707	1.3708
bornyl acetate	0.0141	0.0157	0.0194	0.0182
undecenal	0.0350	0.0351	0.0364	0.0359
nonyl acetate	0.0042	0.0041	0.0058	0.0052
methyl geranate	0.0156	0.0167	0.0164	0.0167

(IR,5R)-(+)-β-pinene (5.1%) : (IS,5S)-(-)-β-pinene (94.9%)
(IR,5R)-(+)-sabinene (15.1%) : (IS,5S)-(-)-sabinene (84.9%)
(4R)-(+)-limonene (98.1%) : (4S)-(-)-limonene (1.9%)
(3S)-(+)-linalool (28.5%) : (3R)-(-)-linalool (71.5%)
(4S)-(+)-terpinen-4-ol (19.7%) : (4R)-(-)-terpinen-4-ol (80.3%)
(4R)-(+)-α-terpineol (22.6%) : (4S)-(-)-α-terpineol (77.4%)

Also in 1998, Mondello et al. applied chiral GC analysis using a 25 m x 0.25 mm; 0.25 mm film thickness diethyl t-butylsilyl-β-cyclodextrin capillary column to separate six major optically active components of lemon oil. They found that the enantiomeric distribution of these six components was as follows:

(+)-β-pinene (6.3%) : (-)-β-pinene (93.7%)
(+)-sabinene (14.9%) : (-)- sabinene (85.1%)
(+)-limonene (98.4%) : (-)-limonene (1.6%)
(+)-linalool (42.0%) : (-)-linalool (58.0%)
(+)-terpinen-4-ol (24.7%) : (-)-terpinen-4-ol (75.3%)
(+)-α-terpineol (24.8%) : (-)-α-terpineol (75.2%)

The following year, Dugo et al. (1999) reported that the range of enantiomeric distribution of five constituents of lemon oil produced throughout the year in Sicily was as follows:

(IR,5R)-(+)-pinene (4.2-7.0%) : (IS,5S)-(-)-β-pinene (93.0-95.8%)
(IR,5S)-(+)-sabinene (12.5-15.3%) : (IS,5S)-(-)-sabinene (84.7-87.5%)
(4R)-(+)-limonene (98.0-98.5%) : (4S)-(-)-limonene (1.5-2.0%)
(4S)-(+)-terpinen-4-ol (13.7-26.9%) : (4R)-(-)-terpinen-4-ol (73.1-86.3%)
(4R)-(+)-α-terpineol (18.0-35.8%) : (4S)-(-)-α-terpineol (64.2-82.0%)

More recently, Dugo et al. (2001) compared the enan–tiomeric distribution of seven constituents of lemon oil produced by various processes. These results are seen summarized in Table XIV.

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G. Dugo, K. D. Bartle, I. Bonaccorsi, M. Catalfamo, A. Cotroneo, P. Dugo, G. Lamonica, H. McNair, L. Mondello, P. Previti, I. Stagno d'Alcontres, A. Trozzi and A. Verzera, *Advanced analytical techniques for the analysis of citrus essential oils. Part 2. Volatile fraction LC-HR GC and MD GC*. Essenz. Deriv. Agrum., 69, 159-217 (1999).

G. Dugo, L. Mondello, A. Cotroneo, I. Bonaccorsi and G. Lamonica, *Enantiomeric distribution of volatile components of citrus oils by MD GC*. Perfum. Flavor., 26(1), 20-35 (2001).Table XIV. Enantiomeric distribution of selected compounds in lemon oil

Oxygen Heterocyclic Compounds

Because of their method of manufacture, cold-pressed oils contain small amounts of non-volatile oxygen hetero–cyclic compounds. The level of these non-volatile components in cold-pressed lemon oil is ca. 6-10%.

Barrett and Nelson (1998) examined the coumarin and psoralen level of 395 samples of coastal lemon oil and found that the majority had the following levels"

herniarin (0-10 ppm)
bergapten (0-10 ppm)
isopimpinellin (0-5 ppm)
citropten (700-1300 ppm)

In contrast, the coumarin and psoralen levels of desert lemon oil (89 samples) were determined to be:

herniarin (<10 ppm)
bergapten (50-350 ppm)
isopimpinellin (35-110 ppm)
citropten (700-1700 ppm)

The authors further concluded that lemon oils whose herniarin, bergapten and isopimpinellin levels are all in excess of 300 ppm can be considered as being adulterated.

This same year, Dugo et al. (1998) used normal phase HPLC to determine the coumarin and psoralen content of lemon oil. They determined that the following non-volatile constituents were normally found in authentic lemon oil:

Table X (cont.). Comparative percentage composition of lemon oil produced from Feminello cultivar over winter (January-April) harvesting season

Compound	January	February	March	April
citronellyl acetate	0.0282	0.0241	0.0289	0.0293
neryl acetate	0.3580	0.2596	0.2665	0.2989
geranyl acetate	0.1795	0.1248	0.1379	0.1559
dodecanal	0.0130	0.0121	0.0134	0.0131
decyl acetate	0.0244	0.0238	0.0232	0.0238
β-caryophyllene	0.1906	0.1481	0.1537	0.1597
trans-α-bergamotene	0.3627	0.3666	0.3554	0.3601
α-humulene	0.0251	0.0200	0.0202	0.0216
(Z)-β-farnesene	0.0329	0.0321	0.0314	0.0320
(Z)-β-santalene	0.0138	0.0136	0.0131	0.0138
γ-muurolene	0.0086	0.0073	0.0079	0.0083
germacrene D	0.0095	0.0091	0.0095	0.0094
valencene	0.0061	0.0047	0.0144	0.0088
bicyclogermacrene	0.1537	0.1586	0.1592	0.1592
α-bisabolene*	0.0507	0.0504	0.0491	0.0487
β-bisabolene	0.5472	0.5600	0.5404	0.5465
γ-elemene†	0.0160	0.0171	0.0159	0.0162
tetradecanal	0.0338	0.0334	0.0345	0.0345
2,3-dimethyl-3-(4-methyl-3-pentenyl) -2-norbornanol	0.0181	0.0181	0.0177	0.0179
campherenol	0.0257	0.0243	0.0238	0.0247
α-bisabolol	0.0279	0.0266	0.0263	0.0270

*correct isomer not identified
†should be germacrene B

Table XI. Percentage composition (range) for the oils of two lemon cultivars over the winter season (January-April) grown in Italy

Compound	Lo Porto	Monachello
heptanal	0.0027-0.0038	0.0070-0.0104
tricyclene	0.0033-0.0036	0.0048-0.0081
α-thujene	0.2589-0.3198	0.4095-0.5289
α-pinene	1.1299-1.3266	1.7985-2.1680
camphene	0.0423-0.0428	0.0495-0.0692
sabinene	1.5495-1.7133	1.7629-2.2871
β-pinene	9.2740-10.8031	10.1647-15.1531
myrcene	1.3289-1.4788	1.3948-1.6860
octanal	0.1287-0.1456	0.0683-0.0890
α-phellandrene	0.0395-0.0422	0.0419-0.0451
δ-3-carene	0.0045-0.0062	0.0037-0.0053
α-terpinene	0.1424-0.2418	0.1966-0.2724
p-cymene	t	t
limonene	61.5571-63.0510	57.9669-62.8777
(Z)-β-ocimene	0.0455-0.0759	0.0340-0.1179
(E)-β-ocimene	0.1231-0.1611	0.0813-0.2009
γ-terpinene	11.3667-12.3426	9.3787-12.3475
cis-sabinene hydrate	0.0892-0.1067	0.0674-0.0895
octanol	0.0024-0.0039	0-0.0024
terpinolene	0.5264-0.5930	0.3956-0.5037
trans-sabinene hydrate	0.0965-0.1373	0.0641-0.0906
linalool	0.2031-0.2766	0.0138-0.1524
nonanal	0.1593-0.2566	0.1812-0.2398
cis-limonene oxide	-	0-0.0018
trans-limonene oxide	0.0043-0.0065	0.0033-0.0061
camphor	0.0151-0.0188	0.0107-0.0145
citronellal	0.1543-0.1918	0.0614-0.0984
borneol	0.0232-0.0379	0.0150-0.0245
terpinen-4-ol	0.0368-0.0431	0.0277-0.0314
α-terpineol	0.3415-0.4707	0.2426-0.3040
decanal	0.0887-0.0949	0.0319-0.0453
octyl acetate	0.0054-0.0073	0.0014-0.0050
nerol + citronellal	0.0340-0.0686	0.0131-0.0319
neral	1.2025-1.7643	0.7046-0.8315
piperitone	0.0053-0.0070	0.0035-0.0049
geraniol	0.0267-0.0341	0.0146-0.0243
carvone	0.0017-0.0029	0-0.0010
geranial + perillaldehyde	2.0537-3.0019	1.2159-1.3984
bornyl acetate	0.0162-0.0186	0.0015-0.0087
undecenal	0.0432-0.0579	0.0195-0.0395
nonyl acetate	0.0061-0.0079	0.0049-0.0070

Table XI (cont.). Percentage composition (range) for the oils of two lemon cultivars over the winter season (January-April) grown in Italy

Compound	Lo Porto	Monachello
methyl geranate	0.0154-0.0190	0.0040-0.0190
citronellyl acetate	0.0265-0.0324	0.0141-0.0348
neryl acetate	0.5500-0.7523	0.2458-0.4637
geranyl acetate	0.3889-0.4144	0.3114-0.4245
dodecanal	0.0168-0.0202	0.0062-0.0093
decyl acetate	0.0296-0.0351	0.0147-0.0218
β-caryophyllene	0.2874-0.3778	0.2202-0.2641
trans-α-bergamotene	0.4650-0.5230	0.2156-0.3321
α-humulene	0.0296-0.0423	0.0217-0.0282
(Z)-β-farnesene	0.0401-0.0424	0.0198-0.0290
(Z)-β-santalene	0.0180-0.0198	0.0089-0.0117
γ-muurolene	0.0076-0.0124	0.0095-0.0146
germacrene D	0.0104-0.0146	0.0056-0.0074
valencene	0.0000-0.0044	0.0041-0.0091
bicyclogermacrene	0.1369-0.1759	0.0339-0.0843
α-bisabolene*	0.0599-0.0748	0.0369-0.0463
β-bisabolene	0.7232-0.7893	0.3139-0.5060
γ-elemene†	0.0203-0.0234	0.0092-0.0144
tetradecanal	0.0322-0.0394	0.0069-0.0198
2,3-dimethyl-3-(4-methyl-3-pentenyl) -2-norbornanol	0.0072-0.0247	0.0115-0.0167
campherenol	0.0279-0.0320	0.0160-0.0209
α-bisabolol	0.0323-0.0365	0.0164-0.0238

*correct isomer not identified
†should be germacrene B
t = trace (<0.0001%)

bergamottin (160-191*)
isoimperatorin†
5-geranoxy-7-methoxycoumarin (180-250)
5-isopentenyloxy-7-methoxycoumarin
citropten (52-142)
8-geranyloxypsoralen (19-36)
5-isopent-2[l]-enyloxy-8-(2[l],3[l]-epoxyisopentyloxypsoralen) (19-37)
oxypeucedanin (89-157)
byakangelicol (66-123)
oxypeucedanin hydrate†
byakangelicin†

*mg/100g of oil
†present although level too low to quantitate

In1999, Dugo et al. used liquid chromatography coupled with atmospheric pressure chemical ionization mass spectrometry to determine the oxygen heterocyclic compounds found in cold-pressed citrus oils. They determined that the mixture of coumarins and psoralens found in lemon oil were as follows:

herniarin (7-methoxycoumarin)
citropten (5,7-dimethoxycoumarin)
5-isopentenyloxy-7-methoxycoumarin
5-geranyloxy-7-methoxycoumarin
bergamottin (5-geranyloxypsoralen)
oxypeucedanin (5-(2[l],3[l]-epoxyisopentyloxy)psoralen)
oxypeucedanin hydrate (5-(2[l],3[l]-dihydroxyisopentyloxy)psoralen)
isoimperatorin (5-isopentenyloxypsoralen)
8-geranyloxypsoralen
imperatorin (8-isopentenyloxypsoralen)
5-geranyloxy-8-methoxypsoralen
byakangelicin(8-(2[l],3[l]-dihydroxyisopentyloxy)-5-methoxypsoralen)
5-isopent-2[l]-enyloxy-8-(2[1],3[l]-epoxyisopentyloxy)psoralen
phellopterin (5-methoxy-8-isopentenyloxypsoralen)
byakangelicol (5-methoxy-8-(2[l],3[l]-epoxyisopentyloxy)psoralen)

This same year, Dugo et al. (1999) reported the same results as noted above for the oxygen heterocyclic com–pounds in lemon oil; however, they added a few more components to this list such as:

Table XII. Comparative percentage composition of cold-pressed peel oils of four lemon cultivars

	Lemon oil ex. Cultivar			
Compound	Bagheria	Eureka	Feminello	Lisbon
α-pinene	2.11	2.27	2.29	2.64
camphene	0.05	0.05	0.06	0.06
β-pinene	12.64	10.54	17.10	14.23
sabinene	1.08	2.20	2.62	2.42
myrcene	1.10	t	1.30	1.61
α-phellandrene	0.26	1.62	0.04	0.04
α-terpinene	0.01	0.02	0.20	0.22
limonene	64.34	69.65	56.60	59.76
β-phellandrene	t	t	0.55	0.37
(Z)-β-ocimene	0.06	0.04	0.10	0.04
γ-terpinene	9.78	8.22	9.08	9.29
p-cymene	0.19	0.04	0.20	0.11
terpinolene	0.47	0.33	0.33	0.38
octanal	t	0.01	0.13	t
heptyl acetate	t	-	t	-
nonanal	0.10	0.04	0.17	-
trans-sabinene hydrate	0.07	0.06	0.14	0.13
citronellal	0.09	0.05	0.07	0.04
decanal	0.05	t	0.08	-
camphor	t	0.01	t	t
1,8-cineole†	t	t	-	-
linalool	0.77	0.19	0.23	0.28
p-menth-1-en-9-ol	t	-	t	-
octanol	0.01	-	t	-
cis-sabinene hydrate	0.03	0.01	0.03	0.02
α-santalene	t	0.01	0.01	t
bornyl acetate	0.01	-	t	-
nonyl acetate	t	-	-	-
α-bergamotene*	0.37	0.21	0.39	0.27
β-caryophyllene	0.18	0.17	0.13	0.16
terpinen-4-ol	0.02	0.02	0.02	-
undecanal	0.01	0.01	t	-
γ-elemene	t	-	t	-
(E)-2-decenal	t	-	t	-
β-santalene	0.01	0.01	t	t
α-humulene	t	0.02	-	-
citronellyl acetate	0.03	0.02	0.03	-
(E)-β-farnesene	0.04	t	0.04	-
neral	0.74	.106	0.77	2.00
δ-muurolene	0.01	t	t	t

Table XII (cont.). Comparative percentage composition of cold-pressed peel oils of four lemon cultivars

	Lemon oil ex. Cultivar			
Compound	Bagheria	Eureka	Feminello	Lisbon
decyl acetate	0.02	-	t	-
α-terpineol	0.27	0.23	0.31	0.27
2,7-dimethyl-2,6-octadienol	0.04	0.01	-	t
β-selinene	0.01	-	t	-
bicyclogermacrene	1.84	1.56	t	-
geranial	0.65	0.98	2.57	4.29
geranyl acetate	0.58	0.16	0.68	0.30
citronellol	t	t	t	t
perillaldehyde	0.03	0.02	0.07	0.09
geranyl propionate	t	0.01	-	t
nerol	0.06	0.02	0.03	0.02
geraniol	0.03	0.01	0.01	0.02
δ-sinensal‡	0.02	-	t	-
p-mentha-1,8-dien-9-ol	0.01	0.01	t	t
α-muurolol	0.01	-	t	-
verbenol*	0.01	-	t	0.01
hexadecanol	t	-	t	t
β-bisabolol	t	-	t	-
neryl acetate	t	0.01	t	t
α-bisabolol	0.02	0.01	0.03	0.03
nootkatone	0.01	-	-	-

*correct isomer not identified
†incorrect identification based on elution order
‡ probably α-sinensal

aurapten(7-geranyloxycoumarin)
7-isopentenyloxycoumarin
bergaptol (50hydroxypsoralen)
pabulen/gosferol(5-(2[l]-hydroxy-3[l]-methylbut-3-enyloxy)psoralen)
heraclenin (9-(2[l],3[l]-epoxyisopentyloxy)psoralen)
heraclenol (8-(2[l],3[l]-dihydroxyisopentyloxy)psoralen)
8-(6[l],7[l]-epoxygeranyloxy)psoralen
5-geranyloxy-8-methoxypsoralen
neobyakangelicol (5-methoxy-8-(2[l]-hydroxy-3-methbut-3-enyloxy)psoralen)
5-isopentenyl-8-(2[l],3[l]-dihydroxyisopentyloxy)psoralen
cnidicin (5,8-diisopentenyloxy)psoralen)
7-methoxy-8-geranyloxypsoralen

Verzera et al. (1999) compared the oxygen heterocyclic compounds of lemon oil produced by Sfumatrice, Pelatrice, FMC and Torchi. Using HPLC as their method of analysis seven major components were quantified. The results of this study can be seen in Table XV.

Using the technique known as fast HPLC Bonaccorsi et al. (1999) developed a method of analysis for the non-volatile oxygen heterocyclic compounds of citrus oils in–cluding lemon. The authors were able to show that the analysis time for these components could be reduced from 20-45 min to a mere 7 min with adequate separation of the following components:

citropten
oxypeucedanin
imperatorin
phellopterin
isoimperatorin
5-isopent-2[l]-enyloxy-7-methoxycoumarin
5-isopent-2[l]-enyloxy-8-(2[l],3[l]-epoxyisopentyloxy)psoralen
8-geranyloxycoumarin
bergamottin
5-geranyloxy-7-methoxycoumarin

It is recommended that citrus oil scientists examine this technique in combination with GC/MS and chiral GC analysis as a support technique for determining oil authenticity.

C. L. Barrett and D. B. Nelson, *Characterization of coumarin and psoralen levels in California and Arizona citrus oils*. In: *Flavor Analysis. Development in isolation and characterization*. Edits., C. J. Mussinan and M. J. Morello, pp. 233-238, ACS Symp. Series No. 705, Amer. Chem. Soc., Washington, DC (1998).

P. Dugo, L. Mondello, E. Cogliandro, A. Cavazza and G. Dugo, *On the genuineness of citrus oils. Part LIII. Determination of the composition*

Table XIII. Comparative percentage composition of nine Italian lemon cultivar oils									
Compound	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
tricyclene	0.03	t	t	t	t	t	t	t	t
α-thujene	0.32	0.33	0.28	0.29	0.27	0.26	0.39	0.33	0.29
α-pinene	1.38	1.50	1.18	1.21	1.12	1.13	1.51	1.41	1.44
camphene	0.04	0.04	0.03	0.03	0.03	0.03	0.04	0.04	0.04
sabinene + β-pinene	8.48	8.49	8.50	8.52	6.66	8.33	9.77	8.44	10.81
6-methyl-5-hepten-2-one	t	t	t	t	t	t	t	t	t
myrcene	1.72	1.75	1.54	1.62	1.65	1.45	1.52	1.54	1.60
α-phellandrene	0.06	0.06	0.12	0.04	0.07	0.10	0.09	0.03	0.11
octanal	0.04	0.03	0.03	0.04	0.03	0.04	0.04	0.03	0.03
δ-3-carene	t	t	t	t	t	0.01	t	t	t
α-terpinene	0.19	0.16	0.19	0.17	0.17	0.19	0.23	0.20	0.15
p-cymene	0.04	0.02	0.04	0.06	0.05	0.08	0.04	0.04	0.06
limonene	72.23	75.99	69.58	72.38	74.34	65.96	67.01	70.20	70.95
(Z)-β-ocimene	0.06	0.04	0.05	0.03	0.04	0.04	0.07	0.20	0.03
(E)-β-ocimene	0.12	0.08	0.11	0.07	0.08	0.10	0.13	0.29	0.07
γ-terpinene	9.27	7.42	9.43	8.70	8.55	9.82	11.11	9.61	7.16
cis-sabinene hydrate	0.07	0.05	0.07	0.07	0.06	0.08	0.08	0.06	0.07
octanol	t	t	t	t	-	t	-	-	-
terpinolene	0.42	0.32	0.42	0.39	0.39	0.46	0.48	0.42	0.31
trans-sabinene hydrate	0.07	0.04	0.10	0.06	0.07	0.09	0.10	0.05	0.06
linalool	0.17	0.13	0.20	0.18	0.18	0.22	0.14	0.13	0.15
nonanal	0.06	0.05	0.11	0.05	0.06	0.11	0.12	0.01	0.09
heptyl acetate	t	t	t	t	t	t	t	t	t
cis-limonene oxide	t	t	t	t	t	t	t	t	t
trans-limonene oxide	t	t	t	t	t	0.01	t	t	t
camphor	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	t
citronellal	0.15	0.08	0.18	0.21	0.17	0.33	0.10	0.07	0.19
borneol	0.01	0.01	0.03	0.02	0.02	0.03	0.02	0.01	0.02
terpinen-4-ol	0.03	0.02	0.03	0.03	0.03	0.04	0.03	0.03	0.02
α-terpineol	0.26	0.16	0.32	0.26	0.26	0.37	0.31	0.21	0.24
decanal	0.04	0.04	0.06	0.04	0.05	0.08	0.22	0.02	0.05
octyl acetate	t	t	t	t	t	t	t	t	t
citronellol + nerol	0.03	0.02	0.07	0.05	0.04	0.07	0.03	0.01	0.03
neral + carvone	0.92	0.48	1.72	1.06	1.05	2.47	1.44	0.41	1.34
piperitone	t	t	t	t	t	t	t	t	t
geraniol	0.02	0.01	0.04	0.03	0.02	0.05	0.05	0.01	0.03
geranial + perillaldehyde	1.51	0.78	2.86	1.76	1.75	4.16	2.42	0.68	2.20
bornyl acetate	t	0.01	0.01	0.01	0.01	0.01	0.01	0.01	t
undecanal	0.01	0.01	0.02	0.02	0.02	0.04	0.02	0.01	0.02
nonyl acetate	t	t	t	t	t	t	t	t	t
methyl geranate	0.01	0.01	0.01	0.01	0.01	0.01	t	0.02	t
citronellyl acetate	0.04	0.03	0.03	0.06	0.05	0.06	0.02	0.06	0.04
neryl acetate	0.30	0.31	0.46	0.53	0.42	0.60	0.39	0.38	0.40
geranyl acetate	0.22	0.17	0.31	0.35	0.34	0.45	0.54	0.49	0.31

Table XIII (cont.). Comparative percentage composition of nine Italian lemon cultivar oils

Compound	1	2	3	4	5	6	7	8	9
dodecanal	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01
decyl acetate	0.03	0.02	0.03	0.02	0.03	0.04	0.03	0.04	0.03
β-caryophyllene	0.22	0.16	0.23	0.31	0.25	0.44	0.28	0.35	0.17
trans-α-bergamotene	0.40	0.32	0.48	0.31	0.45	0.62	0.42	0.61	0.38
α-humulene	0.02	0.02	0.02	0.03	0.02	0.04	0.03	0.03	0.02
(E)-β-farnesene	0.04	0.03	0.04	0.03	0.04	0.06	0.04	0.05	0.03
β-santalene	0.02	0.01	0.02	0.01	0.02	0.02	0.02	0.02	0.01
γ-muurolene	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
germacrene D	0.01	0.01	0.01	0.01	0.01	0.01	0.01	t	0.02
valencene	0.02	0.01	0.02	0.02	0.01	0.06	0.01	0.03	0.01
bicyclogermacrene	0.07	0.04	0.05	0.09	0.05	0.11	0.02	0.02	0.05
(Z)-α-bisabolene	0.04	0.04	0.05	0.03	0.05	0.07	0.05	0.07	0.04
β-bisabolene	0.58	0.47	0.71	0.46	0.67	0.91	0.63	0.88	0.54
(E)-α-bisabolene	0.02	0.01	0.02	0.01	0.02	0.03	0.02	0.03	0.02
tetradecanal	0.01	0.01	0.01	0.02	0.01	0.02	0.01	0.05	0.01
2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	0.02	0.02	0.02	0.02	0.02	0.03	0.02	0.03	0.02
campherenol	0.03	0.02	0.03	0.02	0.03	0.04	0.03	0.04	0.02
α-bisabolol	0.03	0.02	0.03	0.02	0.03	0.05	0.03	0.04	0.02
nootkatone	t	t	t	t	t	0.01	t	t	t

1 = Feminello siracusano
2 = Feminello continella
3 = Feminello Santa Teresa
4 = Feminello fior d'aracio
5 = Feminello dosaco
6 = Feminello incappucciato
7 = Monachello
8 = Interdonato
9 = Fino
t = trace (<0.01%)

Table XIV. Enantiomeric distribution of selected compounds in lemon oil

Enantiomer	Pelatrice	Sfumatrice	Torchi	FMC	CP	Distilled
(S)-(-)-β-pinene	93.6-93.8	93.7	93.6-93.7	93.5-93.6	93.0-95.8	93.4-9.36
(R)-(+)-β-pinene	6.2-6.4	6.3	6.3-6.4	6.4-6.5	4.2-7.0	6.4-6.6
(S)-(-)-sabinene	85.4	85.4	85.3-85.4	85.1-85.5	84.7-87.5	85.4-87.3
(R)-(+)-sabinene	14.6	14.6	14.6-14.7	14.5-14.9	12.5-15.3	12.7-14.6
(S)-(-)-limonene	1.7	1.7-1.8	1.7	1.7	1.5-2.0	1.7
(R)-(+)-limonene	98.3	98.2-98.3	98.3	98.3	98.0-98.5	98.3
(R)-(-)-linalool	54.8-60.2	56.0-61.2	56.4-61.7	55.0-60.6	56.8-71.5	53.1-60.0
(S)-(+)-linalool	39.8-45.2	38.8-44.0	38.3-43.6	39.4-45.0	28.5-43.2	40.0-46.9
(R)-(-)-terpinen-4-ol	81.7-83.7	75.5-76.2	75.2-76.3	78.9-79.4	73.1-86.3	71.5-71.6
(S)-(+)-terpinen-4-ol	16.3-18.3	23.8-24.5	23.7-24.8	20.6-21.1	13.7-26.9	28.4-28.5
(S)-(-)-α-terpineol	74.1-78.3	78.2-78.4	73.5-77.0	77.2-78.6	64.2-82.0	76.4-77.0
(R)-(+)-α-terpineol	21.7-25.9	21.6-21.8	23.0-26.5	21.4-22.8	18.0-35.8	23.0-23.6
(S)-(-)-citronellal	ND	ND	ND	ND	89.0	ND
(R)-(+)-citronellal	ND	ND	ND	ND	11.0	ND

ND = not determined

of the oxygen heterocyclic fraction of lemon essential oils (Citrus lemon (L.) Burm. f.) by normal phase high performance liquid chromatography. Flav. Fragr. J. 13, 329-334 (1998).

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A. Verzera. P. Dugo, L. Mondello, A. Trozzi and A. Cotroneo, *Extraction technology and lemon oil composition*. Ital. J. Food Sci., 11, 361-370 (1999).

I. L. Bonaccorsi, H. M. McNair, L. A. Brunner, P. Dugo and G. Dugo, *Fast HPLC for the analysis of oxygen heterocyclic compound of Citrus essential oils*. J. Agric. Food Chem., 47, 4237-4239 (1999).

Cangerana Oil

Cangerana oil is produced from large trees (*Cabralea cangerana* Saldanha, syn. *C. glaberrima* A. Juss.) that originate in Rio Grande do Sul (Brazil). In 1996, Weyerstahl et al. used a combination of flash chromatography, fractional distillation, GC, GC/MS, ^{1}H-NMR and ^{13}C-NMR to analyze a commercial oil of cangerana. The spicy, woody-odored oil was found to possess the following composition:

safrole (0.5%)
δ-elemene (2.3%)
α-cubebene (0.9%)
α-ylangene (0.3%)
methyl eugenol (0.1%)
α-copaene (2.1%)
β-elemene (1.3%)
α-gurjunene (0.2%)
4,8-α-epoxycaryophyllene (0.2%)
β-caryophyllene (28.6%)
4,8-β-epoxycaryophyllene (0.5%)
γ-elemene (2.4%)
aromadendrene (1.1%)
α-humulene (1.8%)
allo-aromadendrene (0.7%)
γ-muurolene (5.0%)
4,10-epoxymuurolane (t)
4,10-epoxyamorphane (0.1%)
germacrene D (9.3%)
eudesma-4(15),11-diene (0.9%)
γ-amorphene (1.4%)
epi-cubebol + α-muurolene (2.4%)
β-bisabolene (0.4%)
γ-cadinene (2.7%)
cubebol + *trans*-calamenene (0.6%)
δ-cadinene (7.4%)
5,8-cyclocaryophyllan-4-ol (t)
cis-dracunculifoliol (t)
α-calacorene (2.9%)
eudesma-4(15),7(11)-diene[a] + eudesma-3,7(11)-diene[b] (1.1%)
(E)-nerolidol (0.3%)
germacrene B (6.2%)
caryolan-8-ol (1.8%)
spathulenol (0.4%)
caryolan-4-ol (0.1%)
caryophyllene oxide (1.4%)
10-epi-junenol + globulol (0.4%)
trans-dracunculifoliol + salvial-4(11)-en-1-one (0.3%)
humulene epoxide II (0.3%)
5α-H, 6βH, 8βH-cycloeudesma-4(15)-en-1β-ol + ledol (0.2%)
corocalene* (0.4%)
junenol (0.2%)
1αH, 5βH-guaia-6,10(14)-dien-4β-ol + humulene epoxide III (0.6%)

Table XV. Comparative analysis of oxygen heterocyclic compounds in lemon oils produced by four different processes

Compound	Sfumatrice (100)[a]	Pelatrice (59)	FMC (124)	Torchi (81)
bergamottin	2,635	2,955	2,877	2,973
5-geranyloxy-7-methoxycoumarin	2,453	2,711	2,845	2,656
citropten	1,360	1,495	1,473	659
8-geranyloxypsoralen	399	437	454	440
5-isopent-2[I]-enyloxy-8-(2[I], 3[I]-epoxy-isopentyloxy)psoralen	275	324	204	260
oxypeucedanin	1,556	2,200	1,909	863
byakangelicol	992	1,640	1,536	555

[a] number of samples analyzed
[b] mg/kg of oil

clovanol + 10-epi-cubenol (0.6%)
α-muurolol + T-cadinol + T-muurolol (2.4%)
cubenol (0.8%)
β-eudesmol + eudesm-11-en-4α-ol[c] + 10β-hydroxycalamene + α-cadinol (1.8%)
10α-hydroxycalamenene (0.2%)
cadalene (0.2%)
copabornyl acetate (0.2%)
eudesm-7(11)-en-4α-ol[d] (0.5%)

*correct isomer not identified
t = <0.1%
a = syn. selina-4(15),7(11)-diene
b = syn. selina-3,7(11)-diene
c = syn. selin-11-en-4α-ol
d = syn. selin-7(11)-4α-ol

P. Weyerstahl. S. Schneider and H. Marschall, *Constituents of the Brazilian cangerana oil*. Flav. Fragr. J., 11, 81-94 (1996).

Tejpat Oil

Tejpat oil is occasionally found as an item of commerce in India. It is obtained from leaves of *Cinnamomum tamala* (Ham.) Nees et Eberm.

In 1977, Gulati et al. analyzed two samples of lab-produced oils and found that they ranged in composition as follows:

α-pinene (0.26-0.30%)
limonene (0.05-0.10%)
β-phellandrene (0.10-3.95%)
p-cymene (0.64-0.82%)
β-ocimene* (t-0.14%)
γ-terpinene (t-0.22%)
camphor (0.90-3.19%)
linalool (15.28-15.67%)
borneol (1.07-1.18%)
β-caryophyllene (4.00-7.26%)
cadinene* (0-3.06%)
α-terpineol (1.54-1.77%)
geraniol (0.67-1.20%)
benzyl acetate (0.68-0.77%)
benzyl cinnamate (1.81-1.87%)
benzaldehyde (2.00-4.11%)
cinnamaldehyde (41.20-55.19%)
eugenol acetate (2.06-12.45%)
eugenol† + cinnamyl cinnamate (4.23-13.32%)

*correct isomer not identified
† major component of mixed peak

Two years later, Sood et al. (1979) compared the major components found in tejpat oil produced from leaves harvested in two regions of Himachal Pradesh. These oils were found to contain:

α-pinene (0.88-2.63%)
β-pinene (0.41-2.46%)
limonene (1.15-2.68%)
benzaldehyde (1.07-3.88%)
linalool (33.75-50.27%)
benzyl acetate (0.66-2.01%)
linalyl acetate (3.40-5.15%)
α-terpineol (2.88-3.28%)
geraniol (1.69-3.73%)
cinnamaldehyde (8.75-12.76%)
eugenol (0.98-1.57%)
benzyl cinnamate (0.95-2.19%)

According to Husain et al. (1988), tejpat oil is known to occur in two chemotypic forms; eugenol-type and a cinnamaldehyde-type. Nigam and Ahmed (1990) analyzed a leaf oil of *C. tamala* using GC. This oil was determined to contain the following components:

α-pinene (0.13%)
camphene (0.07%)
β-pinene (1.55%)
(Z)-β-ocimene (0.11%)
β-phellandrene (0.97%)
limonene (0.45%)
γ-terpinene (0.05%)
p-cymene (0.84%)
6-methyl-5-hepten-2-one (0.01%)
benzyl alcohol (0.59%)
β-caryophyllene (1.18%)
β-farnesene* (0.94%)
γ-curcumene (0.67%)
β-bisabolene (1.63%)
linalool (19.75%)
linalyl acetate (0.82%)
β-terpineol* (0.82%)
α-terpineol (0.20%)
α-terpinyl acetate (0.45%)
borneol (0.40%)
2-phenethyl alcohol (1.12%)
carvone (0.42%)
perillyl alcohol (0.16%)
cis-carveol (0.04%)
trans-carveol (0.01%)
citronellyl acetate (0.74%)
(E)-cinnamaldehyde (52.86%)
isoeugenol* (4.90%)
methyl isoeugenol* (1.63%)
farnesol* (0.69%)

*correct isomer not identified

Recently, Ahmed et al. (2000) analyzed an oil of *C. tamala* produced from leaves purchased at a local market in Pakistan. The botanical authenticity of this plant material was verified before an oil was produced from it in case it was misidentified in the market. The oil, which

was produced only in 0.03% yield was subject to analysis using GC/MS. The results of this analysis were as fol–lows:

α-pinene (0.5%)
camphene (0.2%)
β-pinene (0.2%)
myrcene (0.1%)
limonene (0.3%)
1,8-cineole (0.3%)
β-phellandrene (0.3%)
butyl 2-methylbutyrate (0.1%)
p-cymene (1.4%)
isoamyl isovalerate (0.4%)
α-cubebene (0.1%)
α-ylangene (0.9%)
α-copaene (0.8%)
β-bourbonene (0.1%)
linalool (13.4%)
β-elemene (0.6%)
terpinen-4-ol (0.7%)
β-caryophyllene (25.3%)
aromadendrene (1.5%)
α-humulene (6.2%)
γ-muurolene (1.6%)
α-terpineol (1.6%)
α-terpinyl acetate (3.2%)
germacrene D (1.0%)
valencene (0.3%)
α-muurolene (0.8%)
α-selinene (0.2%)
geranyl acetate (0.1%)
δ-cadinene (1.4%)
γ-cadinene (0.7%)
cuminaldehyde (0.8%)
nerol (t)
geraniol (0.3%)
cis-calamenene (0.7%)
p-cymen-8-ol (0.1%)
geranyl acetone (1.3%)
α-calacorene* (0.2%)
(E)-3-hexenyl nonanoate[a] (0.2%)
β-ionone[b] (0.2%)
α-calacorene* (0.1%)
isocaryophyllene oxide (1.9%)
caryophyllene oxide (10.3%)
humulene oxide I (0.1%)
humulene oxide II (1.7%)
β-caryophyllene alcohol (0.2%)
cubenol (0.4%)
1-epi-cubenol (0.6%)
globulol (0.9%)
viridiflorol (0.4%)
spathulenol (2.8%)
eugenol (0.1%)
T-cadinol (0.7%)
T-muurolol (0.8%)
α-muurolol (0.3%)
α-cadinol (1.5%)
selin-11-en-4α-ol (0.1%)
caryophylla-2(12),6(13)-dien-5α-ol (0.5%)
caryophylla-2(12),6-dien-5β-ol (0.4%)
dodecanoic acid (1.1%)
benzyl benzoate (0.1%)
tetradecanoic acid (0.2%)
hexadecanoic acid (1.1%)

*compound listed twice
[a] probably (Z)-isomer not (E)-isomer
[b] probably (E)-isomer

It is obvious from this analysis that either the *C. tamala* used in this study exists in other chemotypic forms than that which is normally encountered or it was taxonomically misclassified. According to Husain et al. (1988), the oil content of *C. tamala* leaves ranges from 0.23-0.36%, while the *C. tamala* analyzed by Ahmed had only a 0.03% oil content; however, low oil contents are not unusual for sesquiterpene-rich oils.

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Kanuka Oil

The oil of kanuka, which is produced to a limited extent in New Zealand, is obtained from a myrtaceous shrub or small tree *Kunzea ericoides* (A. Rich). J. Thompson.

In 1997, Perry et al. screened 26 populations of *K. ericoides* obtained from along the coast of North and South Island of New Zealand. The oils were found to have the following major constituents:

α-pinene (67.6%)[a]
β-pinene (0.6%)
p-cymene (5.8%)
1,8-cineole (4.3%)
γ-terpinene (1.6%)
linalool (1.7%)
α-terpineol (0.7%)
β-caryophyllene (0.2%)
trans-calamenene (0.2%)
viridiflorol (2.8%)
leptospermone (0.4%)

[a] average of 26 analyses

A year later, Porter and Wilkins (1998) used both GC and GC/MS to analyze the chemical composition of kanuka oil produced in New Zealand. The results of this analysis are summarized as follows:

α-thujene (0.62%)
α-pinene (55.51%)
camphene (0.08%)
β-pinene (0.63%)
myrcene (0.10%)
α-terpinene (0.08%)
p-cymene (3.41%)
1,8-cineole (3.94%)
limonene (3.94%)
(E)-β-ocimene (0.33%)
γ-terpinene (2.53%)
terpinolene (0.51%)
linalool (1.52%)
terpinen-4-ol (0.24%)
α-terpineol (0.91%)
α-cubebene (0.21%)
α-copaene (0.63%)
α-gurjunene (0.43%)
β-caryophyllene (0.48%)
aromadendrene (0.47%)
cadina-3,5-diene (0.53%)
α-humulene (0.17%)
allo-aromadendrene (0.70%)
δ-amorphene (0.49%)
β-selinene (0.31%)
α-selinene + viridiflorene (2.39%)
α-muurolene (0.20%)
calamenene* (3.02%)
δ-cadinene (0.95%)
cadina-1,4-diene (1.27%)
(E)-nerolidol (1.79%)
spathulenol (1.33%)
caryophyllene oxide (0.16%)
viridiflorol (7.23%)
ledol (1.86%)

*correct isomer not identified

Reichling et al. (1999) used both GC and GC/MS to examine the composition of a commercial sample of kanuka oil obtained in Germany. The constituents identified in the oil are summarized as follows:

α-thujene (1.32%)
α-pinene (58.39%)
β-pinene (0.53%)
p-cymene (7.81%)
limonene (0.18%)
1,8-cineole (6.07%)
γ-terpinene (4.89%)
terpinolene (1.18%)
linalool (1.97%)
terpinen-4-ol (0.39%)
α-terpineol (0.79%)
β-cubenene (0.36%)
β-caryophyllene (0.17%)
aromadendrene (0.34%)
allo-aromadendrene (0.43%)
α-bulnesene (1.10%)
calamenene* (2.92%)

*correct isomer not identified

Hethelyi et al. (2000) determined that the main components of kanuka oil were α-pinene (76.3-88.6%) and 1,8-cineole (6.1-8.6%).

More recently, Chrisoph et al. (2001) analyzed a commercial sample of kanuka oil. They reported that it contained the following constituents:

α-pinene (74.8%)
β-pinene (0.5%)
limonene (1.3%)
1,8-cineole (3.2%)
γ-terpinene (3.4%)
p-cymene (2.9%)
terpinolene (0.8%)
α-copaene (0.4%)
linalool (1.6%)
allo-aromadendrene (0.3%)
viridiflorene (0.6%)
α-terpineol (0.4%)
δ-cadinene (0.6%)
cadina-1,4-diene (0.5%)
trans-calamenene (0.9%)
ledol (0.4%)
(E)-nerolidol (0.7%)
viridiflorol (1.5%)
spathulenol (0.4%)

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