Progress in Essential Oils

Brian M. Lawrence, Consultant

Orange Oil

Using either a combined dialysis/pervaporation system or a combined ultrafiltration/reverse osmosis/dialysis system, Auerbach (1995) compared the composition of a folded orange oil with one prepared by vacuum distillation. The results of this comparative study are presented in **T-1**.

Huang and Wu (1998) used a combination of analytical techniques to examine the composition of cold-pressed orange oil produced from five cultivars ('Hamlin,' 'Jincheng,' 'Taoycheng,' 'Galliangcheng' and 'Washington') grown in China. The results of this study are shown in **T-2**. It is of interest to note that 'Galliangcheng' cultivar was richest in carbonyl compounds, so it probably had the most intense orange aroma.

Sawamura et al. (1999) reported that the major constituents of two samples of cold-pressed orange oil produced from fruit grown at an experimental station in Japan were as follows:

α-pinene (0.5%)
myrcene (1.8%)
limonene (95.8–96.6%)
octanal (0.1–0.3%)
decanal (0.1%)
linalool (0.3–0.4%)
neral (< 0.1–0.1%)
α-terpineol (0.1%)
geranial (0.1%)

In addition, trace amounts (< 0.1%) of β-pinene, γ-terpinene, terpinolene, citronellal, octanol, terpinen-4-ol, geranyl acetate, citronellol, nerol and geraniol were also found in the same oils.

Cold-pressed orange oils produced in Japan from the 'Hamlin,' 'Tarroco,' 'Trovita,' 'Valencia,' 'Omishima Navel' and 'Washington Navel' cultivars were analyzed by Sawamura (2000). The range in composition was found to be as follows:

α-pinene (0.51–0.57%)
β-pinene (0.01–0.05%)
sabinene (0.21–1.77%)
myrcene (1.76–1.87%)
limonene (93.91–96.58%)
(E)-β-ocimene (0.01–0.14%)
terpinolene (t–0.02%)
octanal (0.11–0.40%)
nonanal (0.02–0.03%)
trans-limonene oxide (t–0.01%)
trans-sabinene hydrate (t–0.04%)
citronellal (0.01–0.03%)
α-copaene (0.01–0.02%)
decanal (0.07–0.18%)
β-cubebene (t–0.02%)
linalool (0.13–0.57%)
octanol (0.01–0.02%)
γ-cadinene (0.01–0.02%)
β-caryophyllene (0.01–0.02%)
terpinen-4-ol (0–0.01%)
undecanal (t–0.01%)
(Z)-β-farnesene (0–0.01%)
(E)-β-farnesene (t–0.03%)
neral (0.03–0.09%)
α-terpineol (0.03–0.07%)
germacrene D (0.01–0.02%)
dodecanal (0.01–0.03%)
2,7-dimethyl-2,6-octadien-1-ol (0–0.01%)
geranial (0.06–0.15%)
valencene (0–0.02%)
α-farnesene* (0–0.04%)
δ-cadinene (0–0.03%)
geranyl acetate (0–0.03%)
citronellol (t–0.01%)
perillaldehyde (t–0.01%)

T-1 Comparative percentage composition of folded orange oil

Compound	Crude oil	Fold-1	Fold-2	Fold-3
α-pinene	0.37	0.02	0.24	0.60
sabinene	0.19	0.07	0.17	0.55
β-pinene	nd	0.08	nd	nd
myrcene	1.90	0.56	1.50	3.10
octanal	0.68	nd	6.20	1.40
limonene	95.60	93.40	76.40	86.40
γ-terpinene	0.01	0.12	0.52	nd
linalool	0.53	2.00	10.40	5.80
citronellal	0.09	0.33	0.21	0.14
α-terpineol	0.07	0.16	1.60	0.42
decanal	0.40	2.00	0.63	0.48
neral	0.06	0.63	0.82	0.46
geranial	0.11	0.56	1.20	0.64
Total oxygenated compounds	1.90	5.70	19.50	8.80

Fold-1 = vacuum distillation; Fold-2 = dialysis/pervaporation process; Fold-3 = ultra filtration/reverse osmosis/dialysis process; nd = not determined

nerol (0–0.01%)
p-mentha-1,8-dien-9-ol (0.01%)
elemol (0–0.01%)
octyl propyl ether† (0–0.01%)
β-sinensal (0.01–0.05%)
α-sinensal (t–0.04%)
nootkatone (0–0.01%)

*correct isomer not identified;
†identity requires corroboration;
t = trace (< 0.01%)

In addition, the authors also characterized trace amounts (< 0.01%) of camphene, α-phellandrene, β-phellandrene, (Z)-β-ocimene, γ-terpinene, β-elemene, (E)-2-decenal, decyl acetate, α-muurolene, carvone, geraniol, hexadecanol and an isomer of 2,4-decadienal in one or more of the same orange oils.

Mitiku et al. (2000) compared the composition of hand-pressed oils of the 'Valencia' and 'Hamlin' cultivars of orange grown in Eastern Ethiopia. The oil compositions can be seen summarized in **T-3**. Trace amounts (< 0.01%) of α-thujene, camphene, β-phellandrene, (Z)-β-ocimene, p-cymene, γ-terpinene, isoborneol, nerol, citronellol, geraniol, linalyl acetate, decanol, perillaldehyde, a 2,4-decadienal isomer, undecanal, α-terpinyl acetate, citronellyl acetate, α-cubebene, neryl acetate, (E)-2-undecenal, geranyl acetate, decyl acetate, α-humulene, germacrene D, a nerolidol isomer and caryophyllene oxide. Based on the results shown in **T-3**, it can be seen that 'Valencia' oil would have a more orange-like character because of its high concentration of the low threshold oxygenated constituents.

A commercial sample of sweet orange oil was the subject of analysis by capillary GC and ^{13}C-NMR (Kubeczka and Formacek

Percentage composition of the cold-pressed peel oils of sweet orange cultivars grown in China **T-2**

Compound	'Hamlin'	'Jincheng'	'Taoycheng'	'Galliangcheng'	'Washington'
hexanol	0.03	t	0.03	t	t
(Z)-3-hexenol	0.03	t	0.02	0.03	0.03
α-thujene	t	t	t	t	t
α-pinene	0.52	0.51	0.54	0.54	0.48
camphene	t	t	t	t	t
sabinene	0.66	0.23	0.33	1.15	0.33
myrcene	1.99	1.98	2.02	2.00	1.99
β-pinene	0.04	t	0.02	0.07	0.02
α-phellandrene	0.04	0.04	0.03	0.12	0.05
δ-3-carene	t	0.10	0.13	0.15	0.24
limonene	94.64	94.51	94.63	92.91	94.36
(Z)-β-ocimene	–	–	0.28	0.28	0.29
β-phellandrene	0.28	0.27	0.04	0.04	0.05
(E)-β-ocimene	0.03	0.03	–	–	–
p-cymene	–	–	t	0.02	t
γ-terpinene	0.03	t	0.03	0.06	0.06
octanol	t	0.04	t	0.03	t
cis-sabinene hydrate	t	t	0.02	0.02	0.05
terpinolene	0.03	0.02	0.42	0.47	0.56
linalool	0.17	0.57	0.02	0.05	0.02
trans-sabinene hydrate	t	0.04	t	t	t
nonanal	t	t	–	–	–
citronellal	0.09	0.08	0.08	0.08	0.07
terpinen-4-ol	t	t	t	t	t
decanal	0.10	0.19	0.13	0.27	0.14
α-terpineol	0.03	0.07	0.05	0.07	0.07
citronellol	0.03	t	0.04	t	0.03
nerol	0.02	t	0.02	0.02	0.03
geraniol	0.02	t	0.03	0.02	0.04
neral	0.05	0.11	0.08	0.12	0.08
undecanal	t	t	t	0.02	t
geranial	0.08	0.17	0.12	0.18	0.12
thymol	t	t	t	t	t
citronellyl acetate	t	t	t	0.02	t
perillaldehyde	t	0.02	0.02	0.02	t
α-copaene	0.04	0.03	0.04	0.04	0.04
neryl acetate	0.02	t	t	t	t
β-elemene	0.04	0.04	0.05	0.05	0.04
dodecanal	0.02	0.03	0.03	0.05	0.03
geranyl acetate	0.02	t	t	t	t
β-caryophyllene	0.03	t	t	0.04	0.03
β-farnesene*	t	t	0.03	0.15	0.04
α-humulene	t	t	t	t	t
α-farnesene*	t	t	t	0.14	t
γ-muurolene	0.03	–	0.03	0.04	0.03
valencene	0.05	0.07	0.05	0.05	0.07
γ-elemene	t	t	t	t	t
δ-cadinene	0.04	0.04	0.04	0.04	0.03
β-sinensal	0.02	0.02	0.04	0.05	0.04
farnesol*	0.03	0.02	0.02	0.03	0.03
farnesal*	–	–	t	t	t
α-sinensal	0.02	0.02	0.02	0.03	0.02

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

2002). They found that the oil comprised the following constituents:

α-pinene (0.53%)
β-pinene (0.03%)
sabinene (0.47%)
δ-3-carene (0.07%)
myrcene (1.84%)
limonene (94.39%)
β-phellandrene (0.24%)
(E)-β-ocimene (0.03%)
p-cymene (0.01%)
terpinolene (0.01%)
octanal (0.30%)
nonanal (0.05%)
cis-limonene oxide (0.07%)
trans-limonene oxide (0.04%)
citronellal (0.04%)
α-copaene (0.02%)
decanal (0.29%)
β-cubebene (0.02%)
linalool (0.40%)
octanol (0.10%)
β-caryophyllene (0.02%)
terpinen-4-ol (0.01%)
neral (0.06%)
α-terpineol (0.15%)
dodecanal (0.05%)
valencene (0.06%)
geranial (0.09%)
carvone (0.08%)
geranyl acetate (0.01%)
δ-cadinene (0.01%)
citronellol (0.02%)
nerol (0.02%)
geraniol (0.02%)
β-sinensal (0.04%)
α-sinensal (0.02%)
nootkatone (0.02%)

A hand-pressed sweet orange oil that was produced from fruit grown in Vietnam was analyzed by Minh Tu et al. (2002). The composition of this oil was determined to be as follows:

α-pinene (0.04%)
sabinene (0.2%)
δ-3-carene (0.1%)
myrcene (2.0%)
limonene (94.7%)
β-phellandrene (0.2%)
octanal (0.1%)
decanal (0.1%)
linalool (0.4%)
neral (0.1%)
α-terpineol (0.1%)
geranial (0.1%)

In addition, trace amounts (< 0.1%) of camphene, β-pinene, α-phellandrene, (Z)-β-ocimene, (E)-β-ocimene, 2-methylbutyl butyrate, terpinolene, tetradecane, *cis*-limonene oxide, menthone citronellal, β-cubebene, octanol, terpinen-4-ol, β-caryophyllene, a β-farnesene isomer, α-humulene, α-terpinyl acetate, valencene, germacrene D, neryl acetate, bicyclogermacrene, an α-farnesene isomer, a cadinene isomer, decanol, citronellol, perillaldehyde, octadecane, geraniol, perillyl alcohol, elemol, β-sinensal and α-sinensal were also found in the same oil.

An oil produced from the water distillation of an unidentified orange cultivar grown in India was subjected to a biotransformation study by Singh et al. (2002). The composition of the oil studies was as follows:

α-pinene (0.5%)
myrcene (0.7%)
δ-3-carene (0.3%)
limonene (64.5%)
octanol (0.1%)
linalool (0.6%)
cis-β-terpineol (3.2%)
trans-β-terpineol (1.3%)
terpinen-4-ol (1.0%)
α-terpineol (21.4%)
decanol (0.1%)
decanoic acid (0.1%)
dodecanoic acid (0.1%)

As can be seen, this hydrodistilled peel oil is very different to a cold-pressed oil because of the many hydrolytic reactions that took place during oil isolation.

Although orange oil is extremely rich in monoterpene hydrocarbons, it is the oxygenated constituents that cause the oil to have its characteristic aroma and flavor. As a result, various techniques have been used to concentrate the oxygenated constituents. Shen et al. (2002) used a combination

T-3 Comparative percentage composition of oils from 'Valencia' and 'Hamlin' cultivars of orange grown in Ethiopia

Compound	'Valencia' oil	'Hamlin' oil
α-pinene	0.35	0.41
sabinene	0.21	0.37
β-pinene	0.01	t
myrcene	1.79	1.77
octanal	0.23	0.23
δ-3-carene	0.03	0.01
limonene	96.19	96.57
(E)-β-ocimene	0.02	t
octanol	0.02	t
terpinolene	0.01	0.01
linalool	0.35	0.12
nonanal	0.04	0.03
cis-limonene oxide	0.02	0.03
trans-limonene oxide	0.01	t
citronellal	0.03	0.01
terpinen-4-ol	0.01	t
α-terpineol	0.01	t
decanal	0.16	0.10
cis-carveol	0.02	0.01
trans-carveol	0.02	0.01
neral	0.09	0.04
geranial	0.16	0.08
α-copaene	0.02	0.02
β-elemene	0.02	0.02
dodecanal	0.02	0.01
β-caryophyllene	0.01	0.01
(E)-β-farnesene	0.01	t
tridecanal	0.01	t
β-sinensal	0.03	t
α-sinensal	0.02	t

t = trace (< 0.01%)

of silica gel adsorption followed by fractional extraction by supercritical carbon dioxide at a low temperature. The original cold-pressed oil possessed the following composition:

α-thujene (< 0.01%)
α-pinene (0.46%)
sabinene (0.46%)
β-pinene (0.03%)
myrcene (1.66%)
α-phellandrene (0.03%)
δ-3-carene (0.09%)
α-terpinene (< 0.01%)
p-cymene (< 0.01%)
limonene (94.74%)
(E)-β-ocimene (0.03%)
γ-terpinene (< 0.01%)
terpinolene (0.02%)
octanal (0.22%)
nonanal (0.04%)
decanal (0.38%)
dodecanal (0.11%)
citronellal (0.05%)
neral (0.02%)
geranial (0.03%)
perillaldehyde (0.06%)
β-sinensal (0.03%)
α-sinensal (0.04%)
octanol (0.12%)
linalool (0.40%)
α-terpineol (0.06%)
isodihydrocarveol (0.01%)
cis-carveol (0.05%)
trans-carveol (0.05%)
carvone (0.08%)
cis-limonene oxide (0.08%)
trans-limonene oxide (0.05%)
neryl acetate (0.01%)
geranyl acetate (0.01%)
α-copaene (0.01%)
β-cubebene (0.01%)
β-farnesene* (0.02%)
germacrene D (0.01%)
valencene (0.03%)
α-farnesene* (0.02%)
δ-cadinene (0.02%)

*correct isomer not identified

Shen et al. also found that adsorption removed 75% of the terpene hydrocarbons and through the use of fractional supercritical CO_2 extraction at a 13.1 MPa, 35°C and a throughput of 2 kg/h the decanal in the feed oil was concentrated 20 times that of the feed oil; however, the weight of the feed oil was 487.9 g while the decanal-rich fraction was only 1.08 g. Nevertheless, the technique definitely demonstrated the ability to fractionate a hydrocarbon-rich oil to selectively concentrate desirable oxygenated constituents.

Giamperi et al. (2002) compared the composition of water distilled oils of the peels of blond and blood oranges obtained from the vicinity of Modica (Sicily, Italy). The results of this comparative study are presented in **T-4**.

Ogawa et al. (2002) investigated the use of two novel techniques (capillary liquid chromatography and micellar electrokinetic chromatography) to determine the absolute amounts of selected constituents of Brazilian orange oil. They found that micellar electrokinetic chromatography presented a lower detection limit for compounds such as neral, geranial, α-terpineol, carvone, linalool and α- and β-pinene than capillary liquid chromatography.

Marongiu et al. (2003) compared the composition of a hydrodistilled peel oil of the 'Tarocco' cultivar of orange obtained from Oristano (Sardinia, Italy) with a volatile concentrate of the same batch of peel. The only components characterized in the hydrodistilled oil were:

α-pinene (0.61%)
sabinene (0.37%)
myrcene (2.97%)
octanal (0.59%)
limonene (93.85%)
linalool (0.86%)
decanal (0.75%)

In comparison, the volatile concentrate (which is not an oil) that was produced at 110 bar, 50°C in the extraction vessel, 110 bar and -12°C in first separator and 20 bar and 15°C in second separator was found to contain the following components:

α-pinene (0.81%)
sabinene (0.39%)
myrcene (3.46%)
octanal (0.71%)
α-phellandrene (0.19%)

T-4 Comparative percentage composition of the water distilled peel oils of blond and blood orange of Sicilian origin

Compound	Blond orange oil	Blood orange oil
α-pinene	0.59	0.20
camphene	0.66	0.25
sabinene	–	0.22
β-pinene	0.04	–
octanal	1.44	1.15
myrcene	3.12	2.24
δ-3-carene	0.15	–
limonene	83.62	78.60
trans-sabinene hydrate	0.60	–
octanol	0.60	–
terpinolene	0.11	–
nonanal	0.15	0.26
linalool	2.80	5.87
trans-p-mentha-2,8-dien-1-ol	0.40	0.39
cis-p-mentha-2,8-dien-1-ol	0.20	1.98
trans-limonene oxide	0.20	–
citronellal	0.14	0.28
terpinen-4-ol	–	0.91
α-terpineol	0.82	1.30
decanal	0.05	0.74
linalyl formate	0.20	–
trans-carveol	0.10	0.21
nerol	0.09	0.24
neral	0.27	0.46
carvone	0.20	–
geraniol	–	0.28
perillaldehyde	0.09	0.24
α-cedrene	–	2.74
valencene	0.18	1.13

T-5 Comparative percentage composition of a orange oil analyzed by conventional or fast GC

Compound	Conventional GC analysis	Fast GC analysis
α-thujene	0.03	0.03
α-pinene	0.64	0.58
camphene	0.01	0.01
sabinene	0.52	0.47
β-pinene	0.91	0.81
myrcene	2.03	1.80
octanal + α-phellandrene	0.18	0.16
δ-3-carene	0.16	0.14
α-terpinene	0.02	0.02
p-cymene + limonene†	93.33	93.34
(Z)-β-ocimene	0.01	t
(E)-β-ocimene	0.62	0.60
γ-terpinene	0.59	0.52
cis-sabinene hydrate	0.04	0.04
terpinolene	0.05	0.05
linalool	0.30	0.27
nonanal	0.04	0.03
cis-limonene oxide	0.01	0.01
trans-limonene oxide	0.02	0.02
citronellal	0.04	0.04
terpinen-4-ol	t	0.01
α-terpineol	0.06	0.05
decanal	0.17	0.16
octyl acetate	t	t
nerol	0.02	0.02
neral	0.06	0.06
geraniol	0.01	0.02
geranial	0.13	0.11
perillaldehyde	t	0.02
bornyl acetate	0.01	0.01
undecanal	0.01	0.01
citronellyl acetate	0.01	0.01
neryl acetate	0.04	0.04
α-copaene	0.02	0.02
geranyl acetate	0.03	0.03
β-cubebene + β-elemene	0.03	0.03
dodecanal	0.03	0.03
decyl acetate	0.01	0.01
β-caryophyllene	0.03	0.03
β-copaene	0.02	0.02
trans-α-bergamotene	0.03	0.03
(Z)-β-farnesene	0.02	0.01
α-humulene	t	t
germacrene D	0.02	0.01
α-farnesene*	0.17	0.16
δ-cadinene	0.02	0.02
tetradecanal	0.01	0.01
2,3-dimethyl-3-(4-methyl-3-pentenyl)-2-norbornanol	0.01	0.01
β-sinensal	0.02	0.02
α-sinensal	0.01	0.02
nootkatone	0.02	0.02

†major component of mixture; t = trace (< 0.01%); *correct isomer not identified

δ-3-carene (0.13%)
limonene (91.21%)
(Z)-β-ocimene (0.08%)
(E)-β-ocimene (0.03%)
octanol (0.09%)
terpinolene (0.17%)
linalool (0.97%)
nonanal (0.11%)
citronellal (0.08%)
α-terpineol (0.11%)
decanal (1.13%)
neral (0.06%)
geranial (0.15%)
dodecanal (0.14%)

Mondello et al. (2003) compared the results obtained from a conventional (ca 46 min) and fast (9 min) GC analyses of sweet orange oil of Sicilian origin. The results of this study are presented in **T-5**. These results were also repeated in a follow-up study by Mondello et al. (2004).

Vacuum fractional distillation is one of the commercial techniques used to fold citrus oils. Lopes et al. (2003) showed that five-fold orange oil could be efficiently produced from Brazilian cold-pressed orange oil using a temperature of 80°C, 10 mbar pressure and a 0.25 reflux ratio. A comparison between the original oil and the five-fold oil produced this way is shown in **T-6**. Additional trace amounts (< 0.05%) of α-pinene, sabinene, β-pinene, terpinen-4-ol, geraniol, geranyl acetate and α-humulene were also found in both oils.

The composition of cold-pressed orange oils produced from organically-grown trees and trees grown under standard (normal) agricultural practices in Sicily, Italy, was the subject of study by Verzera et al. (2004). The results presented in **T-7** are the average results of 10 analyses.

Sawamura et al. (2005) analyzed cold-pressed orange oils produced from Chinese cultivars 'Hongjiang,' 'Anliu,' 'Sihui' and 'Washington Navel' all grown in different regions in China. The comparative oil compositions are shown in **T-8**. In addition, trace amounts (< 0.1%) of camphene, β-pinene, α-phellandrene, (Z)-β-ocimene, 1,8-cineole, p-cymene, tetradecane, *cis*-limonene oxide, *trans*-limonene oxide, β-cubebene, octanol, α-cedrene, β-elemene, terpinen-4-ol, β-caryophyllene, undecanal, α-humulene, α-terpinyl acetate, germacrene D, valencene, neryl acetate, carvone, bicyclogermacrene, geranyl acetate, citronellol, β-bisabolene, β-sesquiphellandrene, cuminaldehyde, perillaldehyde, octadecane, *cis*-carveol, geraniol, isopiperitenone, nerol, perillyl alcohol, dihydrocarveol, (E)-2-dodecenal, (Z)-nerolidol, (E)-nerolidol, limonene di-epoxide, elemol, cedrol, (Z,E)-farnesol, β-sinensal and α-sinensal were found in one or more of the cultivar oils.

Njoroge et al. (2005) compared the cold-pressed peel oils of the 'Salustiana,' 'Valencia' and 'Washington Navel' cultivars of orange grown in the Mombasa district (Kenya). The data obtained from this study are presented in **T-9**. Trace amounts (< 0.1%) of ethanol, β-pinene, myrcene, α-phellandrene, β-phellandrene, 1,8-cineole, (Z)-β-ocimene, γ-terpinene, p-cymene, terpinolene, α-pinene oxide, p-menth-1-en-9-al, nonanal, a myrcene oxide, (E,Z)-2,6-nonadienol, perillene, an isomer of *cis*-linalool oxide, *cis*-limonene oxide, *trans*-sabinene hydrate, heptanol, δ-elemene, citronellal, α-cubebene, octanol, α-cedrene, terpinen-4-ol, β-caryophyllene, aromadendrene, dihydrocarveol, *trans*-p-mentha-2,8-dien-1-ol, neral, *cis*-piperitol, dodecanal, germacrene D, geranyl acetate, a carvone oxide isomer, perillyl acetate, limonene di-epoxide, (Z)-nerolidol, globulol, elemol, viridiflorol, cedrol, eugenol, β-sinensol, (Z,E)-farnesol, α-sinensal, undecanoic acid, octadecanal, 1,4,7,10-tetraoxa-cyclodecane, 1,4,7,10,13-pentaoxa-cyclopentadecane, dodecanoic acid and 1,4,7,10,13,16-hexaoxa-cyclo-octadecane were found in one or more of the cultivar oils. It should be pointed out that the tetra-, penta- and hexa oxa-hydrocarbons were only tentatively identified. The authors reported that the mass spectra of these compounds were nearly identical to those of 1-decene, 1-pentadecene and 1-octadecene, respectively.

As valencene, which is the major sesquiterpene hydrocarbon found in Valencia orange oil, has been traditionally used to determine the commercial value of Valencia orange oil, Elston et al. (2006) used multidimensional GC-olfactometry/GC/MS to determine the value of valencene in orange oil. Initially, it was found that the valencene content ranged from 0.30–0.38% in four commercial samples of Valencia orange oil. Through the use of multidimensional GC-olfactometry it was shown (this is hardly earth-shattering) that at the levels found in orange oil valencene do not possess any input on the aroma of orange. Elston et al. did list 37 components that were aroma-active in orange oil. Of these 37, they characterized α-pinene, myrcene, limone, (Z)-β-ocimene, octanal, ethyl heptanoate, a linalool oxide isomer, 1-octen-3-ol, citronellal, decanal, (E)-2-nonenal, linalool, (Z)-2-decenal, (E)-2-decenal, neral, dodecanal, geranial, (E,Z)-2,4-decadienal, citronellol, (E,E)-2,4-decadienal, geraniol, *trans*-4,5-epoxy-(E)-2-decenal, eugenol, wine lactone and β-sinensal. It is a shame that the compounds were not ranked in their comparative importance on the overall orange aroma. Also, it was a major surprise to this reviewer that decanal was not listed as

T-6 Comparative percentage composition of Brazilian cold-pressed orange oil and a five-fold oil produced from it

Compound	Cold-pressed oil	Five-fold oil
myrcene	1.9	0.1
octanal	0.2	t
δ-3-carenen	0.1	t
limonene	95.4	89.3
octanol	t	0.1
terpinolene	t	0.1
linalool	0.4	2.6
nonanal	0.1	0.3
p-menth-2,8-dien-1-ol*	t	0.1
limonene oxide*	t	0.1
citronellal	0.1	0.3
α-terpineol	0.1	0.4
decanal	0.3	1.8
octyl acetate	t	0.1
trans-carveol	t	0.1
nerol	t	0.1
neral	t	0.2
carvone	t	0.1
geranial	0.1	0.5
perillaldehyde	t	0.2
perillyl alcohol	t	0.1
perillyl acetate	t	0.1
α-ylangene	t	0.1
β-cubebene	t	0.1
methyl N-methyl anthranilate	t	0.1
dodecanal	0.1	0.3
β-caryophyllene	t	0.1
trans-α-bergamotene	t	0.2
allo-aromadendrene	t	0.1
valencene	t	0.2
α-muurolene	t	0.1
δ-cadinene	t	0.2
β-sinensal	t	0.1
α-sinensal	t	0.1
nootkatone	t	0.1
hexadecanoic acid	t	0.1

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

Percentage composition of cold-pressed orange oil produced under different agricultural practices **T-7**

Compound	Organic oil	Standard oil
tricyclene	t	t
α-thujene	0.02	0.01
α-pinene	0.65	0.71
camphene	0.01	0.01
sabinene + β-pinene	1.00	0.42
6-methyl-5-hepten-2-one	t	t
myrcene	2.09	2.49
octanal	0.41	0.10
α-phellandrene	0.07	0.03
δ-3-carene	0.31	0.31
α-terpinene	0.01	0.01
p-cymene	t	t
limonene	93.67	94.69
(Z)-β-ocimene	0.01	t
(E)-β-ocimene	0.05	0.03
γ-terpinene	0.26	0.04
cis-sabinene hydrate	0.01	0.01
octanol	0.03	0.01
terpinolene	0.06	0.05
trans-sabinene hydrate	t	t
linalool	0.31	0.32
nonanal	0.06	0.02
cis-limonene oxide	t	0.01
trans-limonene oxide	0.01	0.02
camphor	t	t
citronellal	0.04	0.04
borneol	t	t
terpinen-4-ol	0.01	t
α-terpineol	0.05	0.05
decanal	0.27	0.08
octyl acetate	t	t
cis-carveol	0.01	0.01
nerol + citronellol	0.01	0.01
neral	0.05	0.02
piperitone	t	t
geraniol	0.01	t
geranial	0.13	0.05
perillaldehyde	0.01	0.01
bornyl acetate	0.01	0.01
undecanal	0.01	0.01
nonyl acetate	t	t
α-terpinyl acetate	t	t
citronellyl acetate	t	0.01
neryl acetate	0.02	0.01
α-copaene	0.02	0.02
geranyl acetate	0.02	0.01
β-cubebene + β-elemene	0.02	0.02
dodecanal	0.04	0.01
decyl acetate	t	t
β-caryophyllene	0.02	0.01
γ-cadinene†	0.02	0.02
α-humulene	0.01	0.02
(Z)-β-farnesene + (Z)-β-santalene	0.01	t
γ-muurolene	0.01	t
germacrene D	0.02	0.02
valencene	0.05	t
α-farnesene*	0.03	0.11
δ-cadinene	0.02	0.03
tridecanal	t	t
(Z)-nerolidol	t	0.01
tetradecanal	t	t
β-sinensal	0.02	0.02
α-sinensal	0.01	t
nootkatone	0.01	0.01

*correct isomer not identified; †incorrect identification based on GC elution order; t = trace (< 0.01%)

Comparative percentage composition of cold-pressed oils of orange cultivars grown in China **T-8**

Compound	'Hongjiang' oil	'Anliu' oil	'Sihui' oil	'Washington Navel' oil
α-pinene	0.4	0.5	0.5	0.5
sabinene	0.7	0.8	0.8	0.8
δ-3-carene	0.1	0.1	0.1	0.1
myrcene	2.0	2.0	2.1	2.0
limonene	93.6	94.2	93.6	94.4
β-phellandrene	0.3	0.3	0.3	0.3
(E)-β-ocimene	t	t	t	0.1
terpinolene	t	0.3	t	t
octanal	0.4	t	0.2	0.4
nonanal	0.1	t	t	t
citronellal	0.1	0.1	0.1	t
decanal	0.3	0.2	0.1	0.2
linalool	0.7	0.4	0.4	0.4
(E)-β-farnesene	0.1	0.1	t	t
neral	0.2	0.2	0.2	0.1
α-terpineol	0.1	0.1	0.1	0.1
dodecanal	0.1	t	t	t
geranial	0.3	0.2	0.2	0.2
α-farnesene*	0.1	0.1	0.1	t

*correct isomer not identified

Comparative percentage composition of the cold-pressed oils of three orange cultivars grown in Kenya **T-9**

Compound	'Salustiana' oil	'Valencia' oil	'Washington Navel' oil
α-pinene	0.5	0.3	0.5
sabinene	0.1	0.2	0.2
α-terpinene	1.7	1.5	1.5
limonene	94.6	92.5	90.5
octanal	0.2	0.2	0.3
trans-limonene oxide	0.1	0.3	0.4
trans-linalool oxide*	0.1	0.2	0.2
decanal	0.2	0.2	0.2
linalool	0.4	0.5	0.5
linalyl acetate	t	0.1	0.1
undecanal	0.1	t	t
sabina ketone	0.1	0.2	0.2
α-terpineol	0.1	0.1	0.1
geranial	0.1	t	0.1
carvone	0.2	0.3	0.5
(E,E)-α-farnesene	t	0.1	0.1
cuminaldehyde	t	t	0.1
perillaldehyde	t	t	0.1
cis-carveol	0.1	0.3	0.5
trans-carveol	0.1	0.2	0.3
caryophyllene oxide	t	0.1	0.2
perillyl alcohol	t	0.1	0.1
nonanoic acid	–	0.1	0.1
nerolidol acetate*	t	0.1	0.2
nootkatone	t	0.1	t

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

an aroma-active component of orange oil.

Ranade (2006) reported that a commercial sample of orange oil of unknown origin contained:

α-pinene (0.60%)
β-pinene (0.02%)
sabinene (0.35%)
myrcene (2.50%)
limonene (94.5%)
octanal (0.15%)
nonanal (0.04%)
nonanal† (0.35%)
linalool (0.46%)
neral (0.10%)
valencene (0.15%)
geranial (0.15%)
β-sinensal (0.03%)
α-sinensal (0.03%)

†probably should be decanal

As part of a study on the lipolytic effects of citrus oils, Choi (2006) determined that oils from 'Valencia' and 'Navel' orange of US origin contained the following compositions, respectively:

α-pinene (0.49%, 0.51%)
camphene (< 0.01%, 0.01%)
undecane (0.07%, 0.17%)
β-pinene (0.10%, 0.24%)
myrcene (1.11%, 2.26%)
limonene (95.59%, 95.27%)
γ-terpinene (0.03%, 0.11%)
p-cymene (0.03%, 0.11%)
terpinolene (0.01%, 0.01%)
octanal (< 0.01%, 0.03%)
tridecane (< 0.01%, 0.06%)
tetradecane (0.01%, 0.01%)
linalool oxide* (0.17%, 0.01%)
citronellal (0.15%, 0.02%)
linalool (0.12%, 0.19%)
octanol (< 0.01%, 0.01%)
linalyl acetate (0.03%, 0.01%)
β-elemene (0.01%, 0.01%)
terpinen-4-ol (0.04%, < 0.01%)
citronellyl acetate (0.05%, 0.01%)
neral + geranial (0.02%, 0.04%)
geranyl acetate (0.04%, 0.01%)
citronellol (0.03%, 0.02%)
geraniol (0.01%, < 0.01%)
nerol (0.02%, < 0.01%)
octanoic acid (0.02%, 0.01%)
*correct isomer not identified

Ferhat et al. (2006) described a method of microwave assisted hydrodistillation using a Clevenger-type system with no addition of solvent or water to isolate oil from the orange peel. The oil was compared with the

Comparative percentage composition of orange peel oil produced by microwave hydrodistillation (MHD) and hydrodistillation (HD)

T-10

Compound	MHD	HD
α-pinene	0.5	1.6
sabinene	1.2	1.2
β-pinene	2.4	2.7
myrcene	4.3	5.3
limonene	76.7	78.5
δ-3-carene†	0.1	0.1
γ-terpinene	0.3	0.2
terpinolene	0.2	0.2
linalool	3.1	2.0
trans-limonene oxide	0.3	0.1
citronellal	0.4	0.3
terpinen-4-ol	0.3	0.5
α-terpineol	0.8	0.4
trans-carveol	0.2	0.3
nerol	0.6	0.3
neral	0.4	0.3
geraniol	0.3	0.1
geranial	0.5	0.5
perillyl alcohol	0.1	–
α-copaene	0.1	0.1
β-cubebene	0.1	0.1
β-elemene	0.1	0.1
β-caryophyllene	0.2	0.2
β-gurjunene	0.1	0.1
(E)-β-farnesene	0.1	0.1
germacrene D	0.1	0.1
valencene	0.2	0.4
δ-cadinene	0.1	0.2
β-sinensal	0.2	0.2
α-sinensal	0.7	0.1
octyl acetate	0.1	0.1
hexanol	0.2	–
octanol	1.1	0.5
decanol	0.3	–
nonanal	0.2	0.1
decanal	1.9	1.7
undecanal	0.1	0.1
dodecanal	0.2	0.2

†incorrect identification based on GC elution order

standard Clevenger-type water distillation process used in labs throughout the world. The two oils produced were analyzed by both GC and GC/MS, the results of which can be seen in **T-10**. It would appear that the distillations were not performed to obtain all of the potential oil as the limonene contents of these oils were too low.

Ferhat et al. (2007) compared the production of oil from orange peels by hydrodistillation, microwave assisted hydrodistillation and cold-pressing. The results of this comparative study are summarized in **T-11**.

Rossi et al. (2007) found that a leaf oil of sweet orange of Corsican origin contained the following major components:

α-thujene (4.1%)
α-pinene (1.6%)
sabinene (23.3%)
β-pinene (2.0%)
myrcene (1.8%)
δ-3-carene (4.4%)
p-cymene (4.4%)
limonene (22.8%)
γ-terpinene (1.8%)
linalool (4.1%)

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Comparative percentage composition of the main components of orange peel oil produced by different processes **T-11**

Compound	MHD	HD	CP
α-pinene	0.4	0.5	0.5
sabinene	0.5	0.5	0.5
myrcene	1.6	1.9	1.8
limonene	94.6	95.5	95.1
δ-3-carene†	0.1	0.1	0.1
γ-terpinene	0.1	–	–
octanol	0.2	0.1	–
octyl acetate	0.1	0.1	0.1
decanal	0.3	0.2	0.3
undecanal	–	–	0.1
linalool	0.6	0.3	0.3
citronellal	0.1	–	–
terpinen-4-ol	–	0.1	–
α-terpineol	0.1	0.1	–
nerol	0.1	–	0.1
neral	0.1	–	0.1
valencene	–	0.1	–
δ-cadinene	0.1	–	0.1
β-sinensal	0.1	–	0.1

†incorrect identification based on GC elution order; MHD = microwave hydrodistillation; HD = hydrodistillation; CP = cold-pressed