

# **Progress in Essential Oils**

Brian M. Lawrence, Consultant

#### **Bitter Orange Oil**

Bitter orange peel oil of Chinese origin was analyzed by Lin et al. (1986). Using a combination of GC retention indices, IR and GC/MS, the oil was found to contain the following constituents:

 $\alpha$ -pinene (0.35 percent)  $\beta$ -pinene (0.09 percent) myrcene (1.91 percent) limonene (96.41 percent) linalool (0.16 percent) trans-pinocaveol (t) camphor (t)  $\alpha$ -terpineol (0.03 percent) nerol (t) neral (t) linalyl acetate (0.64 percent) trans-linalool oxide-pyranoid (t) cis-linalool oxide-pyranoid (t) neryl acetate (0.01 percent) geranyl acetate (0.15 percent) nonanal (t) β-caryophyllene (0.03 percent)  $\alpha$ -humulene (t) γ-muurolene (0.03 percent) (Z)-nerolidol (0.03 percent)

t = trace (< 0.1 percent)

Protopapadakis and Papanikolaou (1998) examined the cold-pressed peel oils of four cultivars (Chania, Brazil, Keen and Bittersweet) of bitter orange grown in Crete (Greece). The components identified in these oils can be seen in T-1.

Although the use of fast HPLC methods is not generally very useful in essential oil analysis, because of the occurrence of non-volatile oxygen hetrocyclic compounds in citrus oils an interesting niche for this technique has been found by Bonaccorsi et al. (1999). They found that adequate separation was obtained for the following bitter orange components:

meranzin & isomeranzin bergapten nobiletin 3,3',4',5,6,7,8-heptamethoxyflavone tangeretin osthole epoxybergamottin

Although the authors did not present any quantitative data, from the area count information presented they were able to show that with the exception of the heptamethoxyflavone, which was present at an extremely low level, all of the percent RSDs were 0.7 or less.

Pino and Rosado (2000) analyzed commercial oil of bitter orange produced in Cuba using GC and GC/ MS. The oil composition was found to be as follows:

α-pinene (0.9 percent) sabinene (0.1 percent)  $\beta$ -pinene (0.6 percent) myrcene (4.9 percent) limonene (86.2 percent)  $\gamma$ -terpinene (0.7 percent) terpinolene (0.1 percent) linalool (0.3 percent) terpinen-4-ol (0.1 percent)  $\alpha$ -terpineol (0.4 percent) cis-piperitol (0.3 percent) geranial (0.1 percent) terpinen-4-yl acetate (0.7 percent) neryl acetate (0.5 percent) geranyl acetate (0.5 percent) dodecanal (0.1 percent) trans-a-bergamotene (0.3 percent) dodecanal (0.2 percent) germacrene D (0.2 percent)  $\beta$ -bisabolene (0.5 percent) (Z)-nerolidol (0.2 percent)

Trace amounts (< 0.1 percent) of *cis*-limonene oxide, *trans*-limonene oxide, decanal, octyl acetate, neral,  $\alpha$ -terpinyl acetate, (Z)- $\beta$ -damascenone, (E,E)- $\alpha$ -farnesol and bergapten were also found in the same oil. Comparative percentage composition of oils produced from four cultivars of bitter orange

Compound	Chania	Brazil	Keen	Bittersweet
$\alpha$ -pinene	0.38	0.38	0.39	0.37
β-pinene	0.51	0.23	0.48	0.33
sabinene	0.13	0.10	0.13	0.10
myrcene	1.55	1.62	1.61	1.60
lpha-terpinene	0.22	0.01	0.02	0.02
limonene	89.65	93.00	91.80	88.02
β-phellandrene	0.25	0.26	0.26	0.26
(Z)-β-ocimene	0.02	0.01	0.02	0.02
γ-terpinene	0.04	0.04	0.04	0.04
(E)-β-ocimene	0.26	0.14	0.20	0.18
terpinolene	0.59	0.48	0.54	0.59
linalool	1.97	0.82	1.44	1.05
lpha-terpineol	0.55	0.40	0.50	0.50
nerol	0.13	0.11	0.12	0.13
terpinen-4-ol	0.13	0.08	0.15	0.13
geraniol	0.19	0.09	0.16	0.16
linalyl acetate	0.47	0.19	0.28	0.21
neryl acetate	0.09	0.05	0.09	0.07
geranyl acetate	0.26	0.23	0.25	0.22
trans–linalool oxide*	0.18	0.24	0.22	0.22
<i>cis</i> -linalool oxide*	0.13	0.13	0.14	0.15

\*correct isomer not identified, although most likely the furanoid form

A sample of cold-pressed bitter orange oil of Japanese origin was analyzed by Sawamura (2000) and found to possess the following composition:

a-pinene (0.28 percent)  $\beta$ -pinene (0.03 percent) sabinene (0.29 percent) myrcene (1.73 percent) limonene (96.28 percent)  $\gamma$ -terpinene (0.02 percent) (E)- $\beta$ -ocimene (0.01 percent) terpinolene (0.03 percent) octanal (0.10 percent) nonanal (0.01 percent) trans-sabinene hydrate (0.01 percent) citronellal (0.04 percent) α-copaene (0.01 percent) decanal (0.10 percent) linalool (0.34 percent)  $\gamma$ -cadinene (0.01 percent) citronellyl acetate (0.01 percent) neral (0.03 percent)  $\alpha$ -terpineol (0.05 percent) germacrene D (0.01 percent) dodecanal (0.02 percent) geranial (0.07 percent) δ-cadinene (0.03 percent) citronellol (0.01 percent) α-sinensal (0.01 percent) p-mentha-1,8-dien-9-ol (0.03 percent) β-sinensal (0.01 percent)

Trace amounts (< 0.01 percent) of camphene,  $\alpha$ -phellandrene,  $\beta$ -phellandrene, (Z)- $\beta$ -ocimene, p-cymene, *cis*-limonene oxide, *trans*-limonene oxide,  $\beta$ -cubebene, octanol,  $\beta$ -elemene,  $\beta$ -caryophyllene,  $\alpha$ humulene, decyl acetate, carvone, geranyl acetate, perillaldehyde, *trans*-carveol and an isomer of 2,4-decadienal were also found in this same oil.

Lota et al. (2001) analyzed the peel oils of 26 cultivars and four hybrids of bitter orange. The composition of the oils of seven country-specific cultivars can be seen in T-2. The range of composition for the other peel oils was as follows:

 $\begin{array}{l} \alpha \text{-thujene (0-0.1 percent)} \\ \alpha \text{-pinene (0.2-0.9 percent)} \\ \text{camphene (0-t)} \\ \beta \text{-pinene (t-4.0 percent)} \\ \text{sabinene (0.1-0.7 percent)} \\ \text{myrcene (1.3-1.8 percent)} \\ \alpha \text{-phellandrene (0-t)} \\ \text{limonene (84.2-95.0 percent)} \\ \beta \text{-phellandrene (0.2-0.4 percent)} \\ (Z)-\beta \text{-ocimene (0-t)} \\ \gamma \text{-terpinene (0-4.4 percent)} \\ (E)-\beta \text{-ocimene (0-0.4 percent)} \\ \text{p-cymene (0-0.5 percent)} \\ \text{terpinolene (0-0.2 percent)} \\ \text{octanal (0-0.1 percent)} \end{array}$ 

Compound 1 2 3 4 5 6	7
α-pinene 0.3 0.4 0.5 0.2 0.5 0.3	0.4
β-pinene 0.9 1.2 1.1 0.7 0.7 0.4	t
sabinene 0.2 0.3 0.3 0.2 0.2 0.1	0.4
myrcene 1.6 1.0 1.7 1.5 1.7 1.6	1.7
$\alpha$ -phellandrene t - t t t t	t
limonene 93.8 72.9 93.9 92.5 93.8 94.9	95.1
β-phellandrene 0.2 0.5 0.3 0.3 0.3 0.2	0.2
(Z)-β-ocimene t t	t
γ-terpinene - 0.5 - 0.1 t -	0.5
(E)-β-ocimene 0.3 0.3 0.3 0.3 0.2 0.3	0.3
p-cymene - 0.2 - t	-
terpinolene t	-
octanal 0.1 - 0.1 t 0.1 0.1	0.2
nonanal t t t t	t
<i>cis</i> -linalool oxide <sup>†</sup> t 0.4 t	-
octyl acetate 0.1 - t 0.1 0.1 0.1	0.1
decanal 0.1 - 0.1 0.1 0.1 0.1	0.1
linalool 0.2 5.2 0.1 0.3 0.1 0.1	0.1
linalyl acetate 0.8 5.0 0.4 0.1 0.4 0.6	-
β-caryophyllene 0.2 0.9 0.1 0.1 0.1 0.1	0.1
α-humulene t t -	t
neral t 0.1 t t	0.1
α-terpinyl acetate t	-
α-terpineol 0.1 0.9 0.1 0.5 t t	0.1
germacrene D 0.2 2.1 0.1 0.2 0.2 0.2	0.2
(Z)-α-bisabolene t	0.1
neryl acetate 0.1 0.2 - 0.1 t t	0.2
geranial 0.1 - t - 0.1 t	t
geranyl acetate 0.3 0.6 0.2 0.3 0.2 0.2	-
nerol t	-
geraniol - 0.3	-
(E)-nerolidol 0.2 3.2 0.2 0.3 0.2 0.1	-

<sup>†</sup>furanoid form; t = trace (< 0.1 percent); cultivars: 1. Florida, USA; 2. Spanish; 3. Tunisian; 4. Brazilian; 5. Algerian; 6. Moroccan; 7. Australian

nonanal (0-t) trans-limonene oxide (0-t) octyl acetate (0-0.2 percent) decanal (0-0.4 percent) linalool (t-0.8 percent) linalyl acetate (0-3.9 percent)  $\beta$ -caryophyllene (0-0.2 percent) terpinen-4-ol (0-t)  $\alpha$ -humulene (0-t) neral (0-0.1 percent)  $\alpha$ -terpinyl acetate (0-t)  $\alpha$ -terpineol (0-0.7 percent) germacrene D (0.1-0.8 percent) (Z)- $\alpha$ -bisabolene (0-t) neryl acetate (0-0.1 percent) geranial (0-0.1 percent) geranyl acetate (0.1-0.5 percent) nerol (0-t) geraniol (0-t) (E)-nerolidol (0-0.6 percent)

Mitiku et al. (2001) determined that the enantiomeric distribution of the four major monoterpene hydrocarbons found in cold-pressed bitter orange oil of Japanese origin was as follows:

- (4R)-(+)-limonene (99.55 percent): (4S)-(-)-limonene (0.45 percent) (1R,5R)-(+)-α-pinene (81.94 percent): (1S,5S)-(-)-α-pinene (18.06
- percent) (1R,5R)-(+)-sabinene (71.62 percent): (1S,5S)-(-)-sabinene (28.38 percent)
- (1R,5R)-(+)-β-pinene (80.61 percent): (1S,5S)-(-)-β-pinene (19.39 percent)

Gonzalez de et al. (2002) analyzed the oil produced by the hydrodistillation from Venezuelan bitter orange peel and found that it possessed the following major components:

 $\begin{array}{l} \alpha \text{-pinene} \; (0.64 \; \text{percent}) \\ \text{sabinene} \; (0.52 \; \text{percent}) \end{array}$ 

Comparative percentage composition of the bitter orange oil produced either by conventional or fast GC analysis

## **T-3**

Compound	Conventional GC analysis	Fast GC analysis
$\alpha$ -thujene	0.01	0.01
α-pinene	0.64	0.58
camphene	0.01	0.01
sabinene	0.29	0.27
β-pinene	1.01	0.93
myrcene	1.90	1.75
octanal & $lpha$ -phellandrene	0.20	0.19
δ-3-carene	t	t
$\alpha$ -terpinene	t	0.01
p-cymene & limonene <sup>†</sup>	93.26	93.58
(Z)-β-ocimene	0.01	t
(E)-β-ocimene	0.62	0.52
γ-terpinene	0.08	0.08
<i>cis-</i> sabinene hydrate	t	0.62
terpinolene	0.01	0.01
linalool	0.32	0.31
nonanal	0.03	0.03
<i>cis</i> -limonene oxide	0.01	-
<i>trans-</i> limonene oxide	t	0.02
citronellal	t	t
terpinen-4-ol	t	0.01
lpha-terpineol	0.03	0.04
decanal	0.13	0.13
octyl acetate	0.04	0.05
neral	0.03	0.05
geraniol	0.01	0.02
linalyl acetate	1.04	0.96
geranial	0.05	0.07
perillaldehyde	0.02	0.01
undecanal	0.01	0.01
nonyl acetate	0.01	0.01
$\delta$ -elemene	0.03	0.04
lpha-terpinyl acetate	t	0.01
citronellyl acetate	0.01	0.01
neryl acetate	0.03	0.03
geranyl acetate	0.12	0.12
dodecanal	0.02	0.02
β-caryophyllene	0.06	0.06
$trans-\alpha$ -bergamotene	0.02	0.02
(Z)-β-farnesene	0.02	0.01
germacrene D	0.12	0.11
bicyclogermacrene	0.01	0.01
β-bisabolene	0.01	0.01
tetradecanal	0.01	0.01
2,3-dimethyl-3-(4-methyl-		
3-pentenyl)-2-norbonanol	t	0.01
nootkatone	0.01	0.01

<sup>†</sup>major component of mixture; t = trace (< 0.01 percent)

β-pinene (3.25 percent) myrcene (1.74 percent) limonene (90.04 percent) (E)-β-ocimene (0.94 percent) linalool (2.87 percent)

Kirbaslar and Kirbaslar (2003) analyzed three samples of cold-pressed bitter orange oil produced from plants grown in Turkey. The average composition of these three oils was as follows:

α-pinene (0.42 percent) sabinene (0.17 percent) myrcene (1.80 percent)  $\beta$ -pinene (0.46 percent) octanal (0.11 percent)  $\alpha$ -phellandrene (0.05 percent)  $\delta$ -3-carene (0.01 percent) limonene (94.08 percent) (E)- $\beta$ -ocimene (0.13 percent)  $\gamma$ -terpinene (0.15 percent) linalool (0.39 percent) decanal (0.20 percent)  $\alpha$ -terpineol (0.10 percent) octyl acetate (0.05 percent) linalyl acetate (1.25 percent) geranial (0.10 percent) α-terpinyl acetate (0.03 percent) neryl acetate (0.01 percent) geranyl acetate (0.08 percent) dodecanal (0.11 percent) decyl acetate (0.01 percent)  $\beta$ -caryophyllene (0.10 percent) (E)-nerolidol (0.05 percent)

Trace amounts of  $\alpha$ -terpinene, (Z)- $\beta$ -ocimene, terpinolene, isopulegol, perillaldehyde and  $\alpha$ -humulene were also found in one or more of the Turkish bitter orange oils.

An oil of bitter orange produced in Sicily was subjected to analysis by conventional (ca. 46 min) and fast (9 min) GC by Mondello et al. (2003). The results of this comparative study can be seen in T-3.

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#### **Chamomile Oil and Extract**

The use of chamomile tea [ex Chamomilla recutita (L.) Rausch] as a carminative is not uncommon in Europe and, to a lesser extent, in the United States; however, there are certain allergenic risks associated with drinking this tissane. Ceska et al. (1992) used HPLC to examine the boiling water extracts of commercially available tea bags and a hydro-alcoholic extract of dried chamomile flowers. They determined that two coumarins, namely umbelliferone and herniarin, were found in high concentration in the teas. Furthermore, the authors determined that the antimicrobial properties associated with chamomile were caused by the phototoxity of the two coumarins. In addition, they postulated that herniarin (a strong photosensitizer) was capable of inducing phototoxic reactions in humans and probably was responsible for sporadic allergic reactions associated with chamomile. Finally, Ceska et al. determined that the level of herniarin that could elicit a photosensitization was 0.01-0.001 percent.

With the use of an octakis-(2,6-di-O-methyl-3-O-pentyl)- $\gamma$ -cyclodextrin chiral column, König et al. (1992) were able to chromatographically separate all four enantiomers of  $\alpha$ - and epi- $\alpha$ -bisabolol. They also showed that (-)- $\alpha$ -bisabolol was the main enantiomer in an authentic sample of chamomile oil.

König et al. (1994) also used chiral GC analysis to determine that the predominant isomer of bicyclogermacrene in chamomile oil was (+)-bicyclogermacrene.

An oil of chamomile produced from plants collected in the wild in Poland was analyzed by Gora et al. (1997) and the major compounds were found to be:

 $\begin{array}{l} \mbox{farmesene}^{\circ} (26.6 \mbox{ percent}) \\ \mbox{$\alpha$-bisabolol} (11.5 \mbox{ percent}) \\ \mbox{bisabolol} \mbox{oxide} \mbox{$A$} (6.6 \mbox{ percent}) \\ \mbox{bisabolol} \mbox{oxide} \mbox{$B$} (17.2 \mbox{ percent}) \\ \mbox{$\alpha$-bisabolol} \mbox{oxide} \mbox{$(8.4 \mbox{ percent})) } \end{array}$ 

° correct isomer not identified

Das et al. (2000) analyzed the composition of chamomile oil produced from two cultivars (Prashant and Vallary) grown on the Indo-Gangetic plains (Lucknow, India). They found that the Prashat cultivar was superior in oil quality (T-4) and the prospect for chamomile oil production in this area was very promising.

In a follow-up report, Kumar et al. (2001) compared the oils produced from the flowers (capitula), shoots and roots of the same two cultivars grown in Lucknow. The flower oil compositions were as

Comparative percentage composition of oils of two chamomile cultivars (Vallary and Prashant)

Compound	Vallary oil	Prashant oil
(Z)-3-hexenol	0.3	0.1
α-pinene	0.1	-
camphene	0.1	0.1
6-methyl-5-hepten-2-one	1.2	0.2
myrcene	0.2	0.1
(Z)-3-hexenyl acetate	0.1	0.2
p-cymene	0.1	0.1
limonene	0.1	< 0.1
(Z)-β-ocimene	0.2	0.2
(E)-β-ocimene	0.2	0.2
artemisia ketone	0.6	0.3
γ-terpinene	0.1	0.1
artemisia alcohol	0.6	0.3
linalool	0.2	0.1
camphor	1.1	0.1
isoborneol	0.2	< 0.1
borneol	0.4	0.1
terpinen-4-ol	0.1	<0.1
γ-terpineol <sup>†</sup>	0.1	0.1
nerol	0.1	0.1
pulegone	0.2	0.1
geraniol	0.1	< 0.1
β-bourbonene	0.3	0.1
β-caryophyllene	0.1	0.1
(E)-β-farnesene	8.1	4.9
murrolene*	0.2	0.3
germacrene D	1.1	0.8
α-patchoulene	0.6	0.3
(E,E)-α-farnesene	0.8	0.4
γ-cadinene	0.1	0.1
δ-cadinene	0.4	0.2
(E)-nerolidol	0.4	0.2
spathulenol	0.4	0.2
T-cadinol	0.5	0.5
T-muurolol	0.7	0.2
β-eudesmol	0.6	0.2
α-bisabolol oxide B	12.2	30.9
$\alpha$ -bisabolone oxide A	-	0.2
α-bisabolol	11.3	4.8
chamazulene	2.3	10.9 25 5
$\alpha$ -bisabolol oxide A	28.7	25.5
(Z)-en-yn-bicycloether (E)-en-yn-bicycloether	7.6	4.8
(E)-ell-yll-blcycloetlier	0.4	0.3

described by Das et al. while the shoot and root oil composition can be seen in T-5.

The identification of hexadec-11-yn-15diene has a hitherto not previously characterized constituent of chamomile shoot oil is highly unexpected; therefore, its identity is in question until it is unequivocally corroborated.

Margiatis et al. (2001) examined the composition of callus cultures of *C. recutita*. They found that the volatiles isolated from a culture were dissimilar to those found in the essential oil. The compounds characterized were as follows:

 $\begin{array}{l} gossonorol \left(4.3 \; percent\right) \\ cubenol \left(22.0 \; percent\right) \\ chamomillol \left(64.6 \; percent\right) \\ \alpha\text{-cadinol} \left(3.4 \; percent\right) \\ \alpha\text{-bisabolol} \left(1.4 \; percent\right) \\ l\text{-azulenethanyl acetate} \left(2.6 \; percent\right) \\ \alpha\text{-bisabolyl acetate} \left(1.7 \; percent\right) \end{array}$ 

Povh et al. (2001) compared the major component compositions of a steam distilled oil and an ethanolic solvent extract of dried chamomile flowers obtained from plants grown commercially in Mandirituba (Brazil). The comparative compositions Percentage composition of the shoot and root oils of the Vallary and Prashant cultivars of *Chamomilla recutita* 

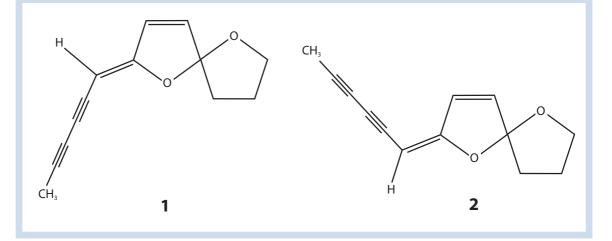
VPVP(Z)-3-hexenal3.11.10.8- $\alpha$ -pinene0.6-0.8-6-methyl-5-hepten-2-one1.60.8-myrcene0.3t0.80.2(Z)-3-hexenyl acetate0.2t0.4 $p$ -cymene0.2t-limonene0.2-t(Z)-3-hexenyl acetate0.2-t0.2-t0.1(Z)-β-ocimene0.2-t0.1(Z)-β-ocimene0.2-10.1-(Z)-β-ocimene0.2-0.2-tartemisia ketone0.3t $\gamma$ -terpinene0.80.2artemisia alcohol0.3t0.30.2-terpinolene0.6-linalool0.50.10.50.10.5borneol0.3-0.31 $\alpha$ -terpineol0.30.310.3-0.40.1nerol1.30.30.30.40.1nerol1.30.30.30.40.1nerol1.30.30.30.40.1nerol1.30.30.3-0.30.4-0.5 <t< th=""></t<>
$\alpha$ -pinene0.6-0.8-6-methyl-5-hepten-2-one1.60.8myrcene0.3t0.80.2(Z)-3-hexenyl acetate0.2t0.40.1p-cymene0.2tlimonene0.2-t0.1(Z)- $\beta$ -ocimene0.2-t0.1(E)- $\beta$ -ocimene0.2-t0.1artemisia ketone0.3t $\gamma$ -terpinene0.80.2artemisia alcohol0.3tterpinolene0.6-0.50.1linalool0.50.10.54.4camphor0.6t0.40.2borneol0.30.1nerol0.30.1nerol1.30.33.516.6pulegone0.40.2geraniol1.70.21.29.0geranyl acetate0.3-0.30.3 $\beta$ -bourbonene0.50.21.21.7 $\beta$ -elemene1.6-2.71.0isocaryophyllene0.80.60.30.4(E)- $\beta$ -farnesene5.437.72.718.4 $\alpha$ -humulene0.80.7
$\alpha$ -pinene0.6-0.8-6-methyl-5-hepten-2-one1.60.8myrcene0.3t0.80.2(Z)-3-hexenyl acetate0.2t0.40.1p-cymene0.2tlimonene0.2-t0.1(Z)- $\beta$ -ocimene0.2-t0.1(E)- $\beta$ -ocimene0.2-t0.1artemisia ketone0.3t $\gamma$ -terpinene0.80.2artemisia alcohol0.3tterpinolene0.6-0.50.1linalool0.50.10.54.4camphor0.6t0.40.2borneol0.30.1nerol0.30.1nerol1.30.33.516.6pulegone0.40.2geraniol1.70.21.29.0geranyl acetate0.3-0.30.3 $\beta$ -bourbonene0.50.21.21.7 $\beta$ -elemene1.6-2.71.0isocaryophyllene0.80.60.30.4(E)- $\beta$ -farnesene5.437.72.718.4 $\alpha$ -humulene0.80.7
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β-bourbonene0.50.21.21.7β-elemene1.6-2.71.0isocaryophyllene0.5-0.80.2β-caryophyllene0.80.60.30.4(E)-β-farnesene5.437.72.718.4α-humulene0.80.7
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β-caryophyllene0.80.60.30.4(E)-β-farnesene5.437.72.718.4 $\alpha$ -humulene0.80.7
(E)-β-farnesene5.437.72.718.4α-humulene0.80.7
α-humulene 0.8 0.7
germacrene D 0.5 14.6 0.7 0.4
$\alpha$ -patchoulene 0.5 0.9 - 0.3
$(E,E)-\alpha$ -farnesene 0.7 0.3
γ-cadinene 0.9 0.8
δ-cadinene 0.2 - 0.5 0.3
(E)-nerolidol 3.5 0.4 1.6 0.3
spathulenol 3.3 0.5 9.4 1.9
caryophyllene oxide 1.1 0.3 t 0.2
chamomillol <sup>a</sup> 1.1 0.5 1.5 4.4
T-cadinol 0.7 0.2 4.9 1.8
T-murrolol 0.4 - 6.4 0.5
$\beta$ -eudesmol 0.4 0.5 1.6 0.1
hexadec-11-yn-15-diene 9.7 3.1 6.2 0.3
$\alpha$ -bisabolol oxide B 0.7 0.2 0.6 0.2
$\alpha$ -bisabolol oxide b 0.7 0.2 0.0 0.2 $\alpha$ -bisabolol 0xide b 0.3 0.3 1.4 0.1
(E,E)-farnesol 0.5 0.5 0.9 0.6 0.1
$\alpha$ -bisabolol oxide A 0.5 0.9 0.0 0.1 $\alpha$ -bisabolol oxide A 0.5 0.3 1.4 0.4
, ,
7 1
isophytol 0.2 0.4 - 0.1 phytol t 0.4 0.9 0.1
phytol t 0.4 0.9 0.1

V= Vallary cultivar, P= Prashant cultivar; t = trace (< 0.1 percent); a = also known as muurol-4-en-7-ol

### Unusual constituents found in chamomile oil 1. (Z)-en-yn-bicycloether or *cis*-spiroether 2. (E)-en-yn-bicycloether or *trans*-spiroether



T-6



are shown in T-6. As can be seen from the results, there are major differences between the oil and the extract, although this should not be unexpected. In a followup paper Povh et al. (2001) examined the parameters for extraction of chamomile flowers using supercritical fluid CO<sub>2</sub>. They monitored the production of the main constituents such as  $\alpha$ -farmesene,  $\beta$ -farmesene,  $\gamma$ -cadinene,  $\alpha$ -bisabolol,  $\alpha$ -bisabolol oxide A,  $\alpha$ -bisabolol oxide B, chamazulene and cis- and trans-bicycloether (see F-1 for structures) as well as yield. They found that the maximum yield obtained was 4.33 percent using 40°C, 200 bar and a solvent flow rate of  $6.67 \times 10^{-5}$  kg/sec.

Hydrodistillation of dried flowers collected in the vicinity of Esphahan (Iran) yielded an oil in 0.65 percent. This oil was the subject of analysis by Pino et al. (2002). The components identified in this oil were as follows:

limonene (0.1 percent) 6-methyl-5-hepten-2-one (0.1 percent) (Z)-3-hexenol (0.1 percent) 3-octanol (0.1 percent) α-ylangene (0.1 percent) linalool (0.1 percent)  $\alpha$ -terpineol (0.1 percent)  $\beta$ -bisabolene (19.6 percent) (Z)-γ-bisabolene (0.5 percent)  $\alpha$ -muurolene (1.1 percent) bicyclogermacrene (0.1 percent) (E,E)- $\alpha$ -farmesene (3.1 percent)  $\delta$ -cadinene (0.1 percent)  $\gamma$ -cadinene (0.1 percent)  $\alpha$ -cadinene (0.2 percent) trans-carveol (0.1 percent) 2-phenethanol (0.2 percent) dendrolasin (0.5 percent)

### Comparative percentage composition of the main constituents of an oil and extract of chamomile

Compound	Oil	Extract
β-farnesene*	52.30	13.48
dihydrolinalyl acetate <sup>†</sup>	3.39	-
γ-cadinene	2.25	0.72
lpha-farnesene*	3.13	0.78
lpha-bisabolol oxide B	4.64	5.54
lpha-bisabolol	6.07	14.74
chamazulene	1.71	0.75
lpha-bisabolol oxide A	1.31	5.53
cis- and trans-bicycloether	3.20	29.36
butyl phthalate <sup>‡</sup>	15.10	-

\*correct isomer not identified; <sup>†</sup>incorrect identification as compound does not occur naturally; <sup>†</sup>incorrect characterization of a natural constituent as this is a synthetic plasticizer contaminant

 $\begin{array}{l} (E)\mbox{-nerolidol} \ (0.2\ percent) \\ (E)\mbox{-}\beta\mbox{-ionone} \ (0.1\ percent) \\ spathulenol \ (3.4\ percent) \\ (Z)\mbox{-}\beta\mbox{-santalol} \ (1.1\ percent) \\ \alpha\mbox{-bisabolol} \ oxide \ B \ (3.08\ percent) \\ \alpha\mbox{-muurolol} \ (0.3\ percent) \\ \alpha\mbox{-bisabolon} \ oxide \ A \ (13.6\ percent) \\ \alpha\mbox{-bisabolol} \ (1.7\ percent) \\ chamazulene \ (2.4\ percent) \\ \alpha\mbox{-bisabolol} \ oxide \ A \ (43.8\ percent) \end{array}$ 

It should be noted that the occurrence of such a high level of  $\beta$ -bisabolene is very unusual. Normally, (E)- $\beta$ -farnesene and (E,E)- $\alpha$ -farnesene are the major sequiterpene hydrocarbons found in chamomile oil, so the identification of  $\beta$ -bisabolene requires corroboration before it can be considered as being an unequivocally characterized constituent of chamomile oil.

The trace constituents present in amounts less than

Comparative percentage composition of different parts of Indian grown *Chamomilla* recutita

Compound	Whole	Disc	Ray	Leaf	Stem	Root
	capitula oil	florets oil	florets oil	oil	oil	oil
(E)-2-hexenol	10.	2.1	0.1	0.4	-	-
(Z)-3-hexenol	0.1	<0.1	0.1	-	-	-
tricyclene α-pinene	0.1 0.5	0.4 0.6	0.2 0.3	- 0.7	- 2.3	-
camphene	0.5	0.0	0.5	-	2.3 3.1	
6-methyl-5-hepten-2-one	5.4	3.4	0.5	0.8	-	-
myrcene	0.1	0.2	0.2	0.2	0.9	-
(Z)-3-hexenyl acetate	0.7	0.6	0.5	-	-	0.2
lpha-phellandrene	<0.1	-	0.1	0.2	0.6	-
p-cymene	0.3	0.3	0.9	1.0	0.4	-
limonene	0.2	0.2	0.3	0.3	23.9	-
(Z)-β-ocimene	0.2	0.2	-	-	-	-
(E)-β-ocimene	0.7	0.6	0.2	0.3	-	0.2
artemisia ketone	10.6 1.6	10.8 1.3	0.8 0.2	1.8 0.5	-	-
γ-terpinene artemisia alcohol	1.0	1.3 1.5	0.2	0.5 -	0.8	-
linalool	0.2	0.1	2.4	- 1.1	- 0.6	0.1
camphor	0.2	1.3	0.3	0.5	1.5	-
isoborneol	-	0.2	0.5	4.4	1.6	-
borneol	1.2	1.2	-	-	-	0.1
terpinen-4-ol	0.1	0.2	0.6	0.5	0.8	-
α-terpineol	0.2	0.1	0.5	1.5	2.7	-
nerol	-	0.1	0.2	3.7	-	-
pulegone	0.5	0.3	-	-	1.3	-
geraniol	-	0.1	1.9	1.5	-	0.1
geranyl acetate	<0.1	0.1	1.0	1.2	2.0	-
α-copaene	0.2	0.2	-	-	-	0.1
β-bourbonene	0.1	0.1	0.7	0.4	0.4	1.8
β-elemene	-	-	-	0.3	-	5.1
isocaryophyllene β-caryophyllene	- 0.1	- 0.2	-	- 0.3	-	0.4 1.8
(E)-β-farnesene	7.4	2.9	- 1.1	0.3 1.8	- 2.9	44.4
α-humulene	7. <del>4</del>	-	-	-	-	2.8
γ-muurolene	-	0.1	-	_	_	0.8
germacrene D	1.2	0.1	-	1.4	-	-
$\alpha$ -patchoulene	0.1	0.1	0.7	2.3	-	0.1
(E,E)-α-farnesene	0.1	0.1	0.4	9.3	-	1.3
γ-cadinene	0.1	0.1	0.5	0.2	-	-
δ-cadinene	0.2	0.1	0.3	-	-	0.8
(E)-nerolidol	0.1	0.2	0.3	0.5	1.2	0.1
spathulenol	-	0.2	0.4	0.5	0.5	0.2
caryophyllene oxide	0.2	0.2	0.3	0.3	-	0.1
chamomillol T-cadinol	- 1.9	- 0.9	- 1.2	1.1 0.6	- 1.7	5.8
hexadec-11-yn-13,15-diene	-	- 0.9	1.Z -	-	-	1.0 2.2
β-eudesmol	- 1.3	1.0	1.6	- 1.0	-	0.3
$\alpha$ -bisabolol oxide B	12.0	13.6	8.6	3.9	2.3	0.6
$\alpha$ -bisabolone oxide	0.6	0.3	3.1	2.1	2.0	-
α-bisabolol	8.0	16.8	0.2	1.8	0.5	-
(E,E)-farnesol	-	-	-	0.3	-	0.2
chamazulene	2.9	1.9	1.4	-	-	-
lpha-bisabolone oxide A	17.4	20.4	8.9	4.0	2.2	0.5
(E,E)-farnesyl acetate	-	-	-	0.2	-	0.2
a(E)-en-yn-bicycloether	0.2	0.1	0.5	1.3	2.6	0.4
isophytol	0.2	0.2	2.7	0.6	1.1	0.4
phytol	-	-	-	2.6	-	0.4

**T-7** 

T-8

Compound	Egyptian oil	Spanish oil
$\alpha$ -pinene	0.02	0.06
sabinene	0.06	0.08
myrcene	0.05	0.11
p-cymene	0.06	0.23
limonene	0.02	0.06
(Z)-β-ocimene	0.06	0.08
(E)-β-ocimene	0.34	0.43
γ-terpinene	0.15	0.26
artemisia ketone	0.47	0.30
artemisia alcohol	0.12	0.04
terpinolene	0.01	0.06
decanoic acid	0.52	-
$\alpha$ -copaene	0.05	0.06
β-elemene	0.10	0.18
isocomene <sup>†</sup>	0.09	0.19
β-caryophyllene	0.13	0.23
(E)-β-farnesene	23.19	30.06
germacrene D	3.01	5.09
(Z,E)-α-farnesene	0.32	0.74
bicyclogermacrene	2.06	1.08
(E,E)-α-farnesene	1.13	4.75
δ-cadinene	0.40	0.20
spathulenol	0.36	0.35
lpha-bisabolol oxide B	4.89	3.92
lpha-bisabolol	2.49	27.79
lpha-bisabolone oxide	5.23	0.45
chamazulene	3.38	5.75
lpha-bisabolol oxide	37.69	2.06
(Z)-spiroether	5.58	10.35
(E)-spiroether	0.67	0.50
spiroether homolog*	0.27	0.53

\*correct isomer not identified

0.1 percent in this same oil were  $\alpha$ -pinene, ethyl 2-methylbutyrate, ethyl isovalerate, hexanal,  $\beta$ -pinene, sabinene, myrcene, 2-heptanone, (E)-2-hexenal, ethyl hexanoate, 1,8-cineole,  $\gamma$ -terpinene, 2,5-dihydro-2,5-diethylfuran, p-cymene, octanal, 2,2,6-trimethylcyclohexanone, 5-methyl-2hexenal, artemisia ketone, 2,6-dimethyl-5heptenal, (Z)-3-hexenol, 3-octanol, yomogi alcohol, nonanal, 2-octanol, 3-octen-2-one,  $\alpha$ -thujone, 1-octen-3-ol, *cis*-linalool oxide (furanoid), 6-methyl-5-hepten-2-ol, furfural, *trans*-linalool oxide (furanoid),  $\delta$ -elemene, artemisia alcohol, 3-nonen-2-one, (E,E)-3,5-octadien-2-one, benzaldehyde, linalool, camphor, terpinen-1-ol, pinocarvone, methyl decanoate,  $\beta$ -elemene,  $\beta$ -caryophyllene, ethyl decanoate, terpinen-4-ol, (Z)- $\beta$ farnesene, ar-curcumene, cadina-1,4-diene, trans-calamenene, geraniol, benzyl alcohol, safrole,  $\alpha$ -calacorene, methylguaiacol and

ledol. The authors noted that the identities of 2,5-dihydro-2,5-dimethylfuran, 2,2,6-trimethylcyclohexanone, 2,6-dimethyl-5-heptenal, 3-nonen-2-one and (E,E)-3,5octadien-2-one were tentative based on mass spectral data only.

Taviani et al. (2002) examined the major components found in the flower head oils of 11 wild populations, one Italian cultivar (Synl) and one Slovak cultivar (Bona) and found that they could be grouped into four previously defined chemotypes and one new one. They found that the  $\alpha$ -bisabolol and chamazulene contents varied from 4.37-51.82 percent and 3.52-20.57 percent, respectively. In addition, four of the wild types were found to possess  $\alpha$ -bisabolol and chamazulene contents similar or higher than the widely cultivated Bona cultivar. Furthermore, the authors found a significant correlation between flower head weight and the  $\alpha$ -bisabolol content of the oil.

Culea et al. (2002) reported that the compounds identified in chamomile oil of different origins in Romania were limonene, carvone, butybutyryl, lactate, a bisabolone oxide isomer, herniarin,  $\alpha$ -terpinyl acetate, palmitic acid, ethyl palmitate, linoleic acid, ethyl linoleate, ethyl linolenate, achillin, eicosane and dioctyl phthalate. It sould be noted, however, that (a) butylbutyryl lactate and dioctyl phthalate are misidentifications as neither compound occurs naturally and (b) as the compounds listed above are supposed to be in elution order from a nonpolar column, their identifications are also questionable.

The oils produced from different parts of *C. recutita* plants grown in Lucknow (India) were analyzed by Das et al. (2002). The plants which were harvested in early winter were separated into whole capitula, disc florets, ray florets, leaves, stems and roots and each was separately hydrodistilled to produce oils in yields of 0.40 percent, 0.45 percent, 0.12 percent, 0.13 percent, 0.03 percent and 0.05 percent, respectively. The composition of each of these oils is presented in T-7.

Using a combination of capillary GC and <sup>13</sup>C-NMR, Kubeczka and Formacek (2002) analyzed two oils of *C. recutita* of Egyptian and Spanish origin. A summary of their compositions can be seen in T-8.

part of an antimicrobial screening program of oils produced in Nepal, Yonzon et al. (2005) determined that a Nepalese sample of chamomile oil possessed the following major constituents:

 $\begin{array}{l} (E)\mbox{-}\beta\mbox{-}farnesene~(36.9\mbox{-}percent)\\ germacrene D~(5.1\mbox{-}percent)\\ bicyclogermacrene~(4.1\mbox{-}percent)\\ a\mbox{-}farnesene~(9.7\mbox{-}percent)\\ bisabolol oxide B~(5.4\mbox{-}percent)\\ bisabolone oxide A~(4.4\mbox{-}percent)\\ chamazulene~(2.4\mbox{-}percent)\\ bisabolol oxide A~(20.9\mbox{-}percent)\\ \end{array}$ 

° correct isomer not identified

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1.	Publication	title:	Perfumer	&	Flavorist
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- 2. Publication number: 0272-2666
- 3. Filing date: September
- 4. Issue frequency: Bi-monthly, except June and October
- 5. Number of issues published annually: 8
- 6. Annual subscription price: \$135.00
- Complete mailing address of known office of publication: 362 South Schmale Road, Carol Stream, DuPage County, IL 60188-2787. Contact Person: Laura Wieringa. Telephone: 630-653-2155
- 8. Complete mailing address of headquarters or general business offices of publishers: Same
- Full names and complete mailing addresses of publisher, editor and managing editor: Publisher: Matt Gronlund, 362 South Schmale Road, Carol Stream, DuPage County, IL 60188-2787; Managing Editor: Jeb Gleason-Allured, 362 South Schmale Road, Carol Stream, DuPage County, IL 60188-2787
- 10. Owner: Allured Publishing Corporation, 362 South Schmale Road, Carol Stream, DuPage County, IL 60188-2787; Stanley E. Allured, Nancy Allured, Janet Ludwig
- 11. Known bondholders, mortgages and other security holders owning or holding 1 percent or more of total amount of bonds, mortgages or other securities: None
- 12. Tax Status (For completion by nonprofit organizations authorized to mail at special rates.) The purpose, function and nonprofit status of this organization and the exempt status for federal income tax purposes: Not applicable
- 13. Publication title: Perfumer & Flavorist
- 14. Issue date for circulation data below: September 2005

15.Extent and nature of circulation Average number of copies each issue Actual number of single issues during preceding 12 months published nearest to filing date 2,025 2,300 Total number of copies (net press run): Α. B. Paid and/or requested circulation. 1. Paid or requested mail subscriptions or form 3541(include advertisers' proof copies/exchange copies): 636 676 Paid in-country subscriptions or form 3541 2. 0 0 3. Sales through dealers and carriers, street vendors, counter sales, and other non-USPS paid distribution 672 689 4. Other classes mailed through the USPS 0 0 С. Total paid and/or requested circulation: 1,308 1,365 Free distribution by mail: D. 0 0 Free distribution outside the mail (carriers or other means): E. 244 50F Total free distribution 244 50 Total distribution: 1,552 1,415 G. Η. Copies not distributed: 473 885 Total: 2,025 2,300 I. Percent paid and/or requested circulation 84.28% 96.47%

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