

# **Progress in Essential Oils**

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#### **Citron Oil**

Citron (*Citrus medica* L.) is the name of a citrus fruit that is larger than lemon, which varies in shape from oval to pearshaped. It has a very thick skin and a somewhat dry pulp containing a small quantity of acidic pulp. Currently, citron is grown commercially along the Tyrrhenian coast from Diamante to Tortora (Calabria, Ital). A limited amount of citron oil is produced commercially in Italy using either the rasping or abrasive whole fruit process.

Capello et al. (1982) used GC-FID to determine that two commercially produced peel oils of citron possessed the following composition:

α-pinene (2.8-3.2%) camphene (0.1%)β-pinene (2.2–2.5%) myrcene (1.5-1.6%)  $\alpha$ -phellandrene (0.1–0.2%)  $\alpha$ -terpinene (t-0.8%) limonene (54.9-56.2%) octanal (t-0.1%) p-cymene (1.9-2.4%) terpinolene (0.3-0.6%) octyl acetate (0.1%) citronellal (0.1%) linalool (0.1-0.2%) decanal (0.1%) terpinen-4-ol (0.1%) neral (1.3-1.6%)  $\alpha$ -terpineol (0.4–0.5%) geranial (2.1-2.7%)  $\beta$ -caryophyllene (0.1–0.6%)  $\alpha$ -bergamotene° (0.3–0.6%) neryl acetate (0.4-0.5%) geranyl acetate (0.6-1.1%) bisabolene<sup>°</sup> (0.5–0.8%) t=trace (<0.05%)

°correct isomer not identified

Trace amounts (<0.05%) of  $\alpha$ -thujene, heptanal,  $\delta$ -3-carene and nonanal were also found in these oils.

Two cold-pressed oils of cedro (the Italian name for citron) were produced

commercially from the 'Diamante' and 'Rugosa' cultivars. A summary of the results of the two analyses performed by Cotroneo et al. (1986) can be seen in **T-1**.

Huet et al. (1986) reported that an Italian oil of citron (known as cedrat in French) was reported to contain the following constituents:

 $\alpha$ -thujene (0.1%)  $\alpha$ -pinene (2.3%) camphene (0.1%)  $\beta$ -pinene (11.5%) sabinene (1.8%) myrcene (1.5%) $\alpha$ -phellandrene (0.2%) limonene (60.6%) p-cymene (0.5%) terpinolene (0.4%) γ-terpinene (7.4%) nonanal (0.1%)octyl acetate (<0.1%) citronellol (0.1%) linalool (0.2%) decanal (0.1%) terpinen-4-ol (0.1%) neral (1.0%)  $\alpha$ -terpineol (0.3%) geranial (1.4%)  $\beta$ -caryophyllene (0.4%)

Compound	'Diamante' oil	'Rugosa' oil
$\alpha$ -thujene	1.0	0.5
α-pinene	2.3	2.3
sabinene	0.3	2.4
β-pinene	2.0	14.8
myrcene	1.5	1.4
lpha-phellandrene	0.1	t
lpha-terpinene	0.4	0.2
p-cymene	0.1	t
limonene	59.4	59.5
γ-terpinene	24.0	11.4
terpinolene	0.8	0.5
linalool	0.1	0.2
nonanal	0.1	0.2
citronellal	0.1	t
terpinen-4-ol	0.1	t
lpha-terpineol	0.3	0.4
decanal	t	0.1
nerol	0.1	0.1
neral	1.0	1.0
geraniol	0.1	0.2
geranial	1.7	1.6
neryl acetate	0.2	0.2
geranyl acetate	0.2	0.2
β-caryophyllene	0.1	0.3
bisabolene <sup>*</sup>	0.5	0.3
t=trace (<0.05%); *correct isomer not identified	1	

### T-1. Comparative percentage composition of the oils of the 'Diamante' and 'Rugosa' cultivars of citron

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 $\begin{array}{l} \alpha\text{-bergamotene}^{\circ}\left(0.5\%\right)\\ neryl \ acetate \ (1.1\%)\\ geranyl \ acetate \ (0.7\%)\\ bisabolene^{\circ}\left(1.1\%\right) \end{array}$ 

° correct isomer not identified

Huet also compared two citron oils produced in Corsica. The results of these analyses are shown in **T-2**. Oil No. 2 was produced from the 'Fox' cultivar of *C. medica*.

Dugo et al. (1986) reported that they examined 441 and 448 samples of citron that were produced from November 1982 to November 1983 and November 1983 to November 1984, respectively. Approximately 50% of the oils were produced by either Pelatrice or Sfumatrice; the main production months were November to January. The production zones were Siracusa, Acireale, Capo d'Orlando and Messina in Sicily, and the types produced were Verdello, Primofiore, Bianchetto and Invernale. Dugo found that the oils produce by Pelatrice or Sfumatrice did not vary much in their mono- and sesquiterpene contents, but did vary to some extent in their oxygenated constituent contents with Pelatrice oils varying more than the Sfumatrice oils.

Dugo et al. (1988) reported the results of their analysis of the 'Diamante' cultivar of *C. medica*. The constituents that were characterized in the oil were:

 $\alpha$ -thujene (0.5%)  $\alpha$ -pinene (2.3%) camphene (0.1%) sabinene (2.1%)  $\beta$ -pinene (13.6%) myrcene (1.5%)octanal +  $\alpha$ -phellandrene (0.2%)  $\alpha$ -terpinene (0.2%) limonene (61.5%) (E)- $\beta$ -ocimene (0.1%)  $\gamma$ -terpinene (12.4%) cis-sabinene hydrate (0.1%)terpinolene (0.5%) *trans*-sabinene hydrate (0.1%)linalool (0.1%) nonanal (0.1%) $\alpha$ -terpineol (0.3%) decanal (0.1%) neral (0.7%) geraniol (0.1%) geranial (1.1%) neryl acetate (0.2%) geranyl acetate (0.2%)  $\beta$ -caryophyllene (0.2%)  $\alpha$ -bergamotene° (0.3%)  $\beta$ -cubebene (0.6%)

valencene (0.1%) bisabolene<sup>°</sup> (0.5%)

° correct isomer not identified

Trace amounts (<0.1%) of p-cymene, (Z)- $\beta$ -ocimene, citronellal, terpinen-4-ol, undecanal, citronellyl acetate and  $\alpha$ -humulene were also found in this oil. In addition, the authors noted that for *C*. *medica* peel oil to be genuine it should possess the following monoterpene hydrocarbon ratios:

 $\begin{array}{l} \alpha \text{-thujene/sabinene} \ (0.26\%) \\ \alpha \text{-thujene/}\beta \text{-pinene} \ (0.04\%) \\ \alpha \text{-pinene/}\beta \text{-pinene} \ (0.17\%) \\ \beta \text{-pinene/}\gamma \text{-terpinene} \ (1.09\%) \end{array}$ 

Poiana et al. (1998) collected fresh fruits of the 'Diamante' cultivar of C. medica from St. Maria del Cedro (Cosenza, Calabria, Italy) and peeled. The peels were chopped using a domestic mincer (probably a blender) and an aliquot was subjected to hydrodistillation. Further aliquots of the minced peels were extracted using a Soxhlet apparatus with pentane as the solvent and supercritical fluid CO<sub>2</sub> using an extractor with an internal diameter of 0.6 m<sup>3</sup>. Using a flow rate of 3.6 kg/hr of  $CO_2$  the authors used a 4 hr extraction pressures at 8 MPa and 25 MPa at 20°-25°C and 40°C, respectively. The oil and extracts were analyzed using GC-FID and GC/MS, the results of which can be seen in T-3.

A peel oil of *C. medica* fruit that were collected from Gorakhpur (Uttar Pradesh, India) was produced by hydrodistillation by Singh et al. (1999). Analysis of the oil using HPLC, GC-FID and GC/MS revealed that it possessed the following composition:

1-methyl-3-isopropylcyclopentane<sup>c</sup> (0.4%)  $\alpha$ -thujene (0.3%) α-pinene (0.8%) sabinene (0.2%)  $\beta$ -pinene (0.9%) octanal (0.3%) myrcene (0.6%)p-cymene (6.5%) 1,8-cineole (0.3%) limonene (32.0%) (Z)- $\beta$ -ocimene (0.2%)  $melonal^{a}\left(0.4\%\right)$ (E)- $\beta$ -ocimene (0.3%)  $\gamma$ -terpinene (2.0%) octanol (0.2%) nonanal (0.2%) linalool (1.2%)

#### T-2. Comparative percentage composition of the main constituents of two citron oils of Corsican origin

Compound	Oil 1	0il 2
$\alpha$ -pinene	1.1	1.2
β-pinene	0.7	1.0
sabinene	-	0.2
myrcene	0.9	0.4
$\alpha$ -terpinene	-	4.2
limonene	51.2	58.7
γ-terpinene	12.9	7.1
linalool	1.1	-
neral	7.4	1.6
geranial	13.3	1.9
bisabolene*	3.2	-
*correct isomer not identif	ied	

cis-limonene oxide (0.6%) trans-limonene oxide (0.3%) isopulegol (0.7%) citronellal (27.5%) iso(iso)pulegol (0.3%) terpinen-4-ol (0.7%)  $\alpha$ -terpineol (1.2%) decanal (0.4%) citronellol (13.0%) geraniol (0.6%) 2,6-dimethyl-5-heptenol (0.5%) geranial (2.3%) citronellic acid (1.8%) p-menthane-2,8-diol<sup>a</sup> (0.9%) citronellyl acetate (0.4%)  $\beta$ -caryophyllene (0.2%) *trans*- $\alpha$ -bergamotene (0.4%)  $\beta$ -bisabolene (0.5%)

<sup>c</sup>fruit or equipment contaminant <sup>a</sup>questionable identification

The fact that the oil was reported to be rich in limonene, citronellal and citronellol leads this reviewer to the conclusion that the fruit used to isolate the oil was not *C. medica*.

The cold-pressed peel oils of the 'Diamante' and Corsican cultivars of C. medica were analyzed using GC-FID, GC/MS and <sup>13</sup>C-NMR by Lota et al. (1999). The comparative oil compositions are presented in T-4. In addition, trace amounts of an allo-ocimene isomer, cis-limonene oxide and citronellyl acetate were characterized in the Corsican oil while citronelly lacetate and  $\alpha$ -bisabolol were characterized in the 'Diamante' oil. Furthermore, it is to note that the 'Diamante' oil produced from fruit produced in Corsica possessed a somewhat different composition to those produced in Italy.

Vekiari et al. (2004) produced an oil from the peel of *C. medica* cv. 'Diamante' fruit grown in Crete (Greece) by hydrodistillation. The constituents identified in the oil by GC-FID and GC/MS were as follows:

 $\alpha$ -pinene (0.2%)  $\beta$ -pinene (1.3%) myrcene (1.3%) δ-3-carene (0.1%) limonene (24.9%) (Z)- $\beta$ -ocimene (0.4%) (E)- $\beta$ -ocimene (0.7%)  $\gamma$ -terpinene (0.3%) terpinolene (<0.1%) linalool (0.5%) nonanal (0.2%) terpinen-4-ol (0.2%)  $\alpha$ -terpineol (0.2%) decanal (0.1%) nerol + citronellol (2.1%) neral (8.1%) geraniol (2.2%) geranial (13.3%) citronellyl acetate (0.1%) $\beta$ -caryophyllene (0.4%) *trans*- $\alpha$ -bergamotene (0.3%)  $\alpha$ -humulene (0.5%) undecanal (0.2%)neryl acetate (1.1%) geranyl acetate (0.6%) farnesene° (0.1%)dodecanal (0.1%) neryl propionate (<0.1%) geranyl propionate (0.1%)

This oil shows some similarity to the 'Diamante' oil of Corsican origin.

Peels of fresh fruits of *C. medica* that were collected from an experimental plantation of the Citrus Research Station (BARI, Jaintapur, Sylhet, Bangladesh) were chopped in a blender and subjected to hydrodistillation for 4 hr by Bhuiyan et al. (2009). The oil was analyzed by GC/MS only, resulting in the authors using the computer to characterize the oil constituents. Unfortunately, the authors knew very little about citron peel oil composition because the results presented were so full of errors, the will not be included in this review.

Gabriele et al. (2009) studied the effect of fruit maturity on the composition of the peel oil of the 'Diamante' cultivar of *C. medica* produced by different non-distillation isolation methods. They chose three maturity states such as small-sized green fruit, large-sized green fruit and mature yellow fruit. Oils produced by different T-3. Comparative percentage composition of the oils and extracts of *Citrus medica* cv. 'Diamante'

Compound	Oil	Pentane extract	SF CO <sub>2</sub> extract 1	SF CO <sub>2</sub> extract 2
$\alpha$ -thujene	0.8-0.9	0.5	0.2	0.2
α-pinene	1.9-2.1	1.3	0.6	0.8
sabinene	0.2-0.3	0.2	0.1	0.1
β-pinene	1.9–2.0	1.4	0.9	1.0
myrcene	1.6	1.5	1.1	0.8
$\alpha$ -phellandrene	0.1	0.1	0.1	0.1
$\alpha$ -terpinene	0.5	0.4	0.2	0.2
p-cymene	0.4–0.5	0.5	0.6	0.6
limonene	59.7–60.8	60.1	53.2	34.6
(Z)-β-ocimene	1.4–1.5	1.5	1.4	0.6
(E)-β-ocimene	1.9-2.1	2.1	2.0	0.6
γ-terpinene	23.4–24.3	24.6	22.1	14.7
terpinolene	1.0-1.1	1.0	1.2	0.6
linalool	0.1	0.1	0.3	0.3
nonanal	0.1	t	0.1	0.2
citronellal	0.1–0.2	0.1	0.2	0.1
terpinen-4-ol	0.1	t	0.2	0.2
$\alpha$ -terpineol	0.1-0.2	t	0.3	0.5
decanal	t-0.1	t	0.1	0.1
<i>trans</i> -carveol	t	t	0.1	t
nerol + citronellol	0.1	0.1	0.3	0.6
neral	0.7–0.8	0.1	1.1	0.4
geraniol	0.1	0.3	0.7	0.6
geranial	1.0	0.2	2.2	1.1
undecanal	t	t	0.2	0.2
$\delta$ -elemene	t	0.1	0.1	0.4
citronellyl acetate	t	0.1	0.2	0.6
neryl acetate	0.2	0.3	1.0	1.8
geranyl acetate	0.2	0.2	0.8	1.6
β-elemene	t	t	0.1	0.1
dodecanal	t	t	0.1	0.2
β-caryophyllene	0.1	0.1	0.5	0.6
<i>trans</i> -α-bergamotene	0.2	0.4	1.7	3.2
(Z)-β-farnesene	t	0.1	0.2	0.4
$\alpha$ -humulene	t	t	0.1	0.2
γ-cadinene	t	t	0.1	0.3
(E,E)-α-farnesene	t	t	0.1	0.3
β-bisabolene	0.2-0.3	0.6	2.6	6.2
germacrene B	t	t	0.1	0.3
(E)-nerolidol	t-0.1	0.1	0.3	1.2
spathulenol	t	t	0.1	0.2
tetradecanal	-	t	0.1	0.2
lpha-bisabolol	t	t	0.2	0.6
pentadecanal	-	t	0.1	0.2
(Z,E)-farnesol <sup>a</sup>	-	t	0.1	0.3
farnesal*	-	0.1	`0.2	0.5
hexadecanal	-	t	0.1	0.2
dimethoxycoumarin isomers*	t	0.5	0.9	10.4
citroptene	-	0.4	0.5	1.2
phytol	-	0.7	0.3	8.9

t-trace (<0.1%); \*correct isomer not identified; \*tentative identification; Extract 1=CO $_2$  at 8 MPa; Extract 2=CO $_2$  at 25 MPa

isolation methods did not vary greatly in compositions that were determined by GC-FID and GC/MS. A summary of these results can be seen in T-5. Trace amounts of camphene, decane, octanal,  $\delta$ -3-carene, *cis*-limonene oxide, trans-limonene oxide, camphor, borneol, p-cymen-8-ol, octyl acetate, perillaldehyde, carvacrol, citronellyl acetate,  $\beta$ -elemene, dodecanal,  $cis-\alpha$ -bergamotene, (Z)- $\beta$ -farnesene, (E)- $\beta$ -farnesene, (Z)- $\beta$ -santalene, bicyclogermacrene, (Z)- $\gamma$ -bisabolene, (E)- $\gamma$ -bisabolene, (E)- $\alpha$ -bisabolene, tetradecanal, camphenol,  $\alpha$ -bisabolol, nootkatone and hexadecanal were also found in these oils. The authors also reported the identification of trace amounts of dihydrolinalyl acetate. To this reviewer's knowledge, this is not a naturally occurring component of any essential oil.

Gabriele et al. also used chiral GC fitted with a diethyl-t-butyl-silyl- $\beta$ -cyclodextrin column to examine the chirality of five constituents of the oils. The results of this chiral study revealed the following composite results:

- $\label{eq:11} \begin{array}{l} (1R,\!5R)\mbox{-}(+)\mbox{-}\beta\mbox{-}pinene \ (90.6\mbox{-}94.6\mbox{-})\mbox{:}(1S,\!5S)\mbox{-}(-)\mbox{-}\beta\mbox{-}pinene \ (5.4\mbox{-}9.4\mbox{-}\%) \end{array}$
- (1R,5R)-(+)-sabinene (63.5–67.9%):(1S,5S)-(-)-sabinene (32.1–36.5%)
- (4R)-(+)-limonene (97.6–97.9%):(4S)-(-)-limonene (2.1–2.4%)
- (3S)-(+)-linalool (52.7–59.0%):(3R)-(-)-linalool (41.0–47.3%)
- (1R)-(+)- $\alpha$ -terpineol (22.6–24.8%):(1S)-(-)- $\alpha$ -terpineol (75.2–77.4%)

Using reversed-phase HPLC, the authors also examined the oxygen heterocyclic components in the various oils. The results of this analysis are presented in **T-6**. It should be noted that the highest

### T-4. Comparative percentage composition of the C.P. peel oils of the 'Diamante' and Corsican cultivars of *Citrus medica*

Compound	'Diamante' oil	Corsican oil
$\alpha$ -thujene	-	0.6
α-pinene	0.1	1.7
β-pinene	0.2	1.7
sabinene	0.1	0.3
myrcene	1.3	1.4
lpha-phellandrene	t	0.1
lpha-terpinene	-	0.5
limonene	70.4	51.9
(Z)-β-ocimene	0.3	0.9
γ-terpinene	t	26.2
(E)-β-ocimene	0.5	1.2
p-cymene	-	0.2
terpinolene	t	1.1
<i>trans</i> -sabinene hydrate	-	0.1
citronellal	0.1	0.1
linalool	0.3	0.3
<i>trans</i> -α-bergamotene	0.4	0.4
β-caryophyllene	0.2	0.2
terpinen-4-ol	0.1	t
neral	7.6	2.8
lpha-terpineol	0.1	0.3
germacrene D	t	0.1
β-bisabolene	0.7	0.6
neryl acetate	0.7	0.7
geranial	14.4	5.3
$lpha$ -bisabolene $^*$	0.1	-
geranyl acetate	0.9	0.8
nerol	0.2	-
geraniol	0.2	-
*correct isomer not identified; t=trace (<0.1%)		

levels of each of the oxygenated heterocyclic compounds were found in the oils obtained by manual abrasion isolation.

The composition of an oil produced by hydrodistillation was compared with an oil produced by cold-pressing and a supercritical fluid  $CO_2$  extract of the 'Diamante' cultivar of *C. medica* by Menichini et al. (2011) using GC/MS only. The results of this comparative study of the oils are reported in **T-7**. Trace amounts (<0.1%) of p-cymene,  $\gamma$ -cadinene, tetradecanal and docosane were found in the oils.

The supercritical fluid  $CO_2$  extract of the citron fruit that were peeled under nitrogen atmosphere was extracted with ca 2 g/min for 6 hr at 40°C and 100 bar was found to contain:

limonene (<0.1%) 2,3-dihydro-3,5-dihydroxy-6-methyl-(4H) pyran-4-one (1.1%) 2,3-dihydrobenzofuran (2.9%) (E)-β-farnesene (0.2% citropten (84.5%)

The authors also found that the cold-pressed oil possessed selective inhibitory activity against acetylcholinesterase, while the hydrodistilled oil exerted the highest inhibitory activity against butyrylcholinesterase, acetylcholinesterase and butyrylcholinesterase are the principal enzymes involved with the hydroxysis of acetylcholine. As a result, cholinesterase inhibitors are important because they are being developed for the symptomatic treatment of Alzheimer's disease and several other neurological disorders.

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### Pimento Leaf and Berry Oil

*Pimento dioica* (L.) Merill, the fruit of which is known as allspice, is a member of the Myrtaceae family, which is indigenous to Jamaica. It can be found widely distributed throughout the island because of natural propagation by birds that eat the fruit pulp and discard the seed. Allspice is the dried berrylike fruit of *P. dioica*, which is the main item of commerce from the tree. However, oils produced commercially from leaves and malformed fruit are the familiar pimento leaf and pimento berry oils.

As the pimento tree is dioecious, each commercial plantation (grove) requires a minimum number of non-fruiting (male) trees to ensure fruit formation from effective pollination. It is difficult to determine the sex of a seedling until it flowers-which can take five to 10 years—and the flower structure can be examined. It is believed by some plantation owners that the sex of a seedling our young tree can be determined from the odor of its leaves. To address this question, Minott and Brown (2007) compared the compositions of the leaf oils of fruiting (female), non-fruiting (male) and young trees of unknown fruiting capability (sex undeclared). For analysis they used GC-FID and retention times to T-5. The effect of fruit maturity on the comparative percentage composition of peel oils of the 'Diamante' cultivar of *Citrus medica* 

Compound	Small green fruit oil	Large green fruit oil	Mature yellow fruit oil
$\alpha$ -thujene	0.3–0.4	0.6	0.4-0.6
α-pinene	0.7–1.1	1.4–1.5	1.0–1.5
sabinene	0.1-0.2	0.2	0.2
β-pinene	0.9-1.2	1.4	1.1–1.4
myrcene	1.1–1.3	1.4–1.5	1.3–1.5
lpha-phellandrene	t–0.1	0.1	t–0.1
lpha-terpinene	0.4	0.4	0.4
limonene	51.3-58.0	56.5-60.6	56.6-60.2
(Z)-β-ocimene	0.8-0.9	1.1–1.2	1.1–1.2
(E)-β-ocimene	1.1–1.3	1.5–1.7	1.6–1.7
γ-terpinene	23.2-24.4	21.2-22.8	20.9-22.5
<i>cis</i> -sabinene hydrate	0.1	t0.1	t–0.1
terpinolene	1.1–1.1	0.9–1.0	0.9–1.0
linalool	0.3–0.4	0.2	0.2
nonenal	t–0.1	0.1	0.1
citronellal	t–0.1	0.1	0.1
terpinen-4-ol	0.1	t-0.1	t–0.1
lpha-terpineol	0.4-0.6	0.3	0.2-0.3
decanal	t–0.1	0.1	0.1
citronellol	t–0.3	t-0.1	t–0.2
neral	1.5–3.8	1.3–1.9	1.1–1.9
geraniol	t–0.2	t-0.1	t–0.2
geranial	2.5-6.3	2.1–3.1	1.8–3.1
undecanal	t–0.1	t–0.1	t
δ-elemene	0.1–0.2	0.1	0.1
neryl acetate	0.1–0.2	0.1-0.2	0.1-0.2
geranyl acetate	0.2–0.4	0.2	0.2-0.2-0.3
β-caryophyllene	0.2	0.1-0.2	0.1
<i>trans</i> -α-bergamotene	0.4–0.5	0.3–0.4	0.3–0.4
lpha-humulene	0.1	t	t
valencene	0.1	t	t
(Z)-α-bisabolene	t–0.1	t	t
$\beta$ -bisabolene	0.5–0.7	0.4–0.5	0.4–0.5
norbanol	t	t–0.1	t
t=trace (<0.05%)			

## T-6. Effect of fruit maturity of the 'Diamante' cultivar of *Citrus medica* on the oxygen heterocyclic contents found in the oils

Compound	Small green	Large green	Mature yellow
	fruit oil	fruit oil	fruit oil
herniarin	1.41–3.22ª	0.21–0.35	0.20–0.91
citropten	6.04–14.72	4.51–5.60	2.78–10.78
byakangelicol	7.60–17.86	5.53–6.62	1.88–9.29
oxypeucedanin <sup>a</sup> g/100 g oil	8.21–21.30	7.15–7.28	2.03-9.01

T-7. Comparative percentage composition of a hydrodistilled and a cold-pressed
oil of the 'Diamante' cultivar of <i>Citrus medica</i>

Compound	Hydrodistilled oil	Cold-pressed oil
$\alpha$ -thujene	1.2	1.1
α-pinene	2.5	2.5
camphene	0.9	t
sabinene	0.4	0.4
β-pinene	2.6	2.2
myrcene	2.1	1.7
$\alpha$ -phellandrene	0.2	t
α-terpinene	1.2	0.3
limonene	35.4	44.5
(E)-β-ocimene	2.4	2.1
γ-terpinene	25.4	26.2
<i>cis</i> -sabinene hydrate	t	0.1
terpinolene	1.5	1.1
linalool	0.6	0.1
nonanal	0.2	-
citronellal	0.3	0.1
terpinen-4-ol	1.5	0.3
α-terpineol	1.1	0.2
decanal	0.1	0.1
nerol	1.0	0.1
neral	4.4	1.1
geraniol	0.3	0.5
perillaldehyde	0.2	-
geranial	5.5	2.9
citronellyl acetate	0.2	0.5
neryl acetate	0.5	0.2
geranyl acetate	0.4	0.3
β-elemene	0.1	0.2
β-cubebene	0.1	-
β-caryophyllene	0.3	0.3
(E)-β-farnesene	0.5	0.1
germacrene D	-	0.1
β-bisabolene	1.2	1.3
α-humulene	0.2	-
δ-cadinene	0.2	-
β-bisabolol	0.4	0.1
eicosane	0.1	-
citropten	0.9	2.5
oxypeucedanin	-	0.2

characterize the major constituents of the oils. The results of the findings of Minott and Brown can be seen in **T-8**. Furthermore, the authors produced polygonal profiles (the compounds listed 1–10 on the table) of the leaf oils from the male and female trees. Examination of the polygonal profiles of the leaf oils of trees of unknown fruiting ability revealed that trees 1, 2 and 4 were female trees and tree 3 was a male tree. This conclusion was supported by the data obtained once the trees flowered. A commercial oil of pimento leaf of Jamaican origin was analyzed by Jirovetz et al. (2007) using GC-FID and GC/MS. The constituents characterized in this oil were:

 $\begin{array}{l} \alpha\text{-thujene}\ (0.1\%)\\ \alpha\text{-pinene}\ (0.2\%)\\ \text{myrcene}\ (0.1\%)\\ \alpha\text{-phellandrene}\ (0.4\%)\\ \text{p-cymene}\ (0.5\%)\\ \text{limonene}\ (0.1\%)\\ \beta\text{-phellandrene}\ (0.3\%)\\ 1,8\text{-cineole}\ (0.3\%) \end{array}$ 

(E)- $\beta$ -ocimene (0.2%) γ-terpinene (0.2%) terpinolene (0.5%) linalool (0.1%) terpinen-4-ol (0.2%)  $\alpha$ -terpineol (0.1%) chavicol (0.1%) eugenol (76.0%)  $\beta$ -elemene (0.2%) methyl eugenol (7.1%)  $\alpha$ -gurjunene (0.1%)  $\beta$ -caryophyllene (6.5%) aromadendrene (0.3%)  $\alpha\text{-humulene}\;(1.4\%)$  $\gamma$ -gurjunene (0.1%) allo-aromadendrene (0.3%)  $\beta$ -selinene (0.6%)  $\alpha$ -selinene (1.0%) (E,E)- $\alpha$ -farmesene (0.1%) $\gamma$ -cadinene (0.2%)  $\delta$ -cadinene (0.4%)  $\alpha$ -cadinene (0.1%) (E)-nerolidol (0.1%)

Trace amounts (<0.1%) of (Z)-3hexenol,  $\beta$ -pinene,  $\gamma$ -elemene and viridiflorol were also found in this oil.

A commercial oil of allspice that was purchased in South Korea was screened against the pine wood nematode by Park et al. (2007). The oil used in this study was found to contain the following constituents:

 $\begin{array}{l} 1,8\text{-cineole}\;(0.1\%)\\ \text{limonene}\;(0.2\%)\\ \beta\text{-caryophyllene}\;(7.7\%)\\ \alpha\text{-humulene}\;(1.0\%)\\ \text{eugenol}\;(86.4\%)\\ \text{methyl eugenol}\;(3.9\%) \end{array}$ 

The major components of a commercial oil of *P. dioica* were determined by Seo et al. (2009) to be as follows:

 $\begin{array}{l} 1,8\text{-cineole}\;(0.1\%)\\ limonene\;(0.2\%)\\ eugenol\;(86.4\%)\\ methyl\;eugenol\;(3.9\%)\\ \beta\text{-caryophyllene}\;(7.7\%)\\ \alpha\text{-humulene}\;(1.0\%) \end{array}$ 

The authors also determined that limonene was 100% dextrorotatory.

*Pimenta dioica* berries, which were obtained from a commercial supplier in Mexico, were steam distilled to produce an oil that was screened for its acaricidal activity against the southern cattle tick (Martinez-Velasquez et al., 2011). Analysis of the oil using both GC-FID and GC/MS revealed that the oil contained the following constituents:

T-8. Comparative average percentage composition of the leaf oils of male, female and some unknown fruiting ability trees of
Pimenta dioica

Compound	Male tree leaf oil	Female tree leaf oil	Unknown fruiting ability tree			
-			1	2	3	4
lpha-thujene	0.28	0.58	0.33	0.32	0.42	0.55
$\alpha$ -pinene	0.55	0.76	0.53	0.56	0.75	0.71
myrcene	0.29	0.52	0.35	0.33	0.37	0.50
$\alpha$ -phellandrene	1.25	2.49	1.72	1.55	1.53	2.44
p-cymene	1.78	1.77	1.40	1.39	2.88	1.56
(E)-β-ocimene	0.05	0.10	<0.01	<0.01	<0.01	0.08
γ-terpinene	0.58	1.17	0.85	0.77	0.67	1.22
terpinolene	1.23	2.35	1.70	1.53	1.46	2.36
terpinen-4-ol	0.74	0.87	0.67	0.69	1.03	0.82
$\alpha$ -terpineol	0.30	0.28	0.28	0.19	0.22	0.42
eugenol	83.68	79.81	83.51	82.25	84.06	81.06
methyl eugenol	0.13	0.08	<0.01	<0.01	<0.01	0.06
β-caryophyllene	1.96	2.30	2.24	2.39	1.41	1.94
$\alpha$ -humulene	0.88	0.98	0.92	1.13	0.60	0.67
oil yield	2.13	2.67	2.86	2.67	2.22	2.85

 $\alpha$ -pinene (0.1%)  $\beta$ -pinene (0.2%) myrcene (0.3%)  $\alpha$ -terpinene (0.1%) 1,8-cineole (4.1%) o-cymene (1.2%)  $\gamma$ -terpinene (0.2%)  $\beta$ -terpineol<sup>†</sup> (0.6%) trans-sabinene hydrate (0.9%)  $\beta$ -caryophyllene (2.5%) terpinen-4-ol (0.5%)  $\alpha$ -humulene (2.7%) borneol (1.1%)eugenol (8.3%)  $\alpha$ -humulene<sup>†</sup> (0.2%)  $\gamma$ -cadinene (0.1%) methyl eugenol (62.7%)

<sup>†</sup>incorrect identification

A summary of the earlier reports on pimento leaf and berry oils (Lawrence, 1980, 1981, 1982, 1990, 1994, 1999 and 2006) shows that although the allspice of Jamaican origin is always rich in eugenol, berry oil produced from fruit collected in Central America, including Mexico, often contains methyl eugenol as the principal constituent.

The dried fruits of *P. dioica* that were obtained commercially were comminuted into a fine powder and either subjected to hydrodistillation for 2 hr or a microwave-assisted hydrodistillation to yield oils of 2.68% and 3.25%, respectively, by Jiang et al. (2013). Separate analyses of the oils revealed that they possessed similar compositions as determined by GC-FID and GC/MS. The main components were found to be eugenol (28.8–30.9%) and methyl eugenol (43.0–44.1%). This composition is not normal and the analysis contains numerous errors, consequently it is only included for completeness of a review.

Vazquez-Cahuich et al. (2013) produced a pimento berry oil in the laboratory from powdered dried fruit that were originally purchased from a local market in Villahermosa (Tabasco, Mexico) using hydrodistillation. Analysis of the oil using GC/MS only showed that it contained:

eugenol (94.9%) methyl (E)-cinnamate (1.1%) caryophyllene oxide (0.7%)

Trace amounts of 1,8-cineole, terpinen-4-ol,  $\beta$ -caryophyllene,  $\beta$ -selinene and  $\alpha$ -selinene were also characterized in the oil. The authors screened the oil for its antimicrobial activity and found that the eugenol-rich oil was moderately antimicrobial.

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