



# Progress in Essential Oils

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## Combava Peel Oil

On occasion, oil produced from the peel of *Citrus hystrix* DC by cold pressing can be found on the international market. Lawrence et al. (1971) reported the results of an analysis of the peel oil of *C. hystrix* of Thai origin. The components characterized in this oil were:

α-pinene (2.5 percent)  
camphene (0.2 percent)  
β-pinene (30.6 percent)  
sabinene (22.6 percent)  
myrcene (1.4 percent)  
limonene (29.2 percent)  
1,8-cineole (1.3 percent)  
γ-terpinene (0.1 percent)  
p-cymene (0.1 percent)  
terpinolene (0.1 percent)  
*trans*-sabinene hydrate (0.6 percent)  
citronellal (4.2 percent)  
α-copaene (0.6 percent)  
linalool (0.5 percent)  
β-cubebene (0.5 percent)  
terpinen-4-ol + β-elemene (0.2 percent)  
β-caryophyllene (0.3 percent)  
citronellyl acetate (0.2 percent)  
α-terpineol (0.7 percent)  
geranial (0.1 percent)  
geranyl acetate + citronellol (0.4 percent)  
δ-cadinene (0.3 percent)  
geraniol (0.1 percent)  
(E)-nerolidol (0.1 percent)  
elemol (0.1 percent)

Sato et al. (1990) also examined the composition of Thai *C. hystrix* peel oil. They found that it possessed the following composition:

α-pinene (1.95 percent)  
camphene (0.14 percent)  
β-pinene (25.93 percent)  
sabinene (20.36 percent)  
myrcene (1.29 percent)  
α-terpinene (0.39 percent)  
limonene (5.27 percent)  
β-phellandrene (0.84 percent)

β-ocimene\* (0.09 percent)  
γ-terpinene (0.90 percent)  
p-cymene (0.25 percent)  
terpinolene (0.30 percent)  
*cis*-linalool oxide (furanoid) (0.34 percent)  
*trans*-sabinene hydrate (0.95 percent)  
citronellal (16.80 percent)  
α-copaene (0.61 percent)  
linalool (1.92 percent)  
*cis*-sabinene hydrate (0.32 percent)  
isopulegol (0.27 percent)  
terpinen-4-ol (3.76 percent)  
citronellyl acetate (1.54 percent)  
α-terpineol (1.70 percent)  
germacrene D (0.36 percent)  
δ-cadinene (0.71 percent)  
geranyl acetate (1.04 percent)  
citronellol (2.91 percent)  
(E)-nerolidol (0.11 percent)  
citronellic acid (0.33 percent)

\*correct isomer not identified

Trace amounts (< 0.03 percent) of 1,8-cineole, elemol, a cadinol isomer, α-eudesmol and an epoxy-isopulegol were also found in this same oil.

Asano (1997) repeated the results of Sato et al. (1990) in a review of the aromatic plants of Thailand.

A peel oil of *C. hystrix* of Malaysian origin was analyzed by Jantan et al. (1996) using GC and GC/MS. The oil was found to contain the following constituents:

α-thujene (0.19 percent)  
α-pinene (1.99 percent)  
camphene (0.15 percent)  
β-pinene (39.25 percent)  
octanal (t)  
myrcene (1.32 percent)

$\alpha$ -phellandrene (0.14 percent)  
 $\delta$ -3-carene (1.44 percent)  
 $\alpha$ -terpinene (0.06 percent)  
limonene (14.16 percent)  
(Z)- $\beta$ -ocimene (t)  
(E)- $\beta$ -ocimene (0.07 percent)  
 $\gamma$ -terpinene (2.36 percent)  
*cis*-furanoid oxide<sup>†</sup> (1.85 percent)  
*trans*-furanoid oxide<sup>†</sup> (0.91 percent)  
terpinolene (1.60 percent)  
nonanal (0.08 percent)  
linalool (1.85 percent)  
*cis*-limonene oxide (0.38 percent)  
*trans*-limonene oxide (t)  
isopulegol (0.51 percent)  
citronellal (11.67 percent)  
p-menth-8-en-1-ol (0.25 percent)  
terpinen-4-ol (8.89 percent)  
 $\alpha$ -terpineol (3.03 percent)  
*cis*-piperitol (0.20 percent)  
citronellol (2.96 percent)  
geraniol (0.68 percent)  
p-menthan-3-ol (0.37 percent)  
 $\delta$ -elemene (0.04 percent)  
citronellyl acetate (0.37 percent)  
neryl acetate (t)  
 $\alpha$ -cubebene (0.10 percent)  
geranyl acetate (0.54 percent)  
 $\beta$ -elemene (0.26 percent)  
 $\beta$ -cubebene (0.07 percent)

$\beta$ -caryophyllene (0.39 percent)  
 $\alpha$ -bergamotene<sup>a</sup> (t)  
 $\alpha$ -humulene (0.12 percent)  
 $\gamma$ -elemene (0.03 percent)  
(Z)- $\beta$ -farnesene (0.24 percent)  
 $\alpha$ -cadinene<sup>†</sup> (0.03 percent)  
(E)- $\beta$ -farnesene (0.06 percent)  
 $\delta$ -cadinene (0.49 percent)  
hedycaryol (0.26 percent)  
(Z)-nerolidol (0.04 percent)  
elemol (0.02 percent)  
 $\beta$ -eudesmol (0.15 percent)  
 $\alpha$ -eudesmol (0.15 percent)  
 $\alpha$ -sinensal (0.08 percent)  
hexadecanoic acid (0.05 percent)  
phytol (0.03 percent)

<sup>a</sup>correct isomer not identified; <sup>†</sup>furanoid form; <sup>‡</sup>incorrect identity based on GC elution order; t = trace (< 0.01 percent)

B.M. Lawrence, J.W. Hogg, S.J. Terhune and V. Podimuang, *Constituents of the leaf and peel oils of Citrus hystrix DC*. Phytochemistry, **10**, 1404-1405 (1971).

A. Sato, K. Asano and T. Sato, *The chemical composition of Citrus hystrix DC (Swangi)*. J. Essent. Oil Res., **2**, 179-183 (1990).

I. Jantan, A.S. Ahmad, A.R. Ahmad, N.A. Mohd Ali and N. Ayop, *Chemical composition of some citrus oils from Malaysia*. J. Essent. Oil Res., **8**, 627-632 (1996).

K-I. Asano, *Thailand in view of her agricultural products, particularly, aromatic plants, spices and a number of citrus*. Koryo, **195**, 53-64 (1997).

## Mace Oil

In 1979, Lawrence reported the results of an analysis of a lab-distilled mace oil produced from the nutmeg arilode known as mace (*Myristica fragrans* Houtt.) of East Indian (Indonesian) origin. The composition of this oil was determined to be as follows:

$\alpha$ -pinene (16.3 percent)  
camphene (0.3 percent)  
valeraldehyde (0.1 percent)  
 $\beta$ -pinene (10.6 percent)  
sabinene (12.5 percent)  
 $\delta$ -3-carene (2.1 percent)  
myrcene (2.2 percent)  
 $\alpha$ -phellandrene (1.7 percent)  
 $\alpha$ -terpinene (7.5 percent)  
limonene (4.6 percent)  
1,8-cineole (0.2 percent)  
 $\beta$ -phellandrene (3.6 percent)  
 $\gamma$ -terpinene (11.6 percent)  
p-cymene (1.4 percent)

Comparative percentage composition of *Rosa centifolia* oil and its headspace

T-1

Compound	Oil	Headspace	Compound	Oil	Headspace
acetaldehyde	t	0.2	$\beta$ -bourbonene	0.09	0.12
acetone	-	t	linalyl acetate	0.17	0.28
methyl acetate	-	0.01	$\beta$ -elemene	0.69	0.72
ethanol	0.07	0.37	citronellyl formate	0.11	0.19
valeraldehyde	0.12	0.20	terpinen-4-ol	0.05	0.07
2-butanol	0.02	0.01	$\beta$ -caryophyllene	0.47	0.56
$\alpha$ -pinene	0.85	3.55	citronellyl acetate	0.73	1.64
isobutanol	0.01	0.04	$\alpha$ -humulene	0.07	t
hexanal	0.02	0.05	(E)- $\beta$ -farnesene	1.30	2.06
$\beta$ -pinene	0.20	0.72	neral	0.21	1.31
sabinene	0.06	0.05	heptadecane	2.00	1.68
myrcene	0.36	1.40	geranal	0.16	0.21
$\delta$ -3-carene	0.03	0.07	(E)- $\beta$ -damascenone	0.15	0.18
heptanal	0.07	0.11	$\alpha$ -terpineol	0.74	0.88
isoamyl alcohol	0.14	0.12	neryl acetate	0.24	0.29
limonene	0.05	0.31	geranyl acetate	0.14	0.17
1,8-cineole	0.09	0.12	citronellol	34.08	33.05
(Z)- $\beta$ -ocimene	0.05	0.06	nerol	5.06	5.44
p-cymene	0.05	0.13	2-phenethyl acetate	1.03	1.24
(Z)-3-hexenol	0.04	0.06	$\alpha$ -cadinene	0.03	0.02
(E)- $\beta$ -ocimene	0.03	0.08	(Z)- $\beta$ -damascenone	0.08	0.10
terpinolene	0.09	0.14	geraniol	14.04	18.11
1-hexen-3-ol	0.06	0.09	isogeraniol	0.12	0.14
hexanol	0.24	0.39	damascone*	0.07	0.08
cis-rose oxide	0.31	0.70	nonadecane	14.83	3.80
trans-rose oxide	0.16	0.51	$\beta$ -ionone*	0.75	0.94
nonanal	0.03	0.07	9-eicosene*	3.47	0.53
tetrahydrolinalool <sup>†</sup>	0.09	0.11	2-phenethyl alcohol	1.72	3.40
acetic acid	0.05	0.13	methyl eugenol	1.42	2.65
butyl tiglate	t	0.05	nerolidol*	0.14	0.16
cis-linalool oxide (furanoid)	0.36	0.72	heneicosane	5.63	0.59
trans-linalool oxide (furanoid)	0.24	0.43	eugenol	0.52	0.81
trans-sabinene hydrate	-	t	docosane	0.99	0.21
citronellal	0.07	0.10	farnesol*	0.24	1.33
$\delta$ -elemene	0.08	0.07	eugenyl acetate	0.14	0.05
$\alpha$ -cubebene	t	t	tricosane	1.16	t
benzaldehyde	0.06	0.09	tetracosane	0.99	-
linalool	2.32	4.67			

t = trace (< 0.01 percent); \*correct isomer not identified; <sup>†</sup>not a naturally occurring constituent

terpinolene (3.7 percent)  
p-cymenene (0.1 percent)  
*trans*-sabinene hydrate (0.2 percent)  
 $\alpha$ -copaene (0.2 percent)  
linalool (0.3 percent)  
*cis*-sabinene hydrate (0.2 percent)  
*cis*- $\mu$ -menth-2-en-1-ol (0.2 percent)  
terpinen-4-ol (14.2 percent)  
 $\beta$ -caryophyllene (0.5 percent)  
*cis*-piperitol (0.1 percent)  
 $\alpha$ -terpineol (1.1 percent)  
 $\alpha$ -terpinyll acetate (0.1 percent)  
germacrene D (0.1 percent)  
 $\alpha$ -muurolene (0.1 percent)  
*trans*-piperitol (0.1 percent)  
 $\delta$ -cadinene (0.1 percent)  
geranyl acetate (0.1 percent)  
safrole (0.2 percent)  
methyl eugenol (0.1 percent)  
elemicin (2.0 percent)  
myristicin (1.3 percent)

landral,  $\beta$ -bisabolene, piperitone,  $\gamma$ -cadinene, zonarene,  $\beta$ -sesquiphellandrene, an  $\alpha$ -bisabolene isomer, cadina-1,4-diene,  $\alpha$ -selinene, p-cymen-8-ol, a calamenene isomer, geraniol, caryophyllene oxide, (E)-methyl isoeugenol, eugenol, (E)-isoelemicin and (E)-isomyristicin were also found in the same oil.

Hener et al. (1991) examined the enantiomeric distribution of three monoterpene hydrocarbons in mace oil. The results of this study can be seen as follows:

(1R,5R)-(+)- $\alpha$ -pinene (20 percent) : (1S,5S)-(-)- $\alpha$ -pinene (80 percent)

(1R,5R)-(+)- $\beta$ -pinene (26 percent) : (1S,5S)-(-)- $\beta$ -pinene (74 percent)

(4R)-(+)-limonene (78 percent) : (4S)-(-)-limonene (22 percent)

Ur-Rahman et al. (1999) produced an oil from a commercial sample of mace obtained from the spice market in Karachi. The oil, which was produced by hydrodistillation, was analyzed by GC/MS. It was found to possess the following composition:

$\alpha$ -pinene (5.3 percent)  
 $\alpha$ -thujene (0.9 percent)  
 $\beta$ -pinene (4.9 percent)

sabinene (2.5 percent)  
 $\delta$ -3-carene (0.9 percent)  
myrcene (1.4 percent)  
 $\alpha$ -phellandrene (0.6 percent)  
 $\alpha$ -terpinene (3.2 percent)  
limonene (2.7 percent)  
1,8-cineole (0.1 percent)  
 $\beta$ -phellandrene (2.8 percent)  
 $\gamma$ -terpinene (5.6 percent)  
p-cymene (2.4 percent)  
terpinolene (2.0 percent)  
p-cymenene (0.1 percent)  
 $\alpha$ -copaene (0.1 percent)  
linalool (0.2 percent)  
trans-p-menth-2-en-1-ol (0.3 percent)  
bornyl acetate (0.2 percent)  
terpinen-4-ol (20.0 percent)  
 $\beta$ -caryophyllene (0.4 percent)  
cis-p-menth-2-en-1-ol (0.2 percent)  
 $\alpha$ -terpineol (3.5 percent)  
 $\alpha$ -terpinyl acetate (0.2 percent)  
geranyl acetate (0.1 percent)  
 $\delta$ -cadinene (0.1 percent)  
safrole (3.4 percent)  
methyl eugenol (13.3 percent)  
(E)-cinnamaldehyde (0.3 percent)  
eugenol (0.7 percent)  
(E)-methyl isoeugenol (2.3 percent)  
elemicin (4.7 percent)  
myristicin (14.4 percent)

B.M. Lawrence, *Major Tropical Spices – Nutmeg and Mace (Myristica fragrans Houtt.)*. In: *Essential Oils 1978*. 50-103, Allured Pub. Corp., Carol Stream, IL (1979).

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A. ur-Rahman, M.I. Choudhary, A. Farooq, A. Ahmed, M.Z. Iqbal, B. Demirici, F. Demirci and K.H.C. Baser, *Antifungal activities and essential oil constituents of some spices from Pakistan*. Third Internat. Electronic Conf. Synth. Org. Chem. (ECSOC-3), Sept 1-30, 10 pages (1999).

### Rose-de-Mai Oil and Extract

Kaiser (1993) reported that the main odorous constituents characterized in the headspace of rose-de-mai (*Rosa centifolia* L.) were (E)-4,8-dimethyl-1,3,7-nonatriene, benzyl methyl ether, cis-rose oxide, trans-rose oxide, rose furan, nerol,  $\beta$ -caryophyllene, geranial, neryl acetate, 2-phenethyl acetate, citronellol, germacrene D, nerol, 2-phenethyl alcohol, geraniol, heptadecane and nonadecane.

Etienne (1993) reported that the absolute of *R. centifolia* contained the following constituents:

benzyl alcohol (1.50 percent)  
linalool + 2-phenethyl alcohol (78.10 percent)  
nerol + nerol + citronellol (8.20 percent)  
geraniol + geranial (3.63 percent)  
citronellyl acetate (0.24 percent)  
geranyl acetate (0.23 percent)  
eugenol (0.84 percent)  
 $\alpha$ -copaene + methyl eugenol (0.54 percent)  
 $\beta$ -caryophyllene (0.15 percent)  
guaiadiene\* (0.13 percent)  
germacrene D (0.16 percent)  
farnesol (0.63 percent)  
pentadecane (0.11 percent)  
nonadecene\* (0.82 percent)  
nonadecane (2.50 percent)  
eicosane (0.20 percent)  
heneicosane (0.50 percent)

\*correct isomer not identified

Jirovetz et al. (2002) compared the composition of headspace of the oil with that of the oil of *R. centifolia*. The oil used was a commercial sample of Moroccan origin. The headspace was obtained using solid phase microextraction on a DVB/Carboxen/PDMS fibre after which it was thermally desorbed and analyzed by GC/MS. The oil was analyzed by a combination of GC and GC/MS. The constituents characterized in the headspace and the oil can be seen in T-1.

R. Kaiser, *Investigation of natural scents as a stimulation in perfumery*. Oral Presentation at Centifolia 93 Oct 28-30, Grasse (1993).

J.J. Etienne, *De nouvelles absolues de roses pour les parfums et les cosmétiques*. Rivista Ital. EPPOS, (Numero Speciale), 418-446 (1997).

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