



Progress in Essential Oils

by Brian M. Lawrence, Consultant

Neroli Oil

In 1977, Kekelidze et al. analyzed an oil produced from bitter orange flowers. They found that it contained the following components:

α -pinene (1.7%)	octanol (8.6%)	α -pinene (0.24%)	terpinen-4-ol (0.22%)
β -pinene (3.1%)	terpinen-4-ol (0.2%)	sabinene (0.33%)	cuminaldehyde (0.10%)
sabinene (15.0%)	phenylacetaldehyde (2.0%)	β -pinene (3.10%)	α -terpineol (6.50%)
myrcene (3.7%)	neral (2.1%)	myrcene (0.34%)	<i>trans</i> -carveol (0.16%)
δ -3-carene (0.8%)	α -terpineol (3.2%)	p-cymene (0.14%)	nerol (0.95%)
α -terpinene (0.2%)	geranial (0.1%)	limonene (9.34%)	neral (0.23%)
limonene (0.3%)	citronellol (0.1%)	<i>cis</i> -linalool oxide — furanoid (1.47%)	carvone (0.14%)
β -phellandrene (t)	nerol (0.2%)	<i>trans</i> -linalool oxide — furanoid (1.27%)	linalyl anthranilate* (16.38%)
β -ocimene* (7.8%)	geraniol (0.1%)	linalool (44.29%)	geranial (0.48%)
γ -terpinene (5.2%)	benzyl alcohol (0.2%)	<i>cis</i> -linalool oxide — pyranoid (0.20%)	geranyl acetate (2.73%)
p-cymene (0.6%)	phenyl ethyl ketone (0.9%)	<i>trans</i> -linalool oxide — pyranoid (0.21%)	nerolidol** (0.60%)
terpinolene (0.1%)	nerolidol* (13.0%)		(Z,E)-farnesol (0.36%)
citronellal (0.6%)	methyl anthanilate (5.0%)		(Z,E)-farnesal (0.10%)
benzaldehyde (0.9%)	farnesol* (5.1%)		
linalool (11.4%)	indole (1.0%)		

*correct isomer not identified

*incorrect identification based on elution order

**correct isomer not identified

As most synthetic linalool contains dihydrolinalool (0.5-2.0%), this impurity can be used to determine adulteration of neroli oil with synthetic linalool. Frey (1988) showed that using the selective ion monitoring mode in GC/MS he could detect down to 50 ppm of dihydrolinalool in neroli oil, which corresponds to the addition of 0.5-1.0% synthetic linalool in the oil. König et al. (1992) proved through chiral GC analysis that the (R)-(-)-linalool enantiomer was predominant in neroli oil. They also found that (S)-(+)-(E)-nerolidol was the main (>98%) enantiomer found in this same oil.

Bitter orange flower oil produced in China was subjected to analysis by GC/MS by Zhu et al. (1993). The components identified in this oil were:

In 1995, Bassaada compared the compositions of two samples of bitter orange flower oil produced from flowers harvested in April. One of the flower samples was harvested from trees grown in Chania, Crete, while two other originated in Tunisia. A summary of the results of this study are shown in T-1.

Casiabiana (1996) determined that the enantiomeric distribution of linalool and linalyl acetate in commercial samples of neroli oil of Tunisian, French, Moroccan and Italian origins was as follows:

(3R)-(-)-linalool (72.0-79.0%):(3S)-(+)-linalool (21.0-28.0%)
(3R)-(-)-linalyl acetate (95.0-98.2%):(3S)-(+)-linalyl acetate (1.8-5.0%)

Hethelyi et al. (1998) used GC and GC/MS to analyze four commercial samples of neroli oil. They found that the compositions of these oils ranged as follows:

α -pinene (t-0.9%) limonene (17.9-20.9%)
 β -pinene (11.9-15.2%) δ -3-carene* (2.6-6.9%)

Comparative percentage composition of bitter orange flower oil from two different origins

T-1

Compound	Greek flower oil	Tunisian flower oil
α-pinene	0.60	0.01
camphene	0.04	0.02
β-pinene	9.80	5.15
sabinene	1.07	4.10
myrcene	1.75	1.77
α-terpinene	0.08	0.07
limonene	8.02	10.87
β-phellandrene	0.20	0.17
(Z)-β-ocimene	0.68	0.55
γ-terpinene	0.11	0.17
(E)-β-ocimene	6.39	3.72
terpinolene	0.35	0.25
<i>trans</i> -linalool oxide*	0.15	0.18
<i>cis</i> -linalool oxide*	0.07	0.05
linalool	32.18	34.41
linalyl acetate	12.05	11.33
β-caryophyllene	0.35	0.23
terpinen-4-ol	0.20	0.61
α-terpineol	4.80	6.58
neryl acetate	1.50	1.76
farnesene*	0.04	0.06
geranyl acetate	4.01	3.42
nerol	1.18	1.43
geraniol	3.15	4.20
nerolidol*	1.46	0.40
farnesol*	5.40	3.88
indole	0.70	0.30

* correct isomer not identified

linalool (37.1-39.2%) neryl acetate (t-1.9%)
 nerol (3.7-5.6%) geranyl acetate (2.8-3.7%)
 linalyl acetate (0.0-8.7%) (E)-nerolidol (2.6-3.8%)
 geraniol (1.0-9.0%) (E,E)-farnesol (1.2-2.3%)

t = trace (<0.1%)
 *incorrect identification based on elution order

The authors also analyzed the volatiles found in two samples of concrete and one absolute of neroli. The results of these analyses are summarized in T-2.

Dugo et al. (1999) used liquid chromatography to separate the hydrocarbons from the oxygenated constituents of neroli oil produced in Italy. Analysis of the oil revealed that it possessed the following composition:

tricyclene (0.01%) α-terpinene (0.52%)
 α-thujene (0.25%) p-cymene (0.71%)
 α-pinene (1.34%) limonene (24.27%)
 camphene (0.04%) (Z)-β-ocimene (0.35%)
 sabinene + β-pinene (20.15%) (E)-β-ocimene (3.74%)
 myrcene (2.43%) γ-terpinene (3.75%)
 α-phellandrene (0.09%) terpinolene (0.55%)
 δ-3-carene (0.54%) δ-elemene (0.02%)

Comparative percentage composition of the volatiles of neroli concrete and neroli absolute

T-2

Compound	Concrete		Absolute
	1	2	
β-pinene	2.6	2.6	2.4
limonene	6.6	6.3	2.8
δ-3-carene	-	-	1.0
linalool	58.8	58.2	55.2
2-phenethyl alcohol	-	1.7	t
nerol	4.7	4.0	3.7
linalyl acetate	5.0	4.9	-
neryl acetate	t	t	1.9
geranyl acetate	2.8	2.8	3.7
(E)-nerolidol	3.8	3.2	2.7
(E,E)-farnesol	2.3	2.0	2.1

t = trace (<0.1%)

β-elemene (0.31%)	citronellal (0.07%)
β-caryophyllene (0.72%)	terpinen-4-ol (1.23%)
<i>trans</i> -α-bergamotene (0.01%)	α-terpineol (1.83%)
α-humulene (0.10%)	<i>trans</i> -piperitol (0.03%)
(Z)-β-farnesene (0.13%)	nerol (0.70%)
germacrene D (0.05%)	neral (0.46%)
(E,E)-α-farnesene (0.08%)	linalyl acetate (9.89%)
δ-cadinene (0.03%)	geraniol (0.65%)
benzaldehyde (0.01%)	indole (0.06%)
6-methyl-5-hepten-2-one (0.11%)	methyl anthranilate (0.10%)
1,8-cineole (0.18%)	α-terpinyl acetate (0.05%)
<i>trans</i> -sabinene hydrate (0.10%)	citronellyl acetate (0.03%)
<i>cis</i> -linalool oxide* (0.02%)	neryl acetate (0.83%)
<i>trans</i> -linalool oxide* (0.01%)	geranyl acetate (1.38%)
<i>cis</i> -sabinene hydrate (0.03%)	methyl N-methyl anthranilate (3.19%)
linalool (15.48%)	(Z)-nerolidol (t)
2-phenethyl alcohol (0.01%)	(E)-nerolidol (1.76%)
<i>cis</i> -p-menth-2-en-1-ol (0.08%)	spathulenol (0.04%)
<i>trans</i> -p-menth-2-en-1-ol (0.19%)	globulol (0.01%)
	α-cadinol (0.02%)
	(Z,E)-farnesol (0.99%)
	(E,E)-farnesol (0.02%)

*furanoid form
 t = trace (<0.01)

According to the European Pharmacopoeia (cited in Braun and Franz 2001), the major nine constituents of neroli oil are as follows:

β-pinene (7.0-17.0%)	geranyl acetate (1.5-4.0%)
limonene (9.0-18.0%)	N-methyl anthranilate (0.1-1.0%)
linalool (18.0-42.0%)	(E)-nerolidol (1.0-9.0%)
linalyl acetate (3.0-16.0%)	
α-terpineol (2.0-7.0%)	
neryl acetate (1.0-3.0%)	

The authors also examined nine neroli oil samples that they obtained either in the commercial trade or the retail trade and found that the oils compositions varied widely revealing that most of the oils had been adulterated to some extent. The oil compositions were as follows:

β -pinene (0.78-14.57%)
 limonene (3.27-18.35%)
 linalool (27.21-55.22%)
 linalyl acetate (1.74-29.26%)
 α -terpineol (2.19-6.49%)
 neryl acetate (0.85-1.88%)
 geranyl acetate (1.37-3.67%)
 N-methyl anthranilate (0.10-3.67%)
 (E)-nerolidol (0-5.60%)

N.A. Kekelidze, M.A. Janikashvili and G. M. Fishman, *The investigation of the essential oils from orange and tangerine flowers*. Subtr. Kult., **4**, 83-85 (1977).

C. Frey, *Detection of synthetic flavorant addition to some essential oils by selected ion monitoring GC/MS*. In: *Flavors and Fragrances: A World Perspective*. Edits., B.M. Lawrence, B.D. Mookherjee and B.J. Willis, pp 517-524, Elsevier, Amsterdam (1988).

W.A. König, B. Gehrke, D. Icheln, P. Evers, J. Donnecke and W.-C. Wang, *New selectively substituted cyclodextrins as stationary phases for the analysis of chiral constituents of essential oils*. J. High Resol. Chromatogr., **15**, 367-372 (1992).

L.-F. Zhu, Y.-H. Li, B.-L. Li, B.-Y. Lu and N.-H. Xia, *Aromatic Plants and Essential Constituents*. South China Institute of Botany, Chinese Academy of Sciences, Hai Feng Publish. Co. distributed by Peace Book Co. Ltd. Hong Kong (1993).

O. Bussaada, *Variation of Essential Oil Yield and Composition of Citrus aurantium var. amara*. M.Sc. thesis Mediterranean Agronomic Institute, Chania, Greece (1995).

H. Casabianca and J.B. Graff, *Chiral analysis of linalool and linalyl acetate in various plants*. Rivista Ital. EPPOS, (Numero Speciale), 227-243 (1996).

E. Hethelyi, M. Palfine-Ledniczky, K. Korany, J. Bernath and R. Banatfy, *GC, GC-MS determination of neroli bigarade oils from orange blossoms*. Olaj Szappan Kosmet., **47**, 222-228 (1998).

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

M. Braun and G. Franz, *Qualität Ältherischer Öle. Chirale Saülen decken Verfälschungen auf*. Pharm. Ztg., **146**(29), 11-17 (2001).

Savory Oil

Sevarda et al. (1985) compared the major component composition of two varieties of *Satureja montana* (savory, or winter savory). They found that the main components of an oil produced from *S. montana* L. var. *domaviana* K. Maly that was produced from plants collected around Beli Rzar (Serbia) were as follows:

α -pinene (3.0%)	linalool (11.2%)
limonene* + 1,8-cineole (2.0%)	β -caryophyllene (50.4%)
p-cymene (14.0%)	geranial (3.8%)

*major component

The main constituents of an oil of *S. montana* ssp. *montana* produced from plants collected in the vicinity of Lim (Serbia) were as follows:

α -pinene (24.0%)	p-cymene (3.0%)
limonene + 1,8-cineole (19.3%)	geranial (23.0%)

It should be pointed out that this is an atypical oil of savory; however, chemotypes are known.

An oil of *Satureja montana* produced from wild plants collected in Italy that was screened by Panizzi et al. (1993) for its antimicrobial properties was analyzed and found to contain the following major constituents:

α -pinene (1.07%)	β -caryophyllene (1.44%)
α -phellandrene (1.41%)	borneol (2.36%)
α -terpinene (1.21%)	carvone (0.41%)
γ -terpinene (10.03%)	thymol (0.31%)
p-cymene (9.83%)	carvacrol (56.82%)

Canales and Sanchez (1997) collected wild plants of *S. montana* from various regions of Spain and determined the p-cymene, 1,8-cineole, carvacrol and thymol contents of lab distilled oils produced from them. The results of these incomplete analyses are shown in T-3.

Slavkovska et al. (1997) examined the variability of oils obtained from *S. montana*. One population of ssp. *pisidica* (Wettst.) Silic collected in the Galicica Mountains (Macedonia) and two populations of *S. montana* ssp. *montana*; one from the gorge of the Beli Rzar River (Serbia) and the other from Zvornik area (Serbia). Within the population of *S. montana* ssp. *pisidica* the oils produced could be grouped into three chemotypes (see T-4), while the oils from the two populations of *S. montana* ssp. *montana* could be subdivided into five chemotypes (see T-5). From a commercial use standpoint only the carvacrol-rich *S. montana* ssp. *pisidica* oils possessed a composition that could prove valuable. In a follow-up report to this study, Slavkovska et al. (2001) used principal component analysis to examine the component variables in the oils of *S. montana* ssp. *montana* and another *Satureja* species (*S. kitaibelli* Jelasnica). Palic et al. (1998) noted that the chemotypes of *S. montana* ssp. *variegata*, *S. montana* ssp. *pisidica* and *S. montana* ssp.

The percentage composition of four components of *Satureja montana* oil produced from plants obtained from several Spanish regions

T-3

Compound	Murcia	Alicante	Oils from Albacete	Valencia	Tarragona
p-cymene	13-14	9-16	8	15	13
1,8-cineole	1.5-2	0-2	-	2	2
carvacrol	22-25	17-23	14	24	25
thymol	3-5	2-4	1.5	6	3

Percentage composition of the oils of the three chemotypes of *Satureja montana* ssp. *pisidica* of Serbian origin

T-4

Compound	Chemotype 1 carvacrol-rich	Chemotype 2 p-cymene rich	Chemotype 3 linalool-rich
α -pinene	-	0.5-1.4	1.2-1.6
camphene	0.5-2.0	0.4-3.6	0.2-2.3
β -pinene	0.1	0.2-1.8	0.1-4.3
myrcene	0.3-1.5	0.6-1.7	0.6-2.9
α -terpinene	1.2-1.5	0.5-1.7	0.2-1.4
limonene	0.2-0.4	0.3-2.4	0.1-1.3
(Z)- β -ocimene	0.5-2.4	0.6-5.3	0.4-6.2
γ -terpinene	1.9-11.0	3.7-13.0	1.5-9.8
p-cymene	19.0-30.5	22.4-75.7	6.7-30.7
<i>trans</i> -sabinene hydrate	0.1-2.9	0.5-6.9	0.4-8.6
linalool	4.3-24.0	0.4-26.0	22.5-68.0
terpinen-4-ol	0.4-0.5	0.2-11.2	0.5-5.2
β -caryophyllene	0.5-3.9	0.3-2.2	0.1-3.4
borneol	0.8-19.0	1.1-9.8	0.9-2.1
p-cymen-8-ol	0.2-13.3	0.5-1.7	0.2-0.9
thymol	0.1-2.2	0.6-3.5	0.1-4.2
carvacrol	26.2-70.5	2.8-20.8	3.3-17.5

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Percentage composition of two oils of five chemotypes of *Satureja montana* ssp. *montana* of Serbian origin

T-5

Compound	Chemotype				
	1*	2	3	4	5
α -pinene	0.7-8.2	0.9-10.3	0.3-8.4	1.9-4.4	0.7-7.1
camphene	0.9-4.1	1.3-3.7	0.3-6.3	4.1-5.7	0.5-3.0
β -pinene	0.1-1.3	0.9-2.0	0.2-5.1	0.6-1.4	0.2-2.6
myrcene	0.1-4.8	0.6-5.2	0.4-5.7	1.1-2.4	0.3-3.3
α -terpinene	0.2-1.9	0.9-4.1	0.3-4.3	0.6	0.3-15.8
limonene	0.3-7.3	0.8-5.9	0.4-9.4	3.0-8.4	1.6-14.3
(Z)- β -ocimene	0.8-8.9	1.0-2.9	0.4-6.2	1.4-9.5	0.5-8.9
γ -terpinene	0.6-14.3	3.7-12.6	0.4-6.5	1.4-3.7	0.6-7.6
p-cymene	14.5-47.7	2.7-21.8	0.5-17.5	2.6-28.8	2.1-18.6
<i>trans</i> -sabinene hydrate	0.4-20.9	14.2-27.1	0.3-15.7	2.4-2.8	0.4-6.9
linalool	0.4-15.5	2.3-18.1	14.4-74.0	0.9-10.5	0.8-16.3
terpinen-4-ol	0.6-11.4	4.3-14.2	0.6-12.1	1.9-2.9	0.5-7.7
β -caryophyllene	-	0-0.5	-	-	-
borneol	4.8-21.4	4.9-10.2	2.3-18.4	21.5-29.6	3.7-7.2
p-cymen-8-ol	1.2-17.9	0.2-5.5	0.3-15.4	0.9	11.3-27.0
thymol	0.7-5.1	0.4-1.6	0.1-2.9	0.8-3.4	0.6-11.0
carvacrol	0.3-1.3	0.2-1.5	0.2-0.6	-	0.5-2.0

*1 = p-cymene-rich; 2 = *trans*-sabinene hydrate-rich; 3 = linalool-rich; 4 = borneol-rich; 5 = p-cymen-8-ol-rich

Percentage composition of *Satureja montana* oils produced from plants harvested at different development stages from three locations in Croatia

T-6

Compound	Biokovo			Brac			Kozjak		
	P (1.9%) ^a	F (1.7%)	S (0.7%)	P (1.7%)	F (1.8%)	S (0.7%)	P (1.6%)	F (1.2%)	S (0.7%)
α-thujene	1.8	1.8	1.2	1.4	1.3	1.3	1.4	1.7	1.5
α-pinene	t	1.0	1.3	0.6	1.4	2.2	0.4	1.3	1.0
myrcene	0.6	0.8	-	0.9	0.6	-	1.1	1.3	1.7
α-terpinene	1.8	1.5	-	1.7	0.9	-	1.0	1.8	-
limonene	-	-	1.0	-	-	1.1	-	1.6	1.6
(Z)-β-ocimene	-	-	-	-	-	-	0.4	0.6	0.6
γ-terpinene	7.2	8.1	-	5.8	4.9	-	6.5	7.6	-
p-cymene	3.0	12.6	28.9	3.8	15.2	25.6	5.0	17.8	19.1
allo-ocimene*	0.9	-	-	0.2	-	-	0.5	0.5	0.5
1-octen-3-ol	0.5	0.7	1.5	0.4	1.0	1.4	0.6	1.3	1.1
trans-sabinene hydrate	0.3	-	1.6	0.3	0.2	1.2	0.3	0.4	0.5
camphor	-	-	0.4	-	-	0.7	-	-	0.5
β-bourbonene	-	-	-	-	-	0.3	-	-	0.6
linalool	24.8	0.5	0.7	0.5	0.6	0.9	0.8	0.7	0.7
methyl thymol	6.8	2.3	5.2	3.2	3.3	12.8	1.5	4.1	3.3
methyl carvacrol	2.0	11.0	7.4	4.6	6.4	5.4	7.0	8.2	4.2
β-caryophyllene	2.7	-	-	1.8	1.3	1.3	-	-	-
aromadendrene	t	t	-	0.2	0.3	0.5	0.2	0.3	-
α-humulene	0.2	-	-	0.2	-	-	-	-	-
neral	-	-	-	-	0.6	-	-	0.7	1.1
α-terpineol	-	0.5	0.6	-	0.3	0.6	-	0.3	0.4
borneol	1.5	4.8	8.1	3.2	6.5	11.5	3.0	5.8	6.2
β-cubebene	-	0.5	-	0.4	-	0.7	0.4	0.4	0.5
geranial	-	-	-	-	0.9	-	0.7	1.1	1.4
β-bizabolene	0.4	2.0	-	0.5	-	1.5	1.0	0.5	0.5
geranyl acetate	-	-	-	-	5.2	-	3.5	2.5	6.7
δ-cadinene	0.1	0.4	0.2	0.3	-	0.2	-	-	-
nerol	-	-	-	-	0.9	0.2	1.5	1.0	1.2
geraniol	t	-	-	-	10.2	-	9.2	9.7	9.5
thymyl acetate	t	0.2	-	0.2	-	-	0.2	-	-
caryophyllene oxide	-	0.4	2.6	-	1.2	2.8	0.5	0.6	2.3
spathulenol	0.3	0.3	0.5	-	0.3	0.7	0.4	0.2	0.7
thymol	15.0	3.9	3.0	11.0	5.4	2.6	20.6	6.1	1.9
carvacrol	25.3	45.7	28.1	52.4	26.2	16.1	30.4	20.8	23.4

P = prior to flowering (July); F = full flowering (September); S = post-flowering — senescence (November); ^aoil content; t = trace (<0.01%); * correct isomer not identified

montana were 1. γ-terpinene/carvacrol and 2. p-cymene/thymol-carvacrol and linalool/α-terpineol-terpinen-4-ol types.

Stoyanova et al. (2000) found that an oil of *S. montana* of Bulgarian origin had the following major constituents:

α-pinene (1.4%)	β-caryophyllene (13.2%)
β-pinene (1.4%)	thymol (32.1%)
limonene (9.5%)	carvacrol (35.0%)
p-cymene (14.5%)	

Milos et al. (2001) examined the seasonal variation in the oil of *S. montana* collected in three different locations along the central part of Dalmatia (Croatia). The oils, which were obtained from plants housed at three distinct development

stages, were analyzed by GC/MS only, the results of which are summarized in T-6. As can be seen, the only trends in oil composition were to increase in p-cymene and, to a lesser extent, borneol contents, and a decrease in the thymol content as the plants matured. Also, although the maximum oil content did not always occur at the full flowering stage, there was not a correlation between oil yield, development stage and carvacrol content.

Hethelyi et al. (2002) used a combination of GC and GC/MS to determine that oils of *S. montana* grown in Hungary possessed the following major constituents:

α-thujene (0.1-0.8%)	1,8-cineole (0.1-0.5%)
α-pinene (0.5-1.3%)	γ-terpinene (18.8-29.4%)
camphene (0.2-1.8%)	thymol (0-8.3%)
β-pinene (0.9-1.9%)	carvacrol (59.7-65.7%)
p-cymene (5.2-8.1%)	β-caryophyllene (0.4-1.1%)

Percentage composition of savory oil produced from plants of different development stages

T-7

Compound	Pre-flowering plant oil	Flowering plant oil
α -thujene	1.77	1.58
α -pinene	0.52	0.99
β -pinene	0.10	t
δ -3-carene	0.40	t
myrcene	2.20	1.42
α -terpinene	3.87	1.71
β -phellandrene	0.44	t
limonene	1.68	t
γ -terpinene	9.49	7.57
p-cymene	7.10	12.36
terpinolene	t	t
allo-ocimene*	0.96	-
1-octen-3-ol	0.55	0.63
terpinen-4-ol**	0.14	t
linalool	0.37	3.15
methyl thymol	5.11	4.99
methyl carvacrol	4.60	5.11
β -caryophyllene	3.75	2.64
aromadendrene	0.38	0.22
neral	-	0.45
α -humulene	0.23	-
borneol	2.36	3.62
ledene or viridiflorene	1.28	0.34
β -cubebene**	0.30	t
geranial	-	0.28
β -bisabolene	0.82	0.02
geranyl acetate	-	3.38
δ -cadinene	0.44	0.23
nerol	-	0.49
geraniol	-	4.15
thymyl acetate	0.17	-
spathulenol	0.34	-
thymol	46.02	35.41
carvacrol	4.52	6.86

t = trace (<0.01%)
 *correct isomer not identified
 **incorrect identity based on elution order

Mastelic and Jerkovic (2003) collected *S. montana* from a sub-Mediterranean area near Sinj (southern Croatia) at two different stages of development (vegetative and flowering). The plant materials were air-dried in the shade for 20 days at room temperature. Oils were obtained from the two different stages by water distillation, to which an internal standard of octyl- β -D-glucoside was added. The oils were then examined by GC/MS (see T-7). The spent plant material was further extracted with boiling water and, once it had been filtered, the combined distillation

water and the second water extraction were pooled and concentrated to 30 mL under reduced pressure at 50°C. The saccharides, proteins and other water-soluble high molecular weight compounds were removed by precipitation with 100 mL ethanol. The aqueous ethanolic solution was further concentrated to 20 mL under reduced pressure to which a few drops of ammonia were added to precipitate acidic materials. Purification of the aglycone containing fraction was performed by flash chromatography over silica gel using an ethyl acetate:ethanol:ammonia (6:3:1 v/v) solvent system. Finally, the glycosides were hydrolyzed with β -glucosidase at 30°C for 70 h from the aqueous solution obtained once the free terpenes and other hydrophobic constituents had been removed by extraction with pentane/methylene chloride. The glycosidically bound volatiles isolated from this Croation savory can be seen in T-8.

As part of a screening study on the effect of essential oils on honey bee diseases, Dellacasa et al. (2003) reported that the oil of a clone of *S. montana* produced in Argentina contained the following major components:

p-cymene (20.8%)	γ -terpinene (11.8%)
carvacrol (31.5%)	thymol (4.2%)

The oil was found to be somewhat effective in the treatment of chalkbrood disease.

- A.L. Sevarda, G.A. Kuznjecova, S. Pavlovic, P. Zivanovic and R. Jancic, *Quantity and composition of essential oil and anatomic structure of Satureja montana L. (Lamiaceae)*. Arh. Farm., **36**(4), 167-175 (1985).
- L. Panizzi, G. Flamini, P.L. Cioni and I. Morelli, *Composition and antimicrobial properties of essential oils of four Mediterranean Lamiaceae*. J. Ethnopharmacol., **39**, 167-170 (1993).
- S. Canales and J.A. Sanchez, *Pharmacognostic active components of ajedra (genus Satureja L.) related to growing soil conditions*. Acta Technol. Legis Med., **6**, 294-300 (1997).
- V. Slavkovska, R. Jancic, S. Milsavljevic and D. Djokovic, *Variability of the essential oil composition of the species Satureja montana L. (Lamiaceae)*. J. Essent. Oil Res., **9**, 629-634 (1997).
- R. Palic, N. Simic, S. Anđelkovic and G. Stojanovic, *Composition of essential oil of selected Balkan's Satureja species and chemotaxonomic implications*. J. Essent. Oil Res. Plants., **1**(2/3), 66-81 (1998).
- A. Stoyanova, A. Georgieva and E. Georgiev, *Essential oil content of winter savory (Satureja montana) and creeping thyme (Thymus serpyllum) natural products*. Izv. Vyssh. Uchebn. Zaved., Pitsheh. Technol., (5/6), 15-16 (2000).
- M. Milos, A. Radonic, N. Bezic and V. Dukic, *Localities and seasonal variations in the chemical composition of essential oils of Satureja montana L. and S. cuneifolia Ten.* Flav. Fragr. J., **16**, 157-160 (2001).
- V. Slavkovska, R. Jancic, S. Bojovic, S. Milosavljevic and D. Djokovic, *Variability of essential oils of Satureja montana L. and Satureja kitaibelii Wierzb. ex Heuff. From the central part of the Balkan peninsula*. Phytochemistry, **57**, 71-76 (2001).
- E.B. Hethelyi, B. Galambosi and J. Bernath, *Investigation of Satureja biflora and other Satureja species essential oils by GC and GC/MS*. Olaj Szappan Kozmet., **51**(2), 62-73 (2002).

Compound	Pre-flowering plant	Flowering plant
(Z)-3-hexenol	9.73	4.27
1-octen-3-ol	1.45	t
α -terpineol	2.98	2.49
thymoquinone	32.93	27.98
methyl salicylate	0.69	0.82
nerol	-	1.07
geraniol	-	4.59
benzyl alcohol	4.37	4.70
2-phenethyl alcohol	1.23	4.03
p-cymen-8-ol	5.74	5.85
eugenol	8.35	17.40
thymol	7.81	4.07
carvacrol	0.87	t
indole	1.25	3.33
3,5,5-trimethyl-3-hydroxy-1-butenyl-2-cyclohexen-1-ol	2.49	5.67
2-methoxy-4-ethyl-6-methylphenol	5.44	4.28

t = trace (<0.01%)

J. Mastelic and I. Jerkovic, *Gas chromatography-mass spectrometry analysis of free and glycoconjugated aroma compounds of seasonally collected *Satureja montana* L.* Food Chem., **80**, 135-140 (2003).

A.D. Dellacasa, P.N. Bailac, M.I. Ponzi, S.R. Ruffinengo and M.J. Eguaras, *In vitro activity of essential oils from San Luis Argentina against *Ascosphaera apis*.* J. Essent. Oil Res., **15**, 282-285 (2003).

Yarrow Oil

Although yarrow oil, which is produced in limited quantities in eastern Europe, is generally considered to be of value because of its richness in chamazulene, the plant *Achillea millefolium* L. ssp. *millefolium* can

be found growing wild throughout the temperate and boreal zones of the northern and southern hemispheres. As with many members of the Asteraceae family, the oils of *A. millefolium* exist in a variety of chemotypic forms. A survey of the literature reveals that the chamazulene-rich forms are not as common as the other chemotypic forms. For example, a sample of yarrow oil produced in Egypt was subjected to GC analysis. Using compound coinjection as the method of component identification, Daw et al. (1994) determined that the oil contained:

β -pinene (2.69%)	linalool (2.30%)
α -phellandrene (0.90%)	camphor (2.40%)
limonene (9.73%)	linalyl acetate (6.30%)
terpinene [*] (2.51%)	cadinene [*] (3.29%)
β -phellandrene (59.68%)	neral (1.50%)
citronellal (0.06%)	geranial (0.16%)
terpinolene (2.90%)	geranyl acetate (0.40%)

^{*}correct isomer not identified

As part of a continuing study on *A. millefolium* Kastner et al. (1996) identified α -longipin-2-en-1-one and 7 β -hydroxy- α -longipin-2-en-1-one in a methylene chloride extract of *A. millefolium*.

Pino et al. (1998) analyzed an oil produced from *A. millefolium* grown in Cuba via GC and GC/MS. This oil was found to possess the following composition:

α -thujene (0.1%)	<i>cis</i> -sabinene hydrate (0.2%)
α -pinene (0.4%)	ethyl heptanoate (0.4%)
camphene (0.4%)	linalool (1.0%)
sabinene (5.4%)	camphor (1.2%)
myrcene (0.1%)	isomenthone (0.2%)
ethyl hexanoate (0.6%)	borneol (9.8%)
<i>p</i> -cymene (0.6%)	terpinen-4-ol (2.8%)
γ -terpinene (0.2%)	isomenthol (0.4%)
1,8-cineole (5.7%)	α -terpineol (2.0%)
terpinolene (t)	

ethyl octanoate (1.5%)	viridiflorene (0.8%)
pulegone (2.4%)	δ -cadinene (0.3%)
bornyl acetate (1.9%)	caryophyllene oxide (20.0%)
thymol (0.5%)	humulene epoxide II (3.2%)
ethyl nonanoate (2.6%)	(<i>E</i>)-isoeugenyl acetate (1.1%)
α -cubebene (0.3%)	14-hydroxy- α -muurolene (0.8%)
β -bourbonene (0.1%)	14-hydroxy- δ -cadinene (0.4%)
β -caryophyllene (5.2%)	2-heptadecanone (0.2%)
α -humulene (0.7%)	methyl hexadecanoate (0.8%)
(<i>E</i>)- β -farnesene (0.2%)	hexadecanoic acid (1.1%)
β -ionone [*] (0.2%)	methyl octadecanoate (0.2%)
germacrene D (0.8%)	

^{*}correct isomer not identified

t = trace (<0.1%)

Although yarrow oil is produced from *A. millefolium* L. ssp. *millefolium*, Chalchat et al. (1999) examined an oil produced from *A. millefolium* ssp. *pannonica* collected from plants growing Serbia and Montenegro. This oil was determined to contained the following constituents:

tricyclene (0.1%)	<i>trans</i> -pinocarveol (0.6%)
α -pinene (1.8%)	<i>cis</i> -verbenol (0.2%)
α -thujene (0.4%)	δ -terpineol (0.3%)
camphene (1.8%)	<i>trans</i> -chrysanthenol (1.3%)
β -pinene (3.7%)	<i>trans</i> -verbenol (0.2%)
sabinene (0.2%)	borneol (15.0%)
δ -3-carene (0.2%)	α -terpineol (2.2%)
dehydro-1,8-cineole (0.2%)	<i>cis</i> -piperitol (0.9%)
1,8-cineole (23.3%)	myrtenol (0.4%)
γ -terpinene (0.4%)	<i>trans</i> -carveol (0.3%)
<i>p</i> -cymene (1.4%)	<i>p</i> -cymen-8-ol (0.1%)
1,2,4-trimethylbenzene (0.1%)	(<i>Z</i>)-jasmone (t)
α -thujone (4.5%)	caryophyllene oxide (1.6%)
β -thujone (0.8%)	nerolidol [*] (1.0%)
camphor (12.7%)	spathulenol (0.1%)
benzaldehyde (0.1%)	α -muurolol (0.1%)
pinocarvone (0.3%)	T-muurolol (0.2%)
<i>trans</i> - <i>p</i> -menth-2-en-1-ol (0.9%)	α -eudesmol (0.2%)
bornyl acetate (3.6%)	β -eudesmol (0.3%)
β -caryophyllene (0.4%)	α -cadinol (0.2%)
terpinen-4-ol (2.5%)	α -selin-11-en-4a-ol (0.7%)
myrtenal (0.3%)	caryophyllen-8-ol (0.7%)
thuj-3-en-10-al (0.3%)	14-hydroxy-9-epi- β -caryophyllene (0.4%)
<i>cis</i> - <i>p</i> -menth-2-en-1-ol (0.8%)	

^{*}correct isomer not identified

t = trace (<0.1%)

As can be seen, this oil bears little resemblance to the chamazulene-rich yarrow oils of commerce.

Orth et al. (1999) compared the composition of oils produced from five *A. millefolium* clones collected in the vicinity of Kalingrad (western Russia) and grown in a botanic garden in Würzburg (Germany). The oils, which were analyzed by GC and GC/MS, could be divided into four chamazulene-rich types and one sesquiterpene hydrocarbon type. A summary of these results can be seen in T-9. The authors also found that the two types had some chiral differences in the enantiomeric distribution of three monoterpenes

hydrocarbons. The sesquiterpene-rich type had the following enantiomeric distributions for the selected monoterpene hydrocarbons:

(1R,5R)-(+)- α -pinene (29-37%):(1S,5S)- α -pinene (63-71%)
 1R,5R)-(+)- β -pinene (0%):(1S,5S)- β -pinene (100%)
 1R,5R)-(+)-sabinene (79%):(1S,5S)-sabinene (21%)

In contrast, the enantiomeric distribution for the same monoterpene hydrocarbons in the chamazulene-rich type was as follows:

(1R,5R)-(+)- α -pinene (3-79%):(1S,5S)-(-)- α -pinene (21-97%)
 (1R,5R)-(+)- β -pinene (0-2%):(1S,5S)-(-)- β -pinene (98-100%)
 (1R,5R)-(+)-sabinene (3-15%):(1S,5S)-(-)-sabinene (85-97%)

Orth et al. (2000) proposed that the enantiomeric distribution of three hydrocarbons in the oil of yarrow could be useful in distinguishing between oils of different taxa. An example of their findings is presented in T-10.

Kalinka et al. (2000) examined the composition of oils produced from *Achillea* species collected from their natural habitats in Siberia. Among the oils examined was one of *A. millefolium*. This oil was found to possess the following composition:

α -pinene (2.67%)	β -cubebene (0.81%)
camphene (0.78%)	camphor (0.18%)
β -pinene + sabinene (28.35%)	α -terpineol (0.58%)
α -terpinene (2.33%)	β -caryophyllene (6.06%)
α -phellandrene (1.31%)	α -humulene (0.97%)
limonene (4.07%)	borneol (2.13%)
1,8-cineole (12.78%)	germacrene D (8.40%)
β -phellandrene (0.94%)	geranyl acetate (0.78%)
γ -terpinene (2.91%)	γ -cadinene (1.16%)
p-cymene (2.06%)	δ -cadinene (1.11%)
terpinolene (0.58%)	germacrenol* (0.05%)
achillenyl acetate (0.05%)	elemol (0.82%)
α -cubebene (t)	selin-11-en-4 α -ol (2.25%)
α -ylangene + β -elemene (0.34%)	caryophyllenol* (0.97%)
	chamazulene (5.14%)

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

Rohloff et al. (2000) analyzed the oils produced from *A. millefolium* cultivated from wild seeds in Norway. They compared the composition of oils produced in the vegetative stage, the early flowering stage and the full flowering stage. Unfortunately, the authors only presented graphical quantitative data so the actual amounts of α -pinene, sabinene, β -pinene, 1,8-cineole, δ -terpinene, linalool, α -thujone, β -thujone, camphor, borneol, terpinen-4-ol, α -terpineol, bornyl acetate, β -caryophyllene, α -humulene, an isomer of α -farnesene, (Z)-nerolidol, (E)-nerolidol and caryophyllene oxide could not be seen. However, it was possible to determine that the oil produced from plants harvested at the full flowering stage contained β -pinene (>16%) and α -thujone (ca. 10%). All of the other constituents were present at levels of less than 6%.

Percentage composition of two oil types of *Achillea millefolium* of Russian origin

T-9

Compound	Type 1 oil	Type 2 oil
α -pinene	0.3-1.3	0.2-0.5
α -thujene	0-0.3	0.1-0.2
β -pinene	2.3-18.6	0.5-1.3
sabinene	0.2-7.5	7.2-17.9
myrcene	0-1.3	0.1-0.2
α -terpinene	0-0.2	0.3-0.5
limonene	0-0.2	0.1
1,8-cineole	0.2-2.1	5.3-6.7
(Z)- β -ocimene	0-0.3	-
γ -terpinene	0-0.5	0.7-0.8
(E)- β -ocimene	0-0.5	0-0.1
p-cymene	0-0.1	-
terpinolene	0-0.1	0.1-0.2
α -cubebene	0-0.1	-
camphor	0-0.3	-
β -bourbonene	0-0.3	0-0.1
1-nonen-3-ol	0-0.2	-
linalyl acetate	-	0.1-0.2
bornyl acetate	0-0.8	-
β -caryophyllene	1.5-11.7	38.6-45.5
terpinen-4-ol	0.4-1.3	3.3-3.7
myrtenal	0-0.1	-
α -humulene	0.2-1.5	4.0-4.8
β -farnesene*	0-0.1	0.1
γ -muurolene	0-0.1	0-0.1
borneol + α -terpineol	0-0.2	-
germacrene D	4.8-7.9	13.9-14.1
(Z,E)- α -farnesene	-	0-0.1
bicyclogermacrene	0.2-1.4	0.5
(E,E)- α -farnesene	0-0.6	0-0.7
δ -cadinene	0.1-0.4	0.3-0.6
β -sesquiphellandrene	0-0.4	-
ar-curcumene	0-0.2	-
myrtenol	0-0.1	-
caryophyllene oxide	0.4-2.8	3.4-4.0
(Z)-nerolidol	-	0-0.1
germacradienol*	0-0.2	0.1
nerolidol*	0-0.5	-
T-muurolol	0-0.2	0.2
α -cadinol	0.1-0.5	0.6
chamazulene	46.2-73.5	0.2-0.5
dimethylvinyl azulene	1.0-2.1	-
phytol	0-0.1	0.1-0.2

Type 1: chamazulene-rich

Type 2: sesquiterpene hydrocarbon-rich

An oil produced from *A. millefolium* harvested in Bulgaria from the wild state was the subject of analysis by Todorova et al. (2001). In this oil the following constituents were identified:

p-cymene (5.5%)	<i>trans</i> -sabinene hydrate (t)
1,8-cineole (36.7%)	<i>cis</i> -sabinene hydrate (t)
artemisia ketone (t)	chrysanthenone (3.5%)

Enantiomeric distribution (%) of three monoterpene hydrocarbons in four oils of different origin

T-10

Enantiomer	1 ^a	2	3	4
(1R,5R)-(+)- α -pinene	18	23	73	87
(1S,5S)-(-)- α -pinene	82	77	27	13
(1R,5R)-(+)- β -pinene	1	1	-	10
(1S,5S)-(-)- β -pinene	99	99	-	90
(1R,5R)-(+)-sabinene	13	23	91	4
(1S,5S)-(-)-sabinene	87	77	9	96

^anumbers relate to oil originComparative percentage composition of *Achillea millefolium* oils of Estonian origin

T-11

Compound	1 (2 ^a)	2 (3)	3 (1)
hexenal	0.1	0.2-0.3	0.1
(E)-2-hexenal	0.1-0.2	0.3	0.3
(Z)-3-hexenol	t-0.1	0.1-0.2	t
tricyclene	0.1	0.1	t
α -thujene	0.3	0.3-0.4	0.1
α -pinene	3.1-8.4	4.0-4.4	6.8
camphene	0.9-2.0	0.9-1.8	0.9
sabinene	8.3	8.7-17.6	2.9
β -pinene	14.9-21.8	16.9-19.9	29.2
1-octen-3-ol	t-0.1	0.2	0.1
myrcene	0.4-0.5	0.2-0.4	0.3
α -terpinene	0.5-0.9	0.7-0.9	0.4
p-cymene	0.6	0.2-1.5	0.8
limonene	0.5-0.8	0.5-0.7	1.0
1,8-cineole	6.9-8.3	13.0-18.3	11.4
(Z)- β -ocimene	0.1	0.1	0.1
(E)- β -ocimene	0.2-0.3	0.1	0.2
γ -terpinene	1.1-1.4	2.9-3.1	1.0
terpinolene	0.1-0.2	0.3	0.1
<i>cis</i> -sabinene hydrate	0.2	0.1-0.2	0.1
linalool	0-t	-	-
β -terpineol*	t-0.1	0.1-0.2	0.1
camphor	1.2-4.3	2.8-4.4	1.1
<i>cis</i> -sabinol	0.2-0.3	0.2	0.2
myrtenal	0.2-0.4	0.2	0.3
borneol	0.6-1.1	1.1-2.1	2.2
terpinen-4-ol	0.7-0.8	1.9-2.4	0.7
myrtenol	0.1	0.3	0.4
α -terpineol	0.7-2.3	1.8-2.2	1.3
piperitone	-	0-2.9	-
bornyl acetate	0.5-2.0	0.1-0.6	0.5
thymol	t-0.1	t-0.3	0.6
sabinyol acetate*	0-0.1	0-0.1	0.4
α -terpinyl acetate	0-0.2	0.1	-
geranyl acetate	0.1-0.2	0-0.1	0.1
α -copaene	0.1-0.3	0.1-0.2	0.2
β -caryophyllene	3.4-5.4	3.3-4.3	6.2
α -humulene	0.5-0.7	0.5-0.7	0.9
allo-aromadendrene	t-0.9	t-0.4	0.3
germacrene D + γ -muurolene	2.7-3.1	0.9-1.6	2.3
α -muurolene + (Z,Z)- α -farnesene + (Z,E)- α -farnesene	0.5	0.2-0.3	0.4

Comparative percentage composition of *Achillea millefolium* oils of Estonian origin
(continued)

T-11

Compound	1 (2 ^a)	2 (3)	3 (1)
(E,E)- α -farnesene + β -bisabolene	0.1-0.5	-	0.1
γ -cadinene	0.3-0.8	0.2-0.5	0.4
δ -cadinene + cuparene	1.0-1.5	0.3-0.4	0.2
(E)-nerolidol	0.5-4.7	1.2-1.9	6.4
caryophyllene oxide	1.0-1.2	1.0-2.4	4.1
viridiflorol	0.1	0.1-0.2	0.4
ledol	0.2	0.2	0.4
guaiol	2.1-11.8	1.0-5.3	0.3
α -muurolol + T-cadinol	0.5	1.1-1.4	1.6
T-muurolol + α -cadinol	0.1	0.2	0.1
caryophyllenol*	0.1-0.4	0.2-0.8	1.5
farnesol* + farnesol*	0.1-1.9	0.2-1.1	0.6
α -bisabolol + (Z,E)-farnesol	0.7-1.2	1.3-2.6	0.7
(E,E)-farnesol	0-1.3	0-0.1	-
chamazulene	12.7-13.3	0.1-3.3	2.2

^anumber of samples
t = trace (<0.1%)
*correct isomer not identified

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trans-sabinol (0.7%)
camphor (8.5%)
sabina ketone (1.0%)
pinocarvone (1.0%)
borneol (2.7%)
terpinen-4-ol (1.0%)
p-cymen-8-ol (1.2%)
 α -terpineol (2.1%)
myrtenal (0.7%)
2-hydroxy-1,8-cineole
(0.6%)

cis-sabinene hydrate acetate
(12.8%)
trans-sabinyl acetate (t)
chavicol (t)
bornyl acetate (0.7%)
cumyl alcohol (1.3%)
ascaridole (2.4%)
eucarvone (1.4%)
p-mentha-1,4-dien-7-ol (t)
(Z)-jasmone (1.4%)
caryophyllene oxide (t)

t = trace (<0.1%)

Oils produced from six samples of *A. millefolium* growing wild in Estonia were analyzed by Orav et al. (2001) via GC and GC/MS and grouped them into

three chemical categories. The data obtained from these analyses is shown in T-11. The authors also compared the compositions of oils produced from the flowers, leaves and stems of two different *A. millefolium* taxa. The results of this study can be found in T-12.

Cornu et al. (2001) collected a series of wild plants found in the summer pastures in central France in June and September at an altitude of 1200 m. The plants were subjected to SPME headspace analysis with GC/MS and retention index component identification. Among the plants collected was *A. millefolium*. A summary of the headspace analysis of this plant over two harvesting periods can be seen in T-13.

Comparative percentage composition of the flower, leaf and stem oils of two taxa of *Achillea millefolium*

T-12

Compound	Sample 1			Sample 2		
	Flower oil	Leaf oil	Stem oil	Flower oil	Leaf oil	Stem oil
tricyclene	t	0.1	t	t	t	t
α -thujene	0.2	0.2	t	t	0.1	0.1
α -pinene	3.8	4.2	3.7	6.7	8.1	6.6
camphene	0.4	1.4	1.2	0.1	0.1	0.2
sabinene	3.4	2.6	1.5	0.7	1.2	0.6
β -pinene	37.1	35.9	34.6	28.9	30.3	29.4
myrcene	0.1	0.1	t	0.3	0.4	0.2
α -terpinene	0.3	0.3	0.4	0.1	0.1	0.1
p-cymene	0.8	1.0	1.5	0.4	1.6	0.8
1,8-cineole	13.5	11.4	5.4	1.7	4.9	1.5
limonene	1.1	3.7	7.3	0.8	2.7	2.2
(Z)- β -ocimene	0.2	0.2	t	0.1	0.1	0.1
(E)- β -ocimene	0.2	0.1	t	0.2	0.2	0.4
γ -terpinene	1.0	0.7	0.6	1.0	0.5	0.3
terpinolene	0.2	0.2	t	0.1	0.1	0.1
linalool	0.1	0.2	t	0.2	t	0.3
camphor	0.4	1.5	1.9	1.0	1.3	1.0
myrtenal	0.2	0.3	1.2	0.3	1.4	1.2
borneol	0.5	1.7	1.7	t	0.2	0.4
terpinen-4-ol	0.6	0.6	0.6	0.1	0.2	t
myrtenol	0.2	0.3	1.0	0.1	1.1	0.9
α -terpineol	1.3	1.1	0.6	0.3	0.3	0.2
α -copaene	0.1	0.2	0.1	0.2	0.2	0.6
β -caryophyllene	7.1	2.7	1.2	9.3	2.5	0.1
α -humulene	1.1	0.3	0.3	1.2	0.4	0.3
germacrene D + γ -muurolene	3.9	0.4	0.1	3.6	0.2	0.3
α -muurolene	0.4	0.1	-	0.6	0.1	0.1
γ -cadinene	0.9	0.6	0.2	0.5	0.1	0.1
δ -cadinene	0.2	t	t	0.3	0.1	t
(E)-nerolidol	1.8	1.3	1.7	t	0.3	0.2
caryophyllene oxide	2.1	2.9	2.5	1.3	4.1	3.1
viridiflorol	0.3	0.3	0.7	0.1	0.3	0.1
ledol	0.2	0.3	0.3	0.1	0.6	0.2
α -muurolol	0.8	0.4	-	0.3	0.4	0.2
α -cadinol	0.7	0.2	-	0.3	0.4	0.2
caryophyllenol*	0.1	0.3	-	0.1	0.2	0.1
(Z,Z)-farnesol	0.4	0.3	-	0.1	-	0.6
α -bisabolol	0.7	0.3	0.3	0.3	0.2	0.4
chamazulene	0.4	0.2	0.1	31.5	6.6	3.8

t = trace (<0.1%)

* correct isomer not identified

Ahmed et al. (2001) used GC/MS to analyze oils produced from both the leaves and stems of *A. millefolium* grown in an experimental farm in Cairo (Egypt). The composition of the leaf oil was as follows:

octane (0.28%)	1-octen-3-ol (0.74%)
3-hexenol* (0.49%)	hexenyl acetate* (1.34%)
nonane (0.58%)	α -terpinene (0.60%)
α -thujene (0.20%)	p-cymene (0.66%)
α -pinene (0.40%)	1,8-cineole (3.25%)
sabinene (20.70%)	(Z)- β -ocimene (0.20%)

(E)- β -ocimene (0.41%)
γ -terpinene (1.23%)
cis-p-menth-2-en-1-ol (1.70%)
nonanone* (0.43%)
trans-p-menth-2-en-1-ol (0.77%)
pinene hydrate* (0.19%)
camphor (0.28%)
borneol (0.58%)
terpinen-4-ol (2.09%)
α -terpineol (0.48%)
decanal (0.30%)
δ -elemene (0.22%)

α -cubebene (5.77%)
eugenol (0.57%)
ylangene* (0.21%)
patchoulene* (0.89%)
bourbonene* (0.27%)
β -elemene (0.45%)
jasmone* (0.59%)
β -caryophyllene (1.79%)
α -humulene (0.27%)
β -gurjunene (2.10%)
α -cadinene** (0.95%)
germacrene D (7.43%)

germacrene B** (6.03%)	patchouli alcohol (0.25%)
α -farnesene* (0.20%)	isocedranol* (3.13%)
γ -cadinene (0.32%)	germacrone (0.42%)
δ -cadinene (2.30%)	(Z,E)-farnesol (0.23%)
(Z)-nerolidol (0.50%)	(Z,Z)-farnesol (0.21%)
(E)-nerolidol (3.28%)	phytol (0.08%)
elemenone* (6.40%)	dibutyl phthalate*** (0.09%)
cedranone* (1.26%)	isophytol (0.10%)
cubenol (0.43%)	docosane (0.07%)
γ -eudesmol (1.37%)	abietol (0.06%)
α -eudesmol (8.18%)	neobietol (0.18%)
α -cadinol (4.62%)	

*correct isomer not identified

**incorrect identification based on elution order

***this is a plasticizer, not a naturally occurring component

In contrast, the stem oil was found to possess the following composition:

octane (0.14%)	α -eudesmol (19.71%)
sabinene (0.20%)	α -cadinol (4.30%)
<i>cis</i> -p-menth-2-en-1-ol (0.06%)	neocedran-5-ol (10.69%)
<i>trans</i> -p-menth-2-en-1-ol (0.07%)	(Z,E)-farnesol (6.37%)
borneol (0.05%)	(Z,Z)-farnesol (0.12%)
terpinen-4-ol (0.18%)	(E,Z)-farnesol (0.12%)
α -terpineol (0.10%)	hexadecanol (0.23%)
δ -elemene (0.44%)	cedrane-8,14-diol (0.22%)
cedrene* (1.24%)	cedrane-8,13-diol (0.23%)
β -caryophyllene (0.44%)	phytol (0.10%)
α -humulene (1.36%)	abietatriene (0.06%)
germacrene D (28.50%)	octadecanol (0.33%)
germacrene B** (8.44%)	isophytol (0.17%)
α -farnesene* (0.10%)	henicosane (0.18%)
δ -cadinene (2.66%)	docosane (0.61%)
(E)-nerolidol (0.88%)	tetracosane (0.62%)
germacrene D-4-ol (5.04%)	hexanedioic acid (0.12%)
γ -eudesmol (5.36%)	pentacosane (0.70%)
α -eudesmol (19.71%)	

*correct isomer not identified

**incorrect identification based on elution order

An oil produced from *A. millefolium* plants that were growing wild in a field station in Pulwama, Jammu and Kashmir (India) and collected during the flowering phase was the subject of analysis by Shawl et al. (2002). The composition of this oil was found to be as follows:

(Z)-3-hexenol (0.3%)	<i>trans</i> -sabinene hydrate (0.1%)
(E)-2-hexenol (0.1%)	<i>cis</i> -linalool oxide* (0.1%)
α -thujene (0.1%)	<i>trans</i> -linalool oxide* (t)
α -pinene (0.7%)	linalool (t)
camphene (1.2%)	<i>cis</i> -sabinene hydrate (0.4%)
sabinene (2.0%)	isochrysanthenone (t)
β -pinene (0.5%)	α -thujone (0.2%)
dehydro-1,8-cineole (t)	<i>trans</i> -p-menth-2-en-1-ol** (0.1%)
myrcene (0.2%)	camphor (28.4%)
p-cymene (0.5%)	<i>cis</i> -p-menth-2-en-1-ol** (0.1%)
1,8-cineole (11.5%)	pinocarvone (0.3%)
(Z)- β -ocimene (0.1%)	isoborneol (0.1%)
benzyl alcohol (t)	borneol (2.9%)
(E)- β -ocimene (0.1%)	terpinen-4-ol (1.9%)
γ -terpinene (0.4%)	myrtenal (0.2%)

Comparative percentage composition of the headspace of *Achillea millefolium* over two harvesting times

T-13

Compound	July harvest	September harvest
1,3,6-trimethylheptatriene	0.3	1.5
tricyclene	0.4	0.5
α -pinene	t	t
camphene	2.7	3.1
sabinene	6.3	5.2
β -pinene	4.1	4.9
myrcene	0.1	t
α -phellandrene	1.6	-
p-cymene	6.5	12.0
1,8-cineole	14.0	7.0
(Z)- β -ocimene	-	t
γ -terpinene	1.5	2.2
p-menth-2-en-1-ol*	1.1	1.1
terpinolene	0.3	0.2
β -thujone	-	1.8
camphor	3.5	7.2
isoborneol	0.9	0.4
borneol	2.2	0.9
α -terpineol	0.4	0.4
bornyl acetate	0.6	0.8
δ -elemene	0.5	0.5
α -copaene	0.9	0.6
β -caryophyllene	39.0	26.0
<i>trans</i> - α -bergamotene	0.4	0.3
α -humulene	-	0.3
(E)- β -farnesene	5.0	2.0
allo-aromadendrene	0.8	0.6
germacrene D	8.0	9.1
bicyclogermacrene	2.0	1.4
(E,E)- α -farnesene	0.7	1.3
γ -cadinene	0.4	-
sesquicineole	-	10.0
(E)- β -farnesene**	2.5	1.7
(E)-nerolidol	2.3	1.0
spathulenol	0.5	0.2
caryophyllene oxide	1.3	0.5

*correct isomer not identified

**incorrect identification based on elution order and the fact that (E)- β -farnesene had already been identified in the headspace volatiles

t = trace (<0.01%)

α -terpineol (0.9%)	linalyl propionate (0.2%)
myrtenol (0.3%)	<i>cis</i> -carvyl acetate** (t)
verbenone (0.1%)	eugenol (0.1%)
<i>trans</i> -carveol (0.3%)	thymyl acetate (0.2%)
cuminaldehyde (t)	α -cubebene (0.4%)
<i>cis</i> -carveol (0.1%)	(Z)-jasmone (0.1%)
pulegone (4.4%)	α -copaene (0.1%)
piperitone (1.6%)	β -cubebene (0.1%)
<i>cis</i> -chrysanthenyl acetate (7.6%)	β -caryophyllene (1.1%)
bornyl acetate (0.2%)	α -bergamotene*** (0.1%)
thymol (0.1%)	(Z)- β -farnesene (4.0%)
	geranyl propionate (0.3%)

Percentage composition of the oils produced from three distinct chemical groups of *Achillea millefolium*

T-14

Compound	Group 1	Group 2	Group 3
α -pinene	3.0-7.4	3.4-5.3	2.1-7.1
camphene	t-2.7	0-1.4	0.6-2.8
sabinene	3.3-14.0	3.4-5.2	2.0-8.5
β -pinene	11.8-31.1	9.7-30.2	5.5-21.3
myrcene	0.2-2.1	0.9-2.2	0.5-1.1
α -terpinene	t-1.2	0-0.7	0.5-1.4
p-cymene	0-1.6	t-1.9	0.7-1.3
1,8-cineole	8.1-17.0	5.3-16.0	4.4-9.0
γ -terpinene	0.3-3.4	0.2-1.7	1.1-3.8
terpinolene	0-0.6	0-t	0-0.9
<i>trans</i> -pinocarveol	-	0-0.7	0-0.4
camphor	t-3.4	0.3-1.8	1.5-4.9
<i>cis</i> -chrysanthenol	0-0.2	0-1.5	0.2-0.4
borneol	t-4.1	0.4-3.8	3.1-6.5
terpinen-4-ol	0.9-3.4	1.2-2.1	1.2-5.5
α -terpineol	0.2-3.0	0.6-2.8	t-1.3
<i>cis</i> -chrysanthenyl acetate	-	0-1.7	0-3.8
bornyl acetate	0.2-4.0	0-1.2	0.3-4.9
α -cubebene	0-0.6	0-0.2	0-0.4
β -bourbonene	0-0.7	0-0.2	0-0.2
β -caryophyllene	3.5-5.9	4.5-8.7	3.4-5.0
α -humulene	t-1.5	0.3-1.4	0-0.9
germacrene D	1.1-4.2	1.9-4.9	1.4-3.8
bicyclogermacrene	0.3-1.0	0-1.2	0.3-0.7
α -muurolene	0-0.4	0-0.7	0-0.4
β -bisabolene	0-0.6	0-0.5	0-0.4
sesquicineole	0-0.4	0-0.4	t-1.8
δ -cadinene	0.6-2.4	0.6-2.4	0.7-2.5
(E)-nerolidol	0.5-7.8	0-5.8	8.2-13.5
spathulenol	0.5-2.4	0.5-1.5	0.3-1.8
caryophyllene oxide	1.4-8.0	1.1-5.4	2.5-5.3
viridiflorol	0-5.1	0-6.5	0.9-3.1
humulene epoxide II	0-0.6	0-0.6	0-0.5
10-epi- γ -eudesmol	0.3-11.0	0-1.6	1.2-8.3
1-epi-cubenol*	0.3-1.9	0-2.2	0.6-2.1
T-cadinol	0-1.2	t-1.0	0.6-1.4
himachalol**	0-1.9	0-3.5	0-1.5
selin-11-en-4 α -ol	0.5-3.9	0-2.2	1.2-7.0
14-hydroxy-9-epi- β -caryophyllene	0.3-1.7	0.4-1.3	0.4-1.7
α -bisabolone oxide A	0-2.2	0-0.3	0.3-3.1
(E,E)-farnesol	0-1.7	0-0.5	0-2.8
chamazulene	0-6.8	9.8-23.2	0-0.1

Group 1: β -pinene/1,8-cineole-types
Group 2: chamazulene-types
Group 3: (E)-nerolidol-types
*tentative identification
t = trace (<0.1%)

γ -muurolene (0.1%)
germacrene D (11.5%)
germacrene*** (1.9%)
bicyclogermacrene (1.1%)
 α -muurolene (0.3%)
(Z)- α -bisabolene (0.2%)
 β -bisabolene (1.1%)

δ -cadinene (0.3%)
 α -cadinene (0.2%)
 α -calacorene (0.1%)
(E)-nerolidol (0.5%)
spathulenol (0.8%)
caryophyllene oxide (0.2%)

humulene epoxide I (0.1%)
humulene epoxide II (0.3%)
hexadecane (0.7%)
 α -muurolol (0.5%)
T-cadinol (0.3%)
 β -eudesmol (0.5%)
 α -eudesmol (0.1%)

α -cadinol (0.2%)
 α -bisabolol (0.3%)
epi- α -bisabolol (0.1%)
heptadecane (0.1%)
(E,E)-farnesol (t)
octadecane (0.2%)
hexaacanal (t)

pentadecanoic acid (0.1%) (E,E)-farnesyl acetone (0.1%)
2-heptadecanone (0.1%) oleic acid (0.1%)
nonadecane (t)

*furanoid form

***cis*- and *trans*- forms should be interchanged

***correct isomer not identified

Mockute and Judzentiene (2002) collected naturally growing *A. millefolium* plants from nine localities in the vicinity of Vilnius (Lithuania). Oils produced from plants from each of the locations were subjected to GC and GC/MS analysis. The authors categorized the oils into three groups; a β -pinene/1,8-cineole group, a chamazulene group and a nerolidol group. The compositional range of each group can be found in T-14. In a follow-up report, the same authors (Mockute and Judzentiene, 2002) analyzed oils produced from 20 samples of *A. millefolium* that were collected from 11 different habitats in eastern Lithuania. On examination of their data, the authors classified the oils into six chemotypic forms as shown in T-15. It is interesting to note that many of the samples (eight of 20) did not contain any chamazulene. Purchasers of yarrow oil should always obtain a sample of the oil before committing to buy it because of its chemical variance and the fact that the plants collected for distillation could be of mixed chemotypes resulting in an unstable composition from year to year.

- Z.Y. Daw, G.E. El-Baroty and A.M. Ebtesam, *Inhibition of Aspergillus parasiticus growth and aflatoxin production by some essential oils*. Chem. Mikrobiol. Technol. Lebensmitt., **16**(5/6), 129-135 (1994).
- U. Kastner, J. Jurenitsch, S. Glasl, B. Follrich, A. Gavanelli, H. Schroder, M. Schubert-Zsilavec, W. Schmidt, E. Haslinger and W. Kubelka, *Longipinen und Achillifolin derivate aus Achillea millefolium—Type "DISA."* Pharmazie, **51**, 503-505 (1996).
- J.A. Pino, A. Rosado and V. Fuentes, *Chemical composition of the leaf oil of Achillea millefolium L. grown in Cuba*. J. Essent. Oil Res., **10**, 427-428 (1998).
- J.-C. Chalchat, M.S. Gorunovic and S.D. Petrovic, *Aromatic Plants of Yugoslavia. I. Chemical composition of oils of Achillea millefolium L. ssp. pannonica (Scheele) Hayak, A. crithmifolia W. et K., A. serbica Nym. and A. tanacetifolia All.* J. Essent. Oil Res., **11**, 306-310 (1999).
- M. Orth, F.-C. Czygan and V.P. Dedkov, *Variation in essential oil composition and chiral monoterpenes of Achillea millefolium s.l. from Kalingrad*. J. Essent. Oil Res., **11**, 681-687 (1999).
- M. Orth, D. Juchelka, A. Mosandl and F.-Ch. Czygan, *Enantiomer Monoterpene in ätherischen Öl von Achillea millefolium s.l.—eine zusätzliche taxonomische Bestimmungshilfe*. Pharmazie, **55**, 456-459 (2000).
- G.I. Kalinka, A.D. Dembitsky and T.P. Berezovskaya, *Chemical composition of essential oils of Achillea species from Siberia*. Khim. Raslit. Syr'ya, (3), 3-18 (2000).
- J. Rohloff, E.B. Skagen, A.H. Steen and T.-H. Iversen, *Production of yarrow (Achillea millefolium L.) in Norway: Essential oil content and quality*. J. Agric. Food Chem., **48**, 6205-6209 (2000).
- M. Todorova, A. Konakchiev and E. Tsankova, *Chemical composition of the essential oils of three Achillea species growing in Bulgaria*. Dokl. Bulgar. Akad. Naak., **54**(6), 25-28 (2001).

Percentage composition of the oils of six chemotypes of *Achillea millefolium* of eastern Lithuanian origin

T-15

Compound	1	2	3	4	5	6
α -pinene	2.2-4.8	1.9-6.3	2.3-4.7	3.7	2.3-2.7	4.6
camphene	0.8-2.3	0.4-1.8	1.1-1.7	2.5	0.9	-
sabinene	0.9-13.0	2.5-4.5	1.5-4.3	3.6	5.4-7.1	3.2
β -pinene	10.2-17.2	6.6-9.5	4.0-12.6	4.5	7.0-9.1	15.1
myrcene	0.5-7.8	0.4-1.1	0.3-0.8	0.6	0.2-1.2	0.9
α -terpinene	t-1.9	0.2-0.6	t-0.7	0.4	0.5-0.6	0.5
p-cymene	0.5-4.0	0.8-2.3	1.0-1.5	0.7	0.7	0.3
1,8-cineole	6.4-11.8	8.8-9.9	5.3-12.5	8.8	4.5-6.4	6.4
γ -terpinene	0-3.3	0.8-2.2	0.2-2.1	1.3	1.5-1.9	0.4
terpinolene	t-0.6	0.1-0.5	0-0.4	0.4	0.3-0.4	0.3
<i>trans</i> -pinocarveol	0-1.1	0-t	0-0.3	-	-	0.7
camphor	0.6-6.6	2.0-5.9	4.1-7.2	13.1	0.9-1.8	0.5
<i>cis</i> -chrysanthenol	0-0.9	2.0-2.4	0.3-3.8	0.4	0-1.2	t
borneol	0.4-8.0	2.0-2.5	11.5-13.2	12.8	2.7-3.7	1.2
terpinen-4-ol	0.9-7.6	1.3-1.9	1.9-4.5	2.2	1.6-2.0	2.3
α -terpineol	0.6-3.0	0.5-0.6	1.4-2.2	0.5	0.4-0.6	2.1
<i>cis</i> -chrysanthenyl acetate	0-t	-	0.8-2.8	0.9	-	-
bornyl acetate	0.6-3.6	1.6-4.7	0.5-1.9	0.7	1.6-2.2	t
α -cubebene	0.1-0.5	0-0.3	0-0.8	0.1	t-0.2	0.2
β -bourbonene	0-0.4	t-0.2	t-0.2	-	0-0.2	0.4
β -caryophyllene	1.5-7.5	2.6-6.5	1.9-3.5	0.7	3.2-5.5	8.0
α -humulene	0.1-1.3	0.4-0.5	0.3-0.7	0.2	0.3-0.4	1.5
germacrene D	0.3-6.3	2.5-3.6	0.9-1.8	0.3	2.5-2.7	4.6
bicyclogermacrene	0.1-1.2	0.2-0.5	0.2-0.4	0.4	0.4-0.6	0.4
α -muurolene	0-0.4	0-0.2	0-0.3	-	0.3-0.5	-
β -bisabolene	0-0.7	0-0.4	0.3	-	0.5-1.2	0.1
sesquicineole	0-1.2	0.2-0.3	0.2	0.1	0.1-0.4	0.4
δ -cadinene	0.7-2.8	0.6-1.3	0.5-0.6	0.3	1.5-1.9	2.1
(E)-nerolidol	2.6-7.8	2.6-6.4	4.6-4.8	2.6	8.5-9.3	6.4
spathulenol	0.5-5.7	0.5-3.3	0.5-1.3	t	0.7-2.7	-
caryophyllene oxide	1.6-7.1	2.8-6.3	1.9-4.7	3.5	2.8-6.0	2.5
viridiflorol	0.4-2.5	1.5-1.8	0.7-1.5	0.2	1.8-5.1	1.0
humulene epoxide II	0-1.1	0-t	-	1.4	0.7-1.1	0.4
10-epi- γ -eudesmol	t-5.5	1.0-2.5	0.9-1.8	1.2	0.5-1.5	t
1-epi-cubenol*	t-2.6	3.1-7.6	0.6-1.5	1.9	1.6-3.0	2.1
T-cadinol	0.3-1.8	0.6-1.1	0.3-0.9	t	0.9-1.6	0.4
himachalol*	0-2.1	0.5-1.1	0.8-2.7	2.1	1.4-2.5	1.1
selin-11-en-4 α -ol	0.8-4.7	2.4-5.9	2.2-4.2	3.6	3.0-5.9	2.0
14-hydroxy-9-epi- β -caryophyllene	0.4-3.8	1.1-1.8	0.3-1.5	1.2	0.2-1.2	1.1
α -bisabolone oxide A	0-2.7	0-4.1	0-1.5	0.6	0.6-5.9	1.1
(E,E)-farnesol	0-0.6	0.2-0.5	0.3-0.8	t	0.7-1.7	-
chamazulene	0-4.8	t-1.2	0-1.3	-	0-0.9	20.1

1 = β -pinene-types (10) 4 = camphor-type (1)
2 = 1,8-cineole-types (3) 5 = (E)-nerolidol-types (2)
3 = borneol-types (3) 6 = chamazulene-type (1)

*tentative identification
t = trace (<0.1%)

A. Orav, T. Kailas and K. Ivask, *Composition of the essential oil from Achillea millefolium L. from Estonia*. J. Essent. Oil Res., **13**, 290-294 (2001).

A. Cornu, A.-P. Carnata, B. Martin, J.-B. Coulon, J.-L. Lamaison and J.-L. Berdague, *Solid-phase microextraction of volatile components from natural grassland plants*. J. Agric. Food Chem., **49**, 203-209 (2001).

Kh.M. Ahmed, S.S. El-Din, S.A. Wahab and E.A.M. El-Krisy, *Study of the coumarin and volatile oil composition from aerial parts of Achillea millefolium L.* Pak. J. Sci. Ind. Res., **44**, 218-222 (2001).

A.S. Shawl, S.K. Srivastava, K.V. Syamasundai, S. Tripathi and V.K. Raina, *Essential oil composition of Achillea millefolium L. growing wild in Kashmir, India*. Flav. Fragr. J., **17**, 165-168 (2002).

D. Mockute and A. Judzentiene, *Chemical composition of the essential oils of Achillea millefolium L. ssp. millefolium (yarrow) growing wild in Vilnius*. *Chemija*, **13**, 97-102 (2002).

D. Mockute and A. Judzentiene, *Chemotypes of the essential oils of Achillea millefolium L. ssp. millefolium growing wild in eastern Lithuania*. *Chemija*, **13**, 168-173 (2002).

Star Anise Oil

In 1990, Kreis et al. used enantiomeric separation on a chiral capillary GC column to determine that the enantiomeric ratios of α -pinene, β -pinene and limonene in star anise oil was found to be:

(1S,5S)-(-)- α -pinene (4-7%) : (1R,5R)-(+)- α -pinene (93-96%)
 (1S,5S)-(-)- β -pinene (26-34%) : (1R,5R)-(+)- β -pinene (66-74%)
 (4S)-(-)-limonene (55-62%) : (4R)-(+)-limonene (38-45%)

Wang et al. (1995) used multidimensional GC on a non-polar column coupled to a chiral phase column to determine that the enantiomeric ratio of limonene in star anise oil was: (4R)-(+)-limonene (39.2%) : (4S)-(-)-limonene (60.8%).

Also in 1995, Ravid et al. determined that the enantiomeric distribution of α -terpineol (0.3%) in a commercial sample of star anise oil was as follows:

(4S)-(+)- α -terpineol (57%) : (4R)-(-)- α -terpineol (43%)

Star anise oil produced in Dongxing (China), which was analyzed by GC/MS (Zhu et al. 1993), was found to contain:

limonene (0.21%)	β -caryophyllene (0.26%)
linalool (1.06%)	(E)-anethole (91.28%)
methyl chavicol (3.46%)	α -bergamotene* (0.25%)
p-anisaldehyde (0.26%)	

*correct isomer not given

Xie and Xu (1997) reported the results of an analysis of a market sample of star anise oil from Guangxi. Using GC/MS as their method of analysis, the following components were identified:

α -pinene (0.24%)	methyl chavicol (4.06%)
β -pinene (0.02%)	(Z)-anethole (0.25%)
camphene (0.03%)	(E)-anethole (92.03%)
myrcene (0.03%)	β -caryophyllene (0.18%)
α -phellandrene (0.35%)	anisaldehyde (0.02%)
β -phellandrene (0.51%)	eudesmene* (0.05%)
limonene (1.43%)	foueniculin (0.34%)

*correct isomer not identified

Using the conditions of 90 bar, 50°C and an extraction time of 510 min, Della Porta et al. (1998) produced a supercritical extract of star anise oil and subjected it to analysis from which the following constituents were identified:

α -thujene (t)	terpinen-4-ol (0.20%)
α -pinene (0.09%)	α -terpineol (0.09%)
β -pinene (0.03%)	methyl chavicol (1.45%)
myrcene (0.04%)	(Z)-anethole (0.15%)
α -phellandrene (0.02%)	(E)-anethole (94.05%)
δ -3-carene (0.09%)	linalyl acetate (0.11%)
α -terpinene (0.01%)	α -cubebene (0.21%)
p-cymene (0.04%)	β -elemene (0.01%)
limonene (1.74%)	β -caryophyllene (0.53%)
(Z)- β -ocimene (t)	<i>trans</i> - α -bergamotene (0.72%)
γ -terpinene (0.06%)	α -humulene (0.02%)
terpinolene (0.02%)	(Z)-b-farnesene (0.01%)
linalool (0.31%)	

t = trace (<0.01%)

If the authors had used the following conditions 300 bar, 50°C and an extraction time of 180 min, then the extract would have had the following composition:

(E)-anethole (60.53%)	methyl linoleate (0.54%)
β -caryophyllene (0.36%)	hencicosane (3.15%)
<i>trans</i> - α -bergamotene (0.53%)	docosane (7.10%)
δ -cadinene (0.11%)	tricosane (7.37%)
spathulenol (0.15%)	tetracosane (4.27%)
α -muurolol (0.07%)	pentacosane (1.70%)
α -cadinol (0.46%)	hexacosane (1.92%)
(E,Z)-farnesol (2.11%)	heptacosane (0.34%)
palmitic acid (0.33%)	octacosane (t)
methyl palmitate (0.80%)	nonacosane (t)

t = trace (<0.01%)

More recently, Kubeczka and Formacek (2002) compared the compositions of star anise oil of Chinese and Vietnamese origins. They used a combination of GC and ¹³C-NMR to perform these analyses, the results of which can be seen in T-16.

- P. Kreis, U. Hener and A. Mosandl, *Chirale Inhaltstoffe ätherischer Öle. III. Stereodifferenzierung von α -pinene, β -pinene and limonen in ätherischen Ölen, Drogen und Fertigarzneimitteln*. Deut. Apoth. Ztg., **130**, 985-988 (1990).
- X-H. Wang, C-R. Jia and H. Wan, *The direct chiral separation of some optically active compounds in essential oils by multidimensional gas chromatography*. J. Chromatogr. Sci., **33**, 22-25 (1995).
- U. Ravid, E. Putievsky and I. Katzir, *Determination of the enantiomeric composition of α -terpineol in essential oils*. Flav. Fragr. J., **10**, 281-284 (1995).
- L-F. Zhu, Y-H. Li, B-L. Li, B-Y. Lu and N-H. Xio, *Aromatic plants and essential constituents*. South China Institute of Botany, Chinese academy of Sciences, Hai Feng Publish. Co. distributed by Peace Book Co., Ltd., Hong Kong (1993).
- L. Xie and S-Y. Xu, *Analysis of star anise oil using GC/MS*. Zhongguo Youzhi, **22**(5), 43-45 (1997).
- G. Della Porta, R. Taddeo, E. D'Urso and E. Reverchon, *Isolation of clove bud and star anise essential oil by supercritical CO₂ extraction*. Lebensm.-Wiss. Technol., **31**, 454-460 (1998).
- K-H. Kubeczka and V. Formacek, *Essential oils analysis by capillary gas chromatography and carbon-13 NMR spectroscopy*. 2nd Edn., 339-347, J. Wiley & Sons, NY (2002). ■

Comparative percentage composition of Chinese and Vietnamese star anise oil

T-16

Compound	Chinese oil	Vietnamese oil
α -pinene	0.62	2.07
β -pinene	0.05	0.22
sabinene	-	0.24
δ -3-carene	0.24	0.93
myrcene	0.08	0.40
α -phellandrene	0.44	0.48
α -terpinene	-	0.15
limonene	0.68	10.44
β -phellandrene + 1,8-cineole	0.42	1.67
γ -terpinene	-	0.14
p-cymene	0.11	0.22
terpinolene	-	0.15
linalool	2.34	1.03
β -caryophyllene + terpinen-4-ol	0.46	0.38
methyl chavicol	6.65	0.56
α -terpineol	0.20	0.23
(Z)-anethole	0.39	0.11
(E)-anethole	86.06	79.93
anisaldehyde	0.62	0.38
foeniculin	0.46	-