



Progress in Essential Oils

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Valerian Root Oil

An oil of valerian root (*Valeriana officinalis* L.) of Chinese origin was the subject of analysis by Zhang et al. (1993). The oil was found to possess the following constituents:

- α -pinene (0.49%)
- camphene (4.74%)
- β -pinene (0.41%)
- p-cymene (0.03%)
- 1,8-cineole (0.01%)
- camphor (0.05%)
- borneol (0.08%)
- isoborneol (0.17%)
- methyl thymol (0.01%)
- bornyl acetate (23.92%)
- isobornyl acetate (6.64%)
- sabinol* (0.62%)
- β -gurjunene † (0.62%)
- β -caryophyllene (0.30%)
- ar-cucumene (0.58%)
- eremophilene (0.30%)
- aromadendrene (0.16%)
- p-mentha-1(7),8-dien-9-ol (0.25%)
- α -santalol† (1.51%)
- mayurone† (0.06%)
- γ -patchoulene† (0.47%)
- cedrenol* (2.42%)
- ledol† (1.74%)
- valeranone (12.43%)
- nootkatone (0.99%)
- farnesal† (7.72%)
- deoxysericealactone† (5.11%)
- 7,10-pentadiynoic acid † (4.42%)
- cyperene† (0.08%)
- β -cedrene† (0.08%)
- α -cedene† (0.03%)

* correct isomer not identified
† incorrect identification based on GC elution order

Nikiforov et al. (1994) analyzed the headspace volatiles of valerian roots using GC/FID and GC/MS and a charcoal trapping medium. The

volatiles that were eluted from the charcoal with CS₂ were as follows:

- acetic acid (0.23%)
- β -bisabolene (0.48%)
- borneol (2.43%)
- bornyl acetate (12.68%)
- bornyl isovalerate (3.93%)
- butyl isovalerate (2.78%)
- γ -cadinene (1.13%)
- cadinol* (0.59%)
- camphene (19.76%)
- camphor (0.14%)
- β -caryophyllene (4.16%)
- cedrene* (0.91%)
- 2,6-dimethyloctanal (1.14%)
- 3,7-dimethyloctanol (1.88%)
- 4,7-dimethylundecane (0.91%)
- dodecanol (1.12%)
- 2-ethyl decanoate † (1.03%)
- 4-ethylheptane (0.47%)
- ethyl isovalerate (0.19%)
- fenchene* (2.01%)
- α -gurjunene (2.17%)
- isoamyl isovalerate (2.48%)
- isovaleric acid (4.48%)
- kessane (2.12%)
- 3-methyldodecane (0.39%)
- 3-methylvaleric acid (1.88%)
- 2-methylundecane (0.16%)
- myrcene (3.16%)
- tetradecanoic acid (1.18%)
- α -pinene (9.74%)
- β -pinene (0.79%)
- prezizaene (1.11%)
- 2-propylheptanol (0.79%)
- sabinene (0.48%)
- α -selinene (1.03%)
- terpinolene (2.14%)
- 2,6,7-trimethyldecane (0.17%)
- valeranone (2.24%)
- valerenal (3.14%)
- zizaene (0.89%)

* correct isomer not identified
† incomplete name

Zhu et al. (1995) reported that the root oil of *V. officinalis* var. *latifolia* Miq. possessed the following composition:

- 2-methylvaleric acid (0.23%)
- α -thujene (0.11%)
- α -pinene (6.76%)
- camphene (16.17%)
- sabinene (0.60%)
- β -pinene (6.48%)
- myrcene (0.99%)
- limonene (1.77%)
- δ -3-carene† (0.04%)
- allo-ocimene* (0.01%)
- camphor (0.05%)
- borneol (0.38%)
- terpinen-4-ol (0.19%)
- myrcenol (0.12%)
- methyl thymol (0.12%)
- bornyl acetate (50.56%)
- trans*-caryyl acetate (5.47%)
- δ -elemene (0.17%)
- dihydrocaryyl acetate (1.62%)
- β -caryophyllene (0.38%)
- α -humulene (0.11%)

* correct isomer not identified
† incorrect identification based on GC elution order

The main constituent of valerian root oil was reported by Deng et al. (1995) to be bornyl acetate. As many of the constituents listed in that analysis appeared to be incorrect the compositional study is not included in this review.

Oils produced from the roots and rhizomes of the 'Shipka' cultivar of *V. officinalis* were produced either by steam distillation or water distillation in Bulgaria by Georgiev et al. (1999). The authors reported that the root and rhizome oils were similar, as can be seen in **T-1**; however, only 41.7% of

Percentage composition of the root and rhizome oils of Bulgarian valerian root

T-1

Compound	Root oil	Rhizome oil
valeric acid	0.4	1.7
α -pinene	1.0	0.3
α -fenchene	1.6	0.5
camphene	1.0	0.6
β -pinene	0.3	0.1
p-cymene	0.1	< 0.1
limonene	0.3	0.2
amyl valerate	< 0.1	0.2
borneol	0.6	0.5
terpinen-4-ol	0.2	0.2
myrtenol	0.6	0.5
bornyl acetate	5.6	6.6
α -terpinyl acetate	0.4	0.4
β -caryophyllene	2.1	2.8
β -guaiene*	5.4	4.7
ar-cucumene	0.8	1.0
(E)- β -ionone	1.3	1.7
bicyclogermacrene	2.3	2.3
kessane	1.8	2.8
spathulenol	4.3	5.9
valeranone	11.6	8.7

* correct isomer not identified

the oil was characterized. In addition they also found that the oil retained in the roots and rhizomes after 3 hr distillation was 32–52% with a valeranone content of 11.6–15.6%. Kornievska et al. (2000) analyzed oils produced from three Ukrainian *Valeriana* species among which was *V. officinalis*. Among the components listed as constituents of the oil were:

- α -pinene (0.76%)
- camphene (2.40%)
- β -pinene (0.12%)
- limonene (0.40%)
- phellandrene* (1.17%)
- bornyl acetate (32.15%)
- β -caryophyllene (3.21%)
- allo-aromadendrene (13.46%)
- myrtenol (3.14%)

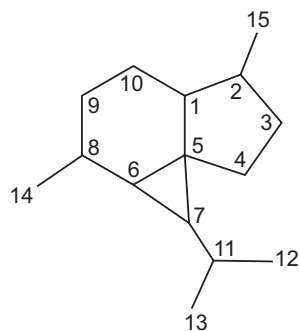
* correct isomer not identified

Oils produced from the 'Shipka' cultivar of *Valeriana officinalis* subsp. *collina* grown in Bulgaria was analyzed by Bos et al. (2000) over two seasons. The root oils were found to range as follows:

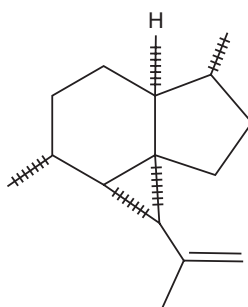
- α -thujene (t-0.1%)
- α -pinene (0.9–2.8%)
- α -fenchene (1.5–4.2%)
- camphene (1.1–2.9%)
- β -pinene (0.3–0.8%)
- p-cymene (0.1–0.2%)
- limonene (0.3–0.8%)
- γ -terpinene (t-0.1%)
- terpinen-1-ol (t-0.1%)
- borneol (0.6–1.0%)
- terpinen-4-ol (0.2–0.3%)
- α -terpineol (t-0.1%)
- myrtenol (0.6–0.7%)
- methyl thymol (0.1%)
- methyl carvacrol (0.1–0.2%)
- bornyl acetate (6.0–7.3%)
- trans-sabinyl acetate (t-0.1%)
- α -terpinyl acetate (0.3–0.5%)
- eugenol (0–2.8%)
- 2,6-dimethoxy-p-cymene (0.1%)
- pacifigorgia-1(6), 10-diene (0.5–0.7%)
- α -calacorene (0.1–0.2%)
- α -humulene (0.1–0.3%)
- valerena-4,7 (11)-diene (4.6–7.2%)
- (E)- β -ionone (0.3%)
- γ -muurolene (0–0.4%)
- ar-cucumene (0.2–1.3%)
- γ -cadinene (0.3–0.8%)
- δ -cadinene (0.3–0.4%)
- kessane (1.4–2.3%)
- pacifigorgiol (1.4–2.1%)
- elemol (0.1–0.2%)
- myrtenyl isovalerate (1.1–1.3%)
- faurinone (0.4–0.8%)

Tamariscane; tamariscene; pacifigorgiane; valerene

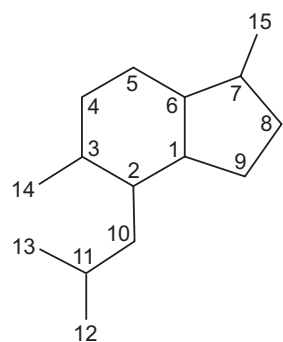
F-1



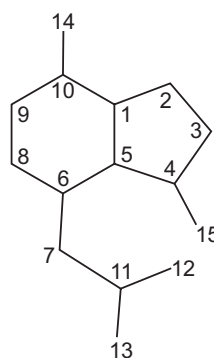
Tamariscane



Tamariscene



Pacifigorgiane



Valerene

maaliol (0.2–0.3%)
 spathulenol (4.1–5.2%)
 hinesol (1.0–1.3%)
 α -eudesmol (0.5%)
 valerianol (0.2%)
 valeranone (6.8–12.0%)
 α -kessyl alcohol (0.1–0.2%)
 valeranal (8.5–9.1%)
 valeranol (0.3–0.4%)
trans-valerenyl acetate (0.6–1.2%)
 α -kessyl acetate (2.1–6.2%)
cis-valerenyl acetate (0.6–0.7%)
 kessanyl acetate (0–0.2%)
 valerenyl isovalerate* (0.4–0.5%)

* correct isomer not identified

t = trace (< 0.1%)

Trace amounts (< 0.1%) of tricyclene, sabinene, isobutyl valerate, α -terpinene, β -phellandrene, 1,8-cineole, terpinolene, linalool, isoamyl isovalerate, isoborneol, thuj-3-en-10-al and (*Z*)-anethole were also found in oils produced over one or two seasons.

Paul et al. (2001) confirmed that δ -elemene, α -ylangene, α -copaene, β -elemene, β -caryophyllene, γ -elemene, allo-aromadendrene, 9-epi- β -caryophyllene, germacrene D, bicyclogermacrene, pacifigorgiol, germacrene B, spathulenol, alismol, and valerianol were components of *V. officinalis* oil. In addition they structurally elucidated valerena-4, 7(11)-diene, pacifigorgia-1,10-diene, pacifigorgia-1(6),10-diene, pacifigorgia-1(9),10-diene, pacifigorgia-2,10-diene, pacifigorgia-2(10),11-diene and tamariscene (see F-1) in the oil. These compounds were found to be the opposite enantiomers of the same compounds found in *Frullania* sp. (liverworts). Paul et al. postulated that tamariscane may be the common biosynthetic precursor of valerianes and pacifigorgianes (see F-1).

Dharmaratne et al. (2002) extracted powdered dried *V. officinalis* roots with methylene chloride and the extract was chromatographed over silica gel with hexane to yield an acidic and a neutral fraction. Reversed phase column chromatography using C-18 silica gel and water/methanol mixture elution resulted in the characterization of valerenic acid, hexadecanoic acid and 3 β , 4 β -epoxyvalerenic acid.

Pavlovic et al. (2004) analyzed an oil produced from the subterranean parts of *V. officinalis* collected from

Comparative percentage composition of valerian root oils produced from two cultivars harvested after 8 and 14 months

T-2

Compound	'Select' oils		'Anthose' oils	
	8 month	14 month	8 month	14 month
α -pinene	1.8	2.1	3.8	2.7
camphene	5.0	5.7	7.2	7.0
β -pinene	1.0	1.2	1.2	1.0
δ -2-carene	-	0.4	0.7	1.4
α -terpinene	0.8	1.0	0.8	0.7
β -phellandrene	1.0	1.2	1.2	1.1
γ -terpinene	0.2	0.2	-	-
terpinolene	0.4	0.3	0.5	0.4
p-cymene	0.2	0.3	0.3	0.2
naphthalene [†]	0.7	0.6	0.8	0.7
α -campholenal	2.3	0.4	1.6	0.9
borneol	0.6	0.6	0.4	0.4
pulegone [†]	0.2	0.3	-	-
terpinen-4-ol	0.2	0.2	-	-
methyl thymol	0.2	0.2	-	-
bornyl acetate	10.7	12.7	9.5	9.2
bornyl isovalerate	-	0.4	-	-
methyl carvacrol [†]	0.3	0.3	-	-
β -caryophyllene ^{††}	-	-	1.3	1.3
δ -elemene	0.6	0.6	0.5	0.5
isocaryophyllene	1.4	2.0	1.0	0.9
isocyclosativene [‡]	0.8	0.4	-	-
β -cubebene	-	-	1.2	1.1
β -elemene	0.4	0.5	0.5	0.5
α -elemene	0.8	0.2	-	-
italicene	0.8	-	-	-
α -gurjunene	3.0	3.5	2.6	4.1
β -caryophyllene	1.5	1.9	-	-
β -gurjunene	-	-	4.1	5.2
α -cedrene	3.7	-	-	-
α -humulene	0.6	0.6	7.4	8.5
aromadendrene [†]	1.1	-	-	-
allo-aromadendrene	1.4	0.7	-	-
6,7-dimethoxy-m-cymene [†]	1.7	11.0	-	-
β -ionone*	0.7	1.0	0.7	0.6
β -selinene	0.8	-	1.5	1.8
δ -selinene [†]	1.7	2.3	1.0	0.9
bicyclogermacrene	1.6	1.9	2.2	1.7
α -selinene	-	-	1.0	0.7
<i>cis</i> -pinocarveol [†]	-	-	0.7	0.6
epi-zonarone	2.9	-	-	-
germacrene D	1.4	1.7	-	-
(E, E)- α -farnesene	-	-	1.5	1.5
γ -cadinene	0.6	-	-	-
γ -selinene [°]	-	1.7	-	1.5
isobornyl 2-methylbutyrate	-	-	0.5	0.4
δ -cadinene	1.5	1.9	-	-
kessyl alcohol	2.1	1.0	0.8	0.7
germacrene B	2.5	2.8	-	-
longipinanol	-	0.4	-	-
spathulenol	2.8	3.3	4.6	4.8
caryophyllene oxide	0.4	0.5	1.0	0.7
benzodiazepine [°]	0.8	1.06	0.7	0.4
1H-cyclopropanol azulen-4-ol [†]	0.4	0.8	0.7	0.7

Comparative percentage composition of valerian root oils produced from two cultivars harvested after 8 and 14 months

T-2
(cont.)

Compound	'Select' oils		'Anthose' oils	
	8 month	14 month	8 month	14 month
15-acetoxyvaleranone	7.1	8.7	5.3	5.7
myrtenyl acetate [†]	1.2	1.4	1.4	1.2
α -cadinol	-	3.5	-	-
valerenol	1.8	1.0	2.2	1.0
valerenal	14.0	11.2	12.3	13.3
valeranone	0.9	0.7	-	-
α -bisabolol	0.6	0.9	-	-
valerenic acid	0.5	5.8	2.0	5.9
(E, E)-farnesol	0.5	0.2	2.6	0.7
eremophilene [†]	-	1.1	-	-
isospathulenol [†]	0.7	0.8	0.8	0.8
<i>cis</i> -valerenyl acetate	2.7	2.6	2.6	0.9
<i>trans</i> -valerenyl isovalerate	1.8	1.9	3.9	4.1

* Correct isomer not identified

[†] Incorrect identification based on GC elution order

[‡] Incorrect identification based on poor computer identification

[§] Impossible as a component of valerian root oil

[¶] Compound listed twice; this one is an incorrect identification

[°] Also known as selina-4(15),7(11)-diene

Tara Mountain (western Serbia) when the plants were in full flower. The composition of this oil was determined to be as follows:

α -pinene (0.8%)
 α -fenchene (4.5%)
 camphene (1.3%)
 β -pinene (0.5%)
 limonene (0.4%)
 myrtenol (0.7%)
 bornyl acetate (11.9%)
 δ -elemene (0.6%)
 α -terpinyl acetate (0.7%)
 β -elemene (0.3%)
 α -humulene (0.9%)
 γ -curcumene (0.9%)
 ar-curcumene (1.1%)
 valencene (2.1%)
 β -bisabolene (0.3%)
 7-epi- α -selinene (1.2%)
 elemol (1.2%)
 germacrene D (0.6%)
 10-epi- γ -eudesmol (1.8%)
 valerianol (57.3%)
 longiborneol acetate (2.2%)

Also, trace amounts (< 0.1%) of isovaleric acid, sabinene, α -terpinene, p-cymene, (Z)- β -ocimene, γ -terpinene, terpinolene, *trans*-pinocarveol, camphor, borneol, terpinen-4-ol, α -terpineol, citronellol, methyl thymol, methyl carvacrol, *trans*-pinocarvyl acetate, citronellyl acetate, eugenol, longicyclene, β -caryophyllene,

2,5-dimethoxy-p-cymene, γ -elemene, *trans*- α -bergamotene, α -guaiene, γ -patchoulene, α -patchoulene, (E)- β -farnesene, α -selinene, β -curcumene, δ -cadinene and epi- α -bisabolol were found in the same oil.

Letchamo et al. (2004) analyzed the oils produced from the subterranean parts of two cultivars ('Select' and 'Anthose') of *V. officinalis* that were harvested at 8 months and 14 months. The results of the comparative analyses are presented in **T-2**. As can be seen from this study, there were a number of misidentifications because the authors appeared to have relied on the computer matching without double-checking whether the component identified had: (a) a retention index that complied with the published data, (b) whether such a compound had been previously found in an essential oil (c) whether such a compound was a possible component of valerian root oil or even (d) whether the compound existed twice in the list of constituents published.

Lopes et al. (2005) used a combination of GC-FID and GC/MS to analyze an oil produced from the dried roots of *V. officinalis* grown in British Columbia (Canada). The composition of this oil was found to be as follows:

methyl isovalerate (0.14%)
 isovaleric acid (13.02%)
 tricyclene (0.12%)
 α -thujene (0.08%)
 α -pinene (3.10%)
 α -fenchene + camphene (10.39%)
 sabinene (0.14%)
 β -pinene (1.43%)
 p-cymene (0.19%)
 limonene + β -phellandrene (2.05%)
 γ -terpinene (0.11%)
 isoamyl isovalerate (0.06%)
 borneol (1.03%)
 terpinen-4-ol (0.15%)
 myrtenol (0.23%)
 methyl carvacrol (0.10%)
 bornyl acetate (16.35%)
 myrtenyl acetate (1.12%)
 δ -elemene (1.14%)
 α -copaene (0.15%)
 β -elemene (0.22%)
 β -caryophyllene (2.52%)
 α -humulene (0.62%)
 (E)- β -farnesene (5.35%)
 allo-aromadendrene (2.46%)
 zingiberene (1.03%)
 α -muurolene (0.14%)
 β -bisabolene (0.87%)
 β -curcumene (0.22%)
 bornyl isovalerate (0.47%)
 δ -cadinene + unknown (0.48%)
 kessane (0.74%)
 spathulenol (1.84%)
 caryophyllene oxide (0.48%)
 β -eudesmol (0.22%)
 valerianol (0.53%)
 epi- α -bisabolol (0.47%)
 valerenal (4.30%)
 valerenol (0.27%)
cis-valerenyl acetate (0.84%)
trans-valerenyl acetate (0.52%)
cis-valerenyl isovalerate (0.06%)
trans-valerenyl isovalerate (0.50%)

In addition, trace amounts (< 0.10%) of hexanal, α -phellandrene, α -terpinene, 1,8-cineole, (Z)- β -ocimene, *cis*-sabinene hydrate, terpinolene, p-cymenene, *trans*-sabinene hydrate, camphene hydrate, isoborneol, α -terpineol, methyl thymol, hexyl isovalerate, eugenol and valerenic acid were found in this oil.

H-L. Zhang, Z-J. Zhang, Y-M. Wang and X-Y Zhao, *Study on the chemical constituents of essential oil from Valeriana officinalis Linn.*, Tianran Chanwa Yanjiu Yu Kaifa, 5(3), 21-27 (1993).

A. Nikiforov, B. Remberg and L. Jirovetz, *Head-space analysis of Valerian roots (Radix Valerianae)*, Sci. Pharm., 62, 331-335 (1994).

Percentage composition of the headspace volatiles of six carnation cultivars

T-3

Compound	1	2	3	4	5	6
methyl 2-methyl-(E)-butenoate	t	-	-	0.3	t	t
ethyl isobutyrate	t	-	-	0.3	0.4	-
hexanal	0.1	0.1	0.7	-	t	-
styrene	t	t	0.2	0.6	0.6	-
α-pinene	t	0.1	0.2	t	0.4	0.1
phenol	-	-	0.3	0.4	-	0.1
camphene	t	t	0.2	-	0.1	-
β-pinene	-	t	0.1	t	-	-
6-methyl-5-hepten-2-one	-	0.1	0.8	t	0.2	-
mycrene	0.1	0.1	0.6	0.1	0.1	-
decane	t	t	t	-	t	0.1
octanal	t	0.2	1.2	t	0.1	t
(Z)-3-hexenyl acetate	0.1	t	0.2	0.1	t	-
δ-3-carene	t	0.1	t	t	-	-
1,4-cineole	-	t	t	0.1	0.1	-
p-cymene	t	0.3	0.2	t	0.1	t
limonene	0.9	1.4	3.3	0.9	2.0	0.1
1,8-cineole	0.2	t	0.2	0.1	0.9	-
methyl benzoate	1.4	0.2	0.8	70.6	32.8	79.1
linalool	0.1	0.2	0.3	-	0.7	t
nonanal	0.1	0.3	4.2	2.1	0.1	2.0
camphor	0.1	0.1	0.1	0.1	0.2	t
menthone	t	t	0.1	t	-	-
benzyl acetate	0.2	0.2	0.1	0.1	0.4	-
ethyl benzoate	0.1	t	0.1	0.2	t	t
creosol ^a	0.1	0.2	t	-	-	-
α-terpineol	0.1	0.1	0.3	t	0.4	-
methyl salicylate	0.1	0.2	0.2	1.4	1.3	0.4
decanal	0.1	0.3	0.7	0.1	0.7	0.1
isopropyl benzoate	t	-	0.1	t	0.2	t
linalyl acetate	0.1	0.1	-	t	0.1	-
2-phenethyl acetate	t	t	0.1	t	0.1	t
ethyl salicylate	-	t	0.1	t	0.1	t
isobornyl acetate	0.1	0.1	t	t	0.2	t
undecanal	t	t	t	-	0.1	t
eugenol	84.1	70.4	60.1	t	0.1	7.6
butyl benzoate	-	1.6	0.1	0.1	0.1	t
vanillin	t	0.4	0.1	-	t	t
α-cedrene	t	0.1	t	t	t	-
β-caryophyllene	0.8	2.2	1.7	10.4	10.8	1.0
isoamyl benzoate	1.4	0.1	t	0.1	0.1	t
(E)-isoeugenol	-	0.2	0.1	-	-	-
α-humulene	t	0.1	0.1	0.2	0.2	t
amyl benzoate	t	0.1	t	0.1	t	t
(Z)-3-hexenyl benzoate	t	-	0.3	-	0.1	t
hexyl benzoate	0.5	1.2	0.2	0.7	0.5	0.5
caryophyllene oxide	3.5	0.2	0.2	2.7	4.6	0.5
hexyl salicylate	t	0.2	-	-	0.2	t
benzyl benzoate	0.2	1.2	1.8	2.4	6.5	t
benzyl salicylate	0.1	-	0.2	-	0.4	t

t = trace (< 0.1%)

^a = also known as 4-methylguaiacol

carnation cultivars: 1. 'Malmaison'; 2. 'Fragrant Ann'; 3. 'Jacqueline Ann'; 4. 'Arnhem Spirit'; 5. 'Dark Pierrot'; 6. 'V.E. Jubilation'

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Carnation Volatiles

Adsorption of the headspace volatiles of six cultivars of carnation (*Dianthus caryophyllus* L.) on Porpak Q followed by characterization by GC/MS was the subject of analysis by Clery et al. (1999). The volatiles characterized in these cultivars can be seen in **T-3**. In addition, trace amounts of p-cresyl methyl ether, carvone, isobutyl benzoate, (Z)-isoeugenol, eugenyl acetate, (E)-3-hexenyl benzoate and amyl salicylate were found in the headspace of one or more of the cultivars.

Schade et al. (2001) examined the fragrance volatiles of developing and senescing carnation flowers (cv. 'Improved White' Sim) using GC/MS. The volatiles that were identified were hexanol, 2-hexanol, (Z)-3-hexenol, nonanal, β -caryophyllene, benzaldehyde, benzyl alcohol, benzyl benzoate, hexanal and (E)-2-hexenal.

El-Ghorab et al. (2006) examined the effect of bioregulators on carnation grown commercially in Egypt for the production of extracts. Oils produced from the freshly picked flowers were subjected to analysis by a combination of GC-FID and GC/MS. The oil obtained from the untreated flowers was found to possess the following main components:

2-phenethyl alcohol (1.36%)
eugenol (18.22%)
hexyl benzoate (0.93%)
(Z)-3-hexenyl benzoate (0.72%)
benzyl benzoate (12.62%)
benzoin (0.67%)
nootkatone (0.69%)
benzyl salicylate (6.85%)
m-cresyl phenylacetate (0.67%)
hexadecanoic acid (28.67%)
eicosene* (7.70%)
linoleic acid (21.20%)

* correct isomer not identified

The identification m-cresyl phenylacetate

and nootkatone requires corroboration as they are not normally found as components in the Caryophyllaceae family. The authors did show, however, that the composition of carnation oil could be drastically altered by the addition of bioregulators to the crop during its life cycle.

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