Progress in Essential Oils

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Hyssop Oil

Renzini et al. (1999) determined that the main constituents of a hyssop oil produced from plants collected in the Piedmont region (Italy) were as follows:

α-pinene (0.6%)
sabinene (1.5%)
β-pinene (11.1%)
myrcene (2.1%)
limonene (12.2%)
pinocamphone (1.7%)
isopinocamphone (43.3%)
β-bourbonene (1.4%)
caryophyllene oxide (0.5%)

Garg et al. (1999) analyzed an oil produced from *Hyssopus officinalis* subsp. *officinalis* grown in an experimental garden in Lucknow (India). The composition of this oil was found to be as follows:

α-thujene (1.0%)
α-pinene (1.8%)
β-pinene (18.4%)
sabinene (1.3%)
limonene (5.6%)
β-phellandrene (4.2%)
(Z)-β-ocimene (0.1%)
pinocamphone (49.1%)
isopinocamphone (9.7%)
α-terpineol (0.5%)
myrtenol (0.7%)
sabinyl acetate* (0.5%)
methyl eugenol (0.2%)
β-caryophyllene (0.3%)
α-humulene (0.1%)
(Z)-β-farnesene (0.3%)
germacrene D (0.7%)
δ-cadinene (0.8%)
γ-elemene† (0.2%)
β-eudesmol (0.2%)
α-eudesmol (0.2%)

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

It is known that three forms of *H. officinalis* subsp. *officinalis* occur: f. *cyaneus* Alef. (blue flowered form), f. *ruber* Mill. (pink flowered form) and f. *albus* Alef. (white flowered form). Chalchat et al. (2001) analyzed the composition of 12 oils produced from plants grown in Serbia. The results of this study are summarized in **T-1**. As can be seen from the results, the oils were grouped into five types depending upon the contents of major components. Also, it could be seen that the flower color (f. type) had no influence on the oil composition whatsoever.

Fraternale et al. (2004) analyzed oils produced from *H. officinalis* grown in two different locations near Urbine (Italy). The main components of the two oils are presented in **T-2**.

An oil of *H. officinalis* subsp. *angustifolius* produced from plants collected in the vicinity of Kose (Turkey) was the subject of analysis by Özer et al. (2005). The components characterized in this oil were:

α-thujene (0.1%)
α-pinene (0.8%)
camphene (0.1%)
sabinene (1.0%)
β-pinene (10.6%)
myrcene (2.5%)
α-terpinene (0.1%)
p-cymene (0.3%)
limonene (0.5%)
1,8-cineole (7.2%)
γ-terpinene (0.2%)
linalool (0.1%)
camphor (0.2%)
pinocamphone (19.6%)
pinocarvone (36.3%)
borneol (0.3%)
isopinocamphone (5.3%)
α-terpineol (0.8%)
myrtenal (0.3%)
pulegone (0.3%)
piperitone (0.4%)
thymol (< 0.1%)
piperitenone oxide (0.1%)
α-copaene (0.1%)
β-bourbonene (0.1%)
β-caryophyllene (0.4%)
β-gurjunene (0.1%)
γ-muurolene (0.3%)
germacrene D (1.0%)
bicyclogermacrene (0.6%)
γ-cadinene (0.1%)
δ-cadinene (0.2%)
spathulenol (0.9%)
caryophyllene oxide (0.1%)

Oils of hyssop of Bulgarian origin were found by Stoyanova and Grozeva (2006) to contain the following major components:

α-pinene (0.8–1.8%)
β-pinene (9.2–14.2%)
sabinene (2.0%)
myrcene (3.0%)
limonene (0.8%)
1,8-cineole (9.0–12.0%)
pinocamphone (38.4–46.5%)
linalool (1.8%)
camphor (8.0–12.5%)
geraniol (2.0–3.0%)
borneol (2.1–3.0%)

G. Renzini, F. Scazzocchio, M. Lu, G. Mazzanti and G. Salvatore, *Antibacterial and cytotoxic activity of* Hyssopus officinalis *L. oils.* J. Essent. Oil Res., 11, 649–654 (1999).

S.N. Garg, A.A. Naqvi, A. Singh, G. Ram and S. Kumar, *Composition of essential oil from an annual crop of* Hyssopus officinalis *grown in Indian plains.* Flav. Fragr. J., 14, 170–172 (1999).

J-C. Chalchat, D. Adamovic and M.S. Gorunovic, *Composition of oils of three cultivated forms of* Hyssopus officinalis *endemic in Yugoslavia:*

Percentage composition of *Hyssopus officinalis* oils **T-1**

Compound	1(4)[a]	2(2)	3(2)	4(2)	5(2)
α-pinene	0.1–0.2	0.1–0.3	0.1–0.3	0.2–0.3	0.1–0.5
α-thujene	t–0.1	0.1–0.2	t–0.1	0.1	0.1
camphene	t–0.1	t–0.1	t	0.1	t–0.1
β-pinene	2.5–4.9	3.8–6.8	3.7–6.5	5.0–6.3	1.2–13.2
sabinene	0.6–1.2	0.9–1.5	0.9–1.2	1.2–1.4	0.9–1.8
myrcene	0.8–1.3	0.9	0.1–1.3	0.9–1.0	1.2–1.8
α-terpinene	0–t	t–0.1	t	t	–
limonene	0.6–0.8	0.7–0.8	0.7–0.8	0.7–0.8	0.9–1.0
β-phellandrene	1.4–2.9	0.7–1.2	2.1–2.2	0.6	1.2–7.9
(Z)-β-ocimene	t–0.1	t–0.1	0–t	0.2	t–0.2
γ-terpinene	t	0–t	t–0.1	0–0.1	t
(E)-β-ocimene	0.1–0.4	t	t–0.1	0.4–0.5	0.1–0.6
p-cymene	t–0.1	0.2–0.7	0.2–0.3	0.5–0.6	0.1
myrtenyl methyl ether	1.0–2.8	2.5–3.9	2.1–2.8	1.1–1.4	3.9–6.1
α-thujone	t–0.1	0.1–0.2	0.1	t	0.1
β-thujone	0.2–0.3	0.1	0.1–0.2	0.3	t
menthone	t–0.4	0.1	t–0.3	0.1	0.1
cis-sabinene hydrate	0.1–0.2	1.6–4.0	1.2–1.8	2.5–3.3	0.1–0.2
pinocamphone	40.8–48.7	2.4–6.6	15.5–21.3	63.3–64.9	1.3–2.2
isopinocamphone	21.8–30.0	59.6–59.9	39.5–42.0	5.8–9.1	29.0–44.3
pinocarvone	0.4–2.6	0.1–0.2	0.1–3.0	0.1–0.4	15.8–16.9
linalool	0.8–0.9	0.1–1.1	1.0–1.3	0.8	1.3–2.1
β-caryophyllene	0.7–1.3	0.2–1.1	1.3	0.7–0.8	0.8–2.7
terpinen-4-ol	0.1	0.5–1.0	0.4–0.5	0.7–0.9	0.1–0.2
myrtenal	0.2–0.3	0.1–0.2	0.2–0.3	0.3–0.4	0.2–0.4
allo-aromadendrene	0.8–1.3	1.1–1.3	1.3–1.4	0.3	0.5–1.3
menthol	0.1–0.3	0.1–0.2	0.1–0.3	0.1–0.2	0.1–0.2
α-humulene	0.1–0.4	t–0.3	0.5–0.6	0.2–0.3	0.4–0.6
methyl chavicol	0.1–0.2	0.1–0.2	0.2	0.1–0.2	0.1–0.2
germacrene D	2.4–2.8	2.2–2.3	1.8–4.1	1.2–1.7	0.5–6.2
carvone	0.3	0.3	0.3	0.3	0.2–0.4
bicyclogermacrene	1.1–2.8	1.5–1.6	1.0–3.0	0.3–0.6	0.1–4.3
myrtenol	1.4–3.3	0.8–2.2	1.7	2.4–2.7	0.7–0.9
2-hydroxypinocamphone	t–0.1	0.2	0.1–0.3	t	0.1–0.3
caryophyllene oxide	0.1–0.2	t–0.2	0.2–0.3	0.2	0.4
cuminyl alcohol	0.1–0.3	0.2	0.2–0.3	t–0.1	0.2
methyl eugenol	0.1–0.3	0.2–0.3	0.2–0.4	0.2	0.3
elemol	0.9–3.5	3.2–4.3	4.4–4.7	0.5–0.6	1.6–5.1
spathulenol	0.7–1.5	1.4	1.3–2.0	0.9–1.2	1.2–2.0
γ-eudesmol	0.1–0.4	0.2–0.3	0.2–0.4	t–0.1	0.1–0.4
α-muurolol	0.1–0.3	0.1–0.2	t–0.3	0.1	t–0.3
α-eudesmol	0.1–0.4	0.3	0.3–0.4	t–0.1	0.1–0.3
β-eudesmol	0.1–0.4	0.2	0.3–0.5	t	0.1–0.4
α-cadinol	0.1–0.4	0.1	0.1–0.3	0.1–0.2	0.1–0.3

t = trace (< 0.1%); [a] = number of oils; 1 = oils rich in pinocamphone > isopinocamphone; 2 = oils rich in isopinocamphone; 3 = oils rich in isopinocamphone > pinocamphone; 4 = oils rich in pinocamphone; 5 = oils rich in isopinocamphone > pinocarvone

f. albus *Alef., f.* cyaneus *Alef. and f.* ruber *Mill.* J. Essent. Oil Res., **13**, 419–421 (2001).

D. Fraternale, D. Ricci, F. Epifano and M. Curini, *Composition and antifungal activity of two essential oils of hyssop (*Hyssopus officinalis *L.).* J. Essent. Oil Res., **16**, 617–622 (2004).

H. Özer, F. Sahin, H. Kilç and M. Güllüce, *Essential composition of* Hyssopus officinalis *L. subsp.* angustifolius *(Bieb.) Arcangeli from Turkey.* Flav. Fragr. J., **20**, 42–44 (2005).

A. Stoyanova and E. Grozeva, *Bulgarian essential oils for the future: A review.* Indian Perfum., **50**, 42–45 (2006).

Osmanthus Extracts

Osmanthus extracts are obtained from the highly scented flowers of *Osmanthus fragrans* Lour. The most commonly encountered flowers are the golden-yellow ones; however,

T-2 Percentage composition of two Italian oils of *Hyssopus officincalis*

Compound	Oil 1	Oil 2
α-pinene	2.1	2.1
sabinene	0.4	0.4
β-pinene	10.5	10.8
myrcene	3.5	1.6
α-phellandrene	7.4	9.6
α-terpinene	1.9	0.7
linalool	0.2	7.9
camphor	0.3	5.3
pinocamphone	34.0	18.5
isopinocamphone	3.2	29.0
α-terpineol	0.1	0.5
linalyl acetate	2.9	0.8
β-caryophyllene	5.6	2.4
α-humulene	3.2	1.9
germacrene D	5.1	3.3
α-cadinene	2.9	< 0.1
cis-calamenene	0.8	< 0.1
β-cadinene	3.8	2.4
bicyclogermacrene	1.6	1.4
spathulenol	2.3	1.4

silvery-white flowers and orange-red flowers of *O. fragrans* can also be found.

The enantiomeric distribution of (E)-α-ionone in osmanthus absolute was found (Werkhoff et al. 1991) to be: (+)-(E)-α-ionone (99.8%): (-)-(E) α-ionone (0.2%).

Zhu et al. (1993) compared the headspace composition of *O. fragrans* var. *thunbergii* Makino (golden-flower), *O. fragrans* var. *latifolius* Makino and *O. fragrans* var. *aurantiacus* Makino flowers grown in China. The results of this study are summarized in **T-3**.

Ohloff (1994) reported that osmanthus absolute contained a trace amount of jasmolactone.

The floral volatiles identified in an isopentane extract of *O. fragrans* by Omura et al. (2000) were as follows:

- (Z)-3-hexenol (0.90%)
- nonanal (0.56%)
- *trans*-linalool oxide† (1.44%)
- *cis*-linalool oxide† (2.99%)
- linalool (5.12%)
- *trans*-linalool oxide‡ (0.41%)
- *cis*-linalool oxide‡ (0.89%)
- (E)-α-ionone (0.32%)
- (E)-β-ionone (3.38%)
- γ-decalactone (6.55%)
- C_{23}–C_{38} alkanes (46.95%)
- C_{11}–C_{22} aliphatic acids (4.83%)
- methyl esters of C_{16}–C_{18} acids (4.16%)

†furanoid form; ‡pyranoid form

Furthermore, the authors observed that although osmanthus flowers emitted a strong scent and possessed a vivid color, the flowers were not attractive to many insects. They found that this insect repellency was mainly due to γ-decalactone.

Kaiser (2002) reported that of the flowering plants found the varieties of *O. fragrans* possess the greatest diversity in aroma constituents derived from carotenoid degradation reactions. The carotenoid-derived constituents that were characterized as components of an absolute of osmanthus were as follows:

- (E)-β-ionone (7.6%)
- dihydro-β-ionone (6.4%)
- dihydro-β-ionol (3.0%)
- (E)-α-ionone (0.6%)
- *cis*- and *trans*-theaspirane (0.7%)
- 4-hydroxy-β-ionone (0.3%)
- 4-oxo-β-ionone (0.1%)
- 4-oxo-β-ionol (0.3%)
- 4-oxo-dihydro-β-ionone (0.8%)

In addition, (E)-β-damascenone (0.02%), dihydroactinodiolide, (E,E)-2,5-epoxy-6,8-megastigmadiene (0.02%), (Z,Z)-2,5-epoxy-6,8-megastigmadiene, 2,7-epoxy-4,8-megastigmadiene, two isomers of 2-hydroxytheaspirane, 2,5-epoxy-megastigm-6-en-9-ol, 2,3,5-trimethylphenylpentan-4-ol, 5,8-megastigmadien-4-one (0.01%), (E)-5,7,9-megastigmatrien-4-one (0.02%), dehydro-β-ionone (0.05%), (E)-4,7,9-megastigmatrien-3-one (0.01%), four isomers of 4,6,8-megastigmatrien-3-one (0.04%), 4,6,8-megastigmatrien-2-ol, 4,5-epoxy-α-ionone (0.02%), (E)-4,7-epoxy-5(11),8-megastigmadiene, 4-oxo-dihydro-β-iononyl acetate, four theaspirone isomers, (E)- and (Z)-retroionols, four isomers of 7-oxo-dihydrotheaspiranes, (Z)-retro-γ-ionone and cyclic-β-ionone have also been found as minor constituents (< 0.01%) in this sample of osmanthus absolute. The structures of many of the above listed compounds can be seen in **F-1**.

Ishikawa et al. (2004) examined the enantiomeric distribution of five important aroma constituents of the yellow-flowered osmanthus. The following is a summary of their results:

- (2R,5S)-(-)-*cis*-linalool oxide (furanoid) (99.5%):(2S,5R)-(+)-*cis*-linalool oxide (0.5%)
- (2R,5R)-(-)-*trans*-linalool oxide (furanoid) (> 99.5%):(2S,5S)-(+)-*trans*-linalool oxide (< 0.5%)
- (3R)-(-)-linalool (99.9%):(3S)-(+)-linalool (0.1%)
- (S)-(-)-α-ionone (< 0.1%):(R)-(+)-α-ionone (> 99.9%)
- (R)-(+)-γ-decalactone (93.1%):(S)-(-)-γ-decalactone (6.9%)

Tamogami et al. (2004) repeated this same information; however, in this report they noted that the amounts of these above constituents were as follows:

- *cis*-linalool oxide† (2.57%)
- *trans*-linalool oxide† (9.45%)
- linalool (8.66%)
- (E)-α-ionone (0.67%)
- γ-decalactone (10.94%)

†furanoid form

Kaiser (2005) reported the results of a headspace study on *O. fragrans* flower 'Four Seasons' using Porpak SQ as the adsorbent. The components characterized in this study were as follows:

- α-pinene (1.0%)
- β-pinene (0.2%)
- sabinene (0.2%)
- α-phellandrene (0.1%)
- limonene (0.3%)
- 1,8-cineole (0.1%)
- (E)-β-ocimene (0.8%)
- p-cymene (0.1%)
- octanal (0.1%)
- (E)-4,8-dimethylnona-1,3,7-triene (0.4%)
- (Z)-3-hexenyl acetate (10.0%)
- 6-methyl-5-hepten-2-one (0.2%)
- hexanol (0.2%)
- (Z)-3-hexenol (4.3%)
- nonanal (0.3%)
- (E)-2-hexenol (0.3%)
- *trans*-linalool oxide† (12.5%)
- *cis*-linalool oxide† (17.0%)
- linalool (15.1%)
- (E)-2,3-epoxy-2,6-dimethylnona-6,8-diene (0.2%)
- β-caryophyllene (0.3%)
- cyclic β-ionone (0.2%)
- *trans*-linalool oxide‡ (0.5%)
- *cis*-linalool oxide‡ (1.5%)
- nerol (0.1%)
- dihydro-β-ionone (8.5%)

Comparative percentage composition of the headspace volatiles of three varieties of *Osmanthus fragrans* **T-3**

Compound	var. *aurantiacus*	var. *thunbergii*	var. *latifolius*
ethyl acetate	–	0.5	–
3-methylbutanone	–	0.1	–
2-hexenal*	0.1	–	–
3-hexenol*	0.1	–	–
3-hydroxy-2-butanone	–	2.3	6.0
5-hexen-3-one	–	1.0	–
3,3-dimethylhexane†	–	–	0.2
6-undecanone†	–	–	0.1
myrcene	–	–	0.1
6-methyl-5-hepten-2-one	0.1	–	–
decane	–	0.7	0.3
3-hexenyl acetate*	0.5	–	0.3
limonene	0.1	0.8	–
α-ocimene	–	18.0	9.1
1,8-cineole	0.1	–	–
(Z)-β-ocimene	0.2	–	–
(E)-β-ocimene	9.9	–	–
cis-linalool oxide (furanoid)	4.6	7.8	18.1
trans-linalool oxide (furanoid)	7.5	6.1	14.1
3-cyclohexenylmethanol†	–	2.2	–
linalool	25.7	7.9	15.3
menthone	–	0.9	–
6-methyl-3,5-heptadien-2-one	–	0.5	–
1,6-diacetoxyhexane†	–	–	1.8
6-ethanyldihydro-2,2,6-trimethyl- -2H-pyran-3(4H)-one†	0.6	–	–
5-phenylmethoxypentanol†	–	1.1	0.1
ethyl benzaldehyde†	–	1.4	0.2
menthol	–	0.2	1.0
2-methyl-6-methylene-1,7- octadiene-3-one	0.6	–	–
cis-linalool oxide (pyranoid)	0.5	0.9	2.2
trans-linalool oxide (pyranoid)	1.4	0.6	1.0
(Z)-3-hexenyl butyrate	0.5	–	0.3
(E)-3-hexenyl butyrate	0.2	–	–
α-terpineol	–	–	0.2
hexyl butyrate	–	–	0.2
ethyl benzoate	–	–	0.2
carvone	–	3.2	0.2
benzothiazole	0.3	–	–
2,4-dimethylphenylethanone†	–	–	0.2
8,8-dimethyl-4-methylene-1- oxaspiro[2,5]oct-5-ene†	–	–	0.1
4,6,6-trimethylbicyclo[3.1.1]-hept- 3-en-2-one†	–	–	0.1
(E)-β-ionone	3.0	2.5	2.3
5-hexyldihydro-2(3H)furanone	2.7	–	–
(E)-β-ionone	17.1	19.4	10.5
γ-decalactone	–	1.1	0.1
pentadecane	0.2	–	–
hexadecanoic acid	–	0.1	–
4-oxo-β-ionone	–	0.1	–

*correct isomer not identified; †questionable identification

(E)-α-ionone (0.1%)
geraniol (0.3%)
(E)-β-ionone (3.5%)
(E)-nerolidol (0.3%)
γ-decalactone (7.0%)
γ-dodecalactone (0.1%)

†furanoid form; ‡pyranoid form

In addition, smaller amounts of myrcene (0.03%), (Z)-β-ocimene (0.05%), benzaldehyde (0.05%), *cis*-theaspirane A (0.02%), *trans*-theaspirane B (0.02%), α-humulene (0.05%), methyl salicylate (0.02%), 2-phenethyl alcohol (0.05%) and methyl *cis*-(Z)-jasmonate (0.05%) were also found in the osmanthus headspace.

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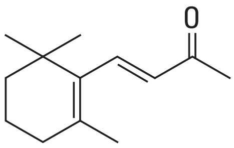
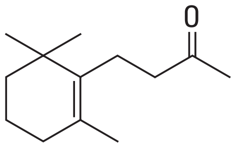
Structures of components of osmanthus absolute F-1


1. β-ionone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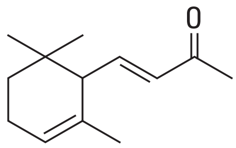
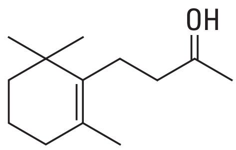
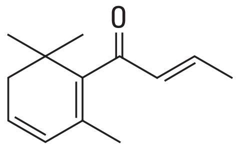
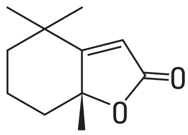
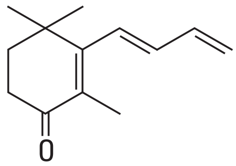
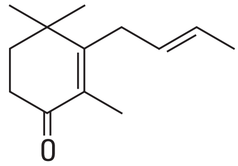

3. α-ionone

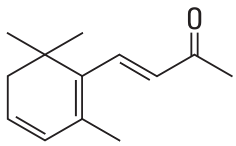

4. dihydro-β-ionol

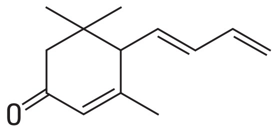

5. β-damascenone

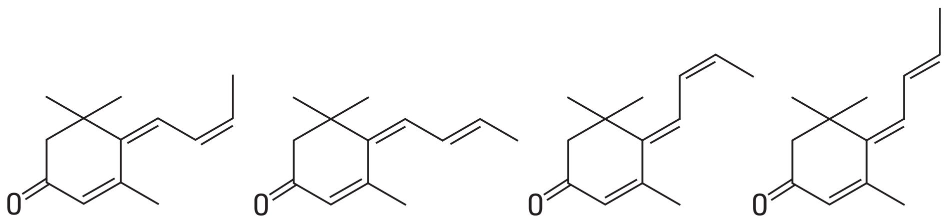

6. dihydroactinodiolide

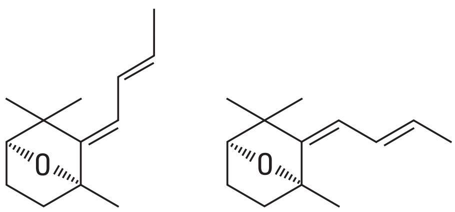

7. 5,7,9-megastigmatrien-4-one

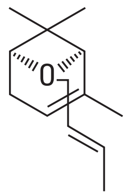

8. 5,8-megastigmadien-4-one

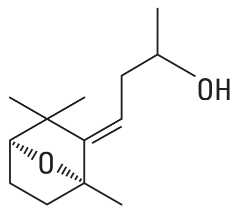

9. 3,4-dehydro-β-ionone

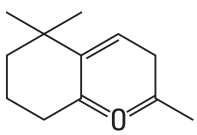

10. 4,7,9-megastigmatrien-3-one

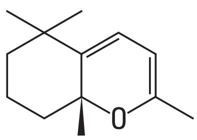

11. four isomers of 4,6,8-megastigmatrien-3-one

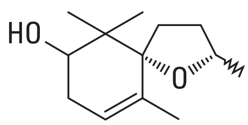

12. from left: (E,E)- and (Z,Z)-2,5-epoxy-6,8-megastigmadiene

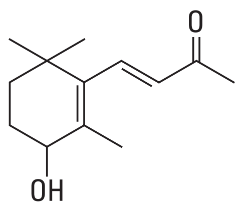

13. 2,7-epoxy-4,8-megastigmadiene

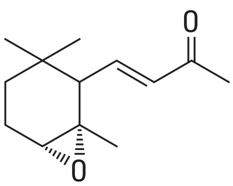

14. 2,5-epoxy-megastigm-6-en-9-ol


15. (Z)-retro-γ-ionone

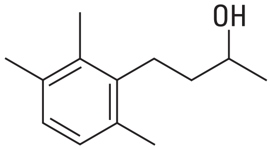

16. cyclic-β-ionone


17. 2-hydroxytheaspirane

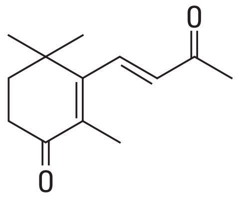

18. 4-hydroxy-β-ionone



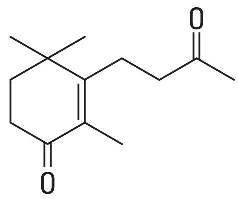
19. 4,5-epoxy-α-ionone



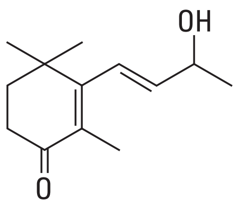
20. 2,3,5-trimethyl-phenylpentan-4-ol



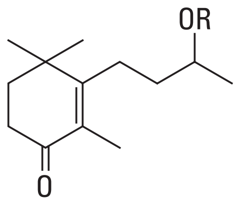
21. 4-oxo-β-ionone



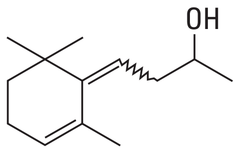
22. 4-oxo-7,8-dihydro-β-ionone



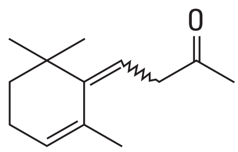
23. 4-oxo-β-ionone



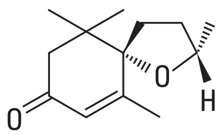
24. 4-oxo-dihydro-β-ionol (R = H) 4-oxo-dihydro-β-ionyl acetate (R = Ac)



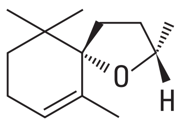
25. retroionol



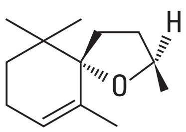
26. retroionone



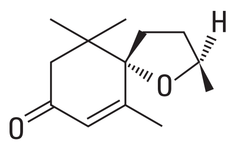
27. theaspirone A



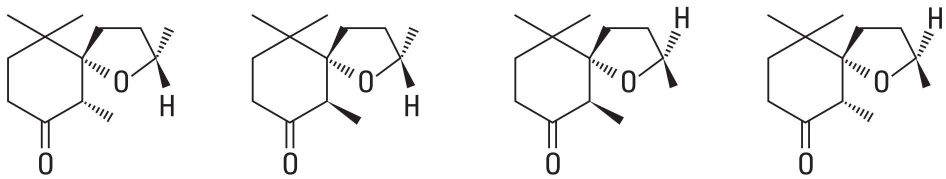
28. theaspirane A



29. theaspirane B



30. theaspirone B



31. 7-oxo-dihydrotheaspirane A and B isomers (left to right: A^1, A^2, B^1, B^2)

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