



Progress in Essential Oils

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Santolina Oil, Hydrosol and Extract

Santolina chamaecyparissus L. (syn. *S. incana* Lam.), which is known as lavender cotton, is a perennial member of the Asteraceae family. It is a semi-woody, tender under-shrub that grows into a many-branched dense mound. It possesses tomentose (densely pubescent) alternate, narrow, pinnate, aromatic leaves that appear to be silvery-green. The flowers are composite, button-shaped yellow heads that are borne on slender stems that rise above the foliage. The aromatic flower heads comprise tubular florets with no ray florets.

Santolina chamaecyparissus is native to the central and western Mediterranean region. Because of its attractiveness, it has become a common garden ornamental. A limited amount of oil is produced annually from this attractive plant.

A commercial sample of santolina oil was screened for its antiplatelet activity by Tognolini et al. (2006). Analysis of the oil using both GC-FID and GC/MS revealed that it possessed the following composition:

- α -pinene (0.4%)
- camphene (0.7%)
- sabinene (7.7%)
- myrcene (8.0%)
- yomogi alcohol (0.9%)
- β -phellandrene (12.8%)
- artemisia ketone (28.2%)
- artemisia alcohol (1.3%)
- camphor (1.9%)
- borneol (1.1%)
- isomenthol (0.2%)
- p-cymen-8-ol (0.3%)
- cryptone (0.3%)
- α -terpineol (1.4%)
- myrtenal (0.4%)
- methyl thymol (0.2%)
- carvone (0.2%)
- linalyl acetate (0.6%)
- neomenthyl acetate (0.2%)
- thymol (3.9%)

- α -longipinene (1.3%)
- α -ylangene (0.2%)
- β -bourbonene (0.2%)
- β -elemene (0.6%)
- β -caryophyllene (0.7%)
- β -copaene (0.2%)
- allo-aromadendrene (0.3%)
- ar-curcumene (1.7%)
- γ -curcumene (1.7%)
- germacrene D (0.6%)
- 1-pentadecene (0.8%)
- β -bisabolene (0.6%)
- spathulenol (2.7%)
- caryophyllene oxide (0.2%)
- vulgarone B (4.8%)

Inouye et al. (2008) compared the composition of santolina oil and its hydrosol produced by steam distillation of flowering plants grown at Saitama (Chichibu District, Japan). Analysis was performed using GC/MS only, and

the results, which were not presented in elution order, can be found in **T-1**. This reviewer questions the analytical data presented in **T-1**. It would appear that the oil should be rich in artemisia ketone, not the hydrosol. Also, the identities of a number of the constituents are questionable.

Supercritical fluid CO₂ extraction of the dried flower heads (ca. 12% moisture) of *S. chamaecyparissus* L. was carried out by Grosso et al. (2009) using a wide range of conditions. Variances such as pressure, temperature, particle size and CO₂ flow rate were examined, and it was found that the optimum yield and composition could be obtained using 8 MPa (80 bar), 40°C, 0.6 mm mean particle size and a 1.1 kg/hr CO₂ flow rate. It was of interest to note that an increase of pressure from 8 MPa to 9 MPa enriched

T-1. Comparative percentage composition of santolina oil and its hydrosol

Compound	Oil	Hydrosol
artemisia ketone	-	25.2
camphor	4.8	16.9
yomogi alcohol	-	8.8
borneol	3.6	8.6
carveol*	-	5.5
epoxy- α -terpinyl acetate	-	4.8
hexenol*	-	3.7
4-heptadienone*	16.4	-
vulgarone A	11.0	-
vulgarone B	9.1	-
spathulenol	6.6	-
sabinene	5.2	-
1-dimethyl-4-hexenyl-4-methylbenzene	3.6	-
myrcene	3.5	-
trimethyl-4-heptadienone*	3.0	-
δ -cadinene	3.0	-
α -longipinene	3.0	-
photonerol	2.8	-

*correct isomer not identified

the extract in sesquiterpenes, while a temperature change from 40°C to 50°C at 9 MPa (90 bar) resulted in an enrichment of alkanes in the extract.

Grosso et al. (2010) used GC-FID and GC/MS to examine the composition of the oil and supercritical fluid CO₂ extract (SFE) of the flower heads of *S.*

chamaecyparissus L. produced in Spain. The SFE conditions used were: particle size = 0.6 mm, pressure = 80 bar, temperature = 40°C, CO₂ flow rate = 1.1 kg/hr and amount of CO₂ consumed = 2.1 kg. The results of the analysis are shown in **T-2**. Trace amounts (<0.05%) of α -calacorene and tricosane were also characterized in the oil and SFE.

The composition of an oil produced from the flowering plants of *S. chamaecyparissus* grown in Spain was the subject of study by Ruiz-Navajas et al. (2012). The constituents characterized using GC-FID and GC/MS were as follows:

sabinene (2.4%)
 myrcene (6.9%)
 β -phellandrene[†] (7.5%)
 artemisia ketone (27.2%)
 camphor (3.9%)
 zingiberene (2.3%)
 ar-curcumene (2.7%)
 spathulenol (2.3%)
 dihydroaromadendrene[†] (18.2%)

[†]incorrect identification

An oil produced from the fresh aerial parts of flowering *S. chamaecyparissus* collected from a location in the vicinity of Algiers (Algeria) was analyzed by Djeddi et al. (2012) using GC-FID and GC/MS. The compounds characterized in this oil were:

santolinatriene (2.2%)
 α -thujene (0.4%)
 α -pinene (1.1%)
 sabinene (4.0%)
 myrcene (1.7%)
 limonene (3.5%)
 1,8-cineole (2.1%)
 santolina alcohol (2.1%)
 p-cymene (8.3%)
 undecane (1.1%)
 camphor (31.1%)
 borneol (1.0%)
 α -terpineol (1.3%)
 carvone (1.5%)
 linalyl acetate (1.0%)
 thymol (1.2%)
 α -ylangene (1.2%)
 β -elemene (0.9%)
 β -caryophyllene (1.1%)
 allo-aromadendrene (0.6%)
 β -ionone* (2.3%)
 germacrene D (0.7%)
 β -bisabolene (1.1%)
 dodecanoic acid (2.3%)
 caryophyllene oxide (0.9%)
 cubenol (17.0%)

*correct isomer not identified

T-2. Comparative percentage composition of the oil and supercritical fluid CO₂ extract (SFE) of the flower heads of *Santolina chamaecyparissus*

Compound	Oil	SFE
α -pinene	0.1	0.2
camphene	0.8	0.9
β -pinene	1.3	1.6
dehydro-1,8-cineole	0.2	t
2-pentylfuran	0.3	t
yomogi alcohol ^a	0.3	0.7
α -terpinene	2.4	1.9
p-cymene	2.0	2.1
1,8-cineole	24.8	38.3
artemisia ketone	0.3	t
<i>trans</i> -sabinene hydrate	3.1	1.7
terpinolene	1.4	2.1
<i>cis</i> -sabinene hydrate	0.8	0.6
nonanol	0.4	0.6
linalool	0.2	0.3
isoamyl isovalerate	0.2	t
α -campholenal	0.3	0.7
<i>trans</i> -p-menth-2-en-1-ol	0.7	-
camphor	7.4	10.7
borneol	8.3	3.8
thuj-3-en-10-al	0.8	0.8
terpinen-4-ol	7.4	1.9
myrtenal	0.4	0.4
α -terpineol	0.2	t
myrtenol	1.7	1.7
<i>trans</i> -carveol	0.2	t
cuminyl alcohol	0.3	0.2
isobornyl acetate	1.5	1.2
thymol	0.1	0.2
carvacrol	0.3	-
(E,E)-2,4-decadienal	0.2	0.4
bicycloelemene	t	0.5
allo-aromadendrene	1.3	1.4
germacrene D	2.7	2.9
bicyclogermacrene	1.4	1.0
γ -cadinene	0.2	0.2
δ -cadinene	0.3	t
spathulenol	3.1	0.6
caryophyllene oxide	1.0	t
globulol	0.1	t
viridiflorol	0.1	t
anhydro-oplopanone	1.0	t
T-cadinol	1.1	0.2
hexadecanoic acid	0.2	t
heneicosane	t	0.3
tetracosane	0.2	0.1
octacosane	0.1	t
hexatriacosane	0.1	0.3

t=trace (<0.05%)

^a(E)-2,5,5-trimethyl-3,6-heptadien-1-ol

Trace amounts (<0.1%) of camphene, benzaldehyde, β -pinene, isomenthol, p-cymen-8-ol, myrtenal, β -bourbonene, β -copaene, a curcumene isomer and (E)-nerolidol were also found in this oil.

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Solidago canadensis Oil

Solidago canadensis L. is known as Canada goldenrod, common meadow or shorthair goldenrod. It is found throughout the United States, except for South Carolina, Georgia, Alabama, Louisiana, Puerto Rico and Hawaii (Weber, 2001). It was introduced into Europe in the 17th century as a horticultural plant, where it has become an extremely invasive weed (Zwoelfer, 1976).

Solidago canadensis is a perennial, herbaceous, aromatic member of the Asteraceae family, which readily forms dense clusters of plants in a variety of environments. In North America, the flowers, which can be found as panicles at the end of each stem, bloom from July to October. Polyploid forms (diploid, tetraploid and hexaploid) are known in North America, although only the diploid form is found in Europe (Weber, 1997). The conspicuous yellow flowers of

Canada goldenrod are self-sterile, and the seeds are readily wind-dispersed. However, the plant more actively spreads through rhizomes. In addition, it is found to possess an allelopathic effect on native vegetation, inhibiting seed germination of some trees found growing in its vicinity. It is believed that this allelopathic effect relates to a mineral imbalance that is somehow caused by goldenrod (Einhellig, 1986).

Abhilasha et al. (2008) revealed that, as a result of chemical analysis, root exudates of *S. canadensis*, including cis-dehydromatricaria ester, were believed to be the cause of its allelopathic effect.

Goldenrod was initially introduced into China as an ornamental plant; however, as a garden escape it is rapidly spreading, becoming a highly invasive weed (Dong, 2006).

Because of its aromaticity, the anatomy of the internal secretory reservoirs in the leaves of *S. canadensis* have been examined. It was found that they are arranged in longitudinal rows, within which is found a colorless oil (Lersten and Curtis, 1989). These reservoirs appear to mimic ducts and are discrete cavities in all organs, including leaves, stems and roots (Curtis and Lersten, 1989).

A survey of the literature reveals that Miller and Eskew (1914) reported that the oil of *S. canadensis* contained α -pinene (85%), borneol (9.2%) and bornyl acetate (3.4%). Other constituents for which no quantitative data were represented were α -phellandrene, limonene and a cadinene isomer.

Bohlman et al. (1980) determined that an extract of the aerial flowering parts of *S. canadensis* contained caryophyllene oxide and three non-volatile clerodane-based diterpenoid compounds.

Kalemba et al. (1990a) analyzed an oil produced from *S. canadensis* collected from alien wild plants in Poland harvested at four different ontogenetic times. Oils were produced from pre-flowering plants (0.30% yield), full-flowering plants (1.44–1.53% yield), the end of flowering plants (1.50–1.90% yield) and post-flowering plants (0.94–1.26% yield) by water distillation (hydrodistillation) in the laboratory. They found that the oils from each of the maturing stages varied quantitatively, but not qualitatively. The components characterized were α -pinene, β -pinene, camphene,

δ -3-carene, α -phellandrene, limonene, γ -terpinene, borneol, α -copaene, α -cubebene, β -caryophyllene, γ -muurolene and some elemene, cadinene and selinene isomers. Furthermore, the authors found that the oil was strongly toxic to the wheat weevil (*Sitophilus granaries*), it paralyzed the lesser grain borer (*Rhyzopertha dominica*) and it was highly repellent toward the confused flour beetle (*Tribolium confusum*), thereby making the oil potentially valuable as a natural insecticide.

In a follow-up report, Kalemba et al. (1990b) analyzed a hydrodistilled oil (1.47% yield) of the dried flowers of *S. canadensis* collected from a natural location in the region of Lodz. The oil, which was analyzed by a combination of GC-FID and GC/MS, was found to contain the following constituents:

α -pinene (13.0%)
camphene (0.5%)
β -pinene (1.9%)
γ -terpinene [†] (4.2%)
α -phellandrene (0.5%)
limonene (12.0%)
δ -3-carene [†] (0.7%)
δ -elemene (1.1%)
γ -muurolene [†] (0.1%)
β -elemene (1.1%)
α -elemene [†] (0.1%)
β -caryophyllene (0.7%)
γ^2 -cadinene [‡] (27.1%)
δ -cadinene (7.5%)
α -copaene [†] (1.2%)
β -selinene (0.4%)
α -cubebene [†] (0.3%)
γ -selinene (2.6%)

[†]incorrect identification based on GC elution order
[‡]questionable identity

Weyerstahl et al. (1993) reexamined the oil, which was produced by Kalemba et al. (1990b), using a combination of analytical techniques, including GC/MS, ¹H-NMR and ¹³C-NMR. Although the quantitative data was produced using GC/MS data only, the components characterized in the oil were as follows:

α -pinene (14.7%)
camphene (0.3%)
sabinene (0.2%)
β -pinene (1.5%)
myrcene (4.2%)
α -phellandrene (0.3%)
limonene (9.3%)
(E)- β -ocimene (0.3%)
bornyl acetate (1.3%)
δ -elemene (0.7%)

α -copaene (0.1%)
 β -bourbonene (0.1%)
 β -elemene (1.5%)
cis- α -bergamotene (0.2%)
cis- β -bergamotene (0.5%)
 β -ylangene (0.7%)
 γ -elemene (0.5%)
trans- α -bergamotene (0.3%)
aromadendrene (0.1%)
trans- β -bergamotene (1.1%)
 γ -muurolene (0.1%)
ar-curcumene (0.8%)
germacrene D (19.8%)
zingiberene (0.5%)
 β -selinene (1.2%)
 α -muurolene (0.7%)
 β -bisabolene (0.4%)
 γ -cadinene (0.2%)
 β -sesquiphellandrene (10.4%)
(E)-nerolidol (0.2%)
germacrene B (0.6%)
spathulenol (0.2%)
junenol^a + A (0.9%)
T-muurolol + T-cadinol (0.3%)
ar-turmerone (0.5%)
 α -cadinol (0.4%)
unidentified^b (1.4%)
4-(6-methyl-5-hepten-2-yl)-cyclohex-2-en-1-one (0.2%)
curlone^c (23.5%)

^aA=unknown with MS similar to curlone

^aalso known as eudesma-4(14)-en-6a-ol

^bunknown with MS similar to ar-turmerone

^calso known as β -sesquiphellandren-9-one

Trace amounts (<0.05%) of p-cymene, terpinolene and α -ylangene were also found in this oil.

Bülow and König (1997) reported that germacrene D was a main component of the oils of *Solidago* species such as *S. gigantea* Ait. and *S. virgaurea* L. subsp. *praecox* and *S. canadensis*. The authors determined that through the use of a 25 m heptakis (2,3-di-O-methyl-6-O-t-butyltrimethylsilyl)- β -cyclodextrin (OV-1701 50%) chiral column the two enantiomers of germacrene D could be separated. They found that the enantiomeric ratio of (+)-germacrene D (the major enantiomer) to (-) germacrene D was dependent upon the plant part (flowers, leaves or stems) from which the oil was obtained. Furthermore, they discussed the acid-catalyzed lability of germacrene D previously reported by Lawrence et al. (1972).

Schmid et al. (1998) confirmed that both the (+)- and (-)-enantiomers of germacrene D in the oil of *S. canadensis* by characterizing two enantioselective germacrene D synthases. They found that the (+)-germacrene D:(-)-germacrene

D ratio was 4:3. In addition, Schmid et al. characterized:

β -caryophyllene (3.0%)
 α -humulene (1.0%)
 δ -cadinene (7.0%)
germacrene A (7.0%)

Other minor sesquiterpenes found were α -ylangene, β -bourbonene and β -gurjunene.

Kasali et al. (2001) reported that an oil off *S. canadensis* produced from plant material of Polish origin contained the

following compounds (using GC/MS only):

(E)-2-hexenol (0.3%)
 α -pinene (2.9%)
camphene (0.4%)
 β -pinene (0.5%)
myrcene (5.1%)
limonene (2.7%)
bornyl acetate (1.8%)
 α -copaene (1.2%)
 β -elemene (1.4%)
 β -caryophyllene (1.5%)
 α -humulene (0.3%)
germacrene D (23.8%)

β -selinene (0.5%)
 bicyclogermacrene (5.0%)
 δ -amorphene (0.9%)
 γ -cadinene (4.1%)
 δ -cadinene (0.4%)
 α -cadinene (0.6%)
 germacrene B (6.3%)
 6-epi-cubenol (2.9%)

In addition, trace amounts of 6-epi- α -cubebene and 6-epi- β -cubebene were characterized for the first time in this oil (**F-1**).

Prosser et al. (2002) determined that the (+)- form of germacrene A was the only form found in *S. canadensis* oil.

Kasali et al. (2002) published a full paper on the information presented in their 2001 poster.

Kalemba and Thiem (2004) used in vitro cultures on *S. canadensis* seeds and raised the resulting plants in an experimental garden at the University of Pozan (Poland). An oil produced by hydrodistillation from the plants harvested during their full flowering period was analyzed by GC-FID and GC/MS. The results of this analysis are as follows:

α -pinene (59.5%)
 camphene (0.8%)
 sabinene (0.7%)
 β -pinene (2.8%)
 myrcene (1.7%)
 limonene (9.7%)

(E)- β -ocimene (0.1%)
cis-verbenol + *trans*-pinocarveol (0.2%)
trans-verbenol (0.5%)
 pinocarvone (0.1%)
 myrtenal (0.1%)
 bornyl acetate (2.0%)
 δ -elemene (0.3%)
 β -bourbonene (0.1%)
 β -cubebene + β -elemene (0.7%)
 β -caryophyllene (1.1%)
 β -gurjunene (0.6%)
 aromadendrene (0.1%)
 α -humulene (0.4%)
 γ -gurjunene (0.1%)
 ar-curcumene (0.1%)
 germacrene D (15.2%)
 β -selinene (0.2%)
 germacrene B (0.1%)
 spathulenol (0.1%)
 caryophyllene oxide (0.1%)
 α -muurolol (0.1%)

Trace amounts (<0.1%) of α -thujene, α -phellandrene, p-cymene, *cis*-sabinene hydrate, linalool, campholenic aldehyde, borneol, terpinen-4-ol, myrtenol, verbenone, carvone, α -copaene, bicyclogermacrene, δ -cadinene, (E)-nerolidol, salvia-4(14)-en-1-one, humulene epoxide II, torilenol (syn. 6,8-cyclo-eudesm-4(14)-en-1-ol), junenol (syn. eudesm-4(14)-en-6 α -ol), α -cadinol, eudesma-4(15),7-dien-1 β -ol and cyclocolorenone were also characterized in this oil.

Ibrahim et al. (2006) obtained *S. canadensis* plants from an experimental farm in Kalubeya (Egypt). An oil produced from these plants (presumably in full bloom) in 0.34% yield was analyzed by GC/MS only. The components characterized in this oil were as follows:

α -pinene (6.1%)
 camphene (0.3%)
 β -pinene (4.6%)
 sabinene (5.7%)
 myrcene (25.6%)
 limonene (16.5%)
 β -phellandrene (11.5%)
 p-cymene (0.3%)
 terpinolene (0.2%)
 δ -elemene (0.5%)
 linalool (0.4%)
 bornyl acetate (1.7%)
 terpinen-4-ol (1.9%)
 β -elemene (0.2%)
 β -cubebene (0.3%)
 α -ylangene (0.3%)
 4,4,5-trimethylcyclononen-1-one[†] (1.1%)
 borneol (0.6%)
 germacrene D (2.9%)
 γ -cadinene (7.3%)
 farnesene^{*} (1.0%)
 α -muurolene[†] (0.3%)
 p-cymen-8-ol (1.1%)
 germacrene D-4-ol (0.3%)
 caryophylla-2(12),5-dien-13-al[†] (0.2%)
 viridiflorol (0.4%)
 muurolol^{*} (0.3%)
 elema-1,3,11(13)-trien-12-ol[†] (1.2%)
 4 α -isopropyl-2-carene[†] (1.0%)
 aromadendrene[†] (0.9%)
 aromadendrene epoxide[†] (0.4%)
 ascaridole[†] (0.3%)

^{*}correct isomer not identified

[†]incorrect identification

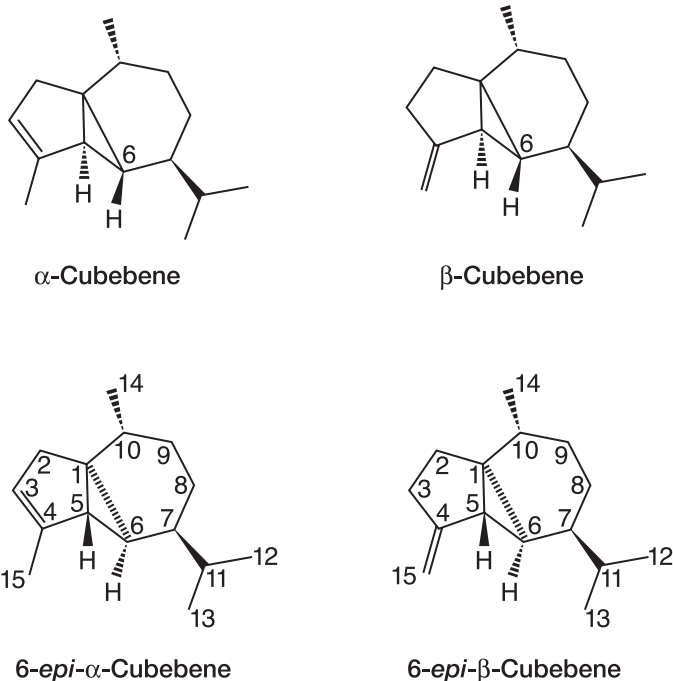
It should be pointed out that this is an atypical oil of *S. canadensis*.

Occasionally, oil of *S. canadensis* is found as an item of commerce.

Mishra et al. (2010) (and a second identified report) analyzed a steam-distilled oil of the roots of *S. canadensis* (origin, Bhimtal, Uttarkhand, India) using GC-FID and GC/MS. The constituents characterized in this oil, which was produced in 0.5% yield, were as follows:

α -pinene (0.3%)
 camphene (0.1%)
 sabinene (0.2%)
 β -pinene (0.7%)
 myrcene (0.6%)
 p-cymene (0.3%)
 limonene (4.3%)
 1,8-cineole (2.9%)

F-1. Cubebene isomers



(E)- β -ocimene (1.1%)
 terpinolene (0.2%)
trans-carveol (2.2%)
 methyl thymol (1.5%)
 methyl carvacrol (2.0%)
 bornyl acetate (1.3%)
 thymol (20.3%)
 carvacrol (5.5%)
 δ -elemene (1.3%)
 α -copaene (6.3%)
 α -ylangene (0.1%)
 isolekene (1.0%)
 β -elemene (0.1%)
trans- α -bergamotene (0.1%)
 γ -muurolene (1.4%)
 γ -curcumene (2.2%)
 germacrene D (2.1%)
 zingiberene (0.2%)
 α -bisabolene^e (1.4%)
 δ -cadinene (0.5%)
 (E)-nerolidol (1.8%)
 β -copaen-4 α -ol (3.5%)
 cubeben-11-ol (3.3%)
 junenol (2.1%)
 1,10-di-epi-cubenol (0.1%)
 β -cedren-9-one (0.2%)
 eremoligenol (0.2%)
 muurola-4,10(14)-dien-1 β -ol (0.5%)
 T-cadinol (0.9%)
 α -muurolol (1.5%)
 14-hydroxy- α -muurolene (0.1%)

^e correct isomer not identified

linalool (0.4%)
trans-sabinene hydrate (0.1%)
 β -cubebene (0.1%)
p-menth-2-en-1-ol^e (0.1%)
 bornyl acetate (3.1%)
 α -bergamotene^e (0.1%)
 β -elemene (0.5%)
 terpinen-4-ol (0.7%)
 β -caryophyllene (0.4%)
 terpinen-1-ol (0.1%)
 γ -elemene (0.1%)
 α -amorphene (0.3%)
 unknown sesqui hydrocarbon (0.4%)
 α -humulene (0.2%)
 α -terpineol (0.3%)

borneol (0.4%)
 germacrene D (21.9%)
 valencene (0.5%)
 bicyclgermacrene (0.5%)
 δ -cadinene (0.3%)
 α -cadinene (0.1%)
 geraniol (0.1%)
 (Z)-nerolidol (0.2%)
 elemol (0.1%)
 spathulenol (0.1%)
 α -cadinol (0.3%)
 α -copaen-8-ol (0.2%)
 (E)-nerolidol (20.8%)

^e correct isomer not identified
^f furanoid form

Amtmann (2010) examined the chemical relationship between the oil of *S. canadensis* and the volatiles of honey produced uniflorally from goldenrod. Using a Likens-Nickerson simultaneous distillation extraction apparatus, an oil was obtained from 200 mg of homogenized flower heads of *S. canadensis*. The oil was analyzed using GC/MS only. The components characterized in this oil were as follows:

α -pinene (14.8%)
 camphene (0.5%)
 β -pinene (2.7%)
 sabinene (2.8%)
 myrcene (10.0%)
 α -phellandrene (10.9%)
 α -terpinene (0.3%)
 limonene (0.9%)
 β -phellandrene (0.3%)
 γ -terpinene (0.4%)
p-cymene (0.6%)
 terpinolene (0.2%)
 (Z)-3-hexenol (0.1%)
 nonanal (0.2%)
cis-linalool oxide^f (0.1%)
trans-linalool oxide^f (0.2%)
 δ -elemene (0.5%)
 decanal (0.2%)

A recent report by Huang et al. (2012) described the analysis and cytotoxic activity of the flower oil of *S. canadensis* collected from invasive wild plants found in the suburbs of Shanghai. Unfortunately, the analysis contained too many errors to be included in this review.

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