

Progress in Essential Oils

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Calendula Oil

Calendula officinalis L., which is known in the United Kingdom as poet's marigold, is also known as pot marigold, Scotch marigold, garden marigold, common marigold, etc. It is a considerably branched annual member of the Asteraceae (Compositae family). A limited amount of oil is produced from this common garden ornamental plant.

Calendula officinalis flowers that were collected from the Institute of Field and Vegetable Crops (Novi Sad, Serbia) were separately subjected to hydrodistillation and supercritical and subcritical fluid CO₂ extraction using a range of pressures. The hydrodistilled oil was produced in 0.05% yield, while the supercritical CO₂ extractions were carried out at 40°C at 100 bar, 200 bar and 300 bar for 3 hrs. In contrast, the subcritical extraction was carried out at 15°C at 60 bar. 90 bar and 120 bar for three hours. The oils and extracts were analyzed by both GC-FID and GC/MS by Petrovic et al. (2007). The composition of the oil was determined to be as follows:

 γ -muurolene (0.7%) ledene (1.6%) α -muurolene (1.2%) γ -cadinene (3.0%) $ledane^{\dagger}$ (2.5%) ledol (0-.7%) cubenol (1.4%) T-cadinol + T-muurolol (9.8%) β-eudesmol (1.6%) α -cadinol (26.5%) hexadecanoic acid (3.0%) 2-pentadecanone (1.7%) heptadecane (1.5%) octadecane (0.9%) nonadecane (1.9%) eicosane (1.2%)

[†]doesn't occur naturally

The composition of the super- and subcritical CO_2 extracts were produced in 4.2% yield with a 2.94% oil content, and 1.87% yield with a 7.23% oil content, respectively, can be seen in **T-1**.

Gazim et al. (2007) analyzed an oil produced from *C. officinalis* that had become acclimatized in the northeast region of Parana State (Brazil) using GC/ MS only. The components characterized in this oil were as follows:

 $\begin{array}{l} \alpha \text{-copaene} \ (1.0\%) \\ \alpha \text{-ionone}^{*\dagger} \ (1.5\%) \\ \alpha \text{-humulene} \ (1.2\%) \\ \text{geranyl acetone} \ (1.6\%) \\ \gamma \text{-muurolene} \ (2.3\%) \end{array}$

T-1. Comparative percentage composition of supercritical and subcritical CO $_2$
extracts of Calendula officinalis

Compound	Supercritical extract ^a	Subcritical extract ^b
thymol	0.6	-
α -cubebene	0.4	0.3
α -copaene	0.6	0.6
α -gurjunene	-	0.4
β-caryophyllene	0.4	-
β-gurjunene	-	0.3
α-humulene	0.9	0.3
<i>cis</i> -muurola-4(14),5-diene	-	0.3
γ-muurolene	1.5	1.4
β-ionone [*]	1.7	1.0
ledene	3.4	2.1
α -muurolene	3.0	3.5
γ-cadinene	9.6	11.4
δ-cadinene	15.7	19.4
cadina-1,4-diene	0.6	0.9
α -cadinene	1.6	2.0
lpha-calacorene	0.4	0.3
ledane [†]	3.8	1.8
ledol	1.0	0.4
cubenol	0.9	0.6
T-cadinol + T-muurolol	8.2	7.6
α -cadinol	19.1	17.6
hexadecanoic acid	0.3	-
2-pentadecanone	0.5	0.8
heptadecane	2.6	2.7
octadecane	1.1	1.2
nonadecane	1.8	2.7
eicosane	1.2	2.2
^a 40°C at 300 bar		
^b 15°C at 90 bar *correct isomer not identified		
*		

[†]does not occur naturally

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β-ionone^{*†} (3.3%) ledene (2.3%) α-muurolene (5.6%) γ-cadinene (8.9%) δ-cadinene (22.5%) α-cadinene (0.9%) α-calacorene (2.3%)

 $\begin{array}{l} caryophyllene \mbox{ oxide } (0.5\%) \\ copaen-4-ol \ (0.6\%) \\ \beta\mbox{-oplopenone } (1.7\%) \\ viridiflorol \ (2.3\%) \\ ledol \ (1.3\%) \\ 1,10\mbox{-di-epi-cubenol} \ (0.9\%) \\ 1\mbox{-epi-cubenol} \ (1.6\%) \end{array}$

T-2. Comparative percentage composition of oils produced from *Calendula officinalis* at selected harvest times

Compound	3	6	9	12
α -thujene	-	0.2	0.1	0.1
α-pinene	0.1	1.6	2.5	2.9
sabinene	0.2	0.7	0.9	0.9
β-pinene	0.1	0.8	1.1	1.4
limonene	0.2	1.4	1.6	2.6
1,8-cineole	11.1	13.1	15.3	22.1
p-cymene	0.1	0.5	0.1	0.1
(E)-β-ocimene	0.2	0.8	1.8	2.0
γ-terpinene	0.1	0.2	0.1	0.1
δ-3-carene [†]	0.3	0.7	0.1	t
nonanal	0.1	2.4	0.4	t
terpinen-4-ol	0.1	-	-	0.1
3-cyclohexen-1-ol [†]	0.1	-	-	t
$lpha$ -phellandrene †	1.1	t	t	t
α -terpineol	0.1	0.4	0.2	0.1
geraniol	10.9	10.3	21.5	44.5
carvacrol	-	0.1	-	0.1
bornyl acetate	-	-	-	t
sabinyl acetate [*]	0.1	0.1	0.1	t
lpha-cubebene	-	0.1	-	1.7
α -copaene	-	-	t	t
lpha-bourbonene	t	-	-	0.2
β-cubebene	-	-	-	t
lpha-gurjunene	-	t	-	0.1
aromadendrene	t	-	-	t
β-caryophyllene	-	t	-	0.9
lpha-ylangene	0.1	0.2	0.3	0.8
α -humulene	1.0	1.3	1.4	1.7
1-epi-bicyclosesquiphellandrene	-	0.1	0.4	0.5
germacrene D	t	0.2	1.3	11.5
allo-aromadendrene	0.1	-	-	0.2
β-selinene	0.3	-	-	0.3
β-gurjunene	0.2	0.4	5.0	5.7
α-muurolene	-	2.5	5.4	0.1
δ-cadinene	0.5	2.4	8.5	23.8
cadina-1,4-diene	0.7	-	0.2	12.2
α-cadinene	1.5	1.6	8.0	10.7
nerolidol*	0.6	1.3	1.1	1.3
palustrol	1.2	0.2	-	0.7
1-endo-bourbonol	0.1	-	t	1.0
β-oplopenone	-	t	-	t
α-cadinol	0.1	6.4	9.4	24.2
T-muurolol	12.5	15.4	18.8	22.5

3 = third week after transplanting oil yield 0.13%; 6 = sixth week after transplanting oil yield 0.48%; 9 = ninth week after transplanting oil yield 0.48%; 9 = ninth week after transplanting oil yield 0.97% *correct isomer not identified

[†]incorrect identification

 $\begin{array}{l} T\text{-muurolol} \ (12.9\%) \\ \alpha\text{-cadinol} \ (20.4\%) \\ cadalene \ (0.8\%) \end{array}$

[°]correct isomer not identified; [†]probably mixed GC peak because of unusual quantitative amount

Seeds of wild populations of *C. officinalis* that were collected in the Eastern Cape Province (South Africa) were initially planted in the nursery of University of Fort Hare after which the seedlings were transplanted into the University of Research Farm by Okoh et al. (2007). Plants were harvested from the third week after transplanting until the 12th week after transplanting. Oils that were produced from the separate harvests were hydrodistilled for three hours. and then analyzed by GC/MS only. The results of these analyses from selected harvest times can be seen in **T-2**.

Okoh et al. (2008) studied the effect of leaf drying on the composition of *C. officinalis* oil produced from plants collected from the wild around the University of Fort Hare (Eastern Cape Province, South Africa). In addition, the authors compared the composition of flower oils with the leaf oils using GC/ MS as their only analytical process. The comparative results of this study are shown in **T-3**.

Gazim et al. (2008) republished the results of an analysis that was first published by the same authors (Gazim et al., 2007), In addition, the authors used a combination of GC-FID and GC/MS to examine the headspace volatiles of powdered dried flowers of *C. officinalis* obtained from Parana State (Brazil). The components identified in the headspace of the flowers were as follows:

 $\begin{array}{l} \beta \mbox{-cyclocitral} (2.1\%) \\ \alpha \mbox{-cubebene} (1.8\%) \\ \alpha \mbox{-copaene} (15.1\%) \\ \beta \mbox{-cubebene} (1.8\%) \\ \alpha \mbox{-gurjunene} (2.7\%) \\ \beta \mbox{-caryophyllene} (2.7\%) \\ \alpha \mbox{-ionone}^* (2.3\%) \\ \alpha \mbox{-ionone}^* (2.3\%) \\ \alpha \mbox{-nunuclene} (3.9\%) \\ \gamma \mbox{-muurolene} (5.3\%) \\ \beta \mbox{-ionone}^* (3.9\%) \\ \alpha \mbox{-muurolene} (6.2\%) \\ \gamma \mbox{-cadinene} (22.1\%) \\ \alpha \mbox{-cadinene} (2.3\%) \end{array}$

°correct isomer not identified

Petrovic et al. (2010) used GC-FID and GC/MS to examine the difference

in composition of oil produced from the head flowers with oils produced from both tubular flowers and ligulate flowers of *C. officinalis*. The comparative oil yields were 0.054% for head flowers, 0.131% for tubular flowers and 0.166% for ligulate flowers. Their compositions are summarized in **T-4**.

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- O.O. Okoh, A.A. Sadimenko and A.J. Afolayan, *The* effects of age on the yield and composition of the essential oils of Calendula officinalis. J. Appl. Sci., **7**, 3806–3810 (2007).

T-3. Comparative percentage composition of the leaf (fresh and dry) and flower oils of *Calendula officinalis*

		B · · · · · · ·	
Compound	Fresh leaf oil	Dried leaf oil	Flower oil
α-thujene	19.2	17.8	26.9
α -pinene	-	2.4	1.8
sabinene	1.1	-	1.8
β-pinene	0.6	6.9	-
myrcene	-	-	1.1
limonene	0.8	-	-
1,8-cineole	-	29.4	1.7
(E)-β-ocimene	0.2	-	-
γ-terpinene	0.4	-	0.7
δ -3-carene [†]	-	0.3	-
nonanal	-	1.0	-
terpinen-4-ol	0.4	-	0.6
4-methyl-3-cyclohexen-1-ol [†]	-	0.6	-
α -terpineol	-	0.6	-
bornyl acetate	0.1	-	-
lpha-cubebene	0.2	-	-
α -copaene	0.3	0.2	0.2
lpha-bourbonene	0.3	0.2	-
β-cubebene	0.4	0.2	0.5
lpha-gurjunene	0.6	-	0.6
aromadendrene	-	0.2	-
β-caryophyllene	1.0	-	1.2
lpha-ylangene	0.2	-	-
α -humulene	1.7	1.2	1.5
1-epi-bicyclosesquiphellandrene	0.4	-	-
lpha-amorphene	0.6	-	0.5
$lpha$ -copaene †	-	-	2.7
allo-aromadendrene	-	-	0.3
β-selinene	0.5	-	-
germacrene D	1.1	0.6	2.8
$lpha$ -cubebene †	-	1.4	0.2
α -muurolene	2.1	1.6	-
γ-cadinene	2.7	2.2	2.2
δ-cadinene	11.4	9.0	13.1
cadina-1,4-diene	0.5	-	0.4
lpha-cadinene	0.6	0.4	0.4
nerolidol [*]	-	-	0.9
palustrol	0.2	-	-
β-gurjunene	2.3	0.5	-
1-endo-bourbonol	0.6	-	0.5
β-oplopenone [*]	0.3	-	-
T-muurolol [‡]	40.9	13.1	24.9
*correct isomer not identified			

*correct isomer not identified †incorrect identification

^tprobably incorrect identification because α -cadinol is normally the major constituent of *Calendula officinalis* oil

- Z.C. Gazim, G.A. Ferreira, C.M. Rezende, C.V. Nakamura, B.P. Dias Filho and D.A. Garcia Cortez, *Chemical compounds of the* Calendula officinalis volatile fraction produced in the Parana State, Brazil. Hort. Braz., 25, 118–121 (2007).
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Dill Weed and Dill Seed Oil

Dill weed oil is obtained by steam distillation of Anethum graveolens L. that is harvested when the plant is in full flower and the primary umbel has started to turn from yellow to orange-brown. Dill is grown commercially primarily in Bulgaria, Canada, Hungary, Poland and the United States. Numerous cultivars of dill are available, such as 'Amat,' Ambrozja,' 'Bouquet,' 'Crown,' 'Diwa,' 'Dukat,' 'Dura,' 'Elefant,' 'Fernleaf,' 'Gewöhnlicher,' 'Hercules,' 'Long Island Mammoth,' 'Lukullus, 'Mammut,' 'Mesten,' 'Sari,' 'Tetra' and 'Vierling,' with 'Long Island Mammoth' and 'Bouquet' being the most popular for dill weed oil production due to their high fresh flower weight and umbel formation (Tucker and De Baggio, 2000).

Dill seed oil, which is mainly produced in India, was once thought to be obtained from *Anethum sowa* Roxb. ex Flemming; however, more recently it has been classified as a synonym of *A. graveolens*. According to Tucker and Debaggio, the most important cultivars are 'Variyali' (dark and pale selections), 'Ghoda' and 'Vizag.'

Dill is an annual member of the Apiaceae (Umbelliferae) family that is native to southeastern Europe. It is propagated by seed; consequently, the volume of dill weed oil can readily fluctuate because of supply and demand. It is estimated that the annual production of dill weed oil is 180–200 metric tonnes.

Gora et al. (1977) determined that the main components of a commercial dill

T-4. Comparative percentage composition of the flower oils of Calendula officinalis grown in Serbia

Compound	Flowerhead oil	Ligulate flower oil	Tubular flower oil
lpha-copaene	-	0.2	0.5
γ-muurolene	-	1.0	0.6
germacrene D	-	-	1.9
β-ionone [*]	1.7	0.3	0.4
ledene	2.0	2.5	4.5
α -muurolene	1.1	2.8	4.8
γ-cadinene	1.5	4.7	8.6
δ-cadinene	5.2	3.2	10.9
cadina-1,4-diene	-	0.3	-
lpha-cadinene	-	1.0	1.8
lpha-calacorene	-	0.4	0.6
β-calacorene	9.0	3.8	4.9
ledol	2.1	1.2	1.4
cadina-3,9-diene	7.0	6.3	11.2
cubenol	3.2	1.9	1.8
T-cadinol + T-muurolol	2.2	6.8	1.2
β-eudesmol	2.1	-	-
α-cadinol	29.1	27.3	21.0
cadalene	2.2	1.1	0.6
heptadecane	1.0	0.4	3.6
octadecane	2.7	0.2	-
nonadecane	1.1	0.4	2.9
eicosane	1.0	0.2	0.7
*correct isomer not identified			

T-5. Percentage enantiomeric distribution of limonene in various plant parts of Anethum graveolens

Plant Part	(-)-4S)-Limonene	(+)-(4R)-Limonene
leaf	30–33	67–70
bud	3–10	90–97
flower	2	98
seed	<1	>99

T-6. Comparative percentage composition of the main constituents of dill weed oil produced in Canada from three cultivars

Oil 1	Oil 2	Oil 3
26.1-34.6	34.2-41.2	28.6-39.6
0.3–1.1	0.3–1.4	0.2-0.9
21.5-28.1	22.5-27.1	23.9-29.8
3.2–3.8	3.4-4.6	3.1–4.3
13.1–13.4	16.5–18.0	12.3–13.1
0.8–1.9	0.5–0.7	0.7–0.9
24.6-27.2	11.3–17.1	14.4–23.5
	26.1–34.6 0.3–1.1 21.5–28.1 3.2–3.8 13.1–13.4 0.8–1.9	26.1–34.6 34.2–41.2 0.3–1.1 0.3–1.4 21.5–28.1 22.5–27.1 3.2–3.8 3.4–4.6 13.1–13.4 16.5–18.0 0.8–1.9 0.5–0.7

*correct isomer not identified Oil 1: from 'Mesten' cultivar; Oil 2: from 'Dukat' cultivar; Oil 3: from 'Hercules' cultivar

oil produced in Poland were limonene (25.8%) and carvone (68.4%). It should be noted that this level of both limonene and carvone is not consistent with the typical levels found in North American *A. graveolens* herb oil.

Faber et al. (1997) used chiral GC to show that the enantiomeric distribution of limonene in various organs of dill was different, as can be seen in **T-5**. In addition, the authors used gas chromatography-combustion-isotopic ratio mass spectrometry (GC-C-IRMS) to study the biosynthetic pathways within the plant. Based on their results, the authors concluded that there was a very close biogenetic relationship between limonene and carvone, and dill ether (3,9-epoxy-p-menth-1-ene) and α -phellandrene.

Bowes et al. (2001) examined the influence of seeding date and cultivars on the oil composition of both dill weed oil and dill seed oil. They found that the earlier the seeding date (May 24) in Truro (Nova Scotia, Canada), versus a later date (June 8), resulted in greater productivity of the 'Mesten' and Dukat' cultivars. Furthermore, the authors recommended that as the composition of the dill weed oils and dill seed oils obtained particularly from the 'Mesten' and 'Dukat' cultivars (as shown in T-6 and **T-7**) was similar to commercially available oils, Nova Scotia could readily become a dill-producing region.

Tomar and Dureja (2001) characterized 2-methoxy-3,4-methylenedioxy-1-propanone as a minor constituent of Indian dill seed.

Five cultivars of dill are grown in Poland. They are 'Ambrosia,' 'Corso,' 'Lucullus,' 'Emerald' and 'Turquoise.' Gora et al. (2002) examined the effect of growth stage of dill herb ('Corso' cultivar) on the composition of oils produced from plants harvested at various ontogenetic stages. The results of this study can be seen in T-8. Examination of the date reveals that the composition of dill herb oil changes with a large reduction in the α -phellandrene and dill ether contents with a corresponding increase in the limonene and carvone, making the dill seed oil more carawaylike than dill-like.

Orav et al. (2003) compared the oil composition of fresh (0.80% yield) and dried (4.3% yield) leaves and stems of *A. graveolens* collected prior to flower development from plants grown commercially in Estonia. The results of this study are shown in **T-9**.

Dill seeds that were purchased form a local market in Gorakhpur (Uttar Pradesh, India) were ground and subjected to hydrodistillation by Singh et al. (2005). In addition, an acetone extract was produced from the same batch of ground dill seeds. Analysis of the oil using GC-FID and GC/MS revealed that it possessed the following composition: $\begin{array}{l} \alpha \text{-pinene} \ (0.1\%) \\ \text{sabinene} \ (0.1\%) \\ \beta \text{-pinene} \ (0.1\%) \\ myrcene \ (0.1\%) \\ limonene \ (16.6\%) \\ \text{sylvestrene} \ (0.1\%) \\ \gamma \text{-terpinene} \ (0.3\%) \\ linalool \ (3.7\%) \\ \text{terpinen-4-ol} \ (0.1\%) \\ \text{dill ether} \ (0.2\%) \\ \textit{cis-dihydrocarvone} \ (2.6\%) \\ \textit{trans-dihydrocarveol} \ (0.1\%) \\ \end{array}$

T-7. Comparative percentage composition of the main components of dill seed oil produced from three cultivars grown in Canada

Compound	Oil 1	Oil 2	Oil 3
α-pinene	0.2-0.3	0.2–0.3	0.3
lpha-phellandrene	3.2–3.3	3.0-4.4	3.2-5.7
p-cymene	0.2-0.3	0.3	0.4-0.7
limonene	28.0-42.0	31.9–39.3	32.2-41.6
dill ether	0.8–1.0	0.5-0.7	0.6-0.9
dihydrocarvone [*]	2.0-2.2	2.2-2.5	2.2-2.7
carvone	50.5-64.0	51.8–59.4	47.6–59.4

*correct isomer not identified; Oil 1: 'Mesten'; Oil 2: 'Dukat'; Oil 3: 'Hercules'

T-8. Comparative percentage composition of oils produced from the 'Corso' cultivar of *Anethum graveolens* harvested at different growth stages

Compound	1	2	3	4	5	6
lpha-thujene	0.41	0.57	0.17	0.22	t	t
α -pinene	2.13	3.08	1.38	1.21	t	t
camphene	0.03	t	t	t	t	t
sabinene	0.92	0.86	0.69	0.68	0.13	0.12
β-pinene	0.12	0.13	t	0.04	t	t
lpha-phellandrene	53.56	10.33	54.20	35.15	0.26	0.99
α -terpinene	0.43	0.56	1.69	0.42	0.67	0.37
limonene	3.52	5.34	7.95	33.69	40.81	36.67
β -phellandrene	7.77	6.36	7.60	4.83	0.06	0.05
p-cymene	6.45	32.75	2.88	4.25	0.28	0.31
1,8-cineole	0.05	t	t	t	t	t
p-cymene	t	0.22	t	0.01	t	t
<i>cis</i> -p-menth-2-en-1-ol	0.04	0.44	t	0.01	t	t
dill ether	11.65	10.53	21.61	10.75	0.19	0.20
<i>cis</i> -dihydrocarvone	0.62	2.13	t	0.14	t	t
<i>trans</i> -dihydrocarvone	0.06	0.80	0.08	1.06	1.94	1.49
carvone	0.16	0.68	t	4.34	54.46	58.73
carvacrol	1.81	6.24	t	0.59	t	t
germacrene D	0.35	0.02	t	0.09	t	t
myristicin	0.03	0.18	t	t	t	t
apiole	t	t	t	t	t	t

t=trace (<0.01%)

stages 1: young-leafed sprouts; 2: vegetative growth prior to umbel formation; 3: beginning of blooming; 4: full flowering stage; 5: green fruit stage; 6: ripe fruit stage

T-9. Comparative percentage composition of the herbage oil of *Anethum* graveolens produced from fresh and dried plant material

Compound	Fresh plant oil	Dried plant oil
α -thujene	0.3	0.4
α -pinene	1.6	2.0
camphene	t	t
sabinene	0.1	0.1
β-pinene	0.1	0.1
myrcene	0.6	0.6
lpha-phellandrene	75.1	75.8
p-cymene	0.6	1.2
limonene	2.8	2.9
β-phellandrene	7.9	7.4
(Z)-β-ocimene	0.1	0.1
(E)-β-ocimene	1.3	1.2
p-cymenene	0.4	0.4
terpinolene	0.4	0.5
dill ether	3.9	2.2
carvone	t	t
bornyl acetate	t	0.1
β-farnesene [*]	0.2	0.2
germacrene D	0.3	0.4
myristicin	0.5	0.3
β-bisabolene	0.1	0.1
phthalide [*]	0.2	0.3
T-cadinol	0.2	0.1
apiole	0.3	0.5
farnesol [*]	0.1	0.3
t=trace (<0.1%) *correct isomer not identified		

 $\begin{array}{l} trans\mbox{-carveol} \ (0.1\%) \\ {\rm carvone} \ (55.2\%) \\ \alpha\mbox{-ylangene} \ (0.1\%) \\ {\rm geranyl} \ {\rm acetate} \ (0.3\%) \\ \beta\mbox{-caryophyllene} \ (0.6\%) \\ \beta\mbox{-bisabolene} \ (0.3\%) \\ \delta\mbox{-cadinene} \ (0.1\%) \\ trans\mbox{-isocroweacin} \ (0.8\%) \\ {\rm dillapiole} \ (14.4\%) \end{array}$

Trace amounts (<0.01%) of isovaleraldehyde, 2-methylbutanal, α -thujene, p-cymene, terpinolene, camphor, isocaryophyllene, α -humulene, germacrene D, β -selinene, *trans*- β -guaiene, elemicin and apiole were also characterized in the same oil.

Analysis of the acetone dill seed extract using the same analytical techniques (Singh et al., 2005) revealed that it contained:

$$\begin{split} & \text{limonene } (0.5\%) \\ & \text{carvone } (3.1\%) \\ & \text{geranyl acetate } (0.3\%) \\ & \beta\text{-caryophyllene } (0.2\%) \\ & \beta\text{-bisabolene } (0.3\%) \\ & \text{dillapiole } (43.2\%) \\ & \text{apiole } (0.1\%) \\ & \text{undecane } (0.5\%) \\ & (E)\text{-anethole } (11.0\%) \\ & \text{eugenol } (0.2\%) \\ & 1\text{-}(4\text{-methoxyphenyl})\text{-}2\text{-propanone } (4.6\%) \\ & 1\text{-}(4\text{-methoxyphenyl})\text{-}1\text{-propanone } (0.2\%) \\ & \text{dodecanal } (0.4\%) \end{split}$$

myristicin (1.5%) p-anisaldehyde (2.7%) T-muurolol (0.2%) foeniculin* (0.9%) heptadecane (0.1%) hexahydrofarnesylacetone (0.1%) hexadecanoic acid (1.3%) linoleic acid (23.1%) 10-nonadecanone (0.1%)

° correct isomer not identified

Trace amounts of *trans*-dihydrocarvone, *trans*- α -bergamotene and neophytadiene were also characterized in the extract.

Zorca et al. (2007) used GC/MS only to examine the composition of dill seed produced in Romania. The authors also compared the oil composition to supercritical CO₂ extracts of the same batch of dill seed produced under variation in CO₂ density. It was found that the higher the CO₂ density, the higher the extraction yield. The comparative results of the analyses are presented in **T-10**.

Moshfekus Saleh-e-in et al. (2008) used GC/MS only to analyze an oil produced in 0.052% yield from the stems of so-called Indian dill grown in Bangladesh. Examination of the data reveals that most of the components purported to be present in the oil were most probably misidentified. However, they did find that apiole (45.6%) was the major constituent of the oil.

A commercial sample of dill oil was screened as a fumigant for its antitermitic activity. The oil, which was analyzed by GC-FID and GC/MS by Seo et al. (2009) and was found to possess the following composition:

α-pinene (0.4%)
myrcene (0.2%)
α-phellandrene (4.9%)
p-cymene (2.3%)
1,8-cineole (1.0%)
limonene (20.5%)
dill ether (4.6%)
trans-dihydrocarvone (0.8%)
cis-dihydrocarvone (0.9%)
carvone (35.6%)

Tibaldi et al. (2010) studied the effect of maturity stage on oil composition of *A. graveolens* grown at the Experimental Centre of University of Torino (Carmagnola, Italy) during the summer. The plants, which were harvested at the pre-blooming stage and at the post-blooming stage, were separately subjected to hydrodistillation. The compositions of the oils, which were analyzed by GC, can be seen in **T-11**.

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T-10. Comparative percentage composition of an oil and $\rm CO_2$ extracts of Anethum graveolens seed of Romanian origin

Compound	Oil	SFE-1	SFE-2	SFE-3
α -thujene	0.2	0.2	0.3	0.1
α-pinene	0.1	0.1	0.1	0.2
camphene	0.2	0.2	0.1	0.1
sabinene	0.1	0.1	0.1	0.1
β-pinene	0.1	-	0.1	0.1
myrcene	0.1	0.1	0.2	0.1
lpha-phellandrene	2.8	3.4	1.6	1.8
p-cymene	0.5	0.3	0.4	0.2
limonene	33.8	30.7	33.3	35.3
1,8-cineole	0.4	0.3	0.1	0.1
γ-terpinene	1.3	1.2	1.0	1.2
p-menth-2-en-1-ol [*]	1.9	1.4	1.3	0.9
linalool	0.9	1.3	1.0	1.0
menthone [†]	0.2	0.3	0.1	0.1
isomenthone [†]	0.1	-	-	0.1
neomenthol [†]	0.1	0.1	0.2	0.2
menthol [†]	0.1	-	-	0.1
dihydrocarvone [*]	1.5	2.8	1.7	1.0
dihydrocarveol*	0.5	0.5	0.5	0.3
carveol*	2.0	2.0	2.0	1.3
carvone	50.0	52.6	53.4	53.7
piperitone	0.2	0.3	0.2	0.1
carvacrol	0.1	0.2	0.2	0.2
thymol	0.3	0.2	0.2	0.1
δ-elemene	0.1	-	0.2	0.3
lpha-cubebene	0.4	0.2	0.1	0.1
α -copaene	0.3	0.3	0.1	0.1
β-elemene	0.1	0.1	0.1	0.2
β-caryophyllene	0.3	0.2	0.1	0.1
α -humulene	0.1	0.1	0.2	0.2
γ-muurolene	0.2	0.2	0.2	0.1
γ-cadinene	0.1	-	-	0.1
germacrene D	0.5	0.4	0.3	0.2
calamenene [*]	0.1	0.1	0.1	0.1
caryophyllene oxide	0.2	0.1	0.1	-
humulene epoxide [*]	0.1	0.1	0.3	0.3
bisabolol oxide [†]	0.1	0.1	0.3	0.3

*correct isomer not identified

[†]either contamination or incorrect identification

SFE-1: CO_2 : 225 g/L, 80 bar at 50°C; SFE-2: CO_2 : 0.403 g/L, 100 bar at 50°C; SFE-3: CO_2 : 0.662 g/L, 120 bar at 40°C; extraction time 120 min for all

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T-11. Comparative percentage composition of oils produced from pre- and post-blooming *Anethum graveolens*

Compound	Pre-blooming oil	Post-blooming oil
α -pinene	2.6	1.0
α-phellandrene	46.4	-
limonene	t	16.1
p-cymene	7.3	-
β-phellandrene	14.1	-
dill ether	22.2	9.2
undecane	t	-
germacrene D	0.3	-
(E)-anethole	t	-
carvone	0.1	29.0
myristicin	0.1	-
apiole	0.1	-
neophytadiene	t	-
phytol	0.4	-
t=trace (0.1%)		

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