Progress in Essential Oils

Brian M. Lawrence, Consultant

Cyperus rotundus Oil and Extract

The roots, or tubers, of purple nutsedge, a pantropical, troublesome weed (*Cyperus rotundus* L.), have been used both for culinary and an oil produced from them for perfume uses since ancient times (Negbi, 1992). The oil, which is produced both in India and China, is obtained by steam distillation of the dried tubers.

Examination of the early literature reveals that oils of Asian origin were reported by Gildemeister and Hoffman (1956) to contain α-pinene, α-cyperone (30–50%), cyperene, cyperol and a trace of 1,8-cineole.

Motl et al. and Trivedi et al. (1963 and 1964) used fractional distillation, alumina column chromatography, UV, IR and ^{1}H-NMR to characterize camphene, β-pinene, limonene, 1,8-cineole, p-cymene, cyperene, β-selinene, α-cyperone, β-cyperone and patchoulenone in a lab-distilled oil of *C. rotundus.* Although the authors did not present any quantitative data they were able to show that the oil was rich in oxygenated sesquiterpenes with α-cyperone being the major component. In addition, the oil was found to contain numerous other non-characterized sesquiterpenoids (**F-1**).

Trivedi et al. (1964) isolated and elucidated a sesquiterpene hydrocarbon from an oil of *C. rotundus* and named it cyperene.

Kapadia et al. (1965) isolated a new sesquiterpene ketone from an Indian oil of *C. rotundus.* It was structurally elucidated to possess a tricyclic skeleton and it was named mustakone.

Hikino et al. (1966a) isolated a sesquiterpene keto-alcohol from *C. rotundus* and named it cyperolone. Hikino et al. (1966b) developed a synthesis for cyperolone to verify their original structural elucidation. The absolute configuration of cyperotundone, another sesquiterpenoid ketone found in the oil of *C. rotundus,* was determined by Hikino et al. (1966c).

Additional examination of the Indian oil of *C. rotundus* by Kapadia et al. (1967) resulted in the characterization of patchoulenone and rotundone (also known as guai-1,5-(11)-dien-2-one).

Further studies on the isolates from *C. rotundus* by Hikino et al. (1967a) resulted in the absolute configuration of cyperolone being determined.

The high-boiling sesquiterpene alcohol fraction of *C. rotundus* was examined by Hikino et al. (1967b) and two new sesquiterpene alcohols were isolated, characterized and their absolute configuration determined. The authors named them cyperol and isocyperol.

Examination of *C. rotundus* oil resulted in the characterization, synthesis and elucidation of the absolute configurations of cyperone, patchoulenone and cyperotundone as constituents by Hikino et al. (1968a).

The column chromatographic separation of *C. rotundus* oil over alumina by Hikino et al. (1968b) using benzene as the eluting solvent afforded the isolation of a sesquiterpene keto-ester, which they named sugeonol acetate.

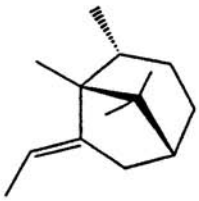
Further column chromatographic separation of the oil of *C. rotundus* resulted in the isolation of two isomeric sesquiterpene alcohols by Hikino et al. (1971). Structural elucidation of both compounds using a combination of spectroscopic techniques allowed their absolute configurations to be determined. The two compounds were named α- and β-rotunol (syn 5α- and 5β-hydroxy-eudesma-3,11-dien-2-one).

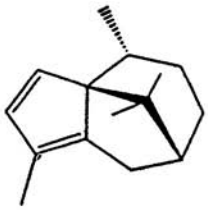
Hikino and Aota (1976) further examined an extract of *C. rotundus* and isolated a sesquiterpene alcohol, which they elucidated to be 4,5-epoxy-eudesm-11-en-3α-ol.

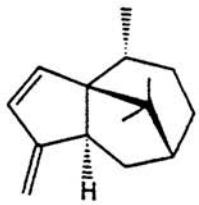
Using a combination of GC/MS and other spectroscopic techniques, two oils of *C. rotundus* were found (Iwamura et al., 1977) to range in composition as follows:

α-copaene (trace)
cyperene (2.1–3.3%)
β-elemene (0.2–0.8%)
β-caryophyllene (0.2–0.6%)
α-humulene (0.2–1.3%)
β-selinene (6.7–13.2%)
δ-cadinene (1.5–2.1%)
calamenene (trace)
cyperotundone (8.5–14.7%)
α-cyperone (29.8–30.1%)

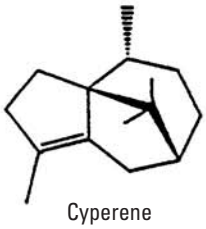
Komai et al. (1977) and Komai and Ueki (1980) became interested in the chemical composition of purple nutsedge tubers because when the plant was found as a weed in plantings of lettuce or rice seedlings it was responsible for inhibition of their growth. Because of the potential alleochemical activity of purple nutsedge, they realized that the tubers must be exuding compounds that were causing the seedling growth inhibition.

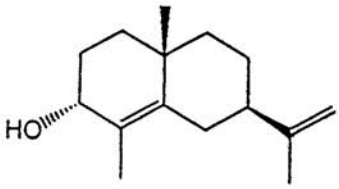

Cyprotene


Cypera-2,4-diene

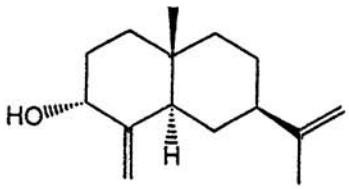


Cypera-2,4(15)-diene

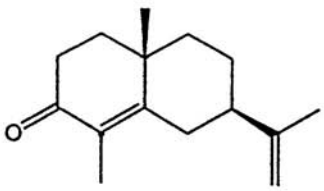

Cyperene



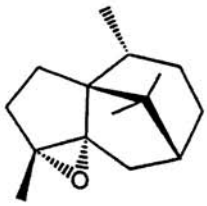
Cyperol (syn. Selina-4,11-dien-3-ol)



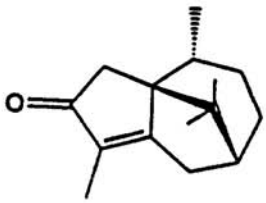
Isocyperol (syn. Selina-4(15),11-dien-3-ol)



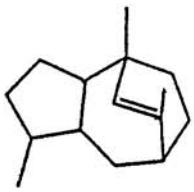
a-Cyperone (syn. Selina-4,11-dien-3-one)

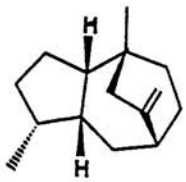


Cyperene epoxide

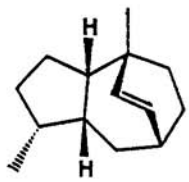


Cyperotundone (syn. cyperenone or articulone)

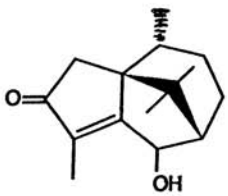

Rotundene (syn. eudesma-3,11-diene)



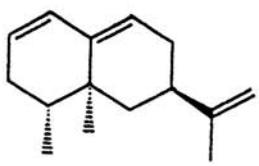
Isorotundene

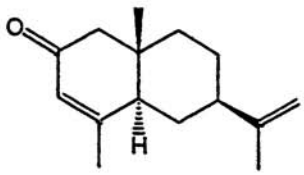


nor-Rotundene

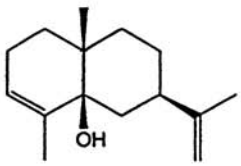


Sugeonol (syn. 6-hydroxycyperotundone)

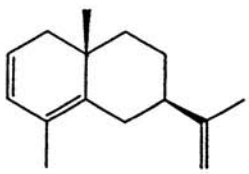

Nootkatene (syn. eremophila-1(10,11-diene)

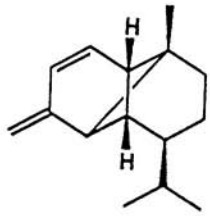


Eudesma-3,11-dien-2-one (syn. rotundone)

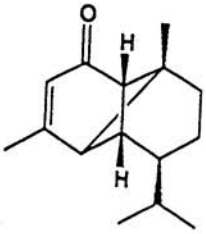


Eudesma-3,11-dien-5-ol

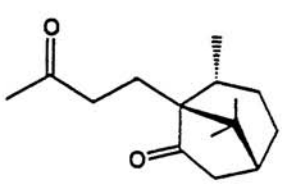

Eudesma-2,4,11-triene



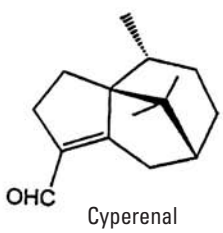
Ylanga-2,4-diene



Mustakone



Cyperadione



Cyperenal

Ramaswami et al. (1988) characterized the presence of cyperene, α-selinene and β-selinene in an oil of *C. rotundus*.

Komai and Tang (1989) analyzed an oil produced from *C.rotundus* tubers collected in Hawaii. They found that the oil contained α-copaene, cyperene, β-elemene, β-caryophyllene, α-humulene, β-selinene, calamenene, δ-cadinene, cyperotundone, α-cyperone, cyperol, patchoulenone, patchoulenol acetate and sugeonyl acetate. The increased level of these two latter compounds made this oil somewhat unique over oils of *C. rotundus* from other geographical sources.

Komai (1991) [and Komai et al. (1993)] reported that a number of chemotypes of *C. rotundus* have been found in the Pacific rim and basin, and the composition of these can be found in **T-1**.

Sugetriol triacetate was isolated from a hexane extract of *C. rotundus* by Yoshizaki et al. (1992). It was characterized by a combination of IR and ^{1}H-NMR. Kalsi et al. (1995) determined that an extract of *C. rotundus* of Punjabi (India) origin contained β-caryophyllene, caryophyllene oxide and caryophylla-6-one.

From a crude hexane extract of *C. rotundus*. Thebtaranonth et al. (1995) isolated and identified patchoulenone, caryophyllene oxide, 4,7-dimethyl-1-tetralone and 10,12-peroxycalamenene.

A methylene chloride extract and an oil of *C. rotundus* of Chadian origin were the subjects of analysis by Mahmout et al. (1997). The results of this study can be found in **T-2**. Furthermore, the authors found that the cyperene and cyperotundone levels in the extract varied throughout the year as 15.2–30.7% and 18.8–25.6%, respectively.

Ohira et al. (1998) analyzed a hexane/water-soluble fractionation of a methanolic extract of *C. rotundus* and found that it contained 4,5-secoeudesmanolide, 10-epi-4,5-secoeudesmanolide, a cyclic acetal cyperolone, musktakone, α-cyperone, nootkatone, α-rotunol and β-rotunol.

An oil produced in the laboratory by Sonwa and König (2001) was analyzed for its sesquiterpenoid content using a combination of GC-FID, GC/MS, ^{1}H-NMR and ^{13}C-NMR. Initially, the known sesquiterpene hydrocarbons such as cyprotene, cypera-2,4-diene, α-copaene, cyperene, α-selinene, rotundene, valencene, ylanga-2,4-diene, γ-gurjunene, *trans*-calamenene, δ-cadinene, γ-calacorene, epi-α-selinene, α-muurolene, γ-muurolene, cadalene and nootkatene were characterized in the oil. Further analysis using silica gel column chromatography followed by silver nitrate coated silica gel column chromatography resulted in the isolation by preparative GC of three new sesquiterpene hydrocarbons. These were isorotundene, cypera-2,4-diene and

Comparative percentage composition of the chemotypes of *Cyperus rotundus* **T-1**

Compound	H oil	O oil	M oil	K oil	OH oil
α-copaene	t	2.0	t	3.6	t
cyperene	-	30.8	7.2	28.7	20.7
β-elemene	0.8	5.2	2.5	6.5	2.6
β-caryophyllene	6.2	3.8	3.3	5.0	3.2
α-humulene	4.5	4.0	1.7	3.9	4.7
β-selinene	18.5	t	17.8	-	-
δ-cadinene	1.0	3.5	2.0	3.8	3.4
calamenene*	3.9	2.6	t	1.5	1.7
cyperotundone	-	13.1	19.4	8.8	25.0
α-cyperone	38.6	-	30.7	-	-
cyperol	7.4	-	5.6	-	-
patchouli acetate	-	t	t	8.0	t
sugeonol acetate	-	t	t	6.9	t

t = trace (<0.1%); *correct isomer not identified

Comparative percentage composition of an oil and extract of *Cyperus rotundus* **T-2**

Compound	Oil	Extract
pinocarveol*	0.2	t
myrtenol	0.1	t
verbenone	0.3	t
ylangadiene*	1.0	0.5
copadiene*	0.9	0.3
α-ylangene	0.2	3.9
α-copaene	0.1	0.4
cyperene	2.9	15.9
δ-cadinene	0.5	0.2
caryophyllene oxide	7.0	6.5
patchoulenone	4.5	2.7
viridiflorol	3.0	1.6
isomustakone	2.3	3.0
mustakone	5.1	5.9
cyperotundone	34.9	15.6
cyperenal	4.6	2.2
sugeonol	0.6	1.2
sugeonol acetate	8.7	12.9

t = trace (<0.1%); *correct isomer not identified

Comparative percentage composition of the oil and its headspace volatiles of *Cyperus rotundus*

T-3

Compound	Oil	Headspace volatiles
hexanal	t	0.1
(Z)-3-hexenol	0.1	0.3
(E)-2-hexenol	0.1	0.2
hexanol	0.7	2.1
α-pinene	0.5	0.8
β-pinene	0.4	1.3
1-octen-3-ol	0.1	0.9
(E)-3-hexenyl acetate	t	0.1
myrcene	t	0.3
(E)-2-hexenyl acetate	t	0.1
hexanoic acid	0.1	0.3
p-cymene	0.1	0.6
limonene	0.4	2.3
1,8-cineole	0.1	1.7
(E)-β-ocimene	t	0.1
octanol	t	0.6
linalool	-	0.3
α-campholenal	0.7	1.2
trans-pinocarveol	5.2	7.4
trans-verbenol	0.9	2.3
pinocamphone	0.2	1.4
pinocarvone	1.0	1.7
isopinocamphone	t	0.8
cis-verbenol	0.6	1.2
p-cymen-8-ol	0.3	0.7
myrtenal	1.6	1.9
α-terpineol	1.1	1.7
myrtenol	0.4	0.9
verbenone	1.2	1.6
trans-carveol	0.6	1.2
carvone	t	0.2
α-cubebene	0.4	0.4
α-ylangene	t	-
α-copaene	11.4	12.1
β-cubebene	0.3	-
cyperene	8.4	11.7
β-elemene	0.3	0.8
α-gurjunene	3.8	3.2
β-bourbonene**	t	-
α-himachalene**	0.4	-
β-caryophyllene	1.4	2.2
aromadendrene	1.2	0.5
γ-elemene	0.1	t
trans-β-bergamotene	t	-
α-bergamotene*	t	-
(E)-β-farnesene	t	-
α-amorphene**	1.5	0.4

Compound	Oil	Headspace volatiles
α-guaiene**	0.6	0.2
rotundene	0.9	0.3
α-humulene	0.4	t
germacrene D	0.2	0.3
allo-aromadendrene	0.2	-
(E,E)-α-farnesene	t	-
α-muurolene	1.3	-
valencene	3.6	5.1
(Z)-α-bisabolene	t	-
δ-cadinene	t	-
β-selinene	0.9	t
γ-cadinene	t	-
cis-calamenene	1.0	0.4
(Z)-nerolidol	0.2	0.1
elemol	0.1	-
α-calacorene	0.3	-
spathulenol	0.4	0.1
caryophyllene alcohol	2.1	1.4
ledol	0.9	0.1
longifolene**	0.1	-
caryophyllene oxide	9.7	7.8
α-cedrene epoxide	0.1	0.2
guaiol	1.2	0.4
globulol	0.9	0.2
humulene epoxide*	3.5	2.9
widdrol	0.7	t
cedrol	0.5	0.1
β-eudesmol	t	-
valerianol	0.6	0.2
valeranone	1.1	0.3
α-muurolol	0.1	-
T-cadinol	0.2	0.1
α-cadinol	0.1	t
mustakone	0.5	t
isomustakone	0.1	t
β-bisabolol	t	-
cyperenone***	0.9	0.2
valerenal	9.8	8.7
(Z)-valerenol	0.4	0.1
eudesm-7(11)-en-4-ol**	1.2	0.5
cyperenal	0.4	0.1
aristolone**	0.2	t
nootkatone	6.7	1.4
phytol	1.2	-

t = trace (<0.1%); *correct isomer not identified; **incorrect identification based on GC elution; ***also known as cyperotundone or isopatchoulenone

nor-rotundene. A new sesquiterpene ketone that was structurally elucidated to be cyperadione was also found in the oil.

Jirovetz et al. (2004) compared the composition and headspace volatiles of the oil of *C. rotundus* roots and tubers produced in the laboratory from plant material collected from Calicut (Kerala, India). Using a combination of GC-FID and GC/MS detailed analyses were performed as can be seen from the results presented in **T-3**. Trace amounts (<0.1%) of (E)-2-hexenal and α-thujene were also found either in the oil or the headspace volatiles.

Chowdhury et al. (2005) reported their results of an analysis of *C. rotundus* oil produced from tubers collected in Bangladesh. The analytical data will not be presented in this review because it contained innumerable errors.

An oil produced from the tubers of *C. rotundus* collected in the region of Monastir (Tunisia) was analyzed by Kilani et al. (2005). The components characterized in this oil were as follows:

α-pinene (1.4%)
β-pinene (3.9%)
borneol (0.3%)
cyprotene (0.2%)
cypera-2,4-diene (2.4%)
α-cubebene (3.4%)
α-copaene (0.9%)
cyperene (30.9%)
β-caryophyllene (0.3%)
α-humulene (0.5%)
rotundene (7.6%)
β-selinene (1.5%)
α-selinene (0.6%)
α-muurolene (1.1%)
trans-calamenene (2.0%)
δ-cadinene (0.7%)
cadina-1,4-diene (0.6%)
elemol (0.2%)
α-calacorene (1.4%)
isorotundene (3.6%)
caryophyllene oxide (0.7%)
humulene epoxide* (0.9%)
isocyperol (2.7%)
cyperol (4.0%)
T-cadinol (1.4%)
T-muurolol (2.2%)
α-muurolol (1.9%)
α-cadinol (2.5%)
mustakone (3.8%)
cyperotundene (8.8%)
(E,E)-farnesol (1.0%)
α-cyperone (4.5%)

*correct isomer not identified

An oil of *C. rotundus* produced from tubers collected in the state of Pará (Brazil) was analyzed by Zoghbi et al. (2008). It was determined to have the following composition.

α-pinene (0.4%)
sabinene (0.1%)
β-pinene (1.5%)
p-menth-3-ene (0.1%)
limonene (0.2%)
1,8-cineole (0.2%)
α-campholenal (0.1%)
trans-pinocarveol (1.1%)
trans-verbenol (0.3%)
pinocarvone (0.7%)
p-mentha-1,5-dien-8-ol (0.1%)
myrtenal + myrtenol (1.7%)
verbenone (0.2%)
trans-carveol (0.1%)
cyprotene (0.2%)
cypera-2,4-diene (0.4%)
cyperene (3.9%)
rotundene (0.7%)
eudesma-1,4,11-triene (1.8%)
β-selinene (5.0%)
α-selinene (2.0%)
epi-cubenol (0.9%)
δ-cadinene (0.6%)
trans-calamenene (0.2%)
cyperene epoxide (1.3%)
caryophyllene oxide (6.9%)
humulene epoxide II (2.4%)
patchoulenone (4.3%)
eudesma-3,11-dien-5-ol (6.2%)
isocyperol (6.7%)
cyperol (5.6%)
cyperotundone (12.1%)
rotundone (4.8%)
eudesm-11-ene-4α,6α-diol (1.3%)
α-cyperone (22.8%)
eudesma-3,11-dien-2-one (1.1%)

Ranade (2009) reported that the major constituents of *C. rotundus* oil were:

cyperene (5–30%)
cyperol (40–45%)
α-cyperone (33–54%)

He also noted that trace amounts of α-pinene, 1,8-cineole and phenol were also components of this oil.

Finally, it is interesting to note that rotundone (920 µg/kg) was found in the leaves of *C. rotundus* by Wood et al. (2008).

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