



Progress in Essential Oils

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Rangpur Lime Oil

Rangpur lime, which is known as 'hime' in Japan, is the name given to the fruit of *Citrus limonia* Osbeck. It is a mandarinlike fruit that is grown in India. Occasionally, an oil of Rangpur lime can be found in commerce. Bricout and Paupardin (1974) determined from an in vitro pericarp that the peel oil volatiles of *C. limonia* were α -pinene, β -pinene, myrcene, limonene, γ -terpinene, p-cymene, terpinolene, linalool, *trans*- α -bergamotene, terpinen-4-ol, α -terpineol, neral, geranial, neryl acetate, geranyl acetate and β -bisabolene. Unfortunately, the authors did not present any quantitative data.

A peel oil of *C. limonia* of Chinese origin was reported by Zhu et al. (1995) to contain the following constituents:

- α -thujene (0.2%)
- α -pinene (0.8%)
- sabinene (0.8%)
- β -pinene (4.8%)
- myrcene (1.4%)
- δ -3-carene (2.0%)
- p-cymene (1.6%)
- limonene (42.5%)
- β -ocimene (0.3%)
- δ -terpinene (1.4%)
- cis*-linalool oxide (0.1%)
- trans*-sabinene hydrate (0.1%)
- terpinolene (0.1%)
- linalool (17.9%)
- dihydrolinalool[†] (0.1%)
- terpinen-4-ol (0.2%)
- α -terpineol (2.5%)
- decanal (0.1%)
- octyl acetate (0.1%)
- nerol (0.5%)
- neral (0.4%)
- geraniol (5.7%)
- linalyl acetate (11.4%)
- geranial (0.5%)
- methyl geranate (0.1%)
- citronellyl acetate (0.2%)
- neryl acetate (0.9%)
- geranyl acetate (1.3%)
- β -caryophyllene (0.1%)
- (E)- β -farnesene (0.2%)
- pentadecane (0.2%)

[†]does not occur naturally

In addition, trace amounts of (Z)-3-hexenol, hexanol, camphene, 6-methyl-5-hepten-1-one, α -fenchyl alcohol, γ -heptalactone, citronellal, isopulegol, lavandulol, nonanol, dodecane, citronellyl formate, geranyl formate, nonyl acetate, α -terpinyl acetate, decyl acetate, an isomer of α -bergamotene and β -bisabolene were found, although their identities were not confirmed.

The cold-pressed peel oil of *C. limonia* fruit that were collected from the Agricultural Crops Research Institute (Giza, Egypt) was analyzed by Haggag et al. (1998) by GC retention times only. The constituents that the oil was reported to contain were as follows:

- α -pinene (26.9%)
- camphene (0.9%)
- β -pinene (18.3%)
- limonene (9.6%)
- p-cymene (0.6%)
- citronellal (0.6%)
- linalool (1.2%)
- nerol (2.7%)
- α -terpineol (1.3%)
- citronellol (7.0%)
- geraniol (3.6%)
- thymol (2.7%)
- carvacrol (1.3%)
- eugenol (8.6%)

A hand-pressed oil of *C. limonia* fruit that were grown at the Hanoi Research Institute of Fruit and Vegetable (Vietnam) was analyzed using GC-FID and GC/MS by Minh Tu et al. (2002). The oil, which was obtained in 0.02% yield, was determined to contain the following constituents:

- α -pinene (1.3%)
- β -pinene (3.7%)
- sabinene (0.9%)
- myrcene (2.0%)
- α -phellandrene (0.1%)
- α -terpinene (0.2%)
- limonene (71.0%)
- (Z)- β -ocimene (0.3%)
- 1,8-cineole (0.3%)

- (E)- β -ocimene (0.1%)
- γ -terpinene (11.9%)
- p-cymene (0.1%)
- terpinolene (0.6%)
- octanal (0.1%)
- menthone (0.1%)
- citronellal (0.6%)
- decanal (0.1%)
- β -cubebene (0.1%)
- linalool (0.1%)
- p-menth-1-en-9-ol (0.1%)
- bergamotene* (0.5%)
- β -caryophyllene (0.5%)
- β -farnesene* (0.1%)
- α -humulene (0.1%)
- neral (0.1%)
- α -terpineol (0.4%)
- germacrene D (0.5%)
- α -farnesene* (0.9%)
- geranial (0.2%)
- bicyclogermacrene (0.1%)
- α -farnesene* (0.7%)
- cadinene* (0.1%)
- citronellol[†] (0.1%)

*correct isomer not identified

[†]incorrect identification based on GC elution order

Three cultivars of *C. limonia*, 'Rangpur Yellow,' 'Rangpur Intermediate' and 'Rangpur Red,' were cold-pressed by Lotta et al. (2002) to yield oils that were analyzed by a combination of GC-FID, GC/MS and ¹³C-NMR. The compounds characterized in these oils were as follows:

- α -thujene (0.3–0.4%)
- α -pinene (1.7–2.4%)
- camphene (0.1%)
- sabinene (1.8–2.1%)
- β -pinene (12.2–14.6%)
- myrcene (1.3–1.4%)
- octanal (0–0.1%)
- α -phellandrene (t–0.1%)
- δ -3-carene (0–t)
- α -terpinene (t–0.2%)
- p-cymene (1.2–5.6%)
- limonene (61.7–62.5%)
- β -phellandrene + 1,8-cineole (0.5–0.8%)
- (Z)- β -ocimene (0–0.1%)
- (E)- β -ocimene (0.2%)
- cis*-sabinene hydrate (0–0.1%)
- trans*-limonene oxide (0–0.2%)

terpinolene (0.2–0.5%)
 nonanal (0–t)
 linalool (0.1–0.2%)
trans-p-menth-2-en-1-ol (0–t)
 terpinen-4-ol (t)
 α -terpineol (0.1–0.4%)
 decanal (0–t)
 octyl acetate (0–t)
 citronellol (t–0.2%)
 neral (0–t)
 linalyl acetate (0–t)
 citronellyl acetate (0–0.2%)
 β -caryophyllene (0.2–0.5%)
trans- α -bergamotene (0.7–0.8%)
 α -humulene (0–t)
 germacrene D (t–1.0%)
 bicyclogermacrene (0–0.1%)
 (E,E)- α -farnesene (0–0.6%)
 β -bisabolene (1.2–1.4%)
 t = (<0.05%)

Rangpur limes were collected from the Passo Patria, Santa Lucia and Capital production regions of the province of Corrientes (Argentina). Oils produced by hand-pressing the peels were analyzed by Acevedo et al. (2012) using GC-FID and GC/MS. The comparative analysis of the oils produced from these different regions can be seen in **T-1**.

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Kewda, or Keora, Oil

Kewda oil is obtained by hydrodistillation of the flowers of *Pandanus fasciculatus* Lam., a member of the Pandanaceae family. Kewda shrubs are found along the coast of Orissa and, to a lesser extent,

Andhra Pradesh (India). It is estimated that the shrub grows wild over an area of ca. 5,000 hectares, from which 3.5 metric tons (ca. 35 million flowers; worth ca \$10 million) are harvested annually from the Ganjam district of Orissa (Rout et al. 2010). About 70–80% of the annual flower harvest takes place during the rainy season. Typically, flowers are 30–35 cm long x 8–10 cm in diameter, each weighing approximately 100 g. Kewda is dioecious, which means that both male and female shrubs exist. It is interesting to note that the female flower is odorless, while the male flower possesses a penetrating characteristic odor. As a result, an attar, a hydrodistilled oil and kewda water have been items of commerce for many years.

Flowers and samples of *P. fascicularis* were collected from the village of Tolu, Khajuribali and Keluapalli (Ganjam district) and separately distilled for 4 hr. The oils produced from each of the village areas were analyzed using GC-FID and

retention indices only by Mishra et al. (2008). The authors found that the oils possessed similar compositions within the following range of constituents:

ethanol (t–0.3%)
 acetic acid (t–0.1%)
 isoamyl alcohol (t–0.1%)
 toluene (t–0.1%)
 hexanol (0.1%)
 α -thujene (0.1–0.5%)
 α -pinene (0.2–0.5%)
 camphene (t–0.1%)
 benzaldehyde (t–0.1%)
 β -pinene (0.1–0.9%)
 myrcene (0.1%)
 α -phellandrene (t–0.1%)
 p-cymene (2.1–2.8%)
 limonene (0.2–0.4%)
 1,8-cineole (t–0.1%)
 α -terpinene^a (0.1–2.1%)
 2-phenethyl methyl ether (67.6–71.6%)
cis-linalool oxide^f (t–0.1%)
 linalool (t–0.1%)
 α -thujone (t–0.1%)
 2-phenethyl alcohol (0.1–0.4%)
cis-p-menth-2-en-1-ol (t–0.1%)

T-1. Comparative percentage composition of Rangpur lime oils produced from fruit obtained from different regions of Argentina

| Compound | Passo Patria oils | Santa Lucia oils | Capital oils |
|--------------------------------|-------------------|------------------|--------------|
| α -thujene | 0.1–0.3 | 0.1–0.3 | t–0.2 |
| α -pinene | 0.5–0.7 | 0.3–0.7 | 0.4–0.6 |
| sabinene | 0.5–0.7 | 0.2–0.8 | - |
| β -pinene | 3.3–3.7 | 2.5–3.3 | 4.4–4.8 |
| myrcene | 1.1–1.3 | 0.6–1.4 | 1.0–1.4 |
| octanal | 0.1–0.2 | t–0.3 | t–0.2 |
| α -terpinene | 0.1–0.3 | 0.1–0.2 | t–0.2 |
| limonene | 62.5–65.3 | 65.0–66.2 | 63.0–66.2 |
| 1,8-cineole | 0.1–0.2 | 0.1–0.2 | t–0.2 |
| (E)- β -ocimene | 0.1–0.3 | 0.1–0.2 | 0.1–0.3 |
| γ -terpinene | 9.8–11.6 | 6.5–9.9 | 11.0–12.0 |
| <i>trans</i> -sabinene hydrate | 0.1–0.2 | 0.1–0.2 | t–0.2 |
| terpinolene | 0.5–0.7 | 0.3–0.5 | 0.4–0.6 |
| linalool | 0.2–0.5 | 0.1–0.3 | 0.2–0.4 |
| nonanal | 0.1–0.2 | t–0.1 | t–0.2 |
| citronellal | 1.0–1.4 | 0.6–1.0 | 0.8–1.4 |
| terpinen-4-ol | 0.1–0.2 | t–0.1 | t–0.2 |
| α -terpineol | 0.5–0.7 | 0.3–0.5 | 0.4–0.8 |
| decanal | 0.1–0.3 | t–0.2 | 0.1–0.3 |
| citronellol | 0.1–0.2 | t–0.2 | t–0.2 |
| geranial | t–0.1 | t–0.1 | t–0.2 |
| perillaldehyde | t–0.3 | t–0.2 | t–0.2 |
| citronellyl acetate | 0.1–0.3 | t–0.2 | t–0.2 |
| β -caryophyllene | 0.8–1.4 | 0.4–0.8 | 0.4–0.8 |
| β -farnesene* | 0.1–0.5 | 0.1–0.3 | 0.1–0.3 |
| germacrene D | 1.0–1.5 | 0.5–0.9 | 0.4–1.2 |
| α -farnesene* | t–0.4 | 0.6–0.8 | 0.6–1.0 |
| β -bisabolene | 4.0–5.4 | 2.2–2.6 | 2.0–3.2 |

* correct isomer not identified; t = trace (<0.5%)

camphor (t-0.1%)
 terpinen-4-ol (10.9–15.7%)
 α -terpineol (1.4–2.1%)
 dihydrocarveol (t-0.1%)
 verbenone (t-0.1%)
 citronellol (t-0.1%)
 piperitone (t-0.1%)
 carvenone (t-0.1%)
 benzyl methyl ketone[†] (0.1%)
 α -amorphene (t-0.1%)
 nerolidol^{*} (t-0.1%)
 α -muurolol (t-0.1%)
 1-heptadecene (t-0.1%)
 benzyl benzoate (0.1%)
 ethyl myristate (t-0.1%)
 2-phenethyl benzoate (0.1–0.5%)
 methyl palmitate (0.1%)
 2-phenethyl phenylacetate (0.1%)
 palmitic acid (t-0.1%)
 ethyl palmitate (t-0.1%)
 phyllocladene (t-0.1%)
 eicosane (t-0.1%)
 methyl linoleate (t-0.1%)
 ethyl oleate (0.1%)
 docosane (t-0.1%)

^{*} correct isomer not identified; ^f furanoid form;

t = trace (<0.1%); ^a should be γ -terpinene;

[†] doubtful identification

Purchit et al (2008a) collected flower samples of selected kedra plants from

the Tulu area of Ganjam district. The spathe and anthers of the flowers were separated and subjected to separate hydrodistillation. The oil yields of the spathe and anthers were 0.026% and 0.018%, respectively. GC-FID analyses revealed that the 2-phenethyl methyl ether and terpinen-4-ol contents of the spathe and anther oil were 79.0% and 8.1%, and 66.6% and 8.7%, respectively.

Purohit et al. (2008b) collected flowers of the screw pine from Tule, Kajurbali and Keluapalli villages, from which they produced oils in 0.015%, 0.016% and 0.014% yields, respectively. Using GC-FID and GC/MS as their method of analysis, each oil was analyzed separately, the results of which were almost identical to those published earlier (Purohit et al. 2008a). The minor differences were that benzyl methyl ketone was not identified in any oil, and (E)-nerolidol and γ -terpinene were the correct isomers. The main difference between the three oils was that the Keluapalli flower oil contained 2-phenethyl methyl ether (71.6%) and terpinen-4-ol (10.9%) as major constituents.

Mohapatra et al. (2009) studied the effect of harvest season, different village flower origins and the physical state of kewda flowers on oil yield and content of the two major constituents. They found that the oil yield of fresh and decayed flowers from the three village origins harvested in the winter season or the rainy season ranged from 0.024–0.095% (this 0.095% yield may be a typographical error) and 0.011–0.037%, respectively. Nevertheless, fresh flowers yield more oil than decayed flowers, while flower buds appear to be devoid of oil. The 2-phenethyl methyl ether and terpinen-4-ol content of oils from fresh flowers harvested during the rainy and winter seasons were 74.9–82.5% and 4.6–8.2%, and 73.4–77.2% and 5.6–7.2%, respectively. In contrast, the 2-phenethyl methyl ether and terpinen-4-ol contents of decayed flowers harvested during the rainy and winter seasons were 52.4–60.1% and 15.3–15.7%, and 46.8–52.6% and 15.5–20.9%, respectively.

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Labdanum and Cistus Oil and Extracts

A commercial sample of labdanum oil was analyzed by Moyler and Clery (1997) using retention indices on a non-polar GC column and GC/MS. The compounds identified during the analysis were:

- furfural (0.5%)
- tricyclene + α -thujene (1.2%)
- α -pinene (40.8%)
- camphene (6.0%)
- benzaldehyde (0.9%)
- sabinene (0.5%)
- myrcene (0.5%)
- α -phellandrene + δ -3-carene (0.3%)
- α -terpinene (0.4%)
- p-cymene (1.9%)
- limonene + β -phellandrene (1.9%)
- 1,8-cineole (2.9%)
- (Z)- β -ocimene (0.2%)
- (E)- β -ocimene (1.1%)
- γ -terpinene (1.0%)
- terpinolene (1.8%)
- linalool (0.5%)
- α -copaene (0.2%)
- β -caryophyllene (0.2%)
- ledene (1.6%)
- δ -cadinene (0.7%)
- ledol (2.6%)
- viridiflorol (1.1%)
- ambrox[†] (0.16%)

[†]three isomers

The authors further noted that this oil contained an additional 220 constituents (31.05% of the oil), but they failed to identify them.

Ranade (2003) reported that an oil of labdanum normally contained the following constituents:

- acetophenone (0.3%)
- benzaldehyde (0.8%)
- borneol (2.5%)
- bornyl acetate (2.5%)
- camphene (0.7%)

- carvomenthone (2.0%)
- 1,8-cineole (12.4%)
- dihydrocarvone* (0.9%)
- fenchone (2.3%)
- furfural (0.7%)
- geraniol (0.8%)
- guaiaacol (3.0%)
- limonene (0.4%)
- linalool (1.9%)
- linalyl acetate (2.0%)
- neral (0.5%)
- 2-nonanone (0.5%)
- α -pinene + β -pinene (44.0%)
- α -phellandrene (0.1%)
- p-cymene (6.3%)
- thujone* (0.1%)

*correct isomer not identified

A comparative study of the oils produced from fresh and dried *C. ladanifer* plants harvested from two locations in Portugal was performed by Gomes et al. (2005). One of the sites where the plants were harvested was a cultivated area, whereas the other site was from wild plants. The comparative compositions of the oils produced from these two sites can be seen in **T-2**. In addition, Gomes et al. reported that the range

in composition of three commercial samples of cistus oil of Spanish origin was as follows:

- α -pinene (12.8–50.4%)
- camphene (1.9–5.0%)
- β -pinene (0.6–4.4%)
- limonene (0.8–1.4%)
- p-cymene (1.9–3.6%)
- 2,2,6-trimethylcyclohexanone (1.5–2.4%)
- p-cymene (0.9–1.6%)
- pinocarvone (0–0.8%)
- bornyl acetate (1.5–3.5%)
- terpinen-4-ol (0.9–2.5%)
- allo-aromadendrene (0.5–1.2%)
- trans-pinocarveol (1.7–4.9%)
- (Z)-tagetone (0–0.3%)
- viridiflorene (1.4–8.0%)
- borneol (0.7–1.6%)
- δ -cadinene (0.3–0.9%)
- myrtenol (0.4–1.2%)
- caryophyllene oxide (0–0.1%)
- globulol (0.4–1.3%)
- viridiflorol (1.1–3.4%)
- verbenone[†] (0.2–0.7%)
- spathulenol (0.1–0.3%)
- ambrox (0.1–0.3%)
- sclareol oxide (0–0.2%)
- 15-nordabdan-8-ol (0.1–0.3%)

[†]incorrect identification based on GC elution order

T-2. Comparative percentage composition of oils produced from fresh and dried cultivated and wild *Cistus ladanifer* plants

| Compound | Cultivated plants | | Wild plants | |
|------------------------------|-------------------|----------------|----------------|----------------|
| | Fresh leaf oil | Dried leaf oil | Fresh leaf oil | Dried leaf oil |
| α -pinene | 2.1 | 4.5 | 0.1 | 0.2 |
| camphene | 0.3 | 0.1 | - | 0.1 |
| p-cymene | 0.2 | 0.3 | 0.1 | - |
| 2,2,6-trimethylcyclohexanone | 2.8 | 2.0 | - | 0.3 |
| pinocarvone | 1.1 | 1.7 | - | 0.7 |
| bornyl acetate | 1.6 | 1.5 | 0.7 | 1.4 |
| terpinen-4-ol | 1.0 | 1.5 | 0.3 | 0.8 |
| allo-aromadendrene | 0.8 | 0.7 | 0.5 | 0.7 |
| trans-pinocarveol | 2.1 | 3.8 | - | - |
| (Z)-tagetone | 0.6 | 0.2 | 4.0 | 2.2 |
| viridiflorene | 1.3 | 1.0 | - | 1.0 |
| borneol | 0.7 | 0.5 | 0.1 | 0.5 |
| (E)-tagetone | - | - | 3.6 | 1.9 |
| δ -cadinene | 1.0 | 1.6 | 0.4 | 1.1 |
| myrtenol | 0.7 | 1.1 | - | 0.7 |
| caryophyllene oxide | 1.8 | 3.6 | 1.7 | 2.5 |
| globulol | 5.0 | 4.3 | 3.1 | 4.0 |
| viridiflorol | 17.4 | 15.1 | 13.6 | 14.9 |
| verbenone [†] | 0.5 | 0.9 | - | 0.1 |
| spathulenol | 0.8 | 0.8 | 0.5 | 0.6 |
| ambrox | 0.8 | 0.7 | 0.8 | 0.8 |
| sclareol oxide | 0.3 | 0.3 | 1.1 | 1.3 |
| 15-nor-labdan-8-ol | 1.7 | 1.8 | 5.2 | 3.5 |

[†]incorrect identification based on GC elution order

A sample of cistus oil produced from plants collected in Corsica was analyzed by Rossi et al. (2007). The main constituents of the oil were found to be as follows:

α -thujene (47.1%)
 camphene (5.0%)
 p-cymene (3.8%)
 limonene (1.6%)
trans-pinocarveol (3.1%)
 borneol (1.0%)
 terpinen-4-ol (1.1%)
 bornyl acetate (2.4%)
 allo-aromadendrene (1.5%)
 globulol (6.2%)

This oil is very unusual and perhaps is not produced from *C. ladaniferus* but from another *Cistus* species found in Corsica.

Cistus ladanifer var. *albiflorus* (the white-petaled cistus) was collected from the Tangier region of northern Morocco by Greche et al. (2009). An oil was produced from the leaves and small branches by hydrodistillation for 4 hr. in 0.3–0.4% yield. Using GC-FID, GC/MS and retention indices the constituents characterized in the oil were as follows:

tricyclene (2.2%)
 α -pinene (5.0%)
 camphene (12.1%)
 β -pinene (0.3%)
 α -terpinene (0.4%)
 p-cymene (1.6%)
 β -phellandrene (0.1%)
 2,2,6-trimethylcyclohexanone (3.6%)
 2,6,6-trimethylcyclohex-1-enone (1.5%)
 p-cymenene (0.5%)
 α -pinene oxide (0.1%)
 p-mentha-1,3,8-triene (0.1%)
 α -campholenal (0.6%)
 camphor (1.7%)
 camphene hydrate (0.7%)
 pinocarvone (0.3%)
 borneol (3.5%)
 terpinen-4-ol (4.1%)
 α -terpineol (0.6%)
 verbenone (0.2%)
trans-carveol (0.1%)
 bornyl acetate (16.5%)
 carvacrol (0.1%)
 cumyl alcohol (0.1%)
 phenylpropionic acid (0.9%)
 α -ylangene (0.4%)
 α -copaene (0.2%)
 β -cubebene (0.1%)
 isoitalicene (0.4%)
 γ -muurolene (0.2%)
 ledene (0.1%)
 δ -cadinene (2.3%)
 ledol (8.0%)
 spathulenol (1.6%)
 caryophyllene oxide (0.1%)
 viridiflorol (19.4%)

guaial (0.5%)
 1-epi-cubenol (1.3%)
 β -eudesmol (1.6%)
 cadalene (2.1%)
 eicosanol (0.6%)
 pentacosane (0.2%)

In addition, trace amounts (<0.1%) of α -thujene, sabinene, α -phellandrene, *cis*-rose oxide, β -thujone, β -fenchyl alcohol, *trans*-rose oxide, isoborneol, myrtenol, cuminaldehyde, carvone, geraniol, thymol, α -cubebene, eugenol, α -gurjunene, β -caryophyllene, β -gurjunene, α -patchoulene, allo-aromadendrene, valencene, α -muurolene, α -calacorene, octadecanol, octadecanal and β -bisabolol were reported as being characterized in this oil.

Greche et al. also produced a resinoid of labdanum gum with ethanol (2.9% yield), a concrete by direct hexane extraction of the whole dried plant (5.0%) and an absolute from the concrete (4.5% yield calculated from the dried plant). In order to facilitate analysis of these three extracts, they were subdivided into free acids by treatment with 0.1 M NaHCO₃ solution and subsequent extraction with diethyl ether to isolate the free acids fraction, with further treatment with 1.0 M NaOH with diethyl ether extraction to isolate the phenols fraction with the residues being the neutral fractions. The resinoid was found to contain free acids (27.8%), phenols (30.7%) and neutral compounds (39.4%) with 3.1% volatiles. The concrete was found to contain free acids (26.3%), phenols (23.8%) and neutral compounds (48.9%) with 4.2% volatiles, while the absolute was found to contain free acids (22.3%), phenols (37.5%) and neutral compounds (44.2%) with 4.2% volatiles. Analyses of the neutral and free acid fractions only of each extract are presented in **T-3** and **T-4**. Labdanoic acid is a significant compound to the fragrance industry because it can be transformed into 12-nor-ambreinolide, which is a precursor of nor-ambreinolide ether (also known as ambrox or ambroxan).

A commercial sample of *C. ladanifer* oil of Spanish origin that was purchased in the United States was analyzed by Costa et al. (2009) using GC-FID and GC/MS. The results of this analysis can be seen as follows:

santene (0.4%)
 tricyclene (0.6%)
 α -thujene (0.1%)
 α -pinene (24.5%)

α -fenchene (2.3%)
 camphene (13.3%)
 benzaldehyde (0.2%)
 sabinene (0.3%)
 β -pinene (2.4%)
 myrcene (0.4%)
 δ -3-carene (1.6%)
 α -terpinene (<0.1%)
 p-cymene (4.2%)
 limonene (4.4%)
 β -phellandrene (0.5%)
 1,8-cineole (0.8%)
 (E)- β -ocimene (0.1%)
 γ -terpinene (0.1%)
cis-sabinene hydrate (0.1%)
 terpinolene (0.1%)
 p-cymenene (<0.1%)
 linalool (0.6%)
 α -thujone (0.1%)
 3-methyl-3-buten-2-one (0.1%)
 β -thujone (<0.1%)
 camphor (1.0%)
trans-pinocarveol (0.2%)
 myrtenyl methyl ether (0.3%)
 pinocamphone (2.7%)
 pinocarvone (0.1%)
 borneol (0.5%)
 isocamphene (6.2%)
 terpinen-4-ol (2.5%)
 p-cymen-8-ol (0.1%)
 α -terpineol (3.9%)
 myrtenol (<0.1%)
 γ -terpineol[†] (0.1%)
 verbenone (<0.1%)
 carvone (0.3%)
 bornyl acetate (6.5%)
 ethyl hydrocinnamate (0.5%)
 α -cubebene (0.3%)
 cyclosativene (0.1%)
 α -copaene (7.6%)
 β -bourbonene (0.2%)
 β -cubebene (0.3%)
 β -elemene (<0.1%)
 cyperene (0.1%)
 dodecanal (0.8%)
 β -caryophyllene (3.2%)
 α -humulene (0.6%)
 9-epi- β -caryophyllene (0.3%)
 cadina-1(6),4-diene (0.1%)
 γ -muurolene (0.2%)
 germacrene D (0.3%)
 β -selinene (<0.1%)
 valencene (0.1%)
 bicyclogermacrene (0.3%)
 α -muurolene (0.1%)
 α -bulnesene (0.1%)
 γ -cadinene (<0.1%)
 δ -cadinene (1.4%)
trans-calamenene (0.1%)
 zonarene (0.1%)
 α -calacorene (<0.1%)
 elemol (0.1%)
 spathulenol (0.2%)
 caryophyllene oxide (0.4%)
 viridiflorol (0.2%)
 humulene epoxide II (0.1%)
 epi-cubenol (0.1%)

T-3. Percentage composition of the main components of the neutral compounds of the resinoid, concrete and absolute of labdanum

| Compound | Resinoid | Concrete | Absolute |
|------------------------------|----------|----------|----------|
| ledene | - | - | 0.4 |
| ledol | 0.9 | - | 1.2 |
| 16-kaarene | 3.7 | 4.6 | 2.3 |
| labd-14-ene-16,18-diol | 7.7 | 6.7 | 6.0 |
| labda-8(20),13(16),14-triene | 2.7 | 4.6 | 1.4 |
| labd-14-ene-8,13-diol | 5.2 | 4.3 | 6.6 |
| labd-14-ene-2,4,13-triol | 1.6 | 1.0 | 1.2 |
| labd-12-en-15,16-diol | 3.8 | 3.2 | 3.2 |
| eicosanol | 1.5 | - | 1.1 |
| octadecane | 2.4 | 4.5 | 2.3 |
| thio-octadecanol | 1.2 | 1.8 | 1.3 |
| pentyltricontane | 1.2 | 18.2 | 1.1 |
| octadecanol | 0.5 | 1.1 | 0.4 |

T-4. Percentage composition of the main components of the free acids of the resinoid, concrete and absolute of labdanum

| Compound | Resinoid | Concrete | Absolute |
|-------------------------|----------|----------|----------|
| phenylpropionic acid | 1.1 | 0.7 | 0.9 |
| cinnamic acid | 1.2 | 2.3 | 0.7 |
| pentadecanoic acid | 1.9 | 2.1 | 2.2 |
| hexadecanoic acid | 1.8 | 0.1 | 0.6 |
| 5-octadecenoic acid* | 2.9 | 2.5 | 0.4 |
| caticic acid | 2.0 | 0.9 | 3.2 |
| labdanolic acid | 1.5 | 0.8 | 0.8 |
| labda-8,14-dienoic acid | 2.4 | 9.8 | 1.1 |
| labda-7,8-dienoic acid | 3.3 | 6.9 | 0.7 |
| labda-8,20-dienoic acid | 1.2 | 4.7 | 0.1 |
| 6-oxo-caticic acid | 2.4 | 2.9 | 3.9 |
| eicosanoic acid | 2.9 | 1.4 | 3.3 |
| labdenoic acid | 2.3 | 1.7 | 12.7 |
| labdanoic acid | 0.5 | 8.9 | 9.0 |
| copaiferic acid | 2.8 | 3.9 | 1.9 |
| octadecanoic acid | 0.9 | 0.8 | 2.9 |
| linoleic acid | 0.8 | 0.5 | 2.5 |

*correct isomer not identified

T-muurolool (0.1%)
 α -muurolool (<0.1%)

¹does not occur naturally

An oil produced by hydrodistillation from the leaves of *C. ladanifer* in 1.4% yield, which were collected from the Chefchaouen region (northwestern Morocco), was analyzed by GC-FID and GC/MS by Viuda-Martos et al. (2011). The results revealed that the oil contained the following constituents:

tricyclene (0.6%)
 α -thujene (1.1%)
 α -pinene (0.8%)
 camphene (2.7%)
 verbenene (0.3%)
 sabinene (1.0%)
 6-methyl-5-hepten-2-one (0.7%)

myrcene (0.7%)
 α -phellandrene (0.4%)
 α -terpinene (2.8%)
 p-cymenene (3.6%)
 1,8-cineole (19.3%)
 (E)- β -ocimene (0.5%)
 γ -terpinene (6.1%)
 cis-sabinene hydrate (1.1%)
 cis-linalool oxide^f (0.5%)
 terpinolene (0.7%)
 p-cymene (1.6%)
 nonanal (0.8%)
 p-mentha-1,3,8-triene (0.6%)
 α -pinene oxide (1.3%)
 trans-pinocarveol (0.9%)
 camphor (0.9%)
 pinocarvone (0.7%)
 borneol (3.9%)
 2-decenal* (0.6%)
 terpinen-4-ol (4.2%)
 p-cymen-8-ol (0.7%)

α -terpineol (0.8%)
 myrtenol (0.5%)
 verbenone (0.4%)
 trans-carveol (0.1%)
 trans-3-carene-2-ol (0.1%)
 cuminaldehyde (0.4%)
 neral (0.2%)
 linalyl acetate (1.3%)
 geraniol (0.1%)
 vitispirane (0.2%)
 bornyl acetate (0.9%)
 carvacrol (0.7%)
 myrtenyl acetate (0.4%)
 isocyclocitral (0.2%)
 α -copaene (0.2%)
 α -ionone* (0.1%)
 aromadendrene (0.2%)
 γ -muurolole (0.1%)
 β -ionone* (0.1%)
 allo-aromadendrene (0.1%)
 α -farnesene* (0.6%)
 α -muurolole (0.1%)
 tridecanal (0.1%)
 δ -cadinene (0.6%)
 α -calacorene (0.2%)
 caryophyllene oxide (1.3%)
 viridiflorol (16.4%)
 humulene epoxide II (3.6%)
 epi-cubenol (3.2%)
 α -muurolool (0.3%)

*correct isomer not identified; ^ffuranoid form

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