

The Essential Oil of Spike Lavender *Lavandula latifolia* Vill. (*L. spica* D.C.)

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The oil isolated from *Lavandula latifolia* Vill. (fam. *Labiatae*) is one of the most important essential oils of Spain.

To obtain a general and proper impression about this oil one should consult the existing literature.¹⁻¹⁰ In addition to the publications cited we will discuss more recent publications, our own investigations and ideas about the subject. The history, botany, isolation methods, physico-chemical properties, chemical composition, olfactive properties and application and production and trade of the essential oil of spike lavender will be treated. Because during the last ten years up to 350 constituents have been detected in the oil, the chemical composition will be discussed in more detail.

History

Some species of the genus *Lavandula* have been known since ancient times. It is probable that *Lavandula* plants were brought over from Greece to the islands of Hyeres, off the French coast near Toulon and Marseille as early as 600 years before Christ. The plants could have spread out from these islands to France, Spain and Italy. The name *lavandula* stems from the Latin verb "lavere" which means (to) wash or (to) purify because some types of these plants are used to per-

fume water for bathing. The eldest known species are *L. stoechas* and *L. spica*. According to Dioscoridis,²⁶ the word "stoechas" stems from the "stoichades", the Greek name for the islands of Hyeres. According to Font Quer⁴⁷ "stoichades" also means, to be or to stand in a line, which could stand for the islands, lying in one line, as well as for the flowers of the *Lavandula* species. The word "spica" originates from Latin, and simply means spike or top of a plant in spike form, that is, the manner in which the flowers of most of the *Lavandula* species are settled on the plants at the end of a stem.

It is possible that in the beginning a type of spike oil was prepared from the flowers of *L. stoechas*. Spike oil has been known throughout history.¹¹⁻²⁰ It is probable that spike or stoechas oil originally was a more fatty oil, whereas so-called "narden" oil was a real essential oil.^{26,27,33} Oil of spike (*Oleum spicae*) had already been mentioned in medical books in the thirteenth century.³² In the early middle ages, spikes (or narden) were used in several medical preparations;²⁹ moreover, the flowers found application to fill sachets for healthy or sick people.³¹

The distillation of spike oil was described in the middle ages by Ryff.¹⁸

Table I. Scheme of Some Important *Lavandula* Species and Subspecies

Main Species	Synonyms	Subspecies & Varieties
<i>L. latifolia</i> Vill.	<i>L. spica</i> D.C. <i>L. spica</i> L. <i>L. vulgaris</i> Lam.	<i>L. tomentosa</i> <i>L. latifolia</i> <i>erigens</i> Jordan & Fourr. <i>L. latifolia</i> <i>inclinans</i> <i>L. latifolia</i> <i>interrupta</i>
<i>L. officinalis</i> Chaix	<i>L. vera</i> D.C. <i>L. spica</i> L. <i>L. angustifolia</i> Mnch. or Mill.	var. <i>fragens</i> Jord. var. <i>delphinensis</i> Jord. var. <i>angustifolia</i> <i>L. intermedia</i> Loisel (hybrid) <i>L. fragens</i> Salisb.
<i>L. hybrida</i> Rev.	<i>L. fragens latifolia</i> Chatenier	hybrid or: <i>L. latifolia</i> Vill & <i>L. officinalis</i> Chaix
<i>L. stoechas</i> L.		<i>L. pedunculata</i> Cav. is subsp. <i>pedunculata</i> (Mill.) Samp.
<i>L. dentata</i> L.		
<i>L. multifida</i> L.		
<i>L. pyrenaica</i> Benth.		
<i>L. viridis</i> L'Herit		
<i>L. burmanni</i> Benth.		

In the sixteenth century different kinds of *Lavandula* were cultivated in England and Germany.^{15,30} Gessner¹⁵ described the steam distillation of the flowers of *spica*, while Porta¹⁶ mentioned the distillation of the flowers of lavender.

The differentiation between spike and lavender oil was made at the end of the sixteenth century.²⁵

Two species of the *Lavandula* genus, namely *L. stoechas* and *L. spica*, probably were known throughout history, but later on the names of *L. vera* and *L. latifolia* have been used.²⁸ The isolation of French spike and/or lavender oil in the eighteenth century has been described in detail by Demachy.²¹ The isolation and yield of spike and lavender oils by steam distillation of the flowers were described for the first time by Cartheuser²² and Lewis.²⁰ Camphor, so-called lavender camphor, was noticed in the oil in 1785 by Arezula²³ and in 1800 by Proust.

The first investigation of lavender oil was made by de Saussure.²⁴

In the nineteenth century the most important constituents of spike oil were determined. Kane²⁵ and Lallemand²⁶ detected d-camphor in the oil. Bruylants²⁷ described the presence of d-borneol and d-camphor. Voiry²⁸ and Bourchardat²⁹ proved

the presence of d- α -pinene, d-camphene, 1,8-cineole and l-linalool in the oil.

Botany

The genus *Lavandula* comprises about thirty species. The taxonomy, however, of this genus is rather complicated because of the existence of several names for one species, and the occurrence of a lot of subspecies and varieties.⁴⁰⁻⁴⁷ The important *Lavandula* species, their synonyms and subspecies are depicted in Table I.

The genus *Lavandula* is subdivided into five sections, namely, *stoechas* and *spica* which grow in Europe, and *pterostoechas*, *chaetostachys*, and *subnuda* which grow in Arabia, Africa, India and the island of Socotora.^{3,41}

Although it was agreed in 1930 (International Congress of Cambridge) that the name *Lavandula spica* D.C. should be eliminated from the botany and further on only the name *Lavandula latifolia* Vill. should be used, most books and reviews of essential oils still use the name *L. spica* D.C.

Because *Lavandula* is one of the most important genera of the family Labiatae with respect to the production of essential oils, it seems worthwhile to describe the botanical features of this

family in more detail. The species of the labiatae are herbs or shrubs, often with a square stem, leaves crosswise opposite each other, mostly hairy, with oil glands which excrete an essential oil in many genera. The flowers are in the axils of the leaves, often in corollas forming a spike at the top. The calyx is two-lipped or five-dentated; the corolla is crooked and mostly has a lower lip with three lobes and an upper lip with two lobes. There are four stamens of which two are longer than the others. The ovary has two capsules, but is, by a secondary outgrowth of the wall, divided into four chambers, of which each carry one seedbud. The fruit falls into four one-seed pieces (nuts).

Lavandula latifolia Vill. is a shrub, sometimes up to 1 meter high, but normally 30 to 60 cm in height. The plant has one main root going 30 to 40 cm into the ground, is ligneous at the feet, and has numerous twigs, also more or less lignified. The twigs bear a lot of leaves. These leaves are somewhat longer than those of other *L.* species, therefore the name of *L. latifolia*, which stems from Latin and means "great-leafy." There exists two types of leaves; the normal ones, which are greyish in colour and about 20 mm long and 2 mm in width; between these leaves another type

appears, more greenish, somewhat shorter and ca 4 mm in width. The leaves are hairy and contain oil glands. The twigs have a top up to 20 cm long, without leaves, bearing the flowers. Each twig-top possesses six to ten groups of sitting flowers, the lower ones on flower-stems, some centimeters outside the flower twig. The flower twig with flowers is a true spike on the plant. The flowers are small and have a violet corolla. The calix is green. The fruits are conically oval with a brilliant brown colour.

L. latifolia grows in Spain between the towns of Burgos and Murcia, in several provinces, but especially in the province of Guadalajara on the sunny side of the hills up to 800 meters high. The plants need a moderate Mediterranean climate, not too warm (below 30°C); they grow in a lime soil.^{46,47}

Isolation

The isolation of spike oil has been described in detail by Argila,⁴³ Guenther,² Gildemeister and Hoffmann,⁶ and Yllera Camino.⁸

Although it is generally accepted that spike oil is obtained by steam distillation of the flowers of *L. latifolia* (see EOA No 4), one should notice that often other particles of the plants, i.e., stems

and leaves, are steam distilled together with the flowers.

The bulk of spike oil in Spain is manufactured in so-called field stills. According to Guenther,² with some modifications, one distillation post usually consists of two stills; while one is in operation, the other one is being emptied of exhausted plants and recharged with fresh material. The stills are of simple construction, made of sheet iron, cylindrical of 2 to 2.5 M³ capacity. They are half-embedded in the ground, usually on a slope, with a fireplace dug into the ground beneath the still. Spent and sun-dried plant material serves as fuel. A perforated grate inserted about 20 cm above the still bottom prevents pyrolyzing of the plants, due to direct contact with the lower still walls surrounded by open flames. The condenser, usually a long pipe, is sunk into a brook or waterhole. After being dried for some time in the sun, the plant bundles (stems with leaves and flowers) are charged into the still, normally about 400 kg, tramped down, and sufficient water, about 150 liters, is poured into the still to fill the space between the still and the bottom grill.

The time of charge lasts thirty minutes, and another thirty minutes are required to get the steam up and start actual distillation, which ends after two hours, discharging needs again about thirty minutes. The complete distillation of one charge requires four hours, when about 125 liters of water have distilled over. The length of distillation depends upon the intensity of the fire, the efficiency of the condenser, and the condition of the plant material (fresh or dried). When the distillation is completed, the still is emptied and recharged. Six charges can thus be completed within twenty-four hours.

The oil, 3.5 to 4 kg, in one charge, is collected in a simple separator, decanted from the distillation water (which often is recycled) and collected in drums. When filled, the drums are transported to the exporters who filter and analyze the oils and make up bulkings for the export trade.

Guenther² mentioned that the difference between the yields from the water and steam distillation probably can be explained by the fact that in the former Spanish stills the lower part of the plant charge often becomes soaked with water, especially when the stills are not provided

Table II. Yields of Spike Oil of Different Origin

Origin	Yield in %	Author
<u>Spain:</u>		
First year	0.82 - 0.97	Argila ⁴³
Second year	1.0 - 1.2	Argila ⁴³
General	0.88	Guenther ²
<u>France:</u>		
Water distillation	0.5 - 0.62	Laval ⁴⁹
Water distillation	0.98	Schimmel ⁴⁸
Steam distillation	1.1	Schimmel ⁴⁸
<u>Italy:</u> (angustifolia)	0.6 - 0.8	Rovesti ⁵⁰
<u>Eritrea:</u> (angustifolia)	0.88 - 0.91	Rovesti ⁵⁰

with grids. The plant charge agglutinates, especially towards the bottom, forming a heavy mass through which the steam can no longer penetrate uniformly, and therefore forms channels. It is then very difficult, if not impossible, to exhaust the material with low pressure steam. The relatively long time (two to three hours) for one charge in the Spanish stills seems to lend support to this theory, lavender plants being exhausted in France with direct steam in less than one hour.

The method employed in Spain⁵¹ is actually a "water *and* steam distillation" which gives good results only if the plant material is separated completely by a grid from the boiling water beneath. If not properly carried out, the method suffers from the drawback of frequently incomplete exhaustion and low yield. The advantage of water and steam distillation, on the other hand, lies in the better solubility of the oils; in fact, Spanish spike oils show good solubility.

Physicochemical Properties

The essential oil of spike lavender is probably the most pronounced oil, for whose properties one can not rely on one or two analyses. In ten publications during the past century more than one-hundred different qualities of oils have been analyzed.

Much work has been done to determine the physicochemical properties, namely specific gravity, optical rotation, solubility in ethanol and the alcohol and ester numbers of the oil. Until 1950 these properties were the main characteristics for the oils of different origin (see Table II). The best oils should have a specific gravity be-

Table III. Physicochemical Properties of Spike Oil (Literature Investigations)

Author (year) Origin	Specific Gravity	Optical Rotation	Refractive Index	Solubility L ₆₅ ° (EtOH)	Alcohol Content % (Number)	Ester Content % (Number)	Acid Number	1,8-Cineole Content
Schimmel (1908) ⁴⁸ : <u>Spain</u>	0.9052 to 0.9078	-0°49' to +0°42'	-	3-3.5	-	(5-11)	-	-
general	0.900 to 0.922	-5°45' to +15°47'	1.464 to 1.467	70°:1.5-2.5	-	(4.6-26.5)	max. 0.8	-
<u>France</u> general	0.900 to 0.920	-4°5' to +6°44'	1.463 to 1.469	70°:1.7-3	30-37	(3-21)	max. 0.8	-
Dorronsoro (1909) ⁵² : <u>Spain</u>	0.903 to 0.9161	-3°28' to +10°54'	1.4650 to 1.4718	2.7-4.3	27.95 to 28.85	-	1.06-3.2	-
Schimmel (1911) ⁵³ : <u>Spain</u>	0.9100	-2°20'	1.468	70°:2	-	(7.0)	3.7	-
<u>Yugoslavia</u>	0.9033 to 0.9022	-0°53' to -0°10'	-	60°:4-5 70°:2-3	-	-	-	-
Tedesko (1927) ⁵⁴ : <u>Spain</u>	0.9123	+2°5'	-	5	32.5	-	-	35.4
general	0.9090 to 0.9046	+1.8° to -2.25°	-	3.5	34-38	-	-	28.5-33
Rutowski (1928) ⁵⁵ : <u>Russia</u>	0.9067 to 0.9111	+1.18° to +2.02	1.4660 to 1.4675	-	-	(86.07 to 108.25	-	36-56.2 (camphor: 12.4-25.5)
Guenther (1929) ¹ : <u>Spain</u> : Guadalajara	0.901	-2°56'	-	3	40.6	2.6	-	-
Palomera	0.900	-4°35'	-	3.5	37.7	2.0	-	-
Palomera	0.901	-4°54'	-	3.5	37.8	2.4	-	-
Campillo	0.908	-1°53'	-	4	32.6	2.3	-	-
Huetor Sant.	0.905	-2°18'	-	3	35.6	1.8	-	-
Granada	0.904	-2°20'	-	3	36.7	2.1	-	-
Moral	0.908	+0°34'	-	3	35.7	2.7	-	-
Murcia	0.908	-1°6'	-	4	32.6	2.4	-	-
general	0.899 to 0.911	+1°9' to -7°25'	1.4624 to 1.4679	3-4.5	34.0-38.5	1.8-3.3	-	-
best quality:	0.900 to 0.905	up to -7°30'	-	3	35-40	-	-	-
lower quality:	0.910 to 0.920	up to +8°	-	2	25-30	-	-	-
Fesneau (1949/1950) ⁵⁷ : <u>Spain</u> general	0.904 to 0.912	-1° to +3°	-	3	30-35	3-5	-	-
Guadalajara I	0.900 to 0.903	-2° to -5°	-	3-3.5	37-43	3-5	-	-
Guadalajara II	00.902	-3°25'	1.465	3	38.7	2.4	0.3	-
Ochoa (1952) ⁵⁸ : <u>Spain</u>	0.906	-2°28'	1.4652	-	26.82	-	-	-
Garcia Araez(1953) ³ : <u>Spain</u> general	0.900 to 0.923	-5° to +12°	1.464 to 1.468	70°:2-3	(95-105)	(4-27)	1-2	-
<u>France</u> general	0.906 to 0.923	-2° to +6°	1.464 to 1.468	70°:1.9-2	(95-110)	(4-22)	0.8-1.2	-
da Costa(1952) ⁵⁹ : <u>Portugal</u>	0.9251	+5°51'	1.4715	2.5	43	(21.8)	1.2	-
Neybergh(1953) ⁶⁰ : <u>Belgian Congo</u>	0.9239	+13°	1.4645	-	19.7	1.75	0	-
Rovesti(1955) ⁵⁰ : <u>Italy</u> Cosio Arroscia	0.8892	-7°71'	1.4629	70°:2.9	0.16	39.33	-	0.28
Monte Fondi	0.8864	-4°74'	1.4612	70°:2.6	0.98	32.11	-	0.87
Monte Pollino	0.9074	-5°46'	1.4628	70°:3.5	1.24	36.42	-	1.38
<u>Eritrea</u> Asmara	0.8801	-7°14'	1.4628	80°:1.5	2.52	17.65	-	1.54
Filfil	0.8783	-11°48'	1.4712	85°:1	1.88	9.47	-	2.62

continued

(Table III continued)

Fenaroli(1963) ⁷ : Spain general	0.894 to 0.918	-6.0° to +8.0°	1.464 to 1.468	1.5-5	-	1-10.5	max. 0.8	-
France general	0.896 to 0.916	-4.0° to +7.0°	1.463 to 1.469	2-4	-	1-10	max. 0.8	-
Yllera Camino (1965) ⁸ : Spain (38 samples)	0.897 to 0.912	-0.024° to -5.045°	-	2.8-4.8	40-60	2-7.5	0.4-0.9	-
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tween 0.900 and 0.905, a negative optical rotation and a high alcohol number; all these properties are mainly due to the amount of l-linalool. A higher specific gravity (0.910-0.920) and a positive optical rotation are mainly caused by the amounts of d-borneol and d-camphor, and result in an oil with lower quality. The amount of d- α -pinene and d-camphene, however, can decrease the specific gravity and increase the negative optical rotation, but results also in a low solubility in aqueous ethanol (65 or 70%). The amount of 1,8-cineole present increases the specific gravity but lowers as well the positive as the negative optical rotation.

Formerly, the total alcohol content was sometimes calculated as borneol, however the most dominant monoterpene alcohol in the oil is linalool (ca 40%), whereas the content of borneol mostly is below 5%. It can be doubted whether before World War II, one was able to determine quantitatively the exact amount of linalool.

The acid number of spike oil is often below 0.8.⁶ Yllera Camino⁴⁵ found an average acid number of 0.5 in thirty-seven samples of the oil analyzed between 1930 and 1936. An acid number of 0.56 means 0.01 maeq. free acids in 1 gr of oil; for a C₅-acid, for example 3-methylbutanoic acid, C₅H₁₀O₂, MW 102, one can calculate 0.1% (1000 ppm) free acid, and for a C₁₀-acid (monoterpenic) about 0.17% (1700 ppm). The ester number of spike oil normally is between 5 and 10 (=5-10 mg KOH per 1 gr of oil); which means for a monoterpene acetate (C₁₂H₂₀O₂) about 2 to 4% ester.^{2,6,45} The dominant esters in the oil are n-hexyl esters, linalyl and bornyl acetate (total 2-3%).

Chemical Composition

The chemical composition of spike lavender oil is rather complicated. Nowadays up to 350 constituents have been detected in the oil. Moreover the main components can vary in their concentrations. The variations in the number of constituents and in their concentrations may be due to

—different plant material (contaminations and subspecies or varieties)

—climatological differences

—differences in the isolation methods (distillation-time, more or less water, forced conditions)

Therefore the outcome of the physicochemical properties and the chemical composition is often based on several oils (see Table III). Thus it is advisable not to rely on the outcome of one or two oils.^{90,91} If one, in the analysis, goes down to 0.01% (100 ppm), about seventy constituents can be detected. On a part per million level (1 ppm), however, over three-hundred components are shown.⁹¹ The main constituents of spike oil can be subdivided into the chemical groups shown in Table IV and discussed further below.

Aliphatic Compounds

During quantitative investigations of spike oil, about ten aliphatic compounds have been identified (see Tables). The most important aliphatic components are n-hexanol and derivatives i.e., methyl ether, acetate and tiglate, octanol-3, oct-1-enol-3 and octanone-3. In a qualitative study, however, over one hundred aliphatic constituents have been determined in the oil.⁹¹ Most of these compounds will be present in spike oil at a part per million or even lower level. Some of these components, however, probably are artefacts caused by mistakes made during collection of the natural product and the isolation of the oil.

The natural product, i.e. plant material, can be contaminated by accident and/or on purpose, with other plants, namely weed and/or other essential-oil-producing plants.^{2,8} The presence of weed in *L. latifolia* plants gives rise to the formation of:

—sulfur compounds, due to the decomposition of S-containing amino acids

—branched chain fatty aldehydes and acids (esters) caused by the degradation of amino acids, mainly leucine and isoleucine (Strecker degradation and oxidation)

—linear aldehydes and acids (esters) caused by the oxidation of fatty oils (unsaturated fatty acid-glycerides)

Table IV. Main Constituents of Spike Oil

Chemical Groups	Approximate Number of Compounds	Approximate Percentage
aliphatic	10	1
monoterpenes	15	7-10
monoterpene alcohols	10	40-50
monoterpene ethers	5	25-30
monoterpene carbonyls	5	12-15
monoterpene esters	5	1-3
sesquiterpenes	10	3-5
sesquiterpenoid derivatives	5	1
benzenoids	5	1

The main mistakes during the isolation of the oil are too little water during the steam distillation and, as mentioned before, open fire in direct contact with plant material both causing pyrolysis, and too long distillation times.² The pyrolysis or "burning" of plant material gives, for instance, rise to the formation of

- aliphatic ketones, e.g. acetone → diacetone alcohol → mesityl oxide
- furan derivatives, due to the degradation of plant sugars
- phenols and pyridines, caused by heating of ligneous materials

Too long distillation times cause the presence of too much high boiling components in the oil, e.g., higher fatty acids (esters) and sesquiterpenes⁹⁰ (see below).

Monoterpenes

Up to fifteen monoterpenes have been detected in spike oil. The total concentration of monoterpenes in the oil is between 5% and 10%. d- α -Pinene, β -pinene and limonene are the most important monoterpenes, their concentrations normally vary between 1% and 3%.

Prager and Miskiewicz⁸⁸ used, for example, the concentration of ocimene to differentiate between spike lavender, lavandin and lavender oils. They found that spike lavender oil contained little (<0.5%), if any, as compared with the other two oils.

Monoterpene Alcohols

So far about ten monoterpene alcohols have quantitatively been identified in spike oil. Their concentrations amount to 40-50% of the oil.^{63,70,76} l-Linalool is dominant of the alcohols with often a concentration of more than 40% in a good quality of spike oil. All other monoterpene alcohols may be present in a total concentration of 3% to 5%,

from which borneol and α -terpineol are the most important. Lavandulol is an olfactively interesting monoterpene alcohol; its concentration in the oil may be 0.5 to 1.0%.

Ter Heide et al.⁹¹ identified in a qualitative study of spike oil more than twenty-five monoterpene alcohols. These alcohols, probably present below 0.1%, will play a role in the overall character of the oil.

In former analysis⁴⁵ the exact content of linalool possibly was difficult to determine; therefore, a part of the linalool present probably was calculated as borneol. Yllera Camino⁴⁵ found the sum of linalool and borneol between 45% and 58% in the oil, however for linalool 17%-27% and for borneol 30%, which percentages are unprobable for a normal spike oil.

Monoterpene Ethers

From the monoterpene ethers 1,8-cineole is a main component in spike oil with a concentration of about 25%. Furthermore cis and trans linalool oxide have been detected in the oil in concentrations between 0.5% and 1.0%. Mugica and Ochoa, however, found for the latter concentrations up to 15%, which seems unlikely.

Monoterpene Esters

About twenty-five monoterpene esters have been detected in spike oil. The most important of these are linalyl acetate (ca 1%), neryl and geranyl acetate, lavandulyl acetate, bornyl acetate and α -terpinyl acetate. Other monoterpene esters, present as trace components, may also play a role for the olfactive quality of the oil.

Monoterpene Aldehydes

The monoterpene aldehydes, about fifteen, are only minor constituents of the oil. Citronellal⁸⁷ and myrtenal^{81,91} may be the most important, in a total concentration less than 0.5%; trace concentrations of citral (neral and geranial), lilac aldehyde, p-menthenals and and p-mentha-dienals have also been identified.⁹¹

Monoterpene Ketones

The monoterpene ketones, about twenty-seven, are olfactively more important for spike oil than the aldehydes. Twenty-seven ketones have been identified in the oil, being d-camphor (ca 15%), cryptone, nopinone, carvone and α/β -thujones olfactively the most important.

Monoterpene Acids

Monoterpene acids are trace components of spike oil, and probably olfactively not very important.

Sesquiterpenes

Seventeen sesquiterpenes so far have been identified in spike oil. Quantitatively the most important sesquiterpenes are caryophyllene, bisabolene and cadinene. The total amount (number and concentration) of sesquiterpenes is dependent on the steam distillation time during the isolation of the oil.⁹⁰

Sesquiterpenoid Alcohols and Epoxide

These alcohols, about eight, are only trace constituents of spike oil. For their concentrations the same holds true as for the sesquiterpenes. These compounds, however, can play a role in the overall fixative properties (long-lastingness) of the oil. The most important sesquiterpenoid oxygen derivative, quantitatively, is caryophyllene oxide, present in the oil in a few promille.

Benzenoids

About sixty benzene derivatives have been identified in spike oil, most of them at a part per million level or even below this concentration.

The most important benzenoids are coumarin (and dihydro derivative), eugenol and possibly thymol and carvacrol. Also the methyl homologues of 2-hydroxy-acetophenone may play a role as trace components. These benzenoids not only are important for their own olfactive properties but may also influence the tenacity (fixation) of other volatiles. Most of the other benzenoids, especially acids (esters) and phenols, probably are present in the oil due to pyrolytic degradation of ligneous plant material.

Nitrogen Containing Compounds

Twenty one nitrogen containing compounds have been detected in spike oil.⁹¹ This group of compounds can be subdivided into (alkyl)-pyridines, acetyl-pyridines, alkyl-pyrazines and -oxazole, and methyl anthranilates. Some of these components may play a role in the overall odour character of the oil, especially because these compounds can possess low threshold values. Most of these compounds, however, can be regarded as degradation-products (pyrolysis) of the plants during the isolation of the oil.

Data of the analyses of spike oils are given in the accompanying table on pages 56-63 at the end of this article.

Olfactive Properties and Applications

Guenther² describes the best, medium and inferior olfactive qualities of spike oils. Better solubility, a lower specific gravity and a higher laevorotation indicate a better quality. These

Table V. USA Import Figures for Lavender and Spike Oils

Year	Total Amount in t/a	Lavandin & Lavender	Estimated Spike Oil
1973	165.0	115.0	50.0
1974	127.0	89.0	38.0
1975	51.5	36.0	15.5
1976	109.5	76.5	33.0
1977	82.3	57.3	25.0
1978	113.7	80.0	33.7
1979	140.0	98.0	42.0

properties are mainly due to the content of monoterpene alcohols and their esters (acetates), and especially l-linalool and its acetates.

According to Garcia Araez,³ the oil of spike is a colour-less or slightly yellow liquid, with a camphoraceous odour, reminiscent of lavandel, especially in dilution, but it is more earthy and heavier than lavender oil. Poucher⁴ mentioned spike lavender oil as brownish-yellow in colour and with a camphoraceous odour reminiscent of rosemary and lavender.

Spike oil has been described by Gildemeister and Hoffmann⁵ as a yellowish liquid whose odour is somewhat camphoraceous with notes of lavandel and rosemary. According to Bauer and Garbe⁶ spike oil is a slightly coloured to yellow liquid with a camphoraceous, lavandel-like odour.

The body of spike oil is olfactively determined by l-linalool, 1,8-cineole and d-camphor, together making up about 80% of the oil. The balance is made by the olfactively less interesting mono- and sesquiterpenes, total 10%-15%. There remains about 5%-10% for the odouristic finishing touch. First of all the oil contains about 5% of other monoterpene alcohols, from which d-borneol is dominant with about 2%, olfactively. However, the most interesting of these alcohols is lavandulol (present ca. 0.5%). Lavandulol has a floral, rose-like, and herbal odour, with a typical spike note.^{9,2} Secondly the oil contains about 2%

esters of monoterpene alcohols, mainly acetates, with linalyl acetate (ca. 1%) as the most important. Finally the trace constituents, like sesquiterpenoids and benzenoids, are olfactively important.

Table V summarizes the olfactive qualities of spike oil, giving the evaporation time and chemical aspects.

For the topnote of spike oil, apart from the monoterpenes and 1,8-cineole, some aliphatic compounds are responsible, as there are n-hexyl methyl ether and -acetate, and 3-octanone and oct-1-enol-3 (together present up to 0.5%). For the middle note, apart from 1,8-cineole, camphor, linalool (lavandulol) and esters, n-hexyl tiglate (up to 0.5%) is olfactively important.

Hexyl tiglate has a soupy, lovage-like odour with a characteristic lavender-like note in the dry-out. The dry-out of a good quality of spike oil is determined by three groups of compounds. The olfactively most important are coumarin and its dihydro derivative, which are present in 0.1% to 0.2%. These compounds afford the oil its sweet aromatic floral dry-out. Secondly, the group of sesquiterpenoids (oxygen derivatives), with caryophyllene oxide as dominant, are olfactively interesting not only for their woody, powdery odour characteristics, but also for their fixative properties which makes the complete oil more long-lasting. Finally, small amounts of eugenol lends its spicy odour note to the oil.

According to Guenther,² oil of spike lavender is widely used for the scenting of soaps. Since the odour of this oil is more camphoraceous, harsher and stronger than that of lavender oil, it gives especially good results in lower priced types of soaps. Spike oil finds use also in many cosmetic and technical preparations: bath salts, room sprays, and disinfectants. In general, it represents a low priced substitute for lavender oil.⁶

Fenaroli⁷ mentioned that spike oil especially finds application in soap compounds, in which it has a good stability and affords a freshness. Moreover, spike oil is used in compositions for

Table VI. Olfactive Qualities of Spike Oil

Odour Aspects	Time Hours	Probably Due To
fresh-green	0-1	n-hexyl derivatives (methyl ether)
harsh terpene-like	1-2	monoterpenes
fresh minty eucalyptol	2-4	1,8-cineole
camphoraceous } fresh floral	4-8	camphor (borneol) linalool (lavandulol)
soupy lovage	8-15	hexyl tiglate
sweet aromatic (hay-like)	8-24	(dihydro) coumarin
spicy	and	eugenol
woody-powdery	more	caryophyllene oxide and other sesquiterpenoids

Spike Lavender Oil

aerosol deodorants, air-refresheners and extrait perfumes. The oil blends excellently with other essential oils, such as rosemary, lavender, eucalyptus and petitgrain.

Reconstitution

The reconstitution or compounding of spike oil, knowing the concentrations of the constituents, seems rather easy but is extremely difficult, and virtually impossible, mainly because the main constituents, l-linalool, d-camphor and d-borneol, are optically active. Moreover an exact copy of the natural product compounded with nature-identical, optically active compounds, will be more expensive than the natural product.

Of course, one can reconstitute a cheaper "coupage" for the original oil, with optically inactive nature-identical chemicals; however, the natural freshness of the oil is missing and the minor olfactively characteristic constituents are diluted.

Production and Trade

Production of spike oil until 1980 can be estimated at 150-200 tons annually, from which the bulk (over 75%) is produced in Spain and Portugal. Very recently Lawrence⁹⁴ stated that at one time more than 200 metric tons of spike lavender oil were produced in Spain. Presently, it is estimated that the recent production has not exceeded 35 metric tons.

The consumption of spike oil in different parts of the world is difficult to define. Most of the consumption is included in the figures of lavender oils.

According to the production figures of lavender and spike oils one may assume the ratio of these oils as 7 to 3. Thus about one third of the oils is spike lavender.

The important figures for the United States during 1973-1980 for the total amount of these oils is depicted in Table VI. The consumption of spike oil until 1980 in different parts of the world can be estimated at 60-75 tons per annum in Western Europe, 40-50 tons in the US and 50-75 tons in the rest of the world.

Acknowledgement

The author is greatly indebted to Mrs. Jayne Meneses for correcting and typing of the manuscript.

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Chemical Composition of Spike Lavender Oil (Literature Investigations)

HYDROCARBONS

isoprene (91),
1-dodecene (91),
toluene (91),

monoterpenes:

α -thujene (87),
 α -pinene (8, 39, 61, 69, 73, 75, 77, 78, 80, 82, 85, 86, 87, 89, 90, 91)
camphene (39, 61, 73, 75, 77, 78, 80, 82, 85, 86, 87, 88, 89, 90, 91)
 β -pinene (61, 73, 75, 77, 78, 80, 82, 85, 86, 87, 88, 89, 90, 91),
myrcene (73, 75, 77, 85, 87, 90, 91),
sabinene (73, 77, 85, 87, 89, 90, 91),
cis-ocimene (72, 81, 87, 88, 91),
trans-ocimene (72, 81, 87, 88),
 α -terpinene (87, 91),
 δ -terpinene (75, 81, 85, 91),
terpinolene (86, 87, 91),
limonene (61, 75, 77, 79, 85, 86, 87, 88, 89, 91),
p-cymene (61, 77, 78, 85, 86, 87, 89, 90, 91),
 α -phellandrene (75, 87, 85),
 β -phellandrene (89).

sesquiterpenes:

β -caryophyllene (73, 87, 88, 90, 91),
 β -selinene (91),
 β -farnesene (81, 90, 91),
 β -cubebene (91),
 α -gurjunene (90, 91),
 γ -cadinene (81, 90, 91),
 δ -cadinene (90, 91),
 α -copaene (91),
 α -cedrene (91),
bergamotene (91),
cis/trans calamene (90, 91),
bisabolene (8),
cis α -bisabolene (87),
trans α -bisabolene (90),
 β -bisabolene (87, 90),
 α -humulene (87),
longifolene (81),
1,6-dimethyl-4-isopropyl-naphthalene (91).

ALCOHOLS**aliphatic:**

ethanol (91),
propanol-2 (91),
butanol-1 (91),
butanol-2 (91),
2-methylpropanol-1 (91),
n-pentanol (91),
3-methylbutanol-1 (91),
1-penten-2-ol (91),
2-methyl-3-buten-2-ol (91),
hexanol (8, 62, 75, 85, 87, 91),
cis-3-hexen-1-ol (91),
heptanol (91),
octanol (8, 85, 91),
octanol-3 (87, 91),
1-octen-3-ol (91),
decanol (91),
diacetone alcohol (91).

monoterpenoid:

linalool (8, 38, 61, 65, 67, 68, 69, 72, 73, 74, 75, 77, 78, 79, 80, 82, 84, 85, 86, 87, 88, 89, 90, 91),
nerol (61, 69, 75, 81, 87, 91),
geraniol (8, 57, 67, 68, 69, 75, 85, 87, 89, 90, 91),
myrcenol (91),
trans-hotrienol (91),
trans-2,7-dimethyl-1,4,6-octatrien-3-ol (91),
 α -terpineol (39, 73, 75, 77, 78, 80, 82, 85, 87, 88, 89, 90, 91)
 β -terpineol (75),
 δ -terpineol (90, 91)
1-terpinen-4-ol (61, 73, 74, 77, 85, 87, 88, 89, 91),
cis-piperitol (91),
trans-piperitol (91),
trans-carvotanacetol (91),
cis-carveol (87, 90),
trans-carveol (75, 87, 90),
p-cymen-8-ol (90, 91)
cis-sabinene hydrate (91),
trans-sabinene hydrate (91),
camphene hydrate (91),
trans-sabinol (91),
myrtenol (75, 90, 91),
isoborneol (86, 87, 89, 91),
isosobrerol (57),
lavandulol (85, 87, 88),
citronellol (61, 87),
trans-pinocarveol (90),
borneol (8, 37, 67, 68, 69, 73, 75, 77, 78, 79, 83, 85, 86, 87, 88, 89, 90, 91),
cuminic alcohol (90, 91)
2,6-dimethyl-6-methoxy-7-octene-2-ol (91),
exo-1,8-epoxy-p-menthan-2-ol (91),
endo-1,8-epoxy-p-menthan-2-ol (91),
1,8-epoxy-2-p-menthen-4-ol (91),
2,2,6-trimethyl-6-vinyl-3-hydroxy-tetrahydropyran (91),
cis-1,8-terpin hydrate (91).

sesquiterpenoid:

nerolidol (91),
 α -cadinol (87, 91),
 β -cadinol (91),
 δ -cadinol (91),
10-epi- α -cadinol (91),
viridiflorol (91),
spathulenol (91),
 α -bisabolol (87, 91).

ETHERS**aliphatic:**

hexyl methyl ether (91).

cyclic:

2-methylfuran (91),
3-methylfuran (91),
2-ethylfuran (91),
3-isopropylfuran (91).

mono- & sesquiterpenoid:

1,8-cineole: (8, 39, 50, 54, 64, 65, 67, 68, 71, 72, 73, 74, 77, 78, 79, 80, 82,

83, 86, 87, 88, 89, 90, 91);
cis-linalooloxide: (85, 87, 90, 91);
trans-linalooloxide: (85, 87, 90, 91);
2,2,6-trimethyl-6-vinyl-tetrahydropyran: (91);
5-isopropyl-2-methyl-2-vinyl-tetrahydrofuran: (91);
(= cis-dihydrolinalooloxide)
trans-dihydrolinalooloxide: (91);
caryophyllene oxide: (73, 77, 87, 90, 91).

benzenoid:

safrole (91);
trans-anethol (91);
thymol methyl ether (91);
carvacrol methyl ether (91).

ESTERS**aliphatic:**

2-propyl formate (91);
hexyl formate (91);
hexyl acetate (8, 81, 91);
hexyl propionate (91);
hexyl butyrate (91);
hexyl isobutyrate (91);
hexyl tiglate (87, 91);
hexyl 2-methylbutyrate (91);
hexyl 3-methylbutyrate (91);
hexyl caproate (91);
hexyl benzoate (91);
ethyl acetate (91);
allyl acetate (91);
2-butyl acetate (91);
octyl acetate (8);
octyl propionate (91);
octyl 2-methylbutyrate (91);
octyl 3-methylbutyrate (91);
methyl butyrate (91);
3-methylbutyl 2-methylbutyrate (91).

terpenoid:

linalyl acetate (61, 64, 65, 67, 68, 69, 71, 72, 73, 74, 75, 77, 78, 79, 80, 82, 84, 85, 86, 87, 88, 91);
geranyl formate (75);
geranyl acetate (87, 85, 91);
geranyl propionate (91);
geranyl butyrate (87);
geranyl 3-methylbutyrate (91);
neryl acetate (87);
neryl propionate (91);
neryl 2-methylbutyrate (91);
neryl 3-methylbutyrate (91);
neryl caproate (91);
lavandulyl acetate (87);

terpenoid:

lavandulyl propionate (91);
lavandulyl butyrate (91);
lavandulyl isobutyrate (91);
lavandulyl 2-methylbutyrate (91);
lavandulyl 3-methylbutyrate (91);
bornyl acetate (75, 86, 87, 91);
isobornyl acetate (75, 85)
iso- or normal bornyl propionate (91);
iso- or normal bornyl isobutyrate (91);

bornyl 2-methylbutyrate (91);
iso- or normal bornyl
3-methylbutyrate (91);
 α -terpinyl acetate (80, 85, 86, 87);
epoxilinalyl acetate (87).

benzenoid:

benzyl benzoate (91);
methyl salicylate (91).

ALDEHYDES

aliphatic:

formaldehyde (61);
acetaldehyde (61, 91);
propanal (61);
butanal (91);
2-methylpropanal (61, 91);
pentanal (91);
3-methylbutanal (91);
2-methylbutanal (91);
hexanal (91);
heptanal (91);
2-ethyl-3-methyl-butanal (91);
octanal (91);
nonanal (91);
decanal (91);
trans 2-methyl-2-buten-1-al (tiglic
aldehyde) (91);
2-methyl-2-penten-1-al (91);
trans 2-hexen-1-al (91);
trans 2-octen-1-al (91);
trans 2-nonen-1-al (91);
cis,trans 2,6-nonadien-1-al (91).

terpenoid:

1,3-p-menthadien-7-al (91);
1,8-p-menthadien-7-al (perilla
aldehyde) (91);
1-p-menthen-7-al (phellandral) (91);
1-p-menthen-9-al (91);
neral (91);
geranial (91);
citronellal (87);
myrtenal (61, 91);
5,5-dimethyl-2-cyclopenten-1-acet-
aldehyde (91);
2,2-dimethyl-3-cyclopenten-1-acet-
aldehyde (91);
1-carboxaldehyde-5-isopropenyl-2-
methyl-1-cyclopentene (91);
 α -campholenic aldehyde (91);
2-formylmethyl-5-isopropenyl-2-
methyltetrahydrofuran (91);
2-(1-formylethyl)-5-methyl-5-vinyl-
tetrahydrofuran (4 isomers)
(= lilac aldehyde) (91);
3-acetyl-2,2-dimethyl-cyclobutane-
acetaldehyde
(= pinone aldehyde) (91).

cyclic:

furfural (57, 87, 91);
3-methylfurfural (91);
5-methylfurfural (91);
1-formyl-2-methyl-1-cyclopentene
(91);
tetrahydropyran-2-yl acetaldehyde
(91).

benzenoid:

benzaldehyde (91);
2,5-dimethylbenzaldehyde (91);
phenylacetaldehyde (91);
2-(p-methylphenyl)-propanal (91);
4-(p-methylphenyl)-pentanal (91);
salicylaldehyde (91);
cuminaldehyde (87, 91)
2-hydroxy-4-isopropylbenzaldehyde
(91);
4-isopropenylbenzaldehyde (91).

KETONES

aliphatic:

acetone (61, 91);
butanone (91);
3-buten-2-one (91);
2-pentanone (91);
3-pentanone (91);
3-methyl-2-butanone (91);
2-hexanone (91);
3-hexanone (91);
4-methyl-2-pentanone (91);
2-methyl-3-pentanone (91);
2-methyl-1-penten-3-one (91);
2-heptanone (91);
3-octanone (8, 73, 77, 85, 87, 91);
2-undecanone (91);
5-hexen-2-one (91);
4-methyl-3-penten-2-one (mesityl
oxide) (91).

terpenoid:

6-methyl-5-hepten-2-one (73, 85, 87,
91);
6-methyl-3,5-heptadien-2-one (91);
cryptone (73, 77, 91);
nopinone (61, 73, 77, 91);
piperitone (91);
4-isopropenyl-3-methyl-2-cyclo-
hexen-1-one (o-isopiperitenone)
(91);
carvone (73, 77, 87, 91);
pinocarvone (91);
carvotanacetone (75);
sabina ketone (91);
camphor (8, 35, 36, 37, 61, 64, 65, 68,
72, 73, 74, 75, 77, 78, 79, 80, 82, 83,
84, 86, 87, 88, 89, 90, 91);
verbenone (61, 91);
isofenchone (91);
camphenilone (91);
 α -thujone (75, 85, 87);
 β -thujone (75, 85, 87);
3-isopropylcyclopentanone (91);
3,4,4-trimethyl-2-cyclopenten-1-one
(91);
3-methyl-2-cyclohexen-1-one (91);
2-hydroxy-3-isopropyl-2-cyclo-
hexen-1-one (91);
6-hydroxy-3-isopropyl-6-methyl-2-
cyclohexen-1-one (91);
2-methyl-3,6-heptadione (91);
2-methoxy-3,5,5-trimethyl-2-cyclo-
hexen-1,4-dione; (91);
geranyl acetone (91);
 α -ionone (91);
 β -ionone (91);
 α -methylionone (91).

benzenoid:

4-methyl-acetophenone (61, 73, 74,
91);
2-hydroxy-acetophenone (91);
2-hydroxy-4-methyl-acetophenone
(91);
2-hydroxy-5-methyl-acetophenone
(91);
2-hydroxy-6-methyl-acetophenone
(91).

ACIDS

aliphatic:

formic acid (57, 62);
acetic acid (91);
propionic acid (91);
butyric acid (91);
isobutyric acid (91);
pentanoic acid (91);
2-methylbutyric acid (91);
3-methylbutyric acid (91);
2,2-dimethylpropionic acid (91);
caproic acid (91);
2,3-dimethylbutyric acid (91);
3,3-dimethylbutyric acid (91);
2-methylpentanoic acid (91);
3-methylpentanoic acid (91);
4-methylpentanoic acid (91);
heptanoic acid (91);
2-ethyl-3-methylbutyric acid (91);
4-methylhexanoic acid (91);
5-methylhexanoic acid (91);
caprylic acid (91);
pelargonic acid (91);
capric acid (91);
tiglic acid (91);
3,3-dimethylacrylic acid (senecioic
acid) (91);
4-methyl-3-pentenoic acid (91)
malonic acid (66);
maleic acid (66);
succinic acid (66, 91)
tartaric acid (66);
citric acid (66);
2-isopropylsuccinic acid (91);
2-isopropylglutaric acid (91);
3,3-dimethyl-1,6-hexanedioic acid
(91);
3-isopropyl-1,6-hexanedioic acid
(91).

terpenoid:

nerolic acid (91);
geranic acid (91);
myrtenic acid (91);
 α -campholenic acid (91);
 γ -campholenic acid (91);
 α -campholitic acid (91);
lilac acid (91);
cyclic:
3-carboxy-4,4-dimethyl-cyclobutane-
1-acetic acid (91);
1-oxo-4,4,5-trimethyl-cyclopentan-3-
acetic acid (91);
2-furancarboxylic acid (91);

benzenoid:

benzoic acid (91);
salicylic acid (91);

phenylacetic acid (91);
3-phenylpropionic acid (91);
3-(o-methoxyphenyl)-propionic acid (91);
cinnamic acid (91);
4-propylbenzoic acid (91);
4-isopropylbenzoic acid (91).

LACTONES

γ-ethyl-δ-butyrolactone (91);
γ-isopropyl-δ-butyrolactone (91);
γ-vinyl-δ-valerolactone (91);
γ-vinyl-δ-hexalactone (91);
4,4-dimethyl-2-buten-1,4-olide (91);
4,4,6-trimethyl-δ-valerolactone (91);
coumarin (57, 62, 73, 77, 87, 90, 91);
dihydrocoumarin (91);
herniarin (90).

PHENOLS

phenol (91);
o-cresol (91);
m-cresol (91);
p-cresol (61, 91);
o-ethylphenol (91);
guajacol (91);
thymol (91);
carvacrol (91);
eugenol (61, 73, 77, 87, 91);
2,4- and/or 2,5-dimethylphenol (91).

NITROGEN CONTAINING COMPOUNDS

pyridine (91);
α-picoline (91);
γ-picoline (91);

2-ethylpyridine (91);
2-ethylpyridine (91);
4-ethylpyridine (91);
4-isopropylpyridine (91);
3-sec. butylpyridine (91);
2,6-lutidine (91);
5-methyl-2-isopropylpyridine (91);
2-methyl-5-isopropylpyridine (91);
5-methyl-2-isopropenylpyridine (91);
2-acetylpyridine (91);
3-acetyl-6-methylpyridine (91);
2-acetyl-5-isopropylpyridine (91);
quinoline (91);
2,3-dimethylpyrazine (91);
2,5-dimethylpyrazine (91);
2,4,5-trimethylloxazole (91);
methyl anthranilate (91);
N-methyl methyl anthranilate (91).

Chemical Composition of Spike Lavender Oil (DBCH Investigations)

	1982	1983	1984	Average
monoterpenes:				
α-thujene	0.05	0.01	t	t
d-α-pinene	1.75	1.63	2.01	1.8
d-camphene	0.60	0.55	0.69	0.6
Beta-pinene	1.64	2.16	2.47	2.1
sabinene	0.49	0.50	0.71	0.6
myrcene	0.57	0.04	0.07	0.2
α-phellandrene	0.13	0.12	0.12	0.1
α-terpinene	0.29	0.07	0.12	0.2
limonene	1.08	1.02	1.11	1.1
cis & trans-ocimene	0.80	0.05	0.07	0.3
p-cymene	0.18	0.27	0.20	0.2
terpinolene	0.25	0.23	0.22	0.2
γ-terpinene	0.26	0.43	0.45	0.4
p-isopropenyltoluene	0.20	0.21	0.16	0.2
sub-total	8.09	7.29	8.40	8.0
sesquiterpenes:				
caryophyllene	1.53	1.36	1.40	1.4
α-humulene	0.43	0.27	0.35	0.4
α-bisabolene	1.85	1.79	2.07	1.9
γ- & δ-cadinenes	0.50	0.70	0.70	0.6
sub-total	4.31	4.12	4.52	4.3
1,8-cineole	25.49	25.93	27.25	26.3
d-camphor	12.49	13.00	13.07	12.8
l-linalool	41.18	43.18	40.60	41.7
cis- & tr. linalooloxide	0.30	0.66	0.46	0.5
linalyl acetate	1.45	0.83	0.92	1.1
d-bornyl acetate	0.27	0.33	0.23	0.3
terpinen-4-ol	0.56	0.56	0.56	0.6
isoborneol	0.26	0.16	0.13	0.2
lavandulol	0.36	0.65	0.63	0.6
α-terpineol	0.99	1.06	1.03	1.0
d-borneol	0.83	0.79	0.88	0.8
nerol	0.03	0.04	0.02	t
geraniol	0.12	0.09	0.09	0.1
sub-total	5.27	5.37	4.95	5.2
coumarin	0.10	0.09	0.07	0.1
dihydrocoumarin	0.03	0.02	0.02	t
caryophyllene oxide	0.03	0.04	0.04	t
eugenol	t	t	t	t
aliphatics:	1.0	1.0	1.0	1.0
butyl acetate				
3-methylbutyl acetate				
n-hexanol				
n-hexyl acetate				
n-octanol				
n-oct-1-enol-3				
octanone-3				
total general	97.99	99.84	99.92	

Approximate Chemical Composition of Different Qualities of Spike Oil

	Poor to Moderate (%)	Moderate to Good (%)	Good to Excellent (%)
l-linalool	30-35	35-40	40-50
l,8-cineole	28-30	26-28	23-26
d-camphor	15-20	13-15	10-13
mono- & sesquiterpenes	15-20	13-15	10-13
other monoterpenoids	2-4	3-5	4-6
other sesquiterpenoids	<0.3	<0.5	<1.0
aliphatics	<0.3	<0.5	<1.0
benzenoids	<0.2	<0.3	<0.5

Chemical Composition of Spike Lavender (DBCH-1984)

<u>Monoterpenes</u>	<u>%</u>	<u>Sesquiterpenes</u>	<u>%</u>
α -pinene	1.6	Beta-bourbonene	0.1
camphene	0.6	α -zingiberene	0.1
sabinene	0.5	α -gurjunene	0.1
Beta-pinene	1.9	Beta-caryophyllene	1.4
myrcene	0.5	α -bergamottene	0.1
limonene	1.1	tr-Beta-farnesene	0.3
Beta-phellandrene	0.3	Beta-cubebene	0.4
Beta-ocimene	0.2	Beta-bisabolene	0.2
γ -terpinene	0.1	germacrene-D	0.2
terpinolene	0.2	α -bisabolene	1.8
p-cymene	0.3	Total	4.7
Total	7.3	<u>l,8-Cineole</u>	25.3
<u>Hexyl esters</u>	<u>%</u>	<u>Camphor</u>	12.7
hexyl isobutyrate	0.1	<u>Linalyl derivatives</u>	<u>%</u>
hexyl butyrate	0.1	linalyl acetate	0.7
hexyl 3 methyl butyrate	0.2	linalyl isobutyrate	0.1
hexyl 2 methyl butyrate	0.1	linalyl 3 methylbutyrate	0.2
Total	0.5	cis-linalol-oxide	0.1
<u>Monoterpene alcohols</u>	<u>%</u>	tr.-linalol oxide	0.1
linalool	43.8	Total	1.2
borneol	1.5	<u>Myrtenal</u>	0.1
α -terpineol	1.0	<u>Carophylleneoxide</u>	0.1
δ -terpineol	0.2	<u>Delta-cadinol</u>	0.1
lavandulol	0.5		
sabinene hydrate	0.1		
tr-pinocarveol	0.1		
terpinene-4-ol	0.4		
geraniol & nerol	0.1		
p-cymene-8-ol	0.1		
Total	47.8		

Chemical Composition Spike Lavender Oil (1980)

Constituents	Wild	Cultivated Plants					
	Growing	1 year	2 years	3 years	4 years	5 years	6 years
α -thujene	0.03	0.17	0.02	0.02	t	t	0.02
α -pinene	1.60	2.82	2.10	1.80	1.60	1.50	1.40
camphene	0.61	0.51	0.41	0.48	0.30	0.35	0.30
Beta-pinene	1.90	3.20	2.70	2.30	2.10	2.00	1.70
sabinene	0.80	1.61	1.41	1.04	1.07	0.98	0.95
myrcene	0.60	0.99	0.89	0.73	0.77	0.64	0.60
α -phellandrene	0.10	0.12	0.05	0.09	0.09	0.10	0.07
α -terpinene	0.04	0.05	0.03	0.04	0.04	t	0.04
limonene	1.00	1.16	0.96	0.95	0.74	0.86	0.77
γ -terpinene	0.60	0.70	0.53	0.58	0.70	0.44	0.66
p-cymene	0.10	0.06	0.07	0.07	0.11	0.07	0.09
terpinolene	0.20	0.22	0.19	0.25	0.21	0.22	0.13
	7.58	11.61	9.36	8.35	7.73	7.16	6.73
1,8-cineole	31.70	42.40	35.10	34.40	32.90	33.10	29.00
linalool	33.20	19.50	31.60	31.80	32.90	33.85	34.20
terpinen-4-ol	0.50	0.53	0.37	0.42	0.47	0.57	0.44
α -terpineol	1.30	2.39	1.73	1.40	2.16	1.65	1.39
borneol	0.80	0.78	0.55	0.70	0.69	0.59	0.79
	35.80	23.20	34.25	34.32	36.22	36.66	36.82
camphor	15.10	14.70	14.40	16.60	14.80	15.30	15.20
bornyl acetate	0.25	0.30	0.25	0.18	0.22	0.26	0.25
cryptone	1.60	2.34	2.52	0.73	1.56	0.22	1.58
caryophyllene	1.30	1.03	1.13	1.60	1.91	1.83	1.69
Total	93.33	95.58	97.01	96.18	95.34	94.53	91.27

J. Carrasco et al. (1980)

Investigation analytique sur l'Huile Essentielle d'Aspic cultivée
Paper No. 112; VIII Int. Congress of Ess. Oils, Cannes, France.

Summary of Groups of Compounds Detected in Spike Oil According to Literature Investigations

Groups	Aliphatic	Monoterpenoid	Sesquiterpenoid	Cyclic	Benzenoid	Total
Hydrocarbons	2	15	17	-	1	35
alcohols	17	34	8	-	-	60
ethers	1	6	1	4	4	16
esters	20	25	-	-	2	47
aldehydes	20	15	-	5	9	49
ketones	16	27	-	-	5	48
acids	34	7	-	3	8	52
lactones	4	2	-	-	3	9
phenoles	-	-	-	-	11	11
nitrogen-containing compounds	-	5	-	-	16	21
Total	114	136	26	12	59	347

Chemical Composition of Spike Oil (Literature Investigations)

	Prager 1979	Kuster 1979	Formacek 1982	de Pascual 1983
terpenes:				
α -thujene	-	0.00-0.17	-	-
d- α -pinene	1.8-6.8	1.69-4.23	0.89	4.21
d-camphene	0.5-2.1	0.36-1.78	0.32	0.23
Beta-pinene	1.2-3.4	1.75-3.56	0.01	0.84
sabinene	-	0.00-0.55	0.22	0.86
myrcene	-	0.39-0.67	-	0.15
α -phellandrene	-	0.02-0.12	+	-
α -terpinene	-	0.04-0.10	-	-
limonene	1.1-2.7	1.00-2.16	1.01	-
cis-ocimene	0.0-0.5	0.00-0.01	-	-
trans-ocimene	0.0-0.4	0.15-0.29	-	-
p-cymene	-	0.26-0.98	0.35	0.91
terpinolene	-	0.13-0.25	-	-
γ -terpinene	-	-	-	-
Beta-phellandrene	-	-	+	-
sub-total	4.6-15.9	5.69-14.87	2.80	7.20
sesquiterpenes:				
α -gurjunene	-	-	-	0.47
caryophyllene	+x	0.31-1.74	-	1.39
δ -cadinene	-	-	-	0.98
Beta-bisabolene	-	0.07-0.20	-	0.40
α -bisabolene	-	0.38-1.61	-	1.90
calemenenes	-	-	-	0.29
Beta-farnesene	-	-	-	0.03
γ -cadinene	-	-	-	0.80
α -humulene	-	0.00-0.46	-	-
sub-total	-	0.70-4.01	-	6.26
1,8-cineole	20.5-31.1	25.40-34.34	28.26	33.65
d-camphor	9.6-16.5	5.94-14.26	12.93	5.31
alcohols:				
linalool	29.8-46.4	37.21-43.75	47.85	26.34
terpinen-4-ol	0.2-2.0 ^x	0.19-0.35	0.67	-
lavandulol	0.2-0.6	0.15-1.50	-	-
α -terpineol	0.7-1.9	0.52-1.96	0.66	1.71
d-borneol	0.4-6.9	1.46-2.54	1.23	4.89
isoborneol	-	0.00-0.39	0.28	-
nerol	-	0.01-0.08	-	-
geraniol	-	0.03-0.19	0.16	0.20
citronellol	-	0.00-0.37	-	-
cis-carveol	-	0.00-0.05	-	0.35
trans-pinocarveol	-	-	-	0.37
p-cymen-8-ol	-	-	-	0.37
myrtenol	-	-	-	0.23
cuminic alcohol	-	-	-	0.23
δ -terpineol	-	-	-	1.02
n-hexanol	-	0.01-0.07	-	-
octanol-3	-	0.00-0.01	-	-
sub-total	31.3-57.8	39.58-51.26	50.85	35.71
ketones:				
6-methyl-hept-5-enone-2	-	0.02-0.07	-	-
octanone-3	-	0.04-0.07	-	-
α -thujone	-	0.02-0.08	-	-
Beta-thujone	-	0.02-0.05	-	-
carvone	-	0.12-0.50	-	-
sub-total	-	0.22-0.77	-	-
esters				
linalyl acetate	0.7-1.8	0.00-1.04	-	-
tr. epoxi-linalyl acetate	-	0.00-0.01	-	-
bornyl acetate	-	0.00-0.69	-	-
lavandulyl acetate	-	0.00-0.30	-	-
α -terpinyl acetate	-	0.00-0.32	-	-
neryl acetate	-	0.10-0.40	-	-
geranyl acetate	-	0.00-0.07	-	-
geranyl butyrate	-	0.03-0.10	-	-
hexyl tiglate	-	0.07-0.79	-	-
oxides:				
cis & tr. linalooloxide	-	0.19-0.48	-	0.53
caryophyllene oxide	-	0.06-0.22	-	1.04
eugenol	-	0.00-0.08	-	-
coumarin	-	0.00-0.20	-	9.04
herniarin	-	0.00-0.20	-	1.07