

Material Review

Woody Notes in Perfumery — Vetiver and Derivatives. Part I

Botanical origin, production, composition and fragrance compounds

by Danute Pajaujis Anonis, Chemist Perfumer

Vetiver is an important woody note in perfumery. The word vetiver, vetyver in French, originated in Tamil:* vettiveru = vetti, worthless + veru, useless.¹ The Indian term for vetiver is Khus, and in Indonesia is known as Akar Wangi.

Botanical origin: Vetiver, *Vetiveria zizanioides* Staph., is a grass growing wild, semi-wild or cultivated in many parts of the world, including India, Haiti, Java, Réunion Islands, China and Indonesia.

Mode of production, yield, type of oil: Vetiver oil is obtained by distillation of *Vetiveria zizanioides* roots. Steam distillation is a more recent method used, and it gives a better yield of the oil. The yield of the dried vetiver Java roots is from 1.5 to 2 percent, and that of vetiver Haiti varies from 1 to 1.5 percent.² A more recent source gives the yield of air-dried vetiver Java roots as 2 to 3 percent.³

The following types of vetiver oil are available commercially:

- . vetiver Java
- . vetiver Haiti
- . vetiver Réunion
- . vetiver China
- . vetiver Indonesian
- . vetiver redistilled
- . vetiver de-ironized
- . vetiver resinoid

Chemical Composition

The chemical composition of vetiver oil was studied in the beginning of the 20th century. Theulier studied the physico-chemical properties of fractionated vetiver oils distilled in France and on Réunion Island. Other investigations were carried out by various researchers, among them Semmler.⁴ In the 1950s, the following components of vetiver oil were identified:⁵

- **α - and β -Vetivone (vetiverone):** Java and Réunion oils were found to contain 7.5 to 35.1 percent of ketones, depending on the method used. The odor of vetiver is due chiefly to ketonic sesquiterpenes. Only two of them, listed above, have been isolated.
- **Vetivenols (vetiverols):** These alcohols were investigated by various researchers in the beginning of the 20th century, but the results were inconclusive. Only by the middle of the century did it become apparent that at least 60 percent of these sesquiterpene alcohols occurring in vetiver Java oil seemed to consist of a mixture of primary alcohols in which a tricyclid alcohol predominates, while bicyclic alcohols amounted only to 10 percent.
- **Vetivenyl vetivenate:** An ester that vetivenic acid forms with vetivenol and which readily hydrolyzes.
- **Palmitic acid.**
- **Benzoic acid:** In considerable amounts.
- **Vetivene:** A colorless and odorless sesquiterpene.

In 1976, a study of the carbonyls of Haitian vetiver oil was done. Seven novel sesquiterpenoid and norsesquiterpenoid carbonyls were characterized. The major isomer of dihydro β -vetivone had “a strong rich woody peppery odor,” while the remaining novel carbonyls were of a general woody type odor. The researchers believed that all of the carbonyls play a role in achieving the complex woody odor of vetiver oil.⁶

Among vetiver oil components characterized in the early 1970s were α - and β -vetivones, the norsesquiterpene khusimone and three biogenetically derived C-12 ketones, which were deemed to be the

*A language spoken by members of the Dravidians, an ethnic group from southeastern India and Ceylon.

most interesting. Spiroketone and khusimone appear to play an important role in the reconstruction of vetiver oil. Therefore, total new syntheses of these compounds were developed.⁷

In the study of vetiver oils of different geographical origins, other researchers reported 95 compounds in the hydrocarbon-rich section, fractionated via distillation of vetiver oil Haiti, and suggested that more than 150 components were present in the total vetiver oil.⁸

In 1977, a synthesis of β -vetivone via base-catalyzed spiroannulation of phenolic tosylates was reported.⁹ A 1978 patent covered a new synthesis of khusimone.¹⁰ 6,6,7-Trimethyl-tricyclo [5.2.2.0^{1,5}] undec-8-en-2-one was given as the starting material for this synthesis. This compound was described as having a woody, sandalwood-like odor with vetiver nuances, thus being of interest. In 1989, another synthesis of khusimone was reported, starting from (S)-6,6-dimethyl-5-methoxycarbonylmethyl-2-cyclohexen-1-one. Overall yield was 6.9 percent.¹¹

In 1996, an analysis of vetiver Haiti was reported that showed that the main constituents were isovalencenol, β -vetivone and khusimol. Because vetiver was a very complex oil, it is a difficult task to separate and elucidate the structure of its components.

Using flashchromatography, two olfactory important fractions were obtained: (1) medium polar (hydrocarbons, ethers and ketones), and (2) polar alcohols and α,β -unsaturated ketones. The combination of distillation and repeated FC yielded several new compounds.

In the medium polar fraction, one of the sesquiterpene hydrocarbons that never isolated before in vetiver oil was *cis*-eudesmadiene. Among other isolated ethers were α -agarofuran and 4,7-epoxy spirovetiva-1,11(12)-diene.

In the polar fraction, the transformation of secondary alcohols to methyl ethers; subsequent split tube distillation gave 30 new sesquiterpene ethers after repeated FC. Olfactory comparison of the odor of several methyl ethers and their corresponding alcohols showed that the odor of the methyl ethers was more earthy, khusimone-vetiver or patchouly-like, while the alcohols had woody odor tonalities with amber, sweet and grapefruit undertones.¹²

Synthetic Compounds

Vetiver has a lasting woody odor with a hint of camphoraceous, earthy and musty undertones. It has points in common with violet-orris and patchouli type odors, and it blends well with sandalwood and rose odors. Vetiver is especially effective with musky odors. Several of the modifiers of vetiver odor are listed in T-1.

Vetiver Imitations

Because of the complexity of vetiver oil it is difficult to reproduce the vetiver odor. Several early imitations were developed; some served in the past to fraud the natural oil. Here are several examples:

Odor characteristics of tridecan-x-ones (x = 2,3,4,5 and 6)

T-1

For lift and freshness	To add floralcy	To add sweetness	Folial/green	Spicy	Woody	Fixatives
Aldehyde C-10 Aldehyde C-11	Aurantiol Jasmine	Coumarin Labdanum resinoid	Hexenyl acetate Methyl octine carbonate	Isoeugenol	Cedarwood Sandalwood	Amyl salicylate Benzophenone
Aldehyde C-12 (MNA)	Lilial	Musk ketone	Phenylacetaldehyde dimethyl acetal			Dimethyl hydroquinone
Bergamot Cassie	Nerol Neroli	Styrax resinoid Tolu balsam				Diphenyl oxide Isobutyl salicylate
Citraldimethyl acetal	Rhodinol	Vanilla resinoid				Musks (various types)
Clary sage Geranonitrile Lavender Orange oil Thyme Ylang ylang	Rose Terpineol Ylang ylang	Vanillin				

Complex of Vetiver Oil No. 1¹³

vetiver oil	50.00
sandalwood	40.50
copaiba balsam	8.00
Rhodinol	0.50
Exaltone	0.01
	<hr/> 99.00

Vetiver Synthetic No. 2

vetiver bourbon	225
cedarwood	150
copaiba balsam	100
solvent	25
	<hr/> 500

Vetiver Synthetic No. 3

cedarwood	145.0
sandalwood	135.0
guaiacwood	75.0
hercolyn Atlas	70.0
vetiver Haiti	55.0
vetiver acetate	40.0
labdanum resinoid	20.0
aldehyde C-18	20.0
methyl ionone	15.0
tolu balsam	12.5
isobutyl quinoline	7.5
cade oil 10%	5.0
	<hr/> 600.0

More recent vetiver specialties approximate the odor of vetiver oil, but leave much to be desired in regard to lastingness and fixative power.

Traditional Fragrance Compounds

Let us take a look at a number of illustrative fragrance compounds containing vetiver or its derivatives.

Aldehydic Chypre¹⁴

clary sage	35
mousse de chene absolute	50
nutmeg	35
patchouli	50
vetiver	95
bergamot	125
orris concrete	25
orange oil	30
sandalwood	90
orange flower absolute	30
jasmin absolute	30
ionone	95
methylionone	90
labdanum absolute	40
coumarin	25
heliotropin	90
vanillin	20
musk ketone	50
aldehyde C-11 10%	75
aldehyde C-10 10%	75
aldehyde C-12 (MNA) 10%	50
	<hr/> 1205

Chypre I¹⁵

mousse de chene	50
bergamot	225
vetiver Bourbon	75
lavender	50

sandalwood EI	70
patchouli	10
cloves	35
jasmin synthetic	100
rose synthetic	80
isobutyl salicylate	70
cinnamic alcohol	50
heliotropin	100
coumarin	50
tonka resinarome	20
aldehyde C-12 (MNA) 10%	15
	<hr/> 1000

Cuir de Russie¹⁶

birch tar oil	60
castoreum 10%	150
rose otto	20
styrax resinoid	100
bergamot	100
sandalwood EI	10
patchouli	5
jasmin absolute	50
musk ambrette	50
musk ketone	80
Exaltolide 10%	20
vetiver acetate	100
tonka resinoid	30
vanilla resinoid	20
vanillin	50
labdanum resinoid	100
clary sage	5
oakmoss decolorized	10
tuberose absolute	7
acetophenone	3
cassie absolute	10
lemon oil	20
	<hr/> 1000

Foin Coupé (New Mown Hay)¹⁷

bergamot	90
coumarin	120
jasmin absolute	100
rose absolute	20
cassie absolute	10
oakmoss absolute	12
anisic aldehyde	18
linalool	20
lavender	36
clary sage	6
vetiveryl acetate	24
hydroxycitronellal	20
methyl anthranilate	6
civet infusion 3%	50
musk tonkin infusion 3%	200
ylang ylang	12
amyl salicylate	60
isobutyl phenylacetate	30
isobutyl benzoate	12
tuberose absolute	20
mimosa absolute	18
ionone a	20
methoxyacetophenone	30
aldehyde C-12 (MNA) 10%	6
sandalwood EI	60
	<hr/> 1000

Traditional illustrative perfume compounds presented in this article were developed before dermatological consideration took effect. Therefore, few components in these formulas would be acceptable today unless a) used in limited percentages or b) spe-

cially processed. In the first case, relevant materials include angelica, cinnamon, cinnamic alcohol, citrus oils and oakmoss, and in the second, bergamot and styrax resinoid. Other components have been completely eliminated, including musk ambrette. We shall discuss this subject matter in greater detail in the second part of this article.

Address correspondence to Danute Pajaujis Anonis, Chemist Perfumer, 98-41 64th Road, #6F, Rego Park, NY 11374-3408.

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Material review

Woody Notes in Perfumery

Vetiver, derivatives and aroma chemicals. Part II

Danute Pajaujis Anonis, Chemist Perfumer

A number of popular fragrances contain vetiver or its derivatives, including Chanel No. 5 (Chanel)



In the first part of this article (*Perfumer & Flavorist*, May 2004),* I discussed various vetiver types, the composition of vetiver oils of different origin, showed a few early vetiver imitations and illustrated a number of traditional perfume compounds. In this article, I shall exemplify additional types of traditional perfume compounds containing vetiver or its derivatives.

Traditional Fragrance Compounds

Fougère No. 6

140	geranium Bourbon
100	vetiver
100	lavender
50	amyl salicylate
50	coumarin
40	cananga
10	patchouly
10	solvent
500	

Lavender Water¹

500	lavender oil French
50	lavender absolute
150	bergamot
30	musk Tonkin infusion 3%
20	civet infusion 3%
10	neroli bigarade
20	orange flower absolute
20	petitgrain Grasse
30	orange oil sweet
50	vetiveryl acetate
5	Tonka resinoid
5	hydroxycitronellal
5	clary sage
5	estragon oil
5	cloves
10	styrax resinoid
20	benzoin resinoid
10	labdanum resinoid
30	vanilla extract 10%
15	methylionone
1000	

Mimosa No. 541²

300	ylang synthetic
375	bergamot synthetic
65	sandalwood E.I.

60	heliotropin
60	vetiver Réunion
50	orris resinoid
45	neroli synthetic
45	rose synthetic
1000	

Opoponax No. 3

200	bergamot
80	benzoin resinoid
50	vanillin
40	vetiver
32	coumarin
35	musk ambrette
25	lemon c.p.
16	castoreum resinoid
8	patchouly
486	

Origan Base No. 290³

200	Raldeine D Giv.
100	cassie synthetic No. 133
100	vetiver Java
80	carnation synthetic No. 117
45	rose synthetic No. 163
40	ylang Bourbon
70	jasmin synthetic No. 51
97	amyl salicylate
40	neroli synthetic No. 75
20	rhodinol
40	opoponax resinoid
23	musk ketone
10	musk ambrette
10	coumarin
50	heliotropin
65	vanillin
6	patchouly
2	aldehyde C-12 (L)
2	mousse de chêne absolute
1000	

*The heading of T-1 (p.32) was a printing error. It should be: "Modifiers of vetiver odor"

Peau d'Espagne⁴

4	birch tar oil rectified
45	castoreum infusion 5%
270	civet absolute 10%
55	sandalwood E.I.
70	bergamot
35	neroli bigarade
7	fleurs d'oranger absolute
55	rose oil Bulgare
50	musk ambrette
35	musk ketone
55	musk Tonkin
10	Tonka absolute
13	coumarin
2	patchouly
14	jasmin absolute
2	cassie absolute
45	tolu resinoid
25	styrax resinoid
20	ylang ylang oil
100	vetiverol
17	cloves
25	lemon c.p.
45	petitgrain Grasse
1000	

Rose V⁵

phenyl ethyl alcohol	425 cm ³
rhodinol	400 cm ³
jasmin synthetic	50 cm ³
nerol	25 cm ³
vetiver	15 cm ³
sandalwood	10 cm ³
neroli oil	10 cm ³
tuberose oil	10 cm ³
rose oil	10 cm ³

Vetiver Compound⁶

vetiver Java	140 g
vetiver Bourbon	200 g
sandalwood	100 g
hydroxycitronellal	120 g
cinnamic alcohol	60 g
jasmin synthetic	90 g
heliotropin	80 g
coumarin	50 g
musk ketone	50 g
musk ambrette	10 g

And a more recent vetiver compound is:

Vetiver Bouquet for Men

400	vetiver Bourbon
150	cedryl acetate
120	methyl ionone
120	Grisambrol 10% (Firmenich)

80	patchouly
30	sandalwood E.I.
20	oakmoss resinoid 50%
10	geraniol
20	musk ketone
950	

Dermatological Consideration

Most of the illustrated traditional perfume compounds were developed before the advent of dermatological considerations. Today, some components in these formulas would (1) be prohibited, (2) have to be

pecially processed or (3) be limited in percentage used, in accordance with IFRA's (International Fragrance Association) recommendations. For example, use of musk ambrette is prohibited.

In the second category are:

- **Citral**: can be used in conjunction with perfume materials preventing sensitization, such as pinene
- **Styrax**: only vacuum distillation or extraction with ethanol should be used
- **Opoponax**: only obtained by extraction of solvents or steam distillation

In the third category are:

- **Bergamot**: because of its phototoxicity, the level in consumer products used on skin exposed to sunshine should not exceed 0.4 percent
- **Cinnamic alcohol**: limited to 8 percent in consumer products
- **Hydroxycitronellal**: limited to 5 percent in fragrance compounds
- **Lemon oil c.p.**: limited to 2 percent in consumer products
- **Oakmoss absolute**: limited to 0.6 percent in consumer products

If older fragrances have to be adapted to today's dermatological requirements, future fragrances are facing even more stringent regulations.

Meschede and Duclos described the changing situations in current and future European legislation:

1. Essential oils and related products in Europe and shipped to Europe will be facing new restrictions. Apparently, the European Parliament will be making most regulating decisions in Brussels. 2. The new legislation requires, among other things, the labeling of 26 substances alleged to be allergens identified by the Scientific Committee for Cosmetics and Non-Food Products (SCCNFP) for all skin products. 3. The new legislation requires among other things a labeling of 26 substances alleged to be allergens identified by the Scientific Committee for Cosmetics and non-Food Products (SCCNFP) for all skin contact products. No differentiation as to the origin of these substances (coming from essential oils or synthetics) was made. The authors listed 59 essential oils illustrating their total percentage of allergens from 0-90. Even vetiver oil would be a "culprit," listed in the 0-5 percent category.⁷

Aroma Chemicals and Specialties

Let us take a look at different aroma chemicals with woody vetiver and multifaceted odors:

- Guaiyl acetate (guai-l(5)-en-ll-ol:acetate) (IFF): woody, vetiver, balsamic
- Isobutyliolone (Bedoukian): woody, vetiver
- α -Isomethyl ionol (Bedoukian): woody, vetiver
- Methyl cedryl ketone (Vertofix Coeur - IFF): woody, vetiver, leather with musky undertones
- Methyl tetrahydroionol acetate (Bedoukian):

vetiver, vetiver acetate-like

- Khusimone (7,7-dimethyl-6-methylene tricyclo [6,2.1.0^{1,5}] undecan-2-one): has been found to be mainly responsible for the characteristic odor of vetiver⁸
- 2-Heptanol, 3,4,5,6,6-pentamethyl (Kohinool - IFF): woody, ambery, vetiver
- Rootanol 100 (BASF): minty, earthy, reminiscent of vetiver roots
- 2,5,10-Trimethyl 1,2,5,9-cyclododecatrien-1-yl methyl ketone and isomers (Trimofix - IFF): amber, woody note with vetiver and tobacco nuances
- Vetynal extra (Givaudan): caryophyllene acetate main component, woody, used as extender of vetiver notes
- Vethymine (2,4-diethoxy-5-methylpyrimidine): earthy, dusty, woody and rooty with strong vetiver, patchouli and agarwood nuances⁹
- Vetyval, vetyvertone (4-cyclohexyl-2-methyl-2-pentanone)¹⁰
- Vetylbois (1,4 dimethoxy-2-terbutyl benzene): woody with vetiver and patchouli nuances¹¹

Vetiver oil and derivatives are valuable perfume materials used in many women's and men's fragrances and in various cosmetic and soap perfumes.

One patent covers isolongifolene esters.¹² These esters "have valuable perfumery properties, particularly as replacements for vetiverol derivatives."

The second patent describes a genus of substituted hexahydro acetoneaphthones having a structure containing a carbon-carbon double bond and carbon-carbon single bonds, possessing multifaceted aromas of sweet, woody, citrusy, vetiver-like, musky, leathery, peppery, hay and green fragrance notes.¹³ They are useful fragrance ingredients for perfumes, colognes, cosmetic powders, soaps and detergents.

An example of a hexahydro acetoneaphthone derivative used to impart a rich, green, woody note of vetiver is given:

Vetiver Fragrance

10 labdanum resinoid

- 50 hexahydro acetone derivative
- 25 cedrol
- 5 cedryl acetate
- 1 isobutyl quinoline
- 2 α -ionone
- 15 caryophyllene
- 2 eugenol

The above fragrance compound may be used in a cologne at a concentration of 2.5 percent in 85 percent aqueous alcohol, and into a handkerchief perfume at a concentration of 20 percent in 95 percent aqueous alcohol. Used in a powder detergent at a concentration of 0.7 percent, it will produce a vetiver aroma with deep green and woody notes.

A third patent describes cyclohexene-3-nitriles, which can be readily synthesized.¹⁴ These nitriles have a good stability in acid and basic conditions; they do not oxidize easily, can withstand higher temperatures, and are suitable in soap, detergents and personal care products.

In the preparation of mixed 3,5-dimethyl- and 2,4-dimethyl-3-cyclohexene nitriles, the odor of combined fractions eight to 18 had a strong green, cuminic note with herbal, cinnamic and woody

background. The 24-h dry-out odor was strong, warm, woody.

Application

Vetiver oil and derivatives are valuable perfume materials used in many women's and men's fragrances and in various cosmetic and soap perfumes. Vetiver root has been employed in the Orient since antiquity. The dried root is used to scent clothes lines, by itself or in a form of sachet.

Vetiver and its derivatives are natural fixatives and are also modifiers of musky odors. Vetiverol is especially suitable as a background note in floral bouquets. Vetiver was used in old-fashioned violet pomade as fixative.

Vetiver gives a covering power and persistence in talcum powder perfumes. Vetiver also finds applications in various types of potpourri, combined with spicy, floral, citrus or lavender notes. In addition, it is a component of perfumes used to impart a pleasant scent to cigar boxes.

It is interesting to note that vetiver is considered an insect repellent, probably due to khusimone, a minor but essential component of vetiver oil with insect repellent power.

Here is an illustrative list of a number of earlier and later fragrances containing vetiver or its derivatives: Arpège (Lanvin), Chanel No. 5 (Chanel), Femme (Ro-

chas), Miss Dior (Dior), Shalimar (Guerlain), Cravache (Piguet), L'O (Lancôme), Mystère de Rochas, Bales-tra (ICR), Anais Anais (Cacherel), Silences (Jacomo), Sheherazade (J. Deprez), Bois des Iles de Chanel (1920s, reappeared in 1993), Paradox (Jacomo), Tiffany (Tiffany & Co.), Paco Energy (Paco Rabanne), Must (Cartier), Eau de Vetiver (Guerlain), L'Eau Cheap et Chic (Moschino), Coriolan (Guerlain), Tiffany for Men Sport Cologne, and the 2004 fragrance Le Baiser du Dragon (Cartier).

Conclusion

Vetiver and its derivatives are likely to remain important woody note components in a broad spectrum of men's and women's fragrances in the foreseeable future. In spite of the pessimistic prognosis of G. and F. Robert, who include vetiver oil among the vanishing raw materials because of its popularity and huge world consumption of natural products, vetiver oil is still available and used in fragrances released in 2004.¹⁵ Vetiver will continue to remain an important ingredient because it is difficult to replace/substitute. The newer vetiver specialties approximate the initial vetiver odor, but leave much to be desired in comparison to vetiver oil's lasting fixative power.

Address correspondence to Danute Pajuajis Anonis, 98-41 64th Road, Rego Park, NY 11374.

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