Natural and Synthetic Musks in Perfumery

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The word "musk" derives probably from the Sanskrit muska (scrotum: from the scrotum-shaped musk bag), Persian mushk, Greek moshkos, late Latin muscus, and old French musc.

The first written record of the use of musk is found in a work compiled by Aetius, a court physician at Byzantine. Musk was probably introduced into Europe by the Arabs.[1]

Natural musk is still highly valued in Asia as folk medicine and as an aphrodisiac. In perfumery, natural musk has been used as a valuable fixative, as well as a modifier.

Of the various types of musks, Musk Tonkin, Musk Nankin, Musk Yunnan, Musk Bengal (less odorous), Musk of Boukharie, and Musk Siberian (having a secondary odor of castoreum—harsh and sour) may be mentioned.[2]

Origin

The deer, *Moschus* moschiferus L., producing Musk Tonkin, lives in the mountains of Tibet and China. The cabardin deers—*Moschus* Siberian and *Moschus* altaicus L.—live in Central Asian, Siberian, Himalayan and Altai mountains.[3]

Musk Tonkin, a brownish greasy secretion with a powerful odor, is produced in a glandular sac beneath the abdomen skin of a mature male deer. The secretion becomes solid granular with a brown-blackish color after drying. The dry glands are called "musk pods" and their contents are called "musk grain."[4]

Musk cabardin is a soft secretion of a reddish color, which becomes powdery in a dry state. Its odor is less refined with an acrid (burnt-leathery) background note.[3]

Chemical Composition

The composition of the pouch's contents varies considerably according to the age and general condition of the animal. It is agreed that the content of the musk pod (sac) serves a purely erogenous purpose.[4]

More recently, it was reported that the glands of the musk deer of India contain, in addition to civetone (9-cis-cycloheptadecen-1-one), the following:[5]

cyclopentadecanone
cyclohexadecanone
cyclohexadecenone
cycloheptadecanone
cycloheptadecenone
cyclooctadecanone
cyclooctadecenone
cyclononadecanone
cyclononadecenone

Quality and Production

The diluted genuine musk varies in odor, ranging from a sharply ammoniac odor to a sweetish-nutty or tart animal scent. Almost all musks were altered at the source, which was a common practice.[4,6]

The musk infusion is prepared the same way as the ambergris infusion, and the amounts used are also identical. Two examples of musk infusions are given by Fouquet:[7]

I	
Alcohol 96°	1 liter
Powdered musk	30 g
Potassium hydroxide 36°	30 g

II	
Alcohol 96°	1 liter
Musk pod cut in small pieces	100 g
Potassium hydroxide 36°	100 g

Statistics of the 1930s showed that pods were needed yearly to meet the demand of musk in the Orient and other parts of the world, about 2,400 kg of musk. Considering the average weight of 60 grams per pod, 40,000 musk deer had to be killed to meet the world demand for musk.[8] This growing demand gave impetus for the development of the first musk substitutes.

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Musk Substitutes

Natural musk, a valuable fixative of high-class fragrances, has been replaced by various aromatics and by other natural isolates.

Research in finding substitutes for natural musk was done in two directions:

1. The development of synthetic musks which are structurally not related to the natural musk, and
2. Isolation and subsequent syntheses of compounds with a musky odor occurring naturally in animal and/or plant sources.

Synthetic Musks, not Structurally Related to Natural Musk: Among various compounds in the first category are nitro musks, indan musks, tetralin musks, isochroman derivatives and coumarin derivatives, to mention the more important commercially available musk types.

The first synthetic nitro musk was musk xylol (2,4,6-trinitro-1,3-dimethyl-5-tertiary butyl benzene). Later, musk ketone (4-tertiary-butyl-3,5-dinitro-2,6-dimethyl acetophenone) and musk ambrette (2,4-dinitro-3-methyl-6-tertiary butyl anisole) were obtained.[9] In the third decade of this century, more stable nitro musks, Moskene M.D. (2,6-dinitro-3-tertiary butyl-4-isopropyl toluene) and Musk Tibetene (2,6-dinitro-3,4,5-trimethyl-1-tertiary butyl benzene) were introduced.[10,11]

Among indan musks, known for their stability, are Celestolide (P&S, now IFF), Phantolide (PFW) and Musk DTI (Firmenich). Tonalid (PFW) is among the first bicyclic musks gaining wide use.

Examples of tetralin and isochroman musks are Versalide* (Givaudan) and Galaxolide (IFF), respectively, the latter achieving commercial success.

Of the coumarin derivatives, Hibiscone (Van Dyck) may be mentioned.

Compounds with Musky Odor in Animal/Plant Sources: In the second category are macrocyclic musks. Already in 1906, Waldbaum had recognized muscone (3-methyl-1-cyclopentanone) as the principal odoriferous component of the Tonquin musk.[12] Later, the natural l-muscone was obtained by saponifying the ether extract from the musk glands with 10% alcoholic potassium hydroxide, and the neutral substances were fractionated by distillation. The isolation was conducted by crystallization of the semicarbazones and by hydrolysis with aqueous oxalic acid solution.[13]

Early Attempts to Synthesize Muscone

Because the production of natural muscone was expensive, various attempts were made at synthesizing muscone and discovering substitutes. This became possible after Ruzicka and collaborators established the structure of muscone. In the course of such research, it was confirmed that macrocyclic compounds with a 14- to 16-membered ring have a musk-type odor.

The early syntheses of muscone yielded a racemic product. A successful synthesis of the optically pure isomer was first achieved in 1951. Since then new methods have been explored and an extremely pure racemic muscone can now be obtained by the cyclization of 3-methyl thapsic acid diester, di-acid chloride or dinitrile.[14]

Ruzicka and coworkers established the synthesis of Exaltone (cyclopentadecanone) and Exaltolide (lactone of 14-hydroxytetradecancarboxylic acid).[15]

Macrocyclic ketones, muscone and cyclopentadecanone are of animal origin. Macrocyclic lactones, such as Ambrettolide (lactone of 16-hydroxy-1-hexadecanoic acid), Exaltolide, and Thibetolide (15-hydroxypentadecanoic acid lactone) are of plant origin, found in ambrette seed and angelica root, respectively.

Ambrettolide, a 17-membered ring macrocyclic lactone with a musk-type odor, was discovered and synthesized by Kerschbaum. The syntheses of both cis- and trans- forms of iso ambrettolide[16] have been achieved.

The macrocyclic musks, manufactured for over sixty years, remain important in perfumery, especially ketones and lactones. A large number of new syntheses have been reported.[17-19] They are based on the development of selective procedures, as well as the use of newly available starting materials. The switch from naturally occurring compounds to synthetic chemicals is significant, for example,

* This product is no longer available because of dermatological reasons.

Formula 1. Musk Base No. 270[23]

	Parts
Musk ambrette	200
Bergamot synthetic	150
Lavender synthetic	100
Oil orange sweet	75
Patchouly	25
Cassia	30
Coumarin	15
Geranium African	15
Oakmoss resinoid	5
Musk xylol	50
Musk ketone	50
Benzyl benzoate	285
	1000

Formula 2. Musk Extract No. 280[24]

	Parts
Red rose synthetic	280
Rose absolute	80
Jasmin liq. S.A.	100
Jasmin synthetic	60
Geranium sur roses Grasse	60
Bergamot	125
Patchouly	25
Tuberose synthetic	50
Tuberose absolute	25
Cassie synthetic	40
Cassie liq. S.A.	20
Product EMA H&C (Fixative base)	10
Benzoin Siam resinoid	40
Civet synthetic, 10%	35
Musk ketone	20
Musk ambrette	15
Exaltone, 10%	15
	1000
Ambergris infusion No. 4	100
Alcohol, 94-96%	8900
	10000

Formula 3. Musk for Extracts I[25]

	Parts
Musk ketone	70
Musk ambrette	20
Benzoin resinoid	20
Labdanum resinoid	100
Ambrette resinoid	20
Tolu resinoid	25
Sandalwood	80
Rose synthetic	280
Bergamot	255
Isobutyl salicylate	40
Heliotropin	90
	1000

Formula 4. Musk for Extracts II[26]

	Parts
Musk ketone	70
Amber synthetic	30
Musk ambrette	20
Sandalwood	80
Bergamot	100
Clary sage	50
Rose absolute	20
Red rose synthetic	260
Isobutyl salicylate	100
Heliotropin	90
Rhodinol	95
α-Ionone	85
	1000

Formula 5. Musk for Soap No. 622[27]

	Parts
Moskene L.G.	100
Musk xylol	50
Spike lavender Spanish	60
Cinnamic aldehyde	50
Clove bourbon	50
Patchouly	30
Bergamot synthetic	130
Cedarwood	100
Benzoin resinoid	80
Benzyl benzoate	350
	1000

Formula 6. Musk for Soap No. 624[28]

	Parts
Musk ambrette	100
Cinnamic aldehyde	30
Terpinyl acetate	180
Terpinolene	100
Terpineol	350
Clove bourbon	80
Cinnamic alcohol	90
Coumarin	40
Benzoin resinoid	30
	1000

cyclodecanone for the synthesis of pentadecanolide; β-methyl substituted α,ω-dicarboxylic acids and their derivatives for the synthesis of macrocyclic dilactones and the ketone d,l-muscone; aleuritic acid to obtain Ambrettolide; acetylenic compound resulting in β-hydroxy macrocyclic lactone; alkyl malonic acid to obtain alkyl macrocyclic ketones; thiophene for macrocyclic keto lactones.

Very few new macrocyclic musk compounds were developed, but among them is 5-cyclohexadecen-1-one, prepared from cyclodecanone or 1,2-divinylcyclododecan-1-ol. Of other types,[18] macrocyclic dilactones of a musk odor with a costus root oil note may be mentioned. Among oxalactones are 10-oxadecanolide (Oxalide, Takasago), 11-oxahexadecanolide (Musk R-1, originally Naarden) with a musk/ambrette seed absolute note, and 12-oxadecanolide (Hibiscolide, RBD).

A study of macrocyclic ketones disclosed that unsaturated macrocyclic ketones have the most intense odors. In general, all unsaturated macrocycles have the lowest threshold values of the compounds investigated, and have "pronounced erogeneous, animal" odors. Of all ketones tested, the most interesting from the point of view of odor are cyclopentadecanone, muscone, cyclohexadec-4-en-1-one, and two previously unknown ketones identified as cyclopentadec-8-en-1-one and 3-methylcyclohexadec-5-en-1-one.[20]

Synthetic Compounds

Musk has distinctive common points with skatole-civet and amber odors and, to a lesser extent, with mushroom and woody odors.[21]

Various types of synthetic musk compounds have been developed for use:

- in fragrances, toilet waters and colognes
- in soaps and powders
- as modifiers in other fragrance types
- as fixatives

Cerbelaud cites the following perfume materials for the development of synthetic musk compounds:[22]

Base Components:

exaltolide
exaltone
muscone
ambrettolide
civettone
undecyl γ-butyrolactone
isobutyl phenyl acetate
labdanum fractions
geranyl methoxy acetate
patchouly
patchoulol
ambergris infusion
sandalwood

For Nuances and Sweetness:

rose oil
rhodinol
geranium
benzophenone
diphenyl oxide
bromelia (ethyl-2-naphthyl ether)

Formula 7. Musk with a Rose Background for Powder No. 5[30]

	Parts
Musk ketone	50
Rosacetol Givaudan (trichlor methyl-phenyl carbinyl acetate)	33
Rose otto	4
Nerol	1
Magnesium carbonate	9
	97

The rose oil is triturated with magnesium carbonate separately, and the mixture is then added to the other previously mixed components.

Formula 8. Musk with a Vetiver Note for Powder No. 6[31]

Moskene Givaudan	50.00%
Exaltone Naef	0.10
Vetiver or vetiverol	20.00
Patchouly	2.00
Coumarin	5.00
Bergamot	10.00
Oakmoss liquid partially decolorized	0.25
Kaolin	10.00
Magnesium carbonate	2.65
	100.00

The rose oil is triturated with magnesium carbonate and kaolin, and then added to the other previously mixed ingredients.

Formula 9. Musk Adouci (Sweetened) No. 1[32]

Cetone D (methyl β-naphthyl ketone)	25.00%
Musk xylol	75.00
	100.00

Formula 10. Musk with a Foliage Note No. 2[33]

Musk ambrette	71.50%
Phenyl acetic acid	14.25
Coumarin	14.25
	100.00

Formula 11. Musk Synthetic No. 9

	Parts
Ambreine	20.00
Peru balsam	30.00
Civet concrete	2.50
Castoreum	2.50
Astrotone	15.00
	70.00

yara yara (methyl β-naphthyl ether)
cetone D (methyl β-naphthyl ketone)
vanillin

Among other components used are bergamot and orange oils, and linalool for the top note; musk ambrette, musk ketone, musk xylol, coumarin and ethyl vanillin as sweeteners; oils of cassia, clary sage, geranium, tuberose absolute, geranyl acetate, dimethyl hydroquinone and heliotropin for nuances; benzoin, oakmoss and olibanum and Tolu resinoids as fixatives. In musk compounds for soap, a spicy note (such as cinnamic alcohol, cinnamic aldehyde or clove oil) may be included.

Earlier conventional musk perfume formulas are given here as examples (Formulas 1-4). Examples of earlier soap perfumer formulas are also given (Formulas 5 and 6). The soaps were usually colored light or dark brown. A more complex musk perfume type for soap was built on a base of synthetic musk and castoreum, rounded out with cedarwood, cassia, geranium, ginger and sandalwood oils, terpineol and benzyl salicylate. For nuances, tetramethyl quinoline, isobutyl quinoline, p-cresol, p-cresyl phenyl acetate, methyl cinnamate, alcohol C-12(L), amyl salicylate, phenyl propyl aldehyde and civet resinoid were added. Labdanum resinoid, cinnamon, clove resinoid, vetiver, Peru and Tolu balsams were used as fixatives. The soap was color brown.[29]

A few conventional musk perfume formulas for powder are also given (Formulas 7 and 8). Earlier synthetic musks serving as fixatives and/or modifiers were based on nitro musks. Formulas 9 and 10 illustrate various approaches.

Other types of musk compounds were built upon a basis of 50% of phenyl acetic acid and various proportions of the three nitro musks, to which the cresyl phenyl acetates and valerianates were added in order to approximate the animal notes of the musk Yunnan or Boukharie. Complemented with labdanum, they were known as Musk Ambré, Musk Oriental, etc.[34]

Later, synthetic musk compounds, serving as fixatives, were developed using labdanum fractions, as well as newer types of macrocyclic musks. Formula 11 may serve as an example.

Macrocyclic musks are of higher odor quality than other synthetic musks which are structurally not related to natural musk. But the latter, being less expensive, have found a wider use in various types of fragrances. While the majority of them have musk-type odors, some possess dual odor characteristics. Cashmeran (IFF), with its floral-musky note, is an example.

Incompatibility

The incompatibility of nitro musks towards alkali can be overcome by dissolving one part of nitro musk in five or six parts of benzyl salicylate. Another incompatibility is with benzaldehyde and sulphuric odors, which cancel the musk odor. Natural or synthetic musks are not recommended in lily of the valley, hyacinth, lilac and violet compounds, especially the latter because ionones and musk odors cancel each other. Musk should also not be used in hair preparations containing camphor or sulphur.[35]

Macrocyclic lactones are stable when exposed to air, but as inner esters "they cannot tolerate excessively alkaline conditions."[36]

From the dermatological point of view, some perfume materials used previously in synthetic musk compounds are presently restricted. They are listed in Table I. Phenyl acetic acid and solvents, such as benzyl benzoate and diethyl phthalate, are no longer used because of dermatological reasons.

Application

Natural musk was a valued material both for its odor and its lasting quality.

It is apparent from the Venetian records of the 13th century that Marco Polo not only brought a sample of musk back with him from Asia, but also dealt in the commodity at the price of about $110.00 for a pound and a half.

Marco Polo also observed that the women of a province north of the Hindu Kush mountains used musk to scent their voluminous silk and linen garments.[37] It is also known

Table I. Restricted musk components

Components	Specially processed	Limited percentage	Used with quenchers
bergamot	*		
cassia		1	
cinnamic alcohol		4	
cinnamic aldehyde			*
cinnamon bark		1	
musk ambrette		4	
Peru balsam	*		
oakmoss		3	

that musk was mixed with mortar to build mosques in the 9th century.[38]

The Chinese used musk to keep moths away from furs and clothing. Musk was also used as medicine, particularly as a cardiac stimulant, for nervous afflictions, asthma, snake bites and against Asiatic cholera. In 16th century Tibet, musk was among the most important medicines.[39] During the same century, musk lozenges called Muscadin were used in Europe. In our times, an experiment with the effect of musk on rats suggested that possibly the musk glands exert a hormonal inhibitory influence on cancer growth.[40]

In fragrances of the Victorian Age, a musk perfume consisted of four parts of musk tincture, two parts of ambergris and one part of rose extract.

Later, natural musk infusions found application in various types of perfume compounds, among them:[41]

Amber	5-10%	of musk infusion 3%
Cyclamen	5-8%	"
Eau de Cologne	3%	"
Fougère	2-5%	of musk tincture 10%
Heliotrope	10%	"
New Mown Hay	10-20%	of musk infusion 3%
Cuir de Russie (Russian Leather)	10-19%	"
Magnolia	4.5-7.5%	of musk tincture 10%
Orchid	10%	of musk infusion 3%
Poppy	2-9%	"

Musk Tonquin infusion was found to be suitable in hair-water and powder perfumes, but its use was not recommended in lipstick, skin oil, brilliantine and hair oil fragrances.[42]

Special fragrances were developed for furs, based on musk tincture, orris, ambrette seed and vanilla tinctures or infusions. They also contained tuberose and orange flower absolute, rose Otto, vetiver, sandalwood, small amounts of clary sage, complemented by a small percentage of a chypre compound.[43]

When natural musk became scarce, it was considered that 1 kg of natural musk infusion No. 1 could be replaced in many instances with 4.5 g of Exaltone 10% (Firmenich) and that 2 g of Exaltone 10% should be equal to 1 kg of musk infusion No. 2.[44] Later, Astrotone (Rhodia) was widely used

in perfumery because of its advantageous price.

Nitro musks were used as fixatives and sweeteners in various types of fragrances, and over 10% could be used even in more delicate perfume compounds. In contrast, macrocyclic musks were used in a much smaller percentage, especially in delicate floral types (0.01% or less).

Musk-type perfume compounds found application in fine fragrances and lotions, the latter containing 1.5 to 3% of perfume oil.

Musk compounds were also used as soap perfumes and as fixatives of other types of soap perfumes, among them:[45]

Lavender (colored white, moss green)
Leather (colored brown)
Lily Milk (white)
Sandalwood (colored brown)

Synthetic musk compounds remain of value in soap perfumes of today.

Musk fragrances became popular again in the 1970s with the revival of animal odors. Musk Jovan, Caswell Massey and others may serve as examples. In some instances, listerine was used as a main component (up to 50%). A musk fragrance was used in crystal clear essence sticks (H. Rubinstein).

The popularity of musk fragrances extended also to the men's line, as attested by Musk for Men (Jovan).

Later, the musk-type fragrance trend abated, but the use of synthetic musk in various types of feminine and masculine fragrances increased.

Oriental fragrances contained both musk and ambergris. Among other fragrance types including musk are Audace, Azzaro, Balestra, Boss, Calèche, Charlie, Cravache, Fidgi, Halston Night, Insolent, Jardanel, Jazz, K de Krizia, Kouros, Loulou, Must, Norell, O', Opium, Weil de Weil and Youth Dew.

In the past, musk was used as a fixative and enhancer at 3 to 5% in fragrance compounds. This percentage has gradually increased to over 10%, and in some fragrances of the 1990s, musk is becoming the main component. Among the latter are[46] Parfum Sacre (Caron) and Trésor (Lancôme), containing 20% and 40% of synthetic musks, respectively.

In some fragrances, macrocyclic musk is used in combination with other synthetic musks.

Because of their stability and lower prices, tetraline, indan and isochroman type musks are used in larger percentages in household and cosmetic perfume compounds. In some instances,[46] up to 40% in fabric softener, and over 30% in some soap and shampoo fragrances.

In the men's line, musk is an established component, as exemplified by Habit Rouge (Guerlain), Halston Limited, Grey Flannel, Calvin for Men, Oleg Cassini for Men, Fahrenheit and other men's fragrances.

Conclusion

Musk has always been an important component of fragrances, enriching them and contributing fixation and diffusion to them.

The natural musk has been replaced by synthetically prepared isolates and other diverse, less expensive aromatic chemicals. It has been a long way from the first synthetic nitro musks at the beginning of this century to the macrocyclic musks and various categories of newer synthetic musks.

Today, there is hardly a fragrance—be it feminine or masculine, cosmetic or household—which would not include macrocyclic or other synthetic musks.

Because of the greater stability and lower prices of the new types of synthetic musks, they are being used more and more boldly in fragrances.

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Suggested Reading

PM Muller and D Lamparsky, *Perfumes: Art, Science and Technology,* London-New York: Elsevier Applied Sciences (1991).

It contains a review of musk chemistry in general, covering the work of well-known researchers, including structure-odor relationships of so-called meta- and ortho musks (Ohloff, Winter and Fehr, Chapter 9); the production aspects of known musk chemicals (Dorsky, Chapter 14); and new achievements in macro- and polycyclic musks (Frater and Lamparsky, Chapter 20).