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Analysis of 7 synthetic musks in cream by supported liquid extraction and solid phase extraction followed by GC–MS/MS



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ABSTRACT

A new method for the simultaneous determination of 7 synthetic musks (musk amberette, musk tibetene, musk moskene, musk ketone, musk xylene, phantolide, and tonalide) in cream by means of supporting liquid extraction (SLE) coupled with LC–Alumina–N SPE, then followed by GC–MS/MS has been established. In this study, 7 synthetic musks are extracted and pre-purified by a mixture solution of water and isopropanol from cream, and separated and purified by tandem columns containing SLE column and LC–Alumina–N SPE column, which were seldom reported before. Ultrasonic and mechanical shaking were applied to improve the extraction efficiency. Different experiment conditions, such as the type of extraction solution, extraction time of ultrasonic and mechanical shaking, the type of SLE and SPE column, and matrix effects were optimized and the recoveries of 7 synthetic musks for each part were above 86.61%. In addition, the use of isotope internal standards was systemically discussed. The method showed satisfactory linearity over the range assayed (5–1000 ng g⁻¹), and the limits of detections (LODs) ranged from 0.15 to 4.86 ng g⁻¹, and the limits of quantifications (LOQs) were ranging from 0.49 to 16.21 ng g⁻¹. The recoveries using this method at three spiked concentration levels (10, 100, and 1000 ng g⁻¹) range from 85.6% to 109%. The relative standard deviation was lower than 9.8% in all case. The proposed analytical method has been successfully applied for the analysis of 7 synthetic musks in commercial cream.

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1. Introduction

Large quantities of synthetic musks are manufactured due to exhibiting a strong, warm, sensual and long-lasting odor, and used in a wide variety of cosmetic products, such as perfumes, skin cream, deodorants, and soaps [1,2]. There are three groups of synthetic musks according to their chemical structure: polycyclic musks (tonalide and phantolide), nitroaromatic musks (musk amberette, musk tibetene, musk moskene, musk ketone and musk xylene) and macrocyclic musk [2]. However two groups of synthetic musks, polycyclic musks and nitroaromatic musks, have been widely applied in cosmetic formulations and then aroused public attention for their potential risks to human health and environment.

In recent years, synthetic musks have been described as a new group of bioaccumulative and persistent xenobiotics [2–5]. It was found that synthetic musks could lead to different types of dermatitis, carcinogenesis and endocrine disorder [3,6–11]. In 1999, Environment Canada issued the “Environmental Protection Act”,

which was clearly required to reduce the use of synthetic musks [12]. In Europe, the Regulation (EC) no. 1223/2009 established the rules as follows: musk amberette, musk tibetene and musk moskene were prohibited and the concentrations of musk ketone, musk xylene, phantolide and tonalide were limited in cosmetics [13]. China and other countries have issued maximum residue limits (MRLs) for synthetic musks based on the Regulation (EC) no. 1223/2009 in cosmetics. In most of the laws and regulations, 7 synthetic musks (musk amberette, musk tibetene, musk moskene, musk ketone, musk xylene, phantolide, and tonalide) were frequently prohibited or limited in cosmetics for their high toxicity and sensitization. In order to guarantee product safety according to regulations, the development of analytical methods for the determination of synthetic musks in cosmetic is mandatory.

Most of the existing analytical methods for synthetic musks were mainly used to analyze environmental samples, such as water [14,15], sewage [16,17], sludge [16], sediment [18], and air [19]. At present, the analytical methods of synthetic musks in perfume and emulsion, since their matrixes are relatively simple, have been developed [20,21]. These methods available for the identification and quantification of musk compounds comprise a sample preparation step, including traditional liquid–liquid extraction with organic solvents (LLE) [22,23], solid phase extraction

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(SPE) [3,24], solid phase microextraction (SPME) [4,20], liquid phase microextraction (LPME) [25,26], stir bar sorptive extraction (SBSE) [27], ultrasound assisted phacoemulsification liquid phase microextraction (USAEME) [28], and molecular imprinted polymers (MIPs) reported recently [29], followed by gas chromatography–mass spectrometry (GC–MS) [3,18,19,20,27], gas chromatography–tandem mass spectrometry (GC–MS/MS) [18], liquid chromatography–mass spectrometry (LC–MS) [30], liquid chromatography–tandem mass spectrometry (LC–MS/MS) [24]. Cream is one of the most widely used cosmetics, which contains not only hydrophilic compounds like alkali, moisturizing, nutritional agent, but also a great quantity of lipophilic compounds, such as paraffin stearic acid, cetyl, and stearyl alcohol [31]. Since synthetic musks are low polarity and lipophilic compounds, it is difficult to completely separate them from the above mentioned lipophilic compounds. The interferent, which could cause the alteration of ionization efficiency in the determination of GC–MS/MS, may lead to serious matrix effects [32,33]. Due to the low selectivity of the extraction techniques, extracts from complex samples like cream have to be subjected to cleanup steps, such as by solid-phase extraction (SPE) [34,35], liquid–liquid extraction with SPME [36], and matrix solid phase dispersion method (MSPD) [37].

The aim of this study is to develop a method based on supported liquid extraction (SLE) and SPE to simultaneously determine 7 synthetics musks (musk amberette, musk tibetene, musk moskene, musk ketone, musk xylene, phantolide, and tonalide) in cream by GC–MS/MS. In this work, water was introduced in the mixed extraction solution to separate synthetic musks from hydrophilic compounds, and the isopropanol in the mixed extraction solution was used to extract synthetic musks from lipophilic compounds. In addition, SLE column and SPE column were selected to adsorb the water and separate the interferent, which could improve the purification effect and reduce the matrix effects. Tandem MS has been selected to enhance selectivity and decrease LODs of the method. The applicability of the method to real samples was tested by 28 cream products, and found that the established method is suitable for routine analysis of 7 synthetics musks in real cream.

2. Experimental

2.1. Apparatus and reagents

The instruments used in the experiment include: a Trace GC Ultra coupled to a TSQ Quantum XLS tandem mass spectrometer (Thermo Fisher Co.), UMX5 high precision electronic balance

(Max=5.1 g, $d=0.1 \mu\text{g}$, METTLER TOLEDO Co.), XS205 electronic balance (Max=81 g, $d=0.01 \text{ mg}$, METTLER TOLEDO Co.), KQ3200E ultrasonic cleaners (Kunming ultrasonic instrument Co., Ltd.), KS 260 Basic concussion instrument (IKA Co.), Vortex-Genie2T vortex mixer (Scientific Industries Co.), nitrogen evaporator (V-EVAPTM111, Organomation Associate, Inc.) and Gradient A10 water purification System (Milli-Q Co. USA). The SPE columns used in the experiment were Supelclean™ LC-Alumina-N SPE Columns (6 mL and 2 g, Supelco, USA), Supelclean™ LC-Florisil SPE Columns (6 mL and 1 g, Supelco, USA), Supelclean™ LC-Si SPE Columns (6 mL and 1 g, Supelco, USA) and ISOLUTE SLE+ Columns (2 mL/15 mL, 5 mL/25 mL, sorbent mass/reservoir volume, Biotage, Sweden).

The studied compounds, their chemical names, CAS numbers, and purity are summarized in Table 1. Cyclohexane (99.98%), acetone (99.8%), hexanes (99.9%), dichloromethane (99.9%), isopropanol (99.9%), isooctane (99.9%) were provided by Fisher Scientific Inc. (Geel, Belgium, UK). Toluene (99.9%) and acetonitrile (99.9%) were respectively purchased from Duksan Pure Chemicals Inc. (Ansan, Kyungkido, KOREA) and Merck KGaA Inc. (Darmstadt, Germany). All above reagents were used without further purification.

The special precautions are required throughout the analytical procedure due to the widespread use of the synthetic musks in many consumer products. All the containers used in the test need to be rinsed by alcohol and acetone 3 times, respectively, before the experiment. Moreover, musk-free gloves were used and the samples were prepared in a fume hood.

Cosmetic samples from national and international brands were purchased from local markets in Beijing. Sealed samples in their original containers were stored at room temperature. The blank samples used in the experiments were found by the method of MSPD [37] from several international brand cream products. The concentrations of 7 synthetic musks (musk amberette, musk tibetene, musk moskene, musk ketone, musk xylene, phantolide, and tonalide) in this blank sample were below the instrumental detection limits (IDLs).

2.2. Extraction and cleanup

0.5 g cream was exactly weighted and placed into a 15 mL PTFE centrifuge tube, then $150 \mu\text{L } 2 \mu\text{g g}^{-1}$ mixed internal standard solution (D15-Musk Xylene and D3-Tonalide) in isooctane was added as a quality control for the entire procedure. 8 mL extraction solution (water: isopropanol=1:2, v/v) was added to the above sample to extract the target compounds by ultrasonic extraction 5 min, shaking extraction 5 min, and then centrifugation for 8000 rpm for 15 min. A 4 mL upper layer extraction solution was

Table 1

Target compounds: chemical names, purity, suppliers, CAS number, retention times and MS conditions.

Abbreviation	Chemical names	Purity	CAS	Retention times (min)	Parent mass (m/z)	Product mass (m/z)	Collision energy (V)	Scan time (s)	Scan width (m/z)
AHMI	Phantolide	10 ng μL^{-1c}	15323-35-0	14.18	229	131,173, 187^a	9	0.25	0.005
MA	Musk amberette	99% ^b	83-66-9	15.94	253	106,120, 223^a	5	0.22	0.005
MX	Musk xylene	99.5% ^b	81-15-2	16.61	282	190,248, 265^a	8	0.15	0.005
AHTN	Tonalide	99% ^f	21145-77-7	16.67	243	159, 187^a ,201	9	0.15	0.005
MM	Musk moskene	10 ng μL^{-1c}	116-66-5	17.22	263	187,216, 221^a	7	0.30	0.005
MT	Musk tibetene	10 ng μL^{-1c}	145-39-1	18.50	251	146,160, 234^a	8	0.35	0.005
MK	Musk ketone	98% ^b	81-14-1	19.67	279	191^a ,247,262	11	0.35	0.005
D15-MX	D15-Musk xylene	100 ng μL^{-1d}	877119-10-3	16.26	294	248,258, 276^a	8	0.12	0.005
D3-AHTN	D3-Tonalide	100 ng μL^{-1e}	–	16.61	246	160, 190^a ,204	9	0.10	0.005

^a Bold figures is quantitative mass.

^b Dr. Ehrenstorfer GmbH (Ausburg, Germany).

^c 10 ng μL^{-1} in cyclohexane from Dr. Ehrenstorfer GmbH (Ausburg, Germany).

^d 100 ng μL^{-1} in acetone from Dr. Ehrenstorfer GmbH (Ausburg, Germany).

^e 10 ng μL^{-1} in isooctane from Dr. Ehrenstorfer GmbH (Ausburg, Germany).

^f Shanghai East's Flavors and Fragrances Co., Ltd.

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