



# Determinations of airborne synthetic musks by polyurethane foam coupled with triple quadrupole gas chromatography tandem mass spectrometer



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## ABSTRACT

Synthetic musk is widely used in various scented consumer products. However, the exposure via inhalation is often ignored due to pleasant smells. In addition, the information regarding the distribution of synthetic musk in air is limited. Hence, this research is aimed to develop a highly sensitive and widely applicable method for the determination of airborne synthetic musk. In this study, polyurethane foam (PUF) and filter were employed for active air sampling. Microwave assisted extraction (MAE) and nitrogen evaporator were performed for sample preparation. A gas chromatography coupled with triple quadrupole tandem mass spectrometer (GC/MS–MS) with specific multiple reaction monitoring (MRM) transition pairs was applied for sample analysis. Compared with using selected ion monitoring (SIM) mode traditionally, the sensitivities were improved in this study about an order at least. In terms of air concentration, as low as  $0.48 \text{ ng m}^{-3}$  can be determined when sampling at  $3.5 \text{ L min}^{-1}$  for 8 h. The method established was further applied to the analysis of synthetic musk compounds in air samples collected in a cosmetics plant. The results showed that the airborne concentrations of gaseous polycyclic musk, gaseous nitro-musk, and particle-phase polycyclic musk were  $6.4 \times 10^2$ ,  $4.0 \times 10^1$  and  $3.1 \times 10^2 \text{ ng m}^{-3}$ , respectively. Meanwhile, Cashmeran, Celstolide, Galaxolide, and Tonalide were found as the dominant musk compounds in the factory investigated.

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## 1. Introduction

Synthetic musk, with relatively lower cost regarding to natural musk, creates a variety of scent. Hence, they have been widely used in many consumer products, such as detergents, air fresheners, and laundry products. Besides, pharmaceutical and personal care products (PPCPs), including cosmetics, shampoos, lotions, and deodorants, all contain different types of synthetic musk.

According to the structures of the compounds, synthetic musk can be divided into different groups, including nitromusk, polycyclic musk, macrocyclic musk, and alicyclic musk [1–4]. Being widely used in the past, nitromusk and polycyclic musk are the two mainly discussed types of synthetic musk in the literatures. Nitromusk constitutes a carbon benzene ring with 2–3 nitro groups. With the health concern and bio-accumulative potential, the usage of nitromusk has been reduced [5]. Instead, Galaxolide (HCHB) and

Tonalide (AHTN), which belong to polycyclic musk, have become most popular thereafter. And, the USEPA has listed Galaxolide as one of the high production volume (HPV) chemicals [6].

As for the adverse health effects, there were studies indicating that synthetic musks might induce asthma as well as act as endocrine disruptors [7–10]. Nitromusk and two of the polycyclic musks, i.e., Tonalide and Galaxolide, have been filed in the Hazardous Substances Data Bank (HSDB<sup>®</sup>) on the National Library of Medicine's (NLM) Toxicology Data Network (TOXNET<sup>®</sup> in the United States) [11].

Studies reported elsewhere have observed that synthetic musk could be detected in consumer products as well as in effluents and sewage [12–21]. The exposure routes include oral intake, dermal exposure, and inhalation. The most discussed way of exposure is through skin contact since the musk-containing products are usually in liquid forms. Besides, pathway of inhalation might be critical because some investigations showed that airborne synthetic musk was ubiquitous over a wide variety of public places, occupational settings, and homes [22–27]. However, due to the lack of sufficient and effective sampling and analysis methods, still very few research has focused on the distributions of airborne synthetic musk [2].

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Solid sorbents, such as PUF, Tenax TA, and XAD, were usually performed for the sampling of synthetic musk in the air. However, various volatile and semi-volatile organic compounds other than synthetic musks can also be adsorbed simultaneously. Hence, the determination is challenging, since the capacity and selectivity of the method might cause concerns.

Gas chromatography/mass spectrometers (GC/MS) or tandem mass spectrometers (MS–MS) with selected ion monitoring (SIM) mode have been applied to analyze synthetic musk in the air. Nevertheless, further applications of specific multiple reaction monitoring (MRM) transition pairs, which will be able to achieve more precise analysis, are still limited elsewhere. Hence, this research aimed to develop a highly selective and sensitive method for the determinations of low-level airborne synthetic musk by using GC/MS–MS with MRM mode.

## 2. Materials and methods

### 2.1. Experimental

#### 2.1.1. Reagents and chemicals

Cyclohexane, acetone, and other solvents with analytical grade were provided by Merck (Taipei, Taiwan). Brand new pre-cleaned-and-ready-to-use polyurethane foam (PUF) plug coupled with glass fiber filter sampling sets (PUF/Glass Fiber Filter, 22 × 100 mm size, 1-section, 76 mm sorbent; SKC 226–126) were from SKC Inc. (USA). GilAir™ Plus (2000 cm<sup>3</sup>–5000 cm<sup>3</sup> min<sup>-1</sup>) and Gillian 3500 (700 cm<sup>3</sup>–3500 cm<sup>3</sup> min<sup>-1</sup>) pumps (Sensidyne, USA) connected with PUF samplers were performed for the method validation and field sampling.

All synthetic musk standards, including musk ambrette (MA), musk ketone (MK), musk moskene (MM), and musk xylene (MX) with 10 µg mL<sup>-1</sup> in cyclohexane as well as Cashmeran (DPMI), Celestolide (ADBI), Galaxolide (HHCB), Phantolide (AHMI), Tonalide (AHTN), and Traseolide (ATII) with 100 µg mL<sup>-1</sup> in cyclohexane were purchased from LGC Standards (London, UK). Table 1 illustrates the physical and chemical properties of synthetic musk studied in this research.

All of the glassware used in the experiment was carefully cleaned with non-scented neutral detergent, rinsed with deionized water and acetone followed by drying in the hood.

#### 2.1.2. Instrumental

The chemical analysis was performed by Agilent 7000B Gas chromatography coupled with triple quadrupole tandem mass spectrometer. The system was operated by the MassHunter software with Wiley and NIST library search.

The 10 synthetic musks studied in this research were analyzed by the following conditions: Column DB-5MS (J & W Scientific, Folsom, CA. 30 m × 0.25 µm id., crosslinked 5% phenyl methyl silicone, 0.25 mm film thickness), splitless mode 2 µL, and injection port temperature of 250 °C. Separation with helium at 1 mL min<sup>-1</sup> was processed by the temperature program set up from 70 to 280 °C. In details, the GC oven was initiated at 70 °C (hold 0.5 min) to 200 °C at 30 °C min<sup>-1</sup> and lifted to 225 °C at 3 °C min<sup>-1</sup> followed by 280 °C at 40 °C min<sup>-1</sup> (hold 1 min). The ion source was with EI mode (70 eV). The mass range was between 50 and 300 amu, and the scan time: 1.6 scans s<sup>-1</sup>. Total analysis time was 15.54 min.

The specialized mode of Agilent 7000B is Multiple Reaction Monitoring (MRM). The basic first stage of instrumental condition was the same with the set up of GC/MS. The difference of the MRM setup was the collision energy. Table 2 shows the collision energy and the two MRM pairs of each synthetic musk compound.

#### 2.1.3. Validation of air sampling method

In this study, active low volume PUF with filter sampling system were applied, according to US EPA Method TO-10A for semi-volatile organic compound [28,29]. Active sampling offers a few benefits of time saving and pumping known volume, compared to passive sampling. The laboratory validation of the method was performed with the guideline of the US EPA Method TO-10A as well. Briefly, by placing 1 mL of 2 µg mL<sup>-1</sup> 10-synthetic-musk-mixture standard solution into an impinger, the sampling efficiency (SE) was determined by two tandem PUF samplers assembled to the impinger with pumping into highly-purified nitrogen at the flow rate of 3.5 L min<sup>-1</sup>. The entire validation was processed in a cleaned hood filled with filtered air (without particles and VOCs), and the sampling time was 8 h. In addition, the experiments were performed in triplicates to estimate the uncertainties of collection efficiencies.

All samples were extracted by microwave-assisted extraction (MAE). The PUFs and glass fiber filters after sampling were placed separately into two GreenChem™ Extraction vessels. 60 mL of 1:1 cyclohexane: acetone (v/v) were required in order to cover the PUF plug (10 cm long) in each vessel and then extracted for 60 min at 85 °C of the Microwave-Accelerated Reaction System, Model MARS-X (CEM Corporation, USA). Compared to traditional extraction methods for synthetic musk, such as Soxhlet extraction (SE), simultaneous distillation-solvent extraction (SDSE), and ultrasound probe (UP), microwave-assisted extraction is effective, efficient, and environmental friendly of less solvent consumption [30,31].

After MAE, nitrogen evaporator with gentle stream was followed, and the extractions were first concentrated to 0.5–1 mL and then rinsed back to 2 mL with cyclohexane for further instrumental analysis.

It is noteworthy that surrogates are normally included in the treatment and analytical procedure to evaluate the loss of the analytes [33]. For example, deuterated musk xylene and AHTN standards are commercially available. However, deuterated AHTN has been reported to undergo partial deuterium to hydrogen exchange during analysis which may cause inaccurate surrogate recovery [34]. Besides, it was reported that no ideal surrogate standard could be found regarding the sampling of musks by PUF and filter [35]. In addition, it has been stated that the procedural calibration might offer higher recoveries than the surrogate corrected recoveries [36]. Nevertheless, regarding the analysis of musks on PUF and glass fiber filter, it was reported that the external recoveries from spiked samples were similar to those obtained by the use of surrogate standard [24]. Hence, instead of surrogates, the spiked samples for recovery evaluation were performed in this study. The target analytes with 2 µg mL<sup>-1</sup> of 10 different musks each were added before the treatments and analytical procedures, while for every batch the spiked samples were treated and analyzed as real samples [35].

#### 2.1.4. Field air sampling

Field air sampling was performed at a cosmetics plant to validate the application of the method developed in this study. The products manufactured include facial wash, lotion, cream, shampoo, body wash, and toner etc. Mixtures of highly concentrated synthetic musk were added as fragrance. All samplers were cleaned and wrapped by aluminum foil in sealed jars individually before sampling. With pumps and tubes appropriately assembled, the samplers were positioned at 1.2–1.5 m high above the ground floor with 0.3–0.5 m away from the reaction tanks. The sampler was face downwards against the dusts. Sampling took approximately 480 min, and the average sampling volume was ca. 1.6 m<sup>3</sup>. Five samples were collected from the plant. After collecting, the samples were foiled, returned to the jars, and shipped back to the lab. Sample preparation was performed a day after the field sampling.

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