

Application News

Gas Chromatograph-Mass Spectrometry / GCMS-TQ™8050 NX

Simultaneous Analysis of Synthetic Musk Compounds in Water Using a Triple Quadrupole GC-MS System

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User Benefits

- ◆ Using a GC-MS/MS, synthetic musk compounds in water can be accurately quantified with high sensitivity.
- ◆ Supporting effective environmental monitoring in water and risk assessment using GC-MS/MS

■ Introduction

Synthetic musk compounds (SMCs) are synthetic organic substances designed as substitutes for natural musk and are widely used in various household products. During everyday activities such as showering and washing, these compounds are released into wastewater. However, current wastewater treatment processes cannot completely remove SMCs, allowing them to enter aquatic ecosystems. As highly bio-accumulative organic compounds, SMCs can cause both acute and chronic toxicity in aquatic organisms. They are also known to cause endocrine disruption when accumulated in the human body¹⁻³⁾. Due to these potential risks, continuous monitoring of SMCs in water is essential.

The main analytical methods for detecting SMCs in water include liquid-liquid extraction (LLE), solid-phase extraction (SPE), and solid-phase microextraction (SPME). Liquid-liquid extraction method requires a large amount of solvent and long preparation time but has the advantage of simultaneously extracting multiple organic compounds⁴⁾.

In this application news, 15 types of SMCs in water were simultaneously analyzed using the gas chromatography method with liquid-liquid extraction method.



Figure 1 GCMS-TQ™ 8050 NX

■ Analytical conditions

In this newsletter, SMCs in water were analyzed using GCMS-TQ8050 NX of SHIMADZU (Figure 1). The instrumental detailed analytical conditions of instrument and the MRM conditions for each compound were shown in Table 1 and 2, respectively.

Table 1 Analytical conditions

GC System	Nexis™ GC-2030
Carrier Gas	: He (99.999 %)
Flow Control Mode	: Liner Velocity
Liner Velocity	: 36.5 cm/s
Injector Temp.	: 280 °C
Injection Mode	: Splitless
Linear Velocity	: 36.5 cm/s
Analytical Column	: SH-Rxi-5MS (30 m × 0.25 mm I.D., 0.25 μm)
Column Temp.	: 60 °C (2 min) → 10 °C/min → 200 °C (10 min) → 10 °C/min → 280 °C (12 min)
MS/MS System	TQ8050 NX
Ionization Method	: EI Mode
Ion source Temp.	: 250 °C
Interface Temp.	: 300 °C
Acquisition Mode	: MRM mode (Table 2)

Table 2 MRM conditions

Peak #	Name	Target Ion	Ref. Ion* 1	Ref. Ion 2
1	DPMI	135.0>119.0	107.0>91.0	135.0>91.0
2	ONTE (Iso-E-super)	191.0>121.2	119.0>91.1	191.0>109.2
3	ADBI	229.0>173.1	244.0>229.2	229.0>57.1
4	AHMI	229.0>187.2	229.0>57.1	229.0>145.1
5	Cyclopentadecanolide	215.0>173.0	258.0>215.0	-
6	Muscione	85.0>67.0	238.0>98.0	-
7	ATII	110.0>81.0	110.0>67.0	124.0>67.0
8	HHCB	243.0>213.2	258.0>243.3	243.0>171.2
9	Musk xylene	282.0>265.0	282.0>77.0	282.0>91.0
10	AHTN	258.0>243.2	243.0>187.2	243.0>57.1
11	Musk moskene	263.0>221.1	263.0>128.1	263.0>201.1
12	Ambrettolide	81.0>79.1	109.0>67.1	81.0>53.1
13	Musk tibetene	266.0>251.2	251.0>160.1	251.0>146.2
14	Musk ketone	279.0>191.1	279.0>117.1	279.0>147.1
15	Ethylene brassylate	98.0>83.1	98.0>70.1	98.0>55.0
ISTD	Phenanthrene-d ₁₀	188.0>160.0	188.0>158.0	-

*Reference Ion

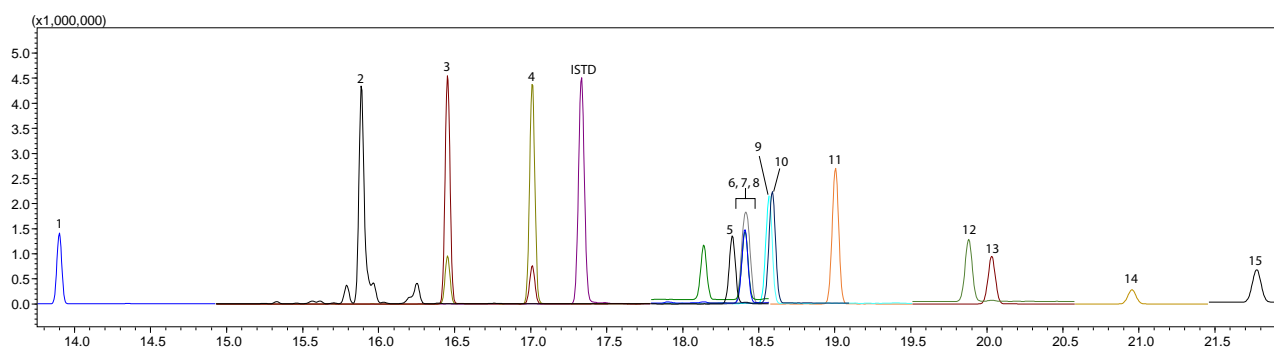


Figure 3 Full chromatogram of 15 SMCs and 1 ISTD

Materials and Method

Solvent A

Solvent A was prepared by mixing n-hexane and Dichloromethane in a 1:1 ratio.

Stock solutions

Each stock solution of the 15 SMCs was prepared at a concentration of 1,000 µg/mL using Solvent A. To prepare a 1,000 ng/mL mixture of the 15 SMCs, 0.1 mL of each stock solution was aliquoted into a 100 mL volumetric flask and then filled with Solvent A.

Working solutions

A 1,000 ng/mL mixed standard solution was added to distilled water to prepare 100 mL of standard solutions with final concentrations of 0.1, 0.2, 0.5, 1.0, 2.0, and 5.0 ng/mL. The working standard solutions were prepared by applying the same pre-treatment procedure used for the samples to these solutions.

Internal standard (ISTD) solution

Solution of Phenanthrene-d₁₀ as internal standard was prepared at 1,000 ng/mL by dilution with solvent A.

Preparation of working solution and sample

All working solutions and water samples were prepared for analysis using GC-MS/MS, following the liquid-liquid extraction (LLE) method (Figure 2). For each sample, 10 mL of solvent A was added to 100 mL of the working solution or water sample. The mixture was then shaken for 10 minutes and the organic solvent layer as the supernatant was taken. To improve extraction efficiency, the process was repeated, and the collected organic solvent layer was evaporated under a nitrogen. The concentrate was reconstituted with 1 mL solvent A and 0.1 mL ISTD solution was added prior to injection into the GC-MS/MS system.

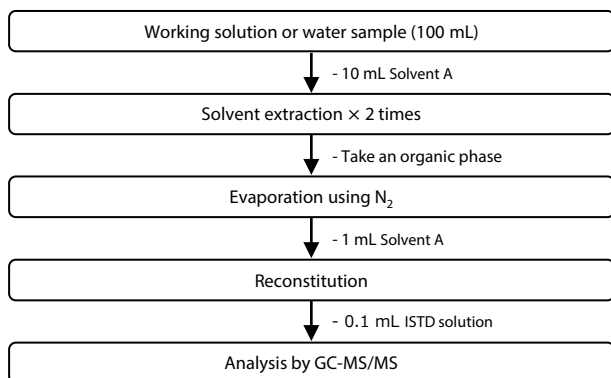


Figure 2 Protocol of preparation

Results

The full chromatogram of 15 SMCs at 5 ng/mL the ISTD at 1 ng/mL is shown in Figure 3. Peaks of chromatogram were identified using the compound numbers listed in Table 2.

Calibration curve

For calibration curve, the coefficient of determination (R^2) for each SMC were higher than 0.996 within the calibration range of 0.1 to 5 ng/mL, as presented in Table 3. The chromatograms and calibration curves of representative compounds are shown in Figure 4.

LOQ and LOD

To determine limit of detection (LOD) and limit of quantitation (LOQ) for the 15 SMCs, spiked samples at 0.2 ng/mL were analyzed in seven replicates. LOD and LOQ were calculated by multiplying the standard deviation by 3.14 and 10, respectively. As a result, the MDLs ranged from 0.003 to 0.020 ng/mL, and the LOQs ranged from 0.01 to 0.07 ng/mL. The calculated values are summarized in Table 3.

Accuracy and Precision

Accuracy and precision were assessed through a recovery test using four replicates spiked solution at 0.2 ng/mL. The recovery values ranged from 86.3 % to 106.4 %, with a relative standard deviation (RSD) of 0.7 % to 4.2 %. The summarized results of the recovery test were shown in Table 3.

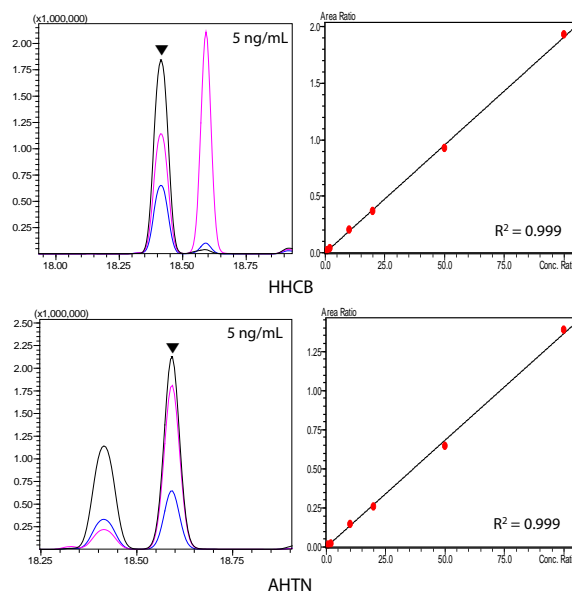


Figure 4 Calibration curves and chromatograms of HHCB and AHTN

Table 3 Retention Time (RT), Linearity, LOD, LOQ, Accuracy and Precision

Name	RT	Linearity (R ²)	LOD (ng/mL)	LOQ (ng/mL)	Accuracy (%)	Precision (%RSD)
DPMI	13.900	0.999	0.010	0.03	99.5	1.8
Iso-E-super (ONTE)	15.887	0.999	0.007	0.02	101.0	1.4
ADBI	16.453	0.999	0.006	0.02	88.3	1.3
AHMI	17.010	0.999	0.008	0.02	90.4	0.9
Cyclopentadecanolide	18.326	0.999	0.008	0.03	94.4	1.7
Muscone	18.409	0.999	0.010	0.03	106.4	1.2
ATII	18.409	0.999	0.020	0.07	99.8	4.2
HHCB	18.414	0.999	0.007	0.02	92.6	0.9
Musk xylene	18.565	0.998	0.003	0.01	95.1	0.8
AHTN	18.592	0.999	0.004	0.01	91.3	0.3
Musk moskene	19.003	0.996	0.009	0.03	86.3	2.1
Ambrettolide	19.879	0.999	0.006	0.02	87.6	1.3
Musk tibetene	20.032	0.998	0.006	0.02	88.6	1.0
Musk ketone	20.954	0.999	0.006	0.02	90.6	0.7
Ethylene brassylate	21.774	0.998	0.010	0.04	93.1	1.2

■ Conclusions

The simultaneous analysis of 15 SMCs in water was performed using the SHIMADZU GCMS-8050 NX system. The coefficient of determination(R²) of the calibration curve for all SMCs were 0.99 or higher. The LOD and LOQ were ranged from 0.003 to 0.020 ng/mL and 0.01 to 0.07 ng/mL, respectively. The recovery test results were between 86.3 % and 106.4 %, with a relative standard deviation (RSD) of 0.7 % to 4.2 %. This analytical method, using a GC-MS/MS system, provided reliable quantitative results for 15 synthetic musk compounds in water samples and demonstrated high sensitivity and efficiency.

■ Reference

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