SHIMADZU APPLICATION NEWS

LA146-E024

● GASCHROMATOGRAPHY MASS SPECTROMETRY

No.M209

Analysis of Musty Odors using SPME-GC/MS

Japan's tap water has a solid reputation for its safety. However, water supplies that are taken from surface water sources, such as dams, lakes and rivers, may generate musty odors during the summer. As a countermeasure, carbon treatment is being used. The substances causing the musty odor are 2-methyl-isoborneol (2-MIB) and geosmin, produced by algae and actinomyces. The odor threshold values for these substances are extremely low, in the order of several ng/L, so pretreatment is required to concentrate samples. In the Japanese goverment's drinking water examination, the solid phase extraction GC/MS and the purge & trap GC/MS methods had been stipulated, and, in 2001, the headspace GC/MS method was added. However, the solid phase extraction GC/MS has disadvantages such as time-consuming and complex extraction procedures and large consumption of organic solvents. As for the purge & trap GC/MS method, the analysis system is expensive and maintenance is difficult.

Solid phase microextraction (SPME) is a pretreatment method that extracts organic compounds from the headspace of liquid or solid samples, concentrates them, thermally desorb them in the GC injector, and introduces them into the column. This pretreatment method is attracting attention because it does not require organic solvents, is fully automated and operation is easy.

■ Analytical Conditions

Autosampler : AOC 5000 (SHIMADZU)
SPME Fiber : PDMS/DVB 65µm(SUPELCO)
Instrument : GCMS-QP2010(SHIMADZU)
GC Column : DB-5(J&W, 30m×0.25mm I.D. df=0.25µm)

AOC-5000	
sample amount	: 10mL+NaCl3g
Incubat Temp.	: 80˚C
Desorb Time	: 3min
Pre Inc Time	: 5min
Extract Time	: 30min (headspace)
GC	
Injection Temp.	: 230˚C
Column Temp.	: 40˚C(3min)-15˚C/min-250˚C(3.0min)
Carrier Gas	: 100kPa
Injection Method	: Splitless (Sampling Time : 3min)
MS	
Interface Temp.	: 250˚C
Ionsource Temp.	: 200˚C
Monitor Ion	: 2-MIB : 95, 107 Geosmin : 112, 182

■ Sensitivity

Fig. 1 shows the SIM chromatograms for each substance at 0.001µg/L and 0.010µg/L. Sufficient sensitivity was verified for a concentration of 0.001µg/L, demonstrating that the SPME method provides sensitivity equivalent to that of the solid phase extraction or purge & trap method.

■ Calibration Curves

Fig. 2 shows the calibration curves for concentrations over a range of 0.001µg/L to 0.050µg/L. Excellent linearity was obtained for both substances, with coefficients of correlation of 0.9995 (2-MIB) and 0.9997 (geosmin).

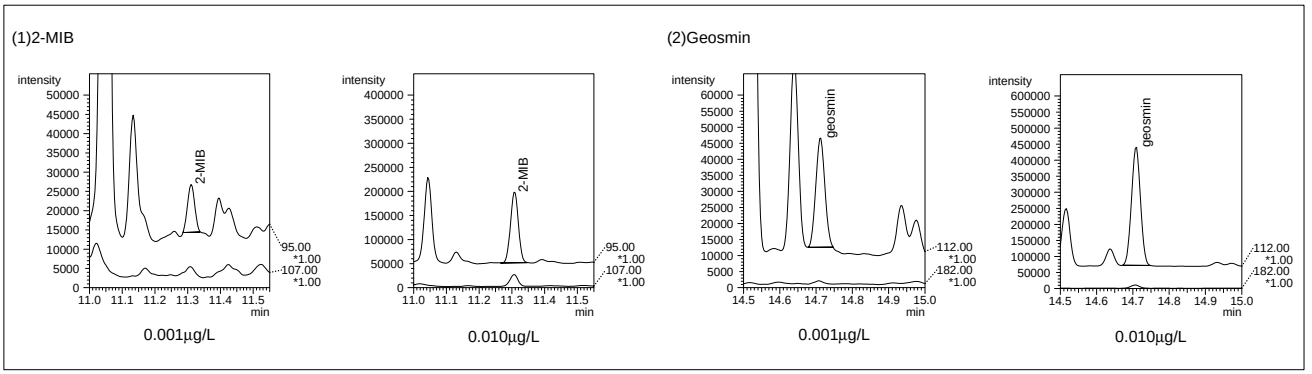

Fig.1 SIM Chromatograms

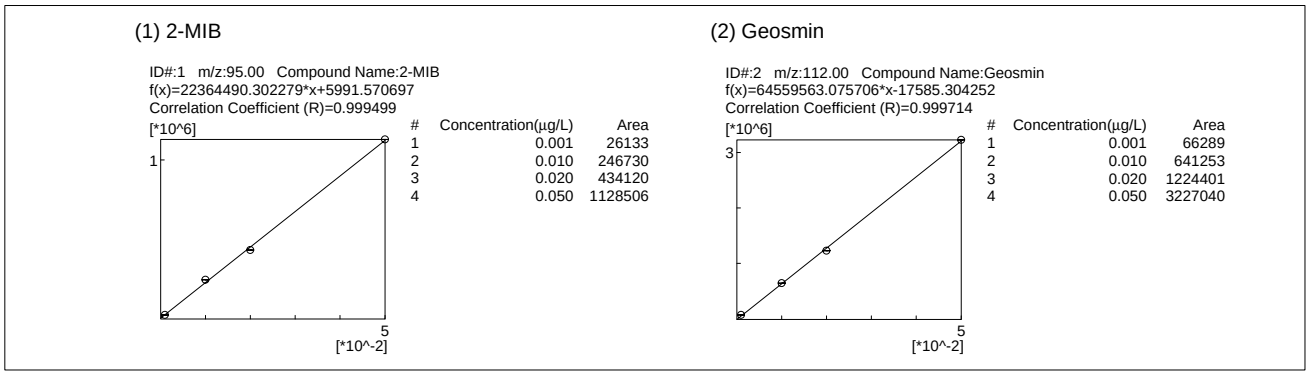

Fig.2 Calibration Curves

■ Reproducibility

In order to check reproducibility, a low-concentration sample (0.001µg/L) was analyzed five times. Table 1 shows the results. Pretreatment conditions such as the temperature and time were controlled by the autosampler, achieving a excellent reproducibility with a CV value at 7%.

Table 1 Reproducibility

	Data 1	Data 2	Data 3	Data 4	Data 5	CV (%)
2-MIB	23993	24463	25919	21435	26133	7.7
Geosmin	69827	67942	68361	58156	66289	7.0

■ Carry Over

In order to check sample carry over, blank water was measured after injecting a high concentration sample (0.050µg/L). In Fig. 3, results are compared for the blank water and low concentration sample (0.001 µg/L). The results show that the carry over is below the lower detection limit.

■ Conclusion

In this Application News, the SPME-GC/MS method was applied to measure the musty odor in water. The results showed good sensitivity and quantitation capability. Although the Drinking Water Examination Method does not accept the SPME-GC/MS method for analyzing musty odors, it has been confirmed that this method is effective.

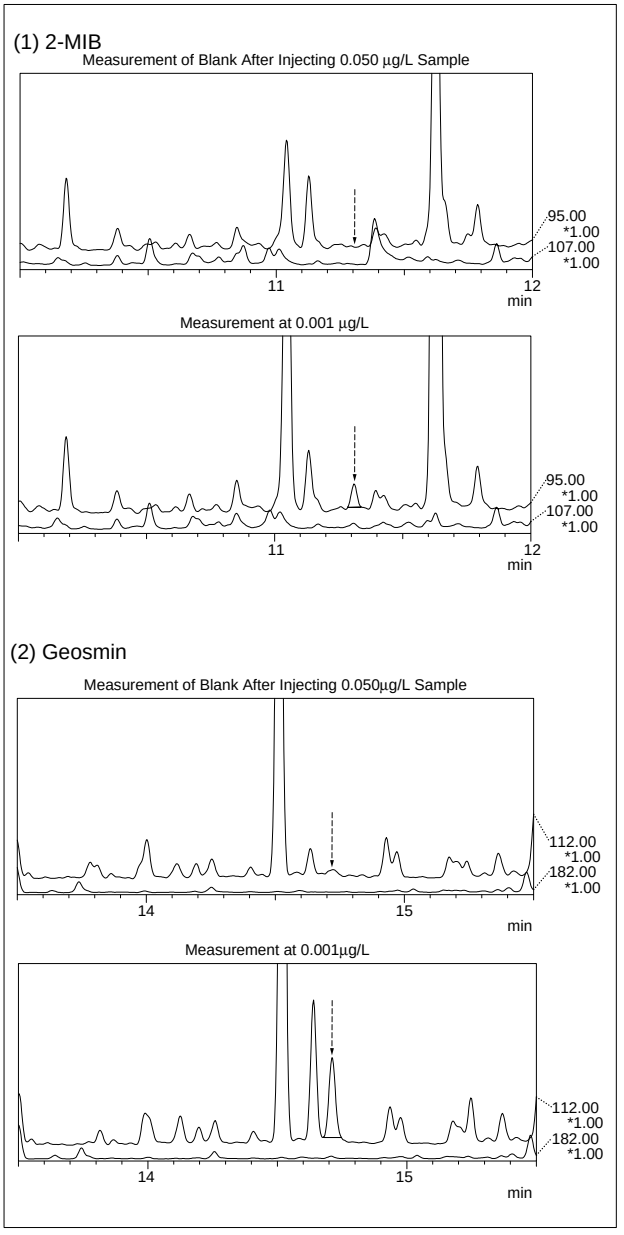

Fig.3 Data Comparison

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

Printed in Japan 3100-02429-10A-IK

Related Solutions

- Price Inquiry
- Product Inquiry
- Technical Service / Support Inquiry
- Other Inquiry