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GC-MS AOC™-6000 Plus/ GCMS-QP2020 NX

Application News

# Analysis of 2-Methylisoborneol and Geosmin in Drinking Water by GC-MS and SPME Arrow

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## User Benefits

- SPME Arrow offers highly durable and sensitive trace analysis.
- Using SPME Arrow not only provides cost savings in the long run but also eliminates the need for the salting-out technique, contributing to overall cost efficiency.

## ■ Introduction

2-methylisoborneol (2-MIB) and geosmin are responsible for earthy and musty odors which can affect the taste of drinking water. Hence, the analysis of these compounds in water sources is important due to their significant impact on water quality and taste. As humans can perceive them at trace levels (ng/L)[1], it is crucial to detect them at this concentration. The Solid Phase Microextraction (SPME) Fiber technique paired with GC-MS has been used to detect these two organic compounds previously; however, salting-out is often required to improve sensitivity. Moreover, if the salt purity is insufficient, it may lead to contaminants co-eluting with the target compounds.

This application news introduces a study utilizing GC-MS with SPME Arrow to effectively detect and quantitate the concentration of geosmin and 2-MIB in drinking water samples. Notably, salting-out is not required, and a limit of quantitation of 1 ng/L still can be achieved, with area %RSD (n=5) for 2-MIB and geosmin being less than 5 %. The spike recovery study at concentrations of 1 ng/L yielded recoveries within 70 % to 130 %.

## ■ Experimental

### Analysis condition

The instruments utilized in this experiment were GCMS-QP2020 NX and AOC-6000 Plus (both from Shimadzu Corporation, Japan), as shown in Figure 1. SPME Arrow in AOC-6000 Plus, designed to enhance the extraction and analysis of volatile and semi-volatile compounds from various matrices, was selected as the sampling technique. This sampling method utilizes a larger adsorbent amount compared to the traditional SPME Fiber, allowing for increased sample capacity and improved sensitivity. SPME Arrow also possesses a thick and sturdy construction, which contributes to its durability and, consequently, cost saving in the long run.

The analytical conditions are presented in Table 1.

**Figure 1:** AOC™-6000 Plus autosampler with GCMS-QP2020 NX

**Table 1:** AOC-6000 Plus and GC-MS analytical conditions for analysis of 2-MIB and geosmin in drinking water

| | |
|---|---|
| **AOC-6000 Plus Autosampler Parameter** | |
| **SPME Arrow** | Smart SPME Arrow 1.10 mm<br>DVB/C-WR/PDMS<br>[P/N: 227-35333-03] |
| **Incubation Temp.** | 80 °C |
| **Incubation Time** | 5 min |
| **Stirrer Speed** | 1000 rpm |
| **Sample Extraction Time** | 15 min |
| **Sample Desorb Time** | 2 min |
| **GC Parameter** | |
| **Carrier Gas** | Helium |
| **Injector Temp.** | 250 °C |
| **Injection Mode** | Splitless |
| **Flow Control Mode** | Linear Velocity, 52.9 cm/s |
| **Oven Temp. Program** | 40 °C (2 min) → 10 °C/min to 120 °C → 30 °C/min to 250 °C (5 min) |
| **Column** | SH-I-5 Sil MS<br>(30 m x 0.25 mm ID x 0.25 µm df)<br>[P/N: 221-75954-30] |
| **MS Parameter** | |
| **Ion Source Temp.** | 200 °C |
| **Interface Temp.** | 250 °C |
| **Acquisition Mode** | SIM<br>2-MIB (95, 108)<br>Geosmin (112, 125) |

### Standard and sample preparations

2-MIB and geosmin were obtained from Merck, while methanol was purchased from Kanto Chemical. Milli-Q water used for the calibration curve was generated from the Millipore Milli-Q Integral 10 Water Purification System.

The standards were prepared by mixing and diluting with methanol to achieve concentrations of 1, 2, 5, 10, and 20 pg/µL. Subsequently, 10 µL of each standard was spiked into a 20 mL screw cap vial containing 10 mL of Milli-Q water, resulting in final concentrations of 1, 2, 5, 10, and 20 ng/L for calibration curve plotting.

Drinking water from 2 different sources were selected as samples (Sample A and Sample B). For sample preparation, 10 mL of the sample was added to a 20 mL screw cap vial, followed by the addition of 10 µL of methanol to the solution.

Once the standard and sample preparations were completed, the vials were then loaded onto the AOC-6000 Plus autosampler for subsequent SPME Arrow extraction and GC-MS analysis.

## ■ Result

### Sensitivity, Repeatability and Linearity

2-MIB and geosmin standards at concentrations of 1, 2, 5, 10, and 20 ng/L were analyzed by the system. The SIM chromatograms of the 2-MIB and geosmin standard at 1 ng/L are displayed in Figure 2. The limit of quantitation (LOQ) for both compounds is 1 ng/L, with signal-to-noise ratios (S/N) greater than 10 (Table 2). The %RSD (n=5) of the area count for both compounds at 1 ng/L was less than 5 % (Table 3). Excellent linearity was achieved, with both compounds having $R^2$ values greater than 0.999 (Figure 3). With SPME Arrow, the salting-out technique is not required to achieve the LOQ at 1 ng/L, which helps save on reagent cost. This also reduces the hassle of sourcing high-purity salt or baking the salt to eliminate contaminants that may co-elute with the targeted compounds.

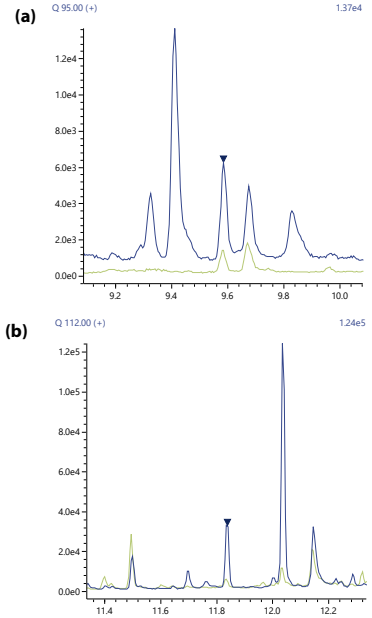


**Figure 2:** SIM chromatograms of: (a) 2-MIB (1 ng/L); (b) Geosmin (1 ng/L)

**Table 2:** Signal-to-noise ratios of 2-MIB and geosmin at 1 ng/L

| Injection | S/N | |
|---|---|---|
| | 2-MIB | Geosmin |
| Injection 1 | 27 | 28 |
| Injection 2 | 16 | 21 |
| Injection 3 | 20 | 35 |
| Injection 4 | 24 | 18 |
| Injection 5 | 21 | 17 |

**Table 3:** Area repeatability %RSD (n=5) of 2-MIB and geosmin at 1 ng/L

| Injection | Area Count | |
|---|---|---|
| | 2-MIB | Geosmin |
| Injection 1 | 7,534 | 26,765 |
| Injection 2 | 7,055 | 26,122 |
| Injection 3 | 7,514 | 26,206 |
| Injection 4 | 7,656 | 27,000 |
| Injection 5 | 7,626 | 26,744 |
| **%RSD** | 3.3 | 1.4 |

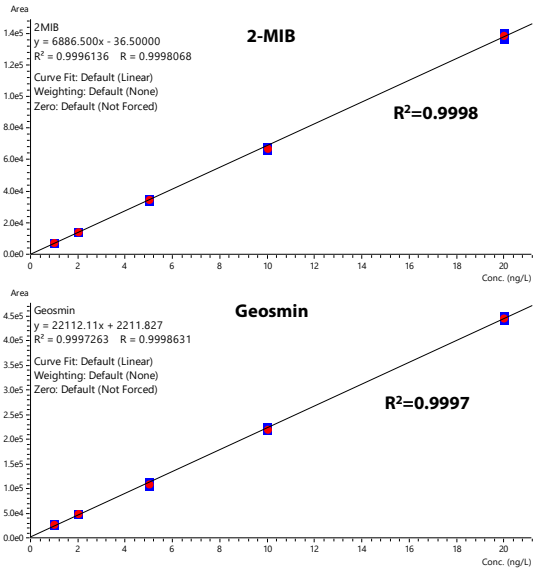


**Figure 3:** Five-point calibration curves of 2-MIB and geosmin

### Quantitation and Recovery

The quantitation results of Sample A and Sample B are displayed in Table 4. To test for recovery, 1 ng/L of the targeted compounds were spiked into the two samples. If the sample yielded a positive result, the recovery would be calculated by subtracting the unspiked result from the spiked result. As shown in Table 4, the recovery for both compounds in the samples were within the range of 70 % to 130 %.

**Table 4:** 2-MIB and geosmin concentration and recovery results in drinking water samples

| Drinking Water Sample | | Conc Result (ng/g) | | % Recovery | |
|---|---|---|---|---|---|
| | | 2-MIB | Geosmin | 2-MIB | Geosmin |
| Sample A | Unspiked | 0.535 | 0.347 | | |
| | 1 ng/L spiked | 1.541 | 1.370 | 101* | 102* |
| Sample B | Unspiked | N.D | 0.137 | | |
| | 1 ng/L spiked | 1.216 | 1.155 | 122 | 102* |

*Percentage recovery was calculated after subtracting unspiked result from spiked result.

## ■ Conclusion

With SPME Arrow technique paired with GC-MS, a method was successfully developed for the analysis of 2-MIB and geosmin in drinking water in trace level. Good sensitivity (LOQ at 1 ng/L), area repeatability and linearity ($R^2$ > 0.999) were achieved without using the salting-out technique. Good recovery at 1 ng/L was obtained (within 70 % to 130 %) for both compounds in drinking water samples.

## ■ Reference

1. Huang et al., Kinetic and mechanistic investigation of geosmin and 2-methylisoborneol degradation using UV-assisted photoelectrochemical, *Chemosphere* 2022



04-AD-0314-EN First Edition: Feb. 2025

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