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Gas Chromatograph-Mass Spectrometry

Application News

# Ultra-Sensitive Analysis of β-cyclocitral in Water Using Solid Phase Micro Extraction and Gas Chromatography–Mass Spectrometry

Hoonju Kim, Jin Jang
Shimadzu Scientific Korea

## User Benefits

- SPME-GC-MS supports effective monitoring of water environments
- Excellent sensitivity, achieving quantification of β-cyclocitral in water within the ultra-trace level
- SPME provides a solvent-free extraction, efficient, and highly sensitive method for detecting β-cyclocitral

## Introduction

Cyanobacterial blooms, commonly known as algal blooms, cause rivers and lakes to turn vividly green due to the excessive growth of cyanobacteria. This phenomenon is caused by factors such as global population growth, industrial pollution, and rising water temperatures caused by climate change.[1)]

Cyanobacterial blooms produce volatile organic compounds (VOCs) like β-cyclocitral and β-ionone, which cause unpleasant odors and reduce the quality of drinking water and aquatic products. This leads to consumer complaints and economic losses in aquaculture and water treatment.

Detecting and quantifying β-cyclocitral (Figure. 1) at trace levels is therefor essential for effective water management. However, conventional solvent extraction methods often fail to detect β-cyclocitral, as it requires acidification or heating to be effectively released from the matrix.[2)]

In contrast, solid phase microextraction (SPME) enables efficient detection by facilitating the thermal desorption of β-cyclocitral from sample matrices onto the fiber. Moreover, SPME reduces solvent use and shortens sample preparation time compared to traditional extraction techniques, making it a suitable approach for the analysis of thermally induced volatile compounds like β-cyclocitral.

This application news describes the suitability of β-cyclocitral analysis using SPME automatic injection (AOC$^{TM}$-6000 Plus) and GCMS-QP2020 NX of Shimadzu. The developed method enables highly sensitive quantitation of β-cyclocitral, achieving detection at the pg/mL level.

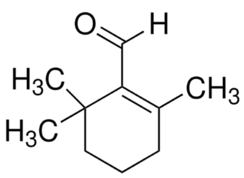

Figure 1. Structural Formula of β-cyclocitral

## Analytical Conditions

β-cyclocitral in water was analyzed using GCMS-QP2020 NX of Shimadzu equipped with the AOC-6000 Plus autosampler as shown in Figure 2. The detailed analytical conditions of instrument was provided in Table 1. For the SPME analysis, 50/30 µm DVB/CAR/PDMS fiber was used, and SH-PolarWax (30 m × 0.25 mm I.D., 0.25 µm) was used as the analytical column.

The AOC-6000 Plus supports various injection techniques, including liquid injection, headspace (HS) injection, and solid-phase microextraction (SPME) injection. Furthermore, sample preparation and preprocessing in the AOC-6000 Plus are automatically carried out during injection into the GC system, improving efficiency and reproducibility.

Figure 2. GCMS-QP2020 NX with AOC$^{TM}$-6000 Plus

Table 1. Analytical Conditions

| | |
|---|---|
| AOC-6000 Plus | |
| SPME Fiber | : 50/30 µm DVB/CAR/PDMS |
| Incubation Temp. | : 60 °C |
| Incubation Time | : 5 min |
| Sample Extract Time | : 20 min |
| Sample Desorb Time | : 10 min |
| Agitation | : On |
| GCMS-QP2020 NX | |
| Analytical Column | : SH-PolarWax (30 m × 0.25 mm I.D., 0.25 µm) |
| Column Temp. | : 50 °C (1 min) → 10 °C/min → 160 °C → 20 °C/min → 240 °C (2 min) |
| Carrier Gas Flow | : He, 1.0 mL/min |
| Injection Mode | : Splitless |
| Injector Temp. | : 260 °C |
| Ion Source Temp. | : 230 °C |
| Interface Temp. | : 250 °C |
| Acquisition Mode | : SIM mode (*m/z* 152, 137) |

## Materials and Preparation

### Materials

β-cyclocitral standard (97 % purity) was purchased from Sigma Aldrich. tert-Butyl methyl ether (MTBE, 99 % purity) used as a solvent was obtained from Samchun Chemical. Distilled water for the calibration curve was prepared using a Millipore Milli-Q water purification system.

### Standard and sample preparation

A stock solution of β-cyclocitral was prepared at a concentration of 1,000 µg/mL in MTBE. Six standard solutions were obtained by diluting the stock solution with MTBE to final concentrations of 1.0, 5.0, 10.0, 20.0, 50.0, and 100.0 ng/mL.
Each calibration standard was prepared by adding 10 µL of the corresponding standard solution to 10 mL of distilled water in 20 mL headspace vial, followed by 4 g of NaCl. The resulting final concentrations of calibration standard solutions were 1.0, 5.0, 10.0, 20.0, 50.0, and 100.0 pg/mL in distilled water.

### Recovery test

To evaluate accuracy and precision, a sample solution with a concentration of 25 pg/mL was prepared and analyzed. The sample was prepared as follows: 10 mL of distilled water was placed in a 20 mL headspace vial, spiked with 10 µL of a 25 ng/mL standard solution, and 4 g of NaCl was added. Four replicate measurements were performed, and the recovery percentage were calculated accordingly. Accuracy was assessed based on the mean recovery value, while precision was determined by calculating the relative standard deviation (%RSD) of the recovery values.

## Results

### Calibration curve

The calibration curve for β-cyclocitral was established using the external standard method over the concentration range of 1.0 – 100.0 pg/mL. Excellent linearity was observed, with the coefficient of determination ($R^2$) better than 0.999. The calibration curves and analytical chromatograms at each concentration level are shown in Figure 3.

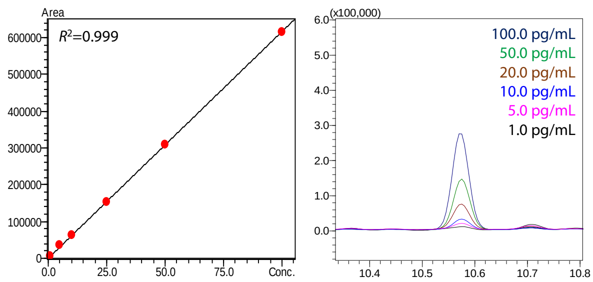


Figure 3. Calibration Curve and Chromatograms of β-cyclocitral

### Accuracy and precision

The analytical method exhibits satisfactory accuracy and excellent precision in recovery test. As summarized in Table 2, the mean recovery was 105.3%, with %RSD of 0.7.

Table 2. Accuracy and Precision of β-cyclocitral (25 pg/mL, *n*=4)

| | Concentration (pg/mL) | Average (pg/mL) | Accuracy (%) | Precision (%RSD) |
|---|---|---|---|---|
| 1 | 26.2 | 26.3 | 105.3 | 0.7 |
| 2 | 26.5 | | | |
| 3 | 26.5 | | | |
| 4 | 26.1 | | | |

## Conclusions

In this application news, SPME-GC-MS method was developed and validated for the ultra-sensitive quantitation of β-cyclocitral in water using the Shimadzu GCMS-QP2020 NX coupled with the AOC-6000 Plus. The method demonstrated quantification capability down to the picogram-per-milliliter (pg/mL) level, excellent linearity ($R^2$ > 0.999). Accuracy and precision were confirmed with a recovery of 105.3% and %RSD of 0.7. These results confirm the reliability and suitability of the SPME-GC-MS method for ultra-trace quantification of β-cyclocitral in environmental water samples.

## Reference

1) Effects of different cultivation conditions on the production of β-cyclocitral and β-ionone in Microcystis aeruginosa, Jessica Aparecide Silva Moretto et al., BMC Microbiology volume 22, Article number: 78 (2022)
2) Analytical Technique Optimization on the Detection of β-cyclocitral in Microcystis Species: Molecules 2020, 25, 832; doi:10.3390/molecules 25040832



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