

Analysis of Trace Levels of 2-MIB and Geosmin in Drinking Water by ChroZen GC-MS/HS-SPME Arrow

- GC/MS Application



Abstract

Geosmin and 2-MIB (2-Methylisoborneol) are known as the compounds that cause earthy or unfavorable taste and odor in drinking water and these are naturally produced by metabolites of bacteria and algae.

In this study, these geosmin and 2-MIB were determined by Headspace-SPME with Gas Chromatography/Mass Spectrometry detection which is described in the regulation for drinking water. The equipment used is ChroZen GC/MS and ChroZen PAL HS-SPME Arrow for the trace level of quantitation for actual water sample.

HS-SPME Arrow has higher sorbent volume than SPME fiber to increase the extraction capacity and enables the detection of trace level of samples. Its robustness provides a long life use as well as cost saving maintenance.

ChroZen GC/MS with ChroZen PAL-HS-SPME Arrow simplifies overall procedure with optimized methods and satisfies the detection limit as well as the recovery efficiency.

Instruments and Software

- ChroZen GC/MS with ChroZen PAL RSI HS-SPME Arrow

Item	Description	Part No.
Oven	ChroZen GC Mainframe Assembly for Mass Spectrometer	6701012500
Inlet	Capillary Inlet Assembly for ChroZen GC	6701012550
Detector	ChroZen MS for ChroZen GC incl. built-in turbomolecular pump - Single Quadrupole - EI source (UEIS: Ultimate Efficiency Ion Source) - Turbo pump(240 L/sec) - Include fore pump and spares kit - Higher Sensitivity (S/N for OFN 2500:1)	6901012110
ChroZen PAL RSI	PAL RSI 850 system without liquid or headspace option	PAL3-RSI-GH-S-9-YL2
	Mounting Kit for ChroZen GC	PAL3-Kit-YL6700
Autosampler	Agitator for the incubation and agitation of up to 6x 20mL vials	PAL3-Agitator
ChroZen PAL RSI Optional Module (SPME Arrow)	Smart SPME Arrow Kit, consisting of: 1 pc SPME Arrow Tool	PAL3-SPME-SArr-Kit
	SPME Arrow Conditioning Module for SPME arrows and SPME fibers	PAL3-SPME-ArrowCond
	Heatex Stirrer for intensive heating and stirring of 1x 20mL vial	PAL3-HeatexStirrer
	Adaptation for split/splitless injector of ChroZen GC	Call
	SPME Arrow Liner ID 1.3mm, Pk of 3 pcs	ARRLIN13-GC7890-3
CDS	YL-Clarity software for single instrument of YL GC	5301011020
	MS module of YL-Clarity(Library as option)	5301011180
	Autosampler control of YL-Clarity	5301011040
	Library(NIST/EPA/NIH 2017 edition)-306,622 spectra	NIST17-MS-LIB
Column	YL-5MS (30m, 0.25 mm, 0.25 µm)	1256120170
Install. Option	Start-up kit includes (Without GC Capillary Column)	1601011110
ACC	Big Universal Trap, 1/8" fttgs, Helium	RMSH-2
	YL PAL System Vial 20CV, 20ml Clear Glass with Label, Pk of 100 Pcs	Vial-20-ND18-CG-100
	YL PAL System Screw Cap 10CV and 20CV, Pk of 100 pcs	Cap-ND18-St-SP15-100



Fig 1.ChroZen GC/MS with ChroZen PAL

Reagents and Standards

Geosmin, 2-MIB Odor Compounds Mix
(200 µg/mL each component in
methanol)

Preparation of Standard Solution

Dilute Geosmin, 2-MIB Odor Compounds
Mix with methanol in the concentration
of 10.0 µg/mL. Transfer this solution into
the vial and cap with no headspace.
Refrigerate it at 4°C and conduct the
analysis within 4 weeks.

Preparation of Water Samples

Transfer 10mL of water sample to a
20mL headspace vial and add 3g of
NaCl.

Use NaCl heated for more than 4 hours
at 450°C furnace to eliminate the
impurities.

GC Conditions	MS Conditions
Column: YL 5ms (30m x 0.25 mm i.d. x 0.25µm)	Ion source 250°C, Transfer Line 280°C
Inlet: splitless, purge on time: 0.1 min	Detection: SIM mode
Oven temperature program: 60°C, 5min, 10°C/min to 270°C, 2min	

Table 1. GC/MS Condition

SPME System Conditions
Preconditioning time: 10min
Incubation time: 2min
Sample Extraction time: 30min
Extraction Temp.: 60°C
Sample Desorb time: 5min

Table 2. SPME System Condition

	Compound	Formula	Molecular Weight	Selected Ions, m/z
1	2-Methylisoborneol	C ₁₁ H ₂₀ O	168.28	95
2	Geosmin	C ₁₂ H ₂₂ O	182.31	112

Table 3. GC/MS Chromatographic Conditions

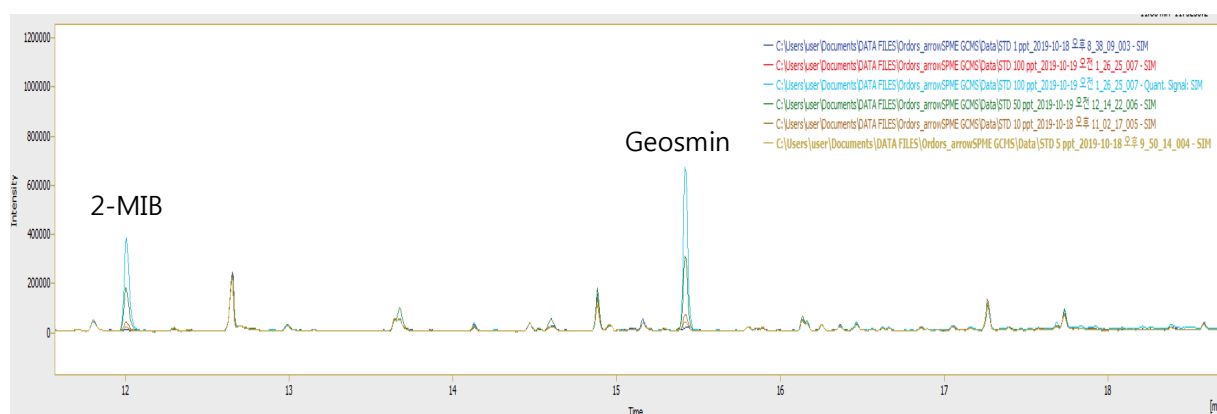


Fig 2. Overlaid Chromatogram of 2-MIB & Geosmin at 1, 5, 10, 50,100ppt

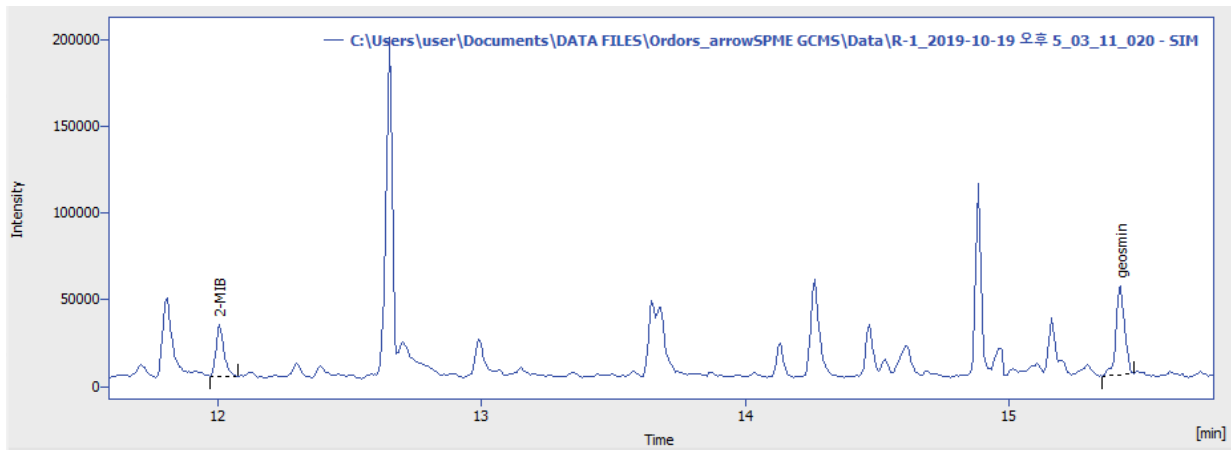


Fig 3. Chromatogram of 2-MIB & Geosmin at 10ppt

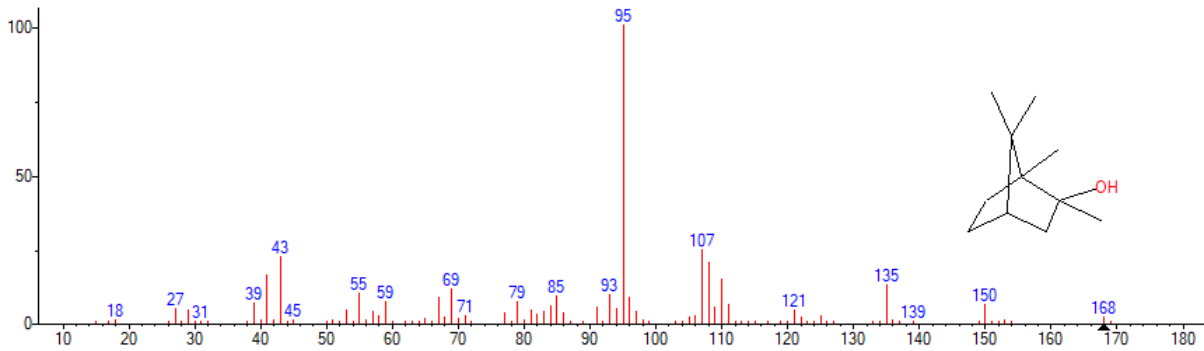


Fig 4. Spectra of 2-MIB

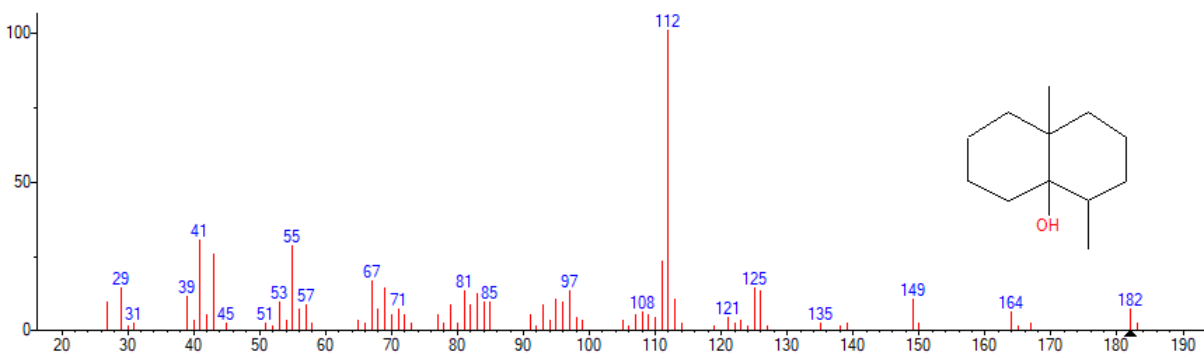


Fig 5. Spectra of Geosmin

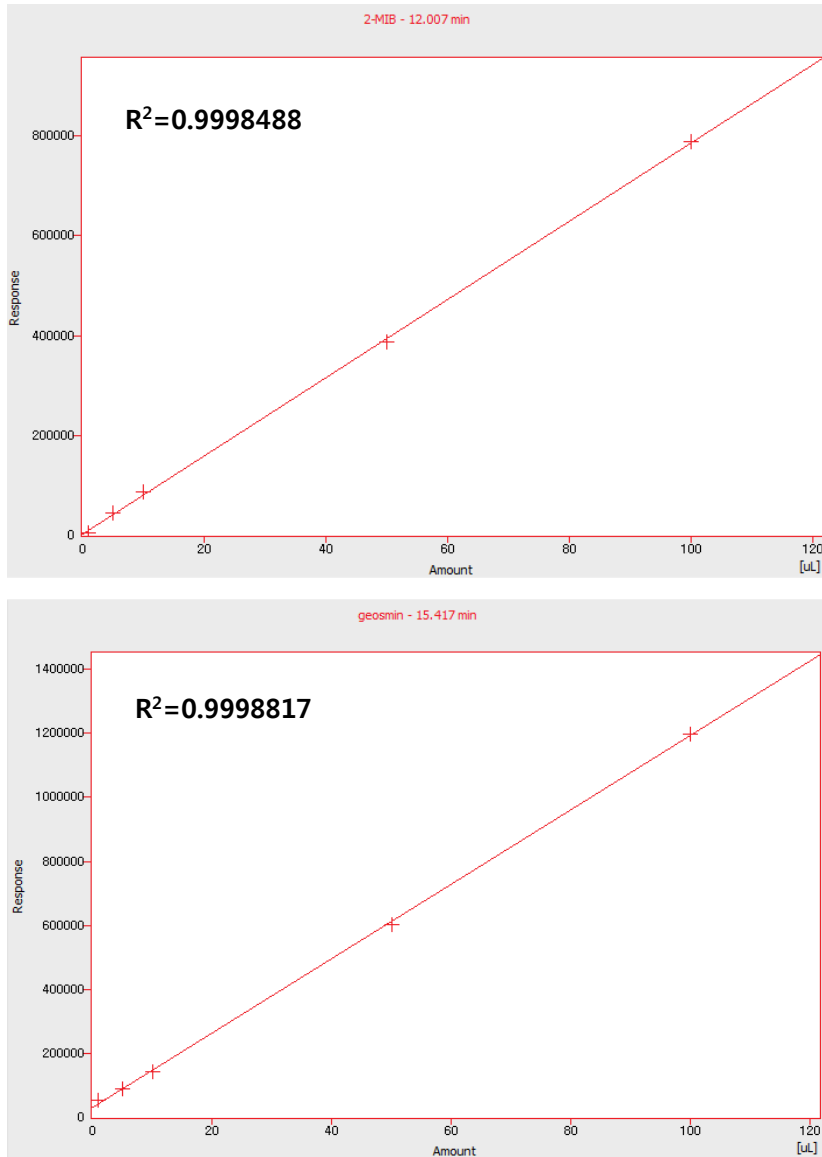


Fig 6. Calibration Curve (1, 5, 10, 50,100ppt) of 2-MIB & Geosmin

Analyte	Sample Name	Resp.	Calc. Conc (ppt)	Accuracy (%)	Precision(%)
2-MIB	Sam-1	41111.8	4.991	101.40	1.45
	Sam-2	42251.7	5.136		
	Sam-3	41836.3	5.083		
Geosmin	Sam-1	89900.3	5.099	100.95	0.89
	Sam-2	88941.6	5.017		
	Sam-3	89056.2	5.026		

Table 4. Recovery of 2-MIB & Geosmin

Analyte	QC Name	Data Conc.(ppt)	STDEV	MDL (ppt)	MRL (ppt)
2-MIB	Sam-1	1.05	0.02	0.057	0.181
	Sam-2	1.01			
	Sam-3	1.02			
	Sam-4	1.01			
	Sam-5	1.02			
	Sam-6	1.00			
	Sam-7	1.00			
Geosmin	Sam-1	1.07	0.04	0.119	0.380
	Sam-2	0.99			
	Sam-3	1.06			
	Sam-4	1.04			
	Sam-5	1.05			
	Sam-6	1.03			
	Sam-7	0.97			

Table 5. Method Detection Limit (MDL) and Minimum Reporting Level (MRL) of 2-MIB & Geosmin

※ MRL is a concentration that is no lower than the lowest level calibration standard and is determined from analysis of seven replicate LFBs(Laboratory Fortified Blanks). The MRL is confirmed if the upper and lower limits of the half range of the prediction interval, calculated as the standard deviation of the replicate LFBs times 3.963, are within 50% of the mean result for the seven replicates.

> Referring Detection Limit/Quantitation Limit Summary Table by EPA

Result

The method detection limit (MDL), the minimum reporting level (MRL) and standard deviation were calculated after analyzing 7 water samples which concentrations are diluted with standards to be similar as MDL according to Standard Test Methods of Drinking Water by National Institute of Environmental Research.

The MDL was calculated after multiplying 3.14 to the standard deviation and the MRL was determined after multiplying 10 to the standard deviation. The MDL in this study was determined to 0.181 ppt for 2-MIB and 0.380 ppt for Geosmin. This value is greater than the MDL (0.001 μ g/L ~ 0.002 μ g/L) indicated in the regulation of drinking water.

To measure the accuracy, CRM(Certified Reference Material) standard was used in this application. The calculation of accuracy described in Standard Test Methods of Drinking Water is indicated in the relative percentage by the difference of concentration of the sample with standards addition and the deionized water with standard addition and the range should be 75% ~ 125%. The accuracy in this study resulted within 102%. The precision should be calculated in the relative standard deviation (RSD) of measured value and the measurement needs to be within 20%. This study resulted within 2%.

Conclusion

In this study, determination of 2-MIB and Geosmin in drinking water was obtained by ChroZen GC/MS coupling with ChroZen PAL HS-SPME Arrow. The SPME Arrow enabled the analysis of trace level sample with much higher sensitivity than SPME Fiber. The analysis was conducted by referring Standard Methods 6040D and Standard Test Methods of Drinking Water and the results were calculated according to Standard Test Methods of Drinking Water. As the result, ChroZen GC/MS with ChroZen PAL HS-SPME Arrow satisfies with the regulation and verifies reliable data.



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Reference

- Standard Methods 6040D
- Standard Test Methods of analysis of Geosmin & 2-MIB in Drinking Water_HS-SPME & GC/MS by National Institute of Environmental Research.