Analysis of Geosmin and 2-Methylisoborneol Utilizing the Stratum PTC and Aquatek 70

Application Note

By: Anne Jurek

Abstract

Geosmin and 2-Methylisoborneol are organic compounds that have a distinct scent. These compounds also have an extremely low odor detection threshold and because of this, many drinking water laboratories require detection levels of below 10ppt. This application note investigates the detection of Geosmin and 2-Methylisoborneol at a 1ppt level utilizing Purge and Trap (P&T) coupled with a Gas Chromatograph and Mass Spectrometer (GC/MS).

Introduction

Geosmin and 2-Methylisoborneol have very poor purge efficiencies. In order to detect these compounds down to the 1ppt level it was necessary to optimize the P&T and GC/MS techniques.

In this study, the GC/MS was run in Selective Ion Monitoring (SIM) mode while the P&T conditions were modified in order to achieve a 1ppt level of detection. A 10% (w/v) salt solution was utilized along with a 25mL purge volume. The Stratum PTC was configured with a #1 trap and a cryofocusing module was utilized for more efficient trapping and injection of the mold compounds.

Experimental-Instrument Conditions

The Stratum PTC and Aquatek 70 autosampler coupled with a cryofocusing module were connected to an Agilent 7890A GC and a 5975 inert XL MS system for analysis. A #1 trap was the analytical trap used. The GC was configured with a J&W Scientific DB-VRX 30m x 0.250mm x 1.4µm column. The MS scanned in the SIM mode. Finally, a 25mL purge volume of 10% (w/v) salt water was used. The GC/MS parameters are outlined in Tables 1 and 2 respectively while Table 3 outlines the P&T conditions.

GC Parameters	
GC:	Agilent 7890A
Column:	J&W Scientific DB-VRX 30m x 0.250mm x1.4um
Oven Program:	40°C for 2.0 min; 16°C/min to 160°C for 0 min; 20°C /min to 240 °C for 5.0 min; 18.5 min. runtime
Inlet:	220°C
Column Flow:	1.02mL/min
Gas:	Helium
Pressure:	12.089 psig

Table 1: GC Parameters

MSD Parameters	
MSD:	Agilent 5975C inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	5.0 min
SIM Ions	95, 107, 108, 112, 125, 126
Dwell Time:	100 msec dwell per ion

Table 2: MS Parameters

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Stratum PTC and Aquatek 70 Parameters Water Parameters			
Variable	**Value**	**Variable**	**Value**
Pressurize Time	0.60 min	Purge Time	12.00
Fill IS Time	0.00 min	Purge Temp	0°C
Sample Transfer Time	0.75 min	Purge Flow	45mL/min
Rinse Loop Time	0.75 min	Dry Purge Time	5.00 min
Purge Loop Time	1.00 min	Dry Purge Temp	20°C
Bake Rinse	On	Dry Purge Flow	45mL/min
Number of Bake Rinses	3	GC Start	End of Desorb
Bake Drain Time	1.50 min	Desorb Preheat Temp	220°C
Bake Drain Flow	250mL/min	Desorb Drain	On
Valve Oven Temp	175°C	Desorb Time	6.00 min
Transfer Line Temp	175°C	Desorb Temp	225°C
Sample Mount Temp	60°C	Desorb Flow	300mL/min
Purge ready Temp	40°C	Bake Time	15.00 min
Condenser Ready Temp	40°C	Bake Temp	230°C
Condenser Purge Temp	20°C	Bake Flow	250mL/min
Standby Flow	45mL/min	Condenser Bake Temp	175°C
Pre-Purge Time	0.0 min	Focus Temp	-100°C
Pre-Purge Flow	0.0mL/min	Inject Time	2.00 min
Sample Heater	On	Inject Temp	200°C
Sample Preheat Time	0.01 min	Standby Temp	150°C
Sample Temp	40°C		

Table 3: Stratum PTC and Aquatek Parameters

Stratum PTC Parameters are in Blue

Calibration

A 50ppb working calibration standard was prepared in methanol. Calibration standards were prepared in a 50mL volumetric flask and filled to volume with 10% (w/v) de-ionized salt water solution. The calibration range was 1.0-100ppt. The standards were transferred to headspace free 40mL vials for analysis. The calibration data was analyzed using Agilent Chemstation software. The calculated linear regression and the %RSD of each compound are outlined in Table 4.

Method Detection Limit (MDL), Carryover, and Precision and Accuracy Study

A statistical determination of the MDL's was determined for both of the compounds by analyzing seven replicate standards of a 1ppt calibration standard. The detection limit is provided in Table 4. Furthermore, seven replicate standards of a 10ppt calibration standard were analyzed in order to determine the precision and accuracy of the experimental conditions. These results are also provided in the Table 4.

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Compound	Calibration Curve %RSD	Calibration Curve Linearity	Spike Level (ppt)	MDL (ppt)	Spike Level (ppt)	Precision (%RSD)	Accuracy (% Recovery)
Geosmin	3.940	1.000	1.00	0.114	10.00	5.80	103.89
2-Methylisoborneol	6.520	1.000	1.00	0.236	10.00	7.38	107.43

Table 4: Experimental Results Summary

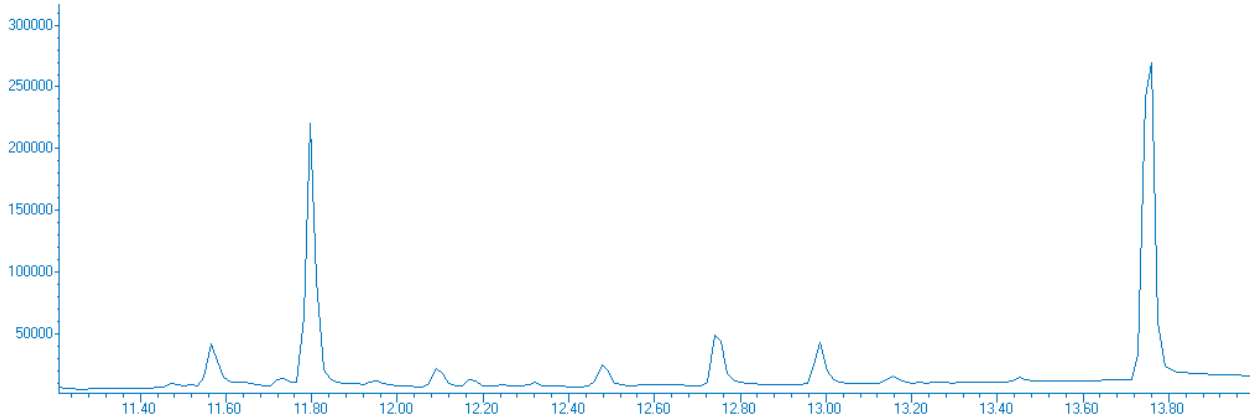
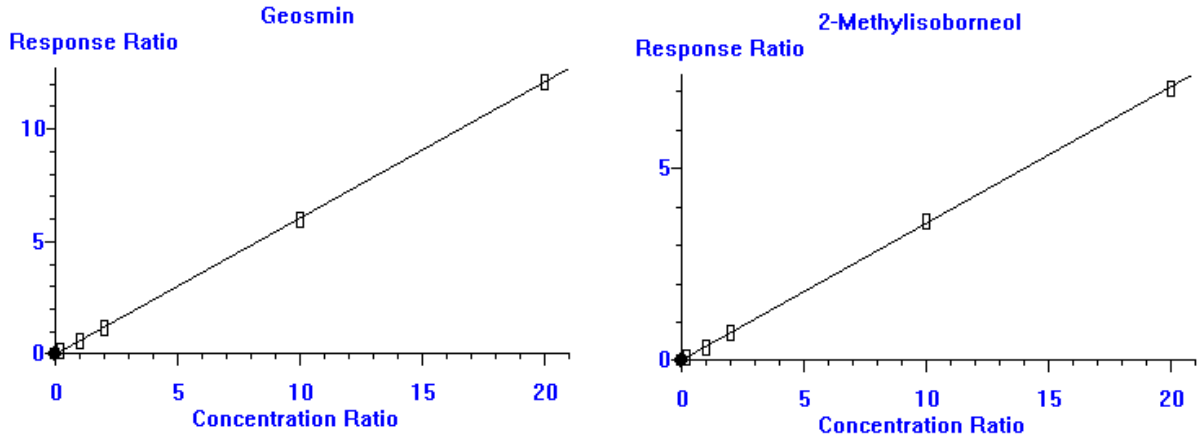


Figure 1: Total Ion Chromatogram of a 50ppt Geosmin and 2-MethylisoborneolStandard



Figures 2 and 3: Calibration Curves of Geosmin and 2-Methylisoborneol

Conclusions

The Stratum PTC and Aquatek 70 configured with a cryofocusing module performed very well in detecting Geosmin and 2-Methylisoborneol. The linearity of the curve was 1.000 for both compounds and the system displayed excellent accuracy and precision results. The 25mL sample volume and 10% (w/v) salt solution aided in increasing the purge efficiency of both the Geosmin and the 2-Methylisoborneol compounds. Finally, by using SIM analysis with the GC/MS and optimizing the purge parameters of the Stratum PTC, a 1ppt detection level of the compounds was achieved.

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