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

Application Note

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 one part per trillion (ppt) 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 in order to aid in compound detection. The Stratum PTC was configured with a #1 trap and three different injection techniques were examined in order to determine the optimum method for injection.

Experimental-Instrument Conditions

The Stratum PTC and Aquatek 70 Autosampler were coupled 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. The Stratum PTC transfer line was configured to the GC column three different ways. The first was through a cryofocusing module, the second was through the Split/Splitless inlet of the GC and the third configuration was a direct column connection. 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 Tables 3 and 4 outline 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
Split: (Used for Split/Splitless Inlet Injection only)	10:1
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

Sales/Support: 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

Stratum PTC and AQUATek 70 Parameters Water Parameters for Cryofocusing Injection			
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 for a Cryofocusing Injection
Stratum PTC Parameters are in Blue

Cryofocusing Parameters are in Green

Stratum PTC and AQUATek 70 Parameters Water Parameters for Split/Splitless Inlet and Direct Column Injections			
Variable	**Value**	**Variable**	**Value**
Pressurize Time	0.60 min	Purge Time	10.00
Fill IS Time	0.00 min	Purge Temp	0°C
Sample Transfer Time	0.75 min	Purge Flow	100mL/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	Start of Desorb
Bake Drain Time	1.50 min	Desorb Preheat Temp	220°C
Bake Drain Flow	250mL/min	Desorb Drain	On

Sales/Support: 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

Valve Oven Temp	175°C	Desorb Time	2.00 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		
Pre-Purge Flow	0.0mL/min		
Sample Heater	On		
Sample Preheat Time	0.01 min		
Sample Temp	40°C		

Table 4: Stratum PTC and Aquatek Parameters for Split/Splitless Inlet and Direct Column Injections

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 and their respective injection techniques are outlined in Tables 5, 6 and 7.

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

A statistical determination of the MDL's was determined for both of the compounds with each injection technique by analyzing seven replicate standards of a 1ppt calibration standard. Furthermore, seven replicate standards of a 10ppt calibration standard were analyzed for each injection method in order to determine the precision and accuracy of the experimental conditions. Finally, three blanks were run after a high, 100ppt calibration standard in order to determine percent carryover for each technique. The detection limits, precision and accuracy data and percent carryover for each compound and their respective injection method are provided in Tables 5, 6, and 7.

Compound	Calibration Curve %RSD	Calibration Curve Linearity	Spike Level (ppt)	MDL (ppt)	Spike Level (ppt)	Precision (%RSD)	Accuracy (% Recovery)	% Carryover
Geosmin	3.94	1.000	1.00	0.114	10.00	5.80	103.89	0.13
2-Methylisoborneol	6.52	1.000	1.00	0.236	10.00	7.38	107.43	0.03

Table 5: Cryofucusing Injection Experimental Results Summary

Compound	Calibration Curve %RSD	Calibration Curve Linearity	Spike Level (ppt)	MDL (ppt)	Spike Level (ppt)	Precision (%RSD)	Accuracy (% Recovery)	%Carryover
Geosmin	3.99	1.000	1.00	0.123	10.00	3.61	94.22	0.44
2-Methylisoborneol	1.70	1.000	1.00	0.206	10.00	4.25	98.39	0.10

Table 6: Split/Splitless Inlet Injection Experimental Results Summary

Sales/Support: 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

Compound	Calibration Curve %RSD	Calibration Curve Linearity	Spike Level (ppt)	MDL (ppt)	Spike Level (ppt)	Precision (%RSD)	Accuracy (% Recovery)	%Carryover
Geosmin	7.18	1.000	1.00	0.220	10.00	2.10	100.01	0.11
2-Methylisoborneol	7.35	1.000	1.00	0.172	10.00	2.77	103.25	0.04

Table 7: Direct Column Injection Experimental Results Summary

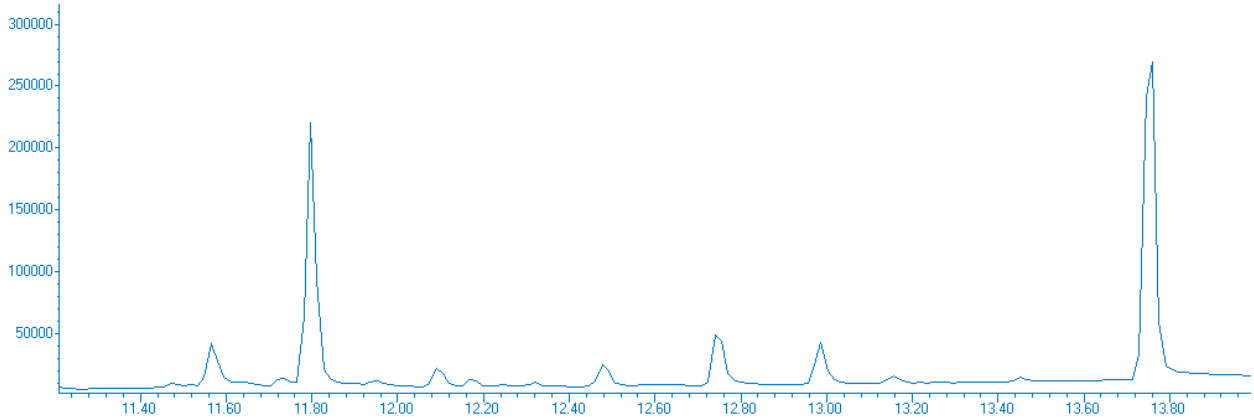

Figure 1: Total Ion Chromatogram of a 50ppt Geosmin and 2-Methylisoborneol Standard Using Cryofocusing Injection

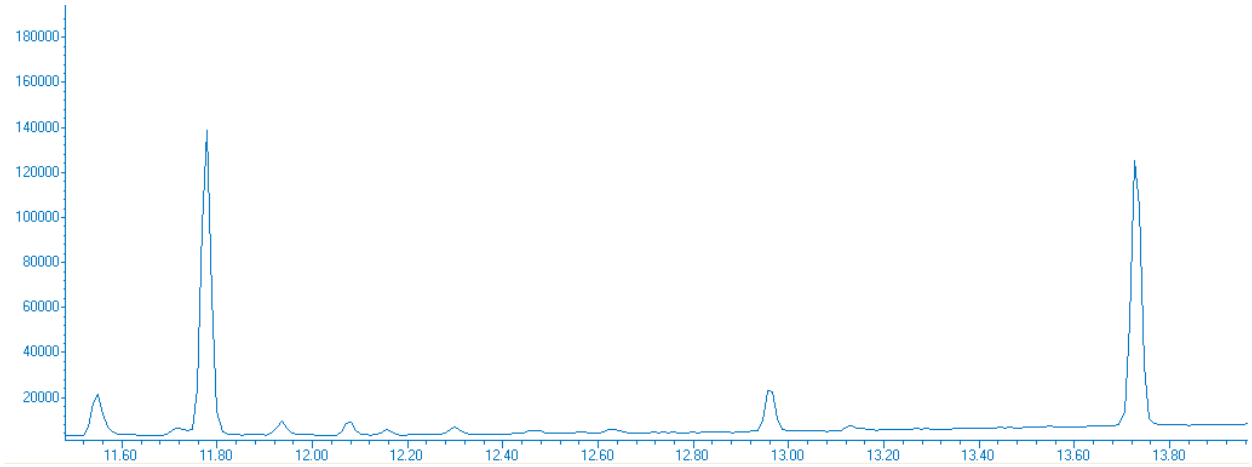

Figure 2: Total Ion Chromatogram of a 50ppt Geosmin and 2-Methylisoborneol Standard using Split/Splitless Inlet Injection

Sales/Support: 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

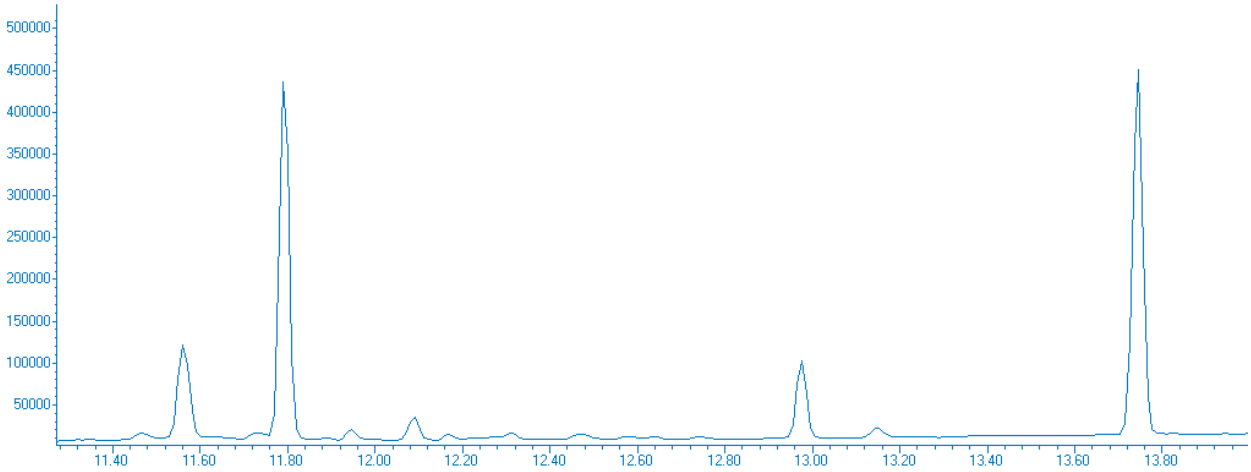

Figure 3: Total Ion Chromatogram of a 50ppt Geosmin and 2-Methylisoborneol Standard Using a Direct Column Injection

Conclusions

The Stratum PTC and AQUATek 70 configured with an Agilent GC/MS system operating in SIM mode performed very well in detecting Geosmin and 2-Methylisoborneol with all three injection techniques. The linearity of all of the curves was 1.000 for both compounds with a %RSD of below 10%. The system displayed excellent accuracy and precision results and the detection limits for the compounds were below 0.250ppt for all three injection methods. 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. Further analysis of the compound responses for all three techniques displayed that the direct column injection configuration had the optimum compound response, thus although the three techniques all showed consistent analytical results and comparable detection limits, I would recommend the direct column injection for Geosmin and 2-Methylisoborneol detection down to a 1ppt level.

Sales/Support: 800-874-2004 · **Main:** 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com