Low Level 2-Methylisoborneol and Geosmin Detection by Purge & Trap Sampling

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Introduction

Algal contamination in drinking water is becoming a common problem. The primary compounds that result from this contamination are 2-Methylisoborneol (2-MIB) and Geosmin. ese compounds cause a musty odor in water and since the odor of these compounds have a very low threshold for detection, small amounts of contamination can cause drinking water to taste and smell unpleasant. This application note will investigate the detection of 2-MIB and Geosmin down to a 1ppt level.

Discussion

Both 2-MIB and Geosmin are volatile tertiary alcohols that can be smelled and/or tasted at part per trillion levels. ue to the polarity of these compounds, it was difficult to purge them out of water. Consequently, developing a purge and trap method was essential in compound detection.

The part per trillion detection requirement also posed challenges for the GCMS analysis. The transfer line was coupled directly to the GC column, so the analytes were transferred onto the column similar to a direct injection. Sensitivity was optimized on the MS by employing the Selective Ion Monitoring (SIM) mode.

Experimental

The Centurion WS and Encon Evolution was the sampling system used for this analysis. The En on Evolution was equipped with a tenax trap (A trap) nd a 25ml sparge vessel. Coupled to the sampling system was a Shimadzu QP2010s GCMS. The GC was configured with a Restek Rxi-1ms 30m x 0.32mm x 0.5µm column and the MS was run in SIM mode. Experimental parameters for the purge and trap and GCMS systems are outlined in Tables 1 and 2 respectively.

Purge and Trap Concentrator	EST Encon Evolution
Trap Type	A
Valve Oven Temp.	150ºC
Transfer Line Temp.	150ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp	39ºC
Purge Time	12 min
Purge Flow	45mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	50mL/min
Dry Purge Time	3.0 min
Desorb Pressure Control	On
Desorb Pressure	5psi
Desorb Time	6.0 min
Desorb Preheat Delay	0 sec.
Desorb Temp.	230ºC
Moisture Reduction Trap (MoRT) Bake Temp.	210ºC
Bake Temp	230ºC
Sparge Vessel Bake Temp.	130ºC
Bake Time	10
Bake Flow	40mL/min
Purge and Trap Auto-Sampler	**EST Centurion WS**
Sample Type	Water
Sample Fill Mode	Syringe
Sample Volume	25mL
Sample Prime Time	3 sec
Loop Equilibration Time	5 sec
Sample Transfer Time	20 sec
Syringe Rinse	On/25mL
Number of Syringe Rinses	2
Sample Loop Rinse	On/25 sec
Sample Loop Sweep Time	40 sec
Number of Sparge Rinses	Syringe/2
Rinse Volume	25mL
Rinse Transfer Time	20 sec
Rinse Drain Time	50 sec
Number of Foam Rinse Cycles	1
Water Heater Temp.	85°C
Internal Standard Vol.	10µl

Table 1: Purge and Trap Parameters

GC/MS	Shimadzu QP2010S
Flow Control mode	Linear Velocity
Pressure	29.2 kPa
Total Flow	43.0ml/min
Column Flow	2.0ml/min
Linear Velocity	51.0 cm/sec
Purge Flow	1.0 ml/min
Column	Rxi-1MS 30m x 0.32mm I.D. x 0.5µm film thickness
Oven Temp. Program	40ºC hold for 2 min, ramp 16ºC/min to 160ºC, hold for 0.0 min, ramp 20ºC/min to 240ºC hold for 3 min
Ion Source Temp.	185ºC
Interface Temp.	180ºC
Solvent Cut Time	3.0 min
Event Time	0.30 sec
ACQ Mode	SIM
SIM ions 174 and 75	3.0 to 8.0 min
SIM ions 95, 107 and 108	8.0 to 9.5min
SIM ions 112, 125 and 126	9.5 to 16.5 min

Table 2: GCMS Parameters

A 50ppb working calibration standard was prepared in methanol and a calibration curve was run from 1ppt to 100ppt. Bromofluorobenzene (BFB) was chosen for the Internal Standard (IS). The concentration of the BFB was held constant at 5ppt throughout the study. The results of the calibration curve are listed in Table 3 and displayed in Figures 1 and 2. Next, a method detection limit (MDL) study was done; seven replicate samples were run at 1ppt in order to determine the MDLs of 2-MIB and Geosmin. Finally, 7 replicate samples were run at 10ppt and at 50ppt in order to show the precision and accuracy of the method at two different calibration levels. The results of the MDL and precision and accuracy studies are also listed in Table 3. A 50ppt chromatogram is displayed in Figure 3.

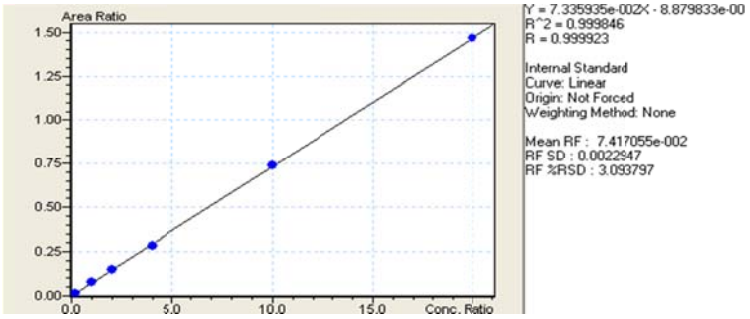

Figure 1: 2-MIB Curve

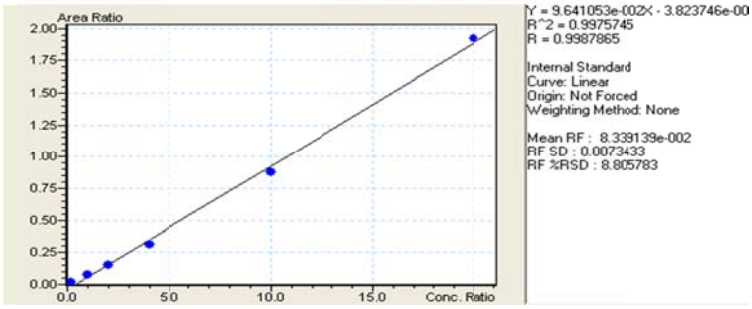

Figure 2: Geosmin Curve

Compound	Curve %RSD	Curve R^2	MDL (1ppt)	Precision, %RSD (10ppt)	Accuracy %Recovery (10ppt)	Precision %RSD (50ppt)	Accuracy %Recovery (50ppt)
Methylisoborneol	3.09	000	0.34	4.83	87.95	3.67	88.03
Geosmin	8.81	998	0.35	3.98	96.12	1.89	113.28

Table 3: Results Summary

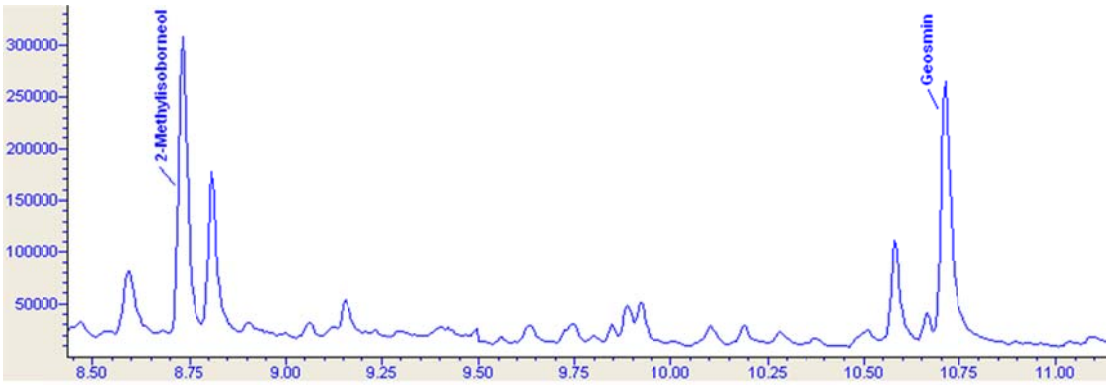

Figure 3: 50ppt Chromatogram of 2-MIB and Geosmin

Conclusions

The EST Analytical Centurion WS and Encon Evolution in conjunction with the Shimadzu QP2010s GCMS proved to be an excellent system for the analysis of 2-MIB and Geosmin. The sensitivity of the system was exceptional and the compound response was linear from 1ppt to 100ppt. Furthermore, the precision and accuracy of the two calibration points verified the reliability of the system.

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