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Determination of Geosmin and 2-MIB in Drinking Water by SPME-PTV-GC/MS

James Chang, Robert Biniakewitz, Gail Harkey,
Thermo Fisher Scientific, Inc.

This application describes the analysis of two key taste and odor compounds in drinking water using solid phase microextraction and gas chromatography-mass spectrometry to achieve detection limits of <0.4 ppt (0.4 ng/L) for each compound.

Geosmin (1,2,7,7-tetramethyl-2-norborneol) and 2-MIB (2-methylisoborneol) are compounds that cause musty, earthy odors in public water supply reservoirs and are produced mainly by blue-green algae (cyanobacteria) and actinomycete bacteria. Although these compounds have not been considered a health concern in public water supplies, they require removal. The odor threshold for these compounds is very low and humans can typically detect them in drinking water at 30 and 10 ng/L (ppt) for geosmin and 2-MIB, respectively (1,2). Therefore, concentrations of geosmin and 2-MIB are monitored in areas where they occur.

This note describes a technique for the analysis of these two compounds using automated headspace/solid phase microextraction (SPME) sampling in conjunction with the Thermo Scientific TRACE GC Ultra™ and Thermo Scientific DSQ™ II single quadrupole mass spectrometer, with assay linearity ranging between 1 and 1000 ppt (ng/L).

Experimental Conditions

Calibration and internal standards were obtained as a Drinking Water Odor Standards Kit (Supelco, Bellefonte, PA). Analytes in the standard solution kit were geosmin, 2-MIB, and 2-isopropyl-3-methoxy-pyrazine (IPMP), which was used as the internal standard. Care was taken during sample preparation to add the same concentration of methanol to all calibrators and samples, since proper sorption onto the SPME fibers is dependent on methanol concentration. A Thermo Scientific TriPlus™ Duo autosampler equipped with a SPME attachment (sold under license from Supelco) was used. The divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) 50/30 mm thick fiber (Supelco, Bellefonte, PA) was preconditioned prior to analyses.

Samples (10 mL) sealed in 20 mL headspace vials with 3g sodium chloride were incubated in the TriPlus oven at 60 °C for 30 minutes before desorption for 4 minutes in the GC inlet. A TRACE GC Ultra with a programmable temperature vaporizing (PTV) inlet was used, and the analytical column was a 5% phenyl 95% methyl silicone 30 m x 0.25 mm ID x 0.25 μm film thickness fused-silica capillary column. The GC was coupled to a DSQ II single quadrupole MS, operated in selected ion monitoring (SIM) mode. Data were acquired and processed with Xcalibur™ 1.4 software and Thermo Scientific EnviroLab™ Forms 2.0.

Results

Excellent linearity was obtained for both geosmin and 2-MIB,

with R2 values of 0.9994 and 0.9995, respectively, over a concentration range from 1 to 1000 ppt. Seven replicates were analyzed for method detection limits according to 40 CFR Part 136, Appendix B and showed the MDLs to be 0.191 and 0.358 ng/L for geosmin and 2-MIB. Average RSDs of the replicate injections were 0.56% for geosmin and 1.6% for 2-MIB. Excellent sensitivity and chromatographic performance were demonstrated across the calibration range for both MIB and geosmin (Figure 1).

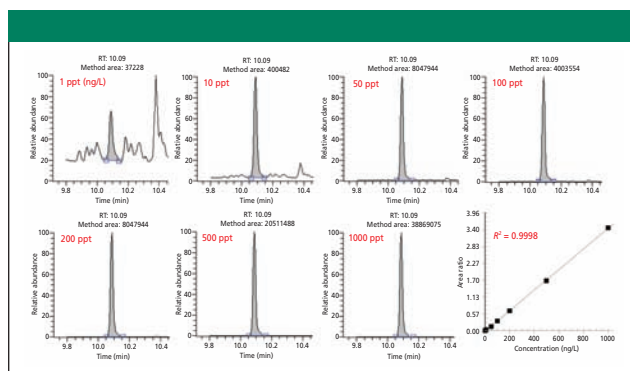


Figure 1: Geosmin, from 1 – 1000 ppt, including calibration curve

Conclusion

SPME configured on the TriPlus Duo, together with the TRACE GC Ultra gas chromatograph and DSQ II single quad MS, offers an easy method for the analysis of trace levels of odorants in drinking water samples. The sensitivity and wide linear dynamic range of the DSQ II GC/MS working in SIM mode permits accurate quantification over a broad concentration range. Method detection limits for geosmin and 2-MIB were less than 0.4 ppt – less than concentrations typically detected by humans. These detection limits were run using 1:10 split injections; lower detection limits may be possible with splitless injections. The optimized incubation geometry of the TriPlus SPME accessory ensures optimal fiber lifetime. Ease of data processing is enhanced by EnviroLab Forms, offering rapid data review and standard report formats.

References

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Thermo Fisher Scientific, Inc.

2215 Grand Avenue Parkway, Austin TX 78728
tel. +1 800 532 4752 fax +1 561 688 8731
analyze@thermo.com, www.thermo.com/gc