Geosmin and 2-Methylborneol by Headspace/Trap and SPME a Comparison of Techniques for Gas Chromatographic/Mass Spectrometry Analysis

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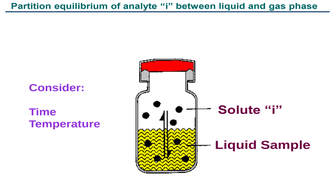
Introduction

Geosmin and 2-Methylisoborneol are of great interest in assessing water quality. The human olfactory sense can detect these compounds in low PPT ranges. Further the presence of these compounds can indicate dangerous Cyanobacteria or other infections in waterways. We have data from 2 techniques that easily achieve 1PPT levels with minimal sample preparation.

Both techniques allow for:

- Aggressive detection limits
- Very simple to use
- Robust to enhance uptime
- Non-detectable carryover
- Inert sample path
- Optimized accuracy
- Excellent repeatability
- Minimum sample prep-time
  - Avoid extractions
  - Eliminate losses
  - Protects analytical system



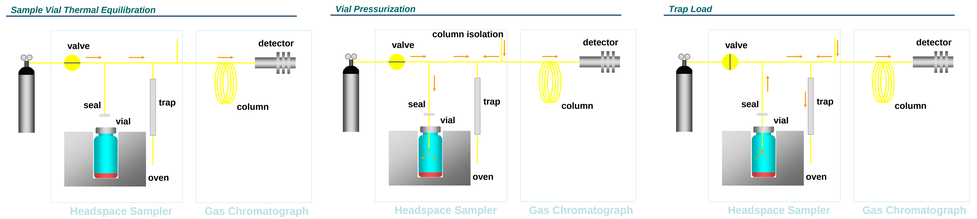
Both Techniques Utilize Headspace (HS) for extraction.
Solid Phase Micro Extraction (SPME) adsorbs compounds from the headspace of a sample onto a fiber, which is injected into the GCMS. HS-Trap collects the entire HS on a trap, which is then ballistically heated and injected into the GCMS.

Instrumentation

PerkinElmer Clarus 690 GC + SQ8T MS +Turbomatrix Headspace Trap

Maximum Capacity = 110 samples
Time to run 45 samples = 19 Hrs

How Automated HS-Trap Works and Method Timing

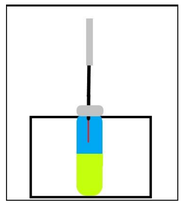


PerkinElmer Clarus 690 GC + SQ8T MS +Turbomatrix Multiprep with SPME

Maximum Capacity = 45 samples/tray (can have multiple trays). Time to run 45 samples = 30 Hrs

How Automated SPME Works and Method Timing

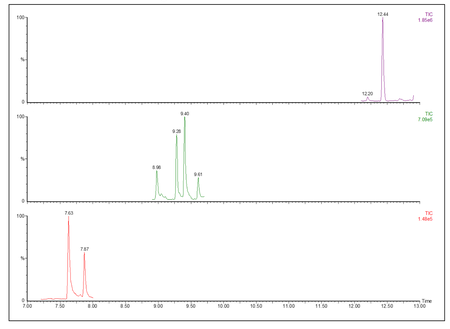
SPME Fiber is Inserted into Vial and Gently Agitated While at Thermal Equilibration



After Equilibration SPME is removed and injected into GC/MS

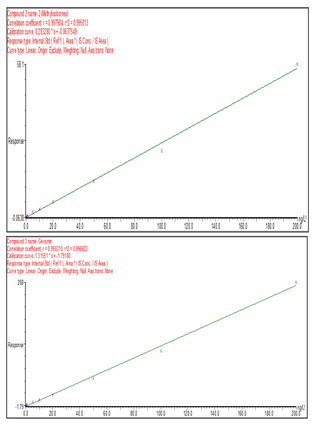
Chromatograms

The selected ion chromatograms of a 200 ng/L 2-MIB and Geosmin standards and a 25 ng/L internal standard. MultiPrep SPME

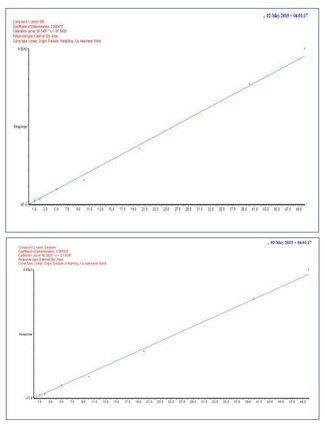


Calibration Curves

SPME



HS-Trap



Linearity, Precision and S/N

Precision (% RSD) was determined by making six analysis from individual vials of a 5ng/L (ppt) standard by the SPME method and 10ng/L (ppt) by the HS-Trap method. The calculations are based on raw data (no internal standard was applied in this calculation).

Technique	Compound	Linearity (r^2) (1-200 ng/L)	Precision %RSD @5 ng/L (SPME) and 10ng/L HS-Trap	Signal : Noise @ reporting limit 1.0ng/L
SPME	MIB	0.9981	14.04%	9:1
SPME	Geosmin	0.9978	4.44%	106:1
HS-Trap	MIB	0.9996	1.97%	106:1
HS-Trap	Geosmin	0.9997	3.26%	176:1

Summary

There is a growing demand for the analysis of trace levels of odorants, such as 2-MIB and Geosmin, in drinking water. The results attained in this study demonstrate the ability of the PerkinElmer SQ8 GC/MS to easily qualify and quantify 2-MIB and Geosmin in drinking water with both SPME and HS-Trap systems. The method has a wide linear range and high sensitivity, enabling accurate detection of these compounds in drinking water samples. While both SPME and HS-Trap are able to meet the required low level determination, the HS-Trap is able to produced better signal to noise levels and superior repeatability. This improved performance enables the HS-Trap to provide better results with enhanced reliability.