AppBrief

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No. 14

Determination of Odor Compounds in Fish Tissue by Stir Bar Sorptive Extraction (SBSE)

Highlights

- Solventless extraction of methyl isoborneol and geosmin with low levels of quantitation
- Avoiding alternate microwave assisted distillation method
- Instrumental setup eliminates the need for liquid nitrogen

Overview

Methyl isoborneol (MIB) and geosmin (GEO) are produced by certain algae and bacteria present in the environment. Increases in these microorganisms can lead to off odor and taste in farm raised fish which can bioaccumulate these compounds over time. Low levels of MIB and GEO cause an unpleasant earthy/musty smell. These compounds have a low odor threshold in the low ng/L range.

Ruan et al. [1] developed a method for determination of MIB and GEO in fish tissue using SBSE. The method was solventless, less labor intensive than the alternate microwave assisted distillation method and provided low levels of quantification for these compounds, MIB (1 ng/L) and GEO (1 ng/L). The method used liquid nitrogen for cryofocusing of the two compounds in the CIS 4 inlet at -120 °C on a glass wool filled liner. SBSE has also been used for the determination of these compounds in water [2-4].

In this AppBrief, we present data using the same methodology but using a Tenax-TA filled CIS 4 liner with Peltier cooling. This setup eliminates the need for liquid nitrogen.

Introduction

Stir Bar Sorptive Extraction (SBSE) is a solventless extraction procedure which can be used for trace level analysis of organic compounds in water. It is performed using a GERSTEL Twister® which is a PDMS coated stir bar. It is simply placed in a water sample, stirred, removed from the sample, dried and placed on the instrument for analysis by thermal desorption GC-MS.

Experimental

Instrumentation

A GERSTEL LabWorks Platform on an Agilent GC with MSD was used for the application. The GERSTEL LabWorks Platform is a universal system for sample introduction and offers unrivaled capabilities and flexibility to solve your critical challenges. Liquid, headspace and thermal desorption are all included without the need for additional bench space.

GERSTEL LabWorks Platform, here on an Agilent 8890 GC with 5977B MSD

Sample Preparation

Samples were prepared by placing one gram of ground salmon tissue and 9 mL of saturated NaCl solution in a 10 mL screw cap vial. The samples were extracted with conditioned Twister stir bars for 2 hours at 1000 rpm. The Twister stir bars were removed from the vial, rinsed in water, and blotted dry with a lint-free tissue. Two stir bars from the same sample were placed in an empty notched glass TDU tube and capped with a transport adaptor for analysis.

The samples were analyzed by thermal desorption GC-MS with analyte trapping on a glass wool filled liner at -120 °C.

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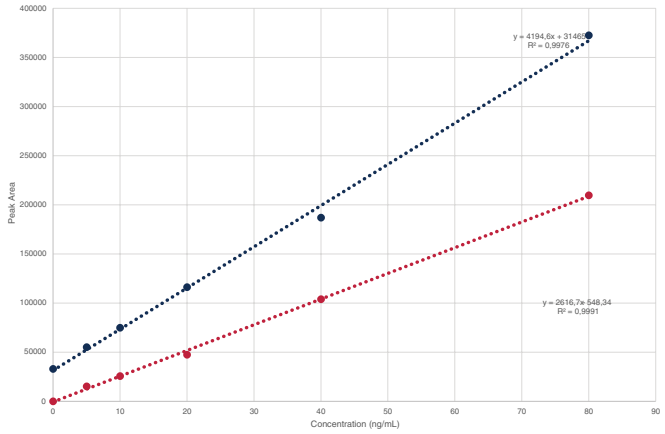
Results and Discussion

Seven samples were analyzed. The concentration of MIB and GEO in the samples was measured using a 6-point calibration curve.

Sample 1 had the highest concentration for MIB, while Sample 5 had the lowest. Samples 5 and 6 were the only samples that contained GEO.



Calibration curves for MIB (blue) and GEO (red)

Sample results

Sample	MIB [ng/g]	GEO [ng/g]
1	0.60	ND
2	0.43	ND
3	0.47	ND
4	0.30	ND
5	0.29	0.23
6	0.54	0.35
7	0.53	ND

In the paper describing this method [1], the authors utilized liquid nitrogen trapping at -120 °C on a glass wool filled liner. This is the preferred temperature since it ensures all compounds are trapped, leaving no doubt that any important compounds are missed. When the analytes to be determined are known, it is acceptable to optimize the method using higher trapping temperatures and/or adsorbents to speed up analysis time and eliminate the need for liquid nitrogen.

Area counts for alternate trapping of MIB and GEO

	Tenax 10 °C		Glass Wool -120 °C	
	MIB	GEO	MIB	GEO
1	17,470	70,069	22,160	61,564
2	17,855	67,201	23,371	62,733
3	19,198	78,164	21,974	72,467
Ave	18,174	71,811	22,502	65,588
RSD	5	8	3	9

The table shows data for MIB and GEO extracted from water by SBSE at a concentration level of 10 ng/mL and trapped at -120 °C on a glass wool filled liner and at 10 °C on Tenax-TA filled glass liner.

The results are equivalent for the two trapping methods used.

Summary

- GERSTEL Twister extraction allows extraction of MIB and Geosmin from fish tissue
- Simple and "green" extraction with no solvents
- Multiple samples can be processed simultaneously for increased throughput using the GERSTEL 20 stir plate
- Trapping can be accomplished without the need for liquid nitrogen

References

[1] E. Ruan, J. Aalhus, S. Summerfelt, J. Davidson, B. Swift and M. Juarez, *Determination of off-flavor compounds, 2-methyl isoborneol and geosmin, in salmon fillets by stir bar sorptive extraction-thermal desorption coupled with gas chromatography-mass spectrometry*, J. Chrom. A (**2013**) 1321: 133-136.

[2] D. Benanou, F. Acobas, M.R. de Roubin, F. David and P. Sandra, *Analysis of off-flavors in the aquatic environment by stir bar sorptive extraction-thermal desorption-capillary GC/MS/Olfactometry*, Anal Bioanal Chem (**2003**) 376:69-77.

[3] N.Ochiai, K.Sasimoto, M.Takino, S.Yamashita, S.Daishima, A.Heiden and A.Hoffmann, *Determination of trace amounts of off-flavor compounds in drinking water by stir bar sorptive extraction and thermal desorption GC-MS*, Analyst (**2001**) 126:1652-1657.

[4] S.Nakamura, N.Nakamura and S.Ito, *Determination of 2-methylisoborneol and geosmin in water by gas chromatography-mass spectrometry using stir bar sorptive extraction*, J Sep Sci (**2001**) 24:674-677.