



Sample Preparation Products

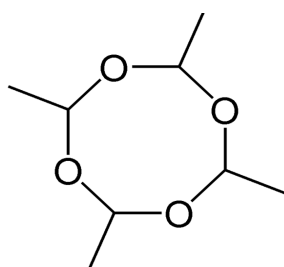
Application Note: ENV611

Extraction of Metaldehyde, Geosmin and 2-Methylisoborneol from Water

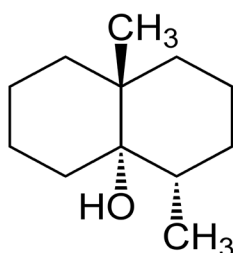
Introduction

Metaldehyde is a molluscicide (snail/slug killer) whose use has markedly increased in recent years as a result of changing agricultural practices. This has resulted in a number of exceedences of the 0.1µg/l EU pesticide standard in treated drinking waters.

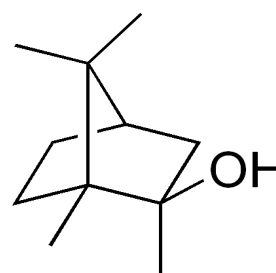
Geosmin and 2-methylisoborneol (MIB) are naturally occurring alicyclic alcohols which possess potent earthy/musty taste and odour tainting properties. They can occur in raw and potable waters as metabolites derived from various micro-organisms including actinomycete fungi and blue-green algae.



Metaldehyde



Geosmin



MIB

This Application Note describes a solid phase extraction procedure for the recovery of these compounds from raw and treated drinking waters, prior to analysis by GC-MS. The method uses TELOS™ ENV 200mg/6ml SPE Columns.

Sample Considerations

Analytes

Metaldehyde, geosmin and 2-methylisoborneol (MIB).

Matrix

Raw and treated surface and groundwaters.

Sample Preparation

Columns

TELOS ENV 200mg/6ml SPE Columns, part number 690-200M-006T.

Sample Pre-Treatment

The procedure is based upon a one litre sample but smaller volumes may be used if high concentrations of the analytes are likely to be present. No sample preservation is necessary but samples should be stored in the dark at 2-8°C for a period not exceeding 14 days.

Samples containing significant levels of particulates must be pre-filtered through a glass microfibre membrane prior to solid phase extraction.



Extraction of Metaldehyde, Geosmin and 2-Methylisoborneol from Water

Column Conditioning

On a suitable vacuum manifold, condition the column with ethyl acetate (2 column volumes), methanol (2 column volumes) and ultra pure water (1 column volume). Do not allow the pre-conditioned column to dry out.

Sample Application

Extract the analytes by filtering the samples under vacuum through the TELOS ENV SPE columns at a flow rate of approximately 30-35ml/min. Dry the loaded columns under vacuum (typically about one hour). The sorbent becomes a universal light brown colour when thoroughly dry.

Analyte Elution

Place the dried columns onto a suitable elution rig with collection tubes in place. Add 2ml of ethyl acetate to each column and allow to soak for at least 5 minutes. Drain the solvent to the level of the sorbent, then add a further 2ml of ethyl acetate and allow to elute.

Evaporation and Reconstitution

Carefully evaporate the eluates to ~100µl using nitrogen and a water bath at 40°C. Add 2ml of iso-octane to each sample, evaporate to ~500µl and add 50µl of a 2ng/µl solution of naphthalene-d8 in iso-octane to act as an internal standard. Centrifuge at 2500rpm for 4 minutes to remove precipitated material and carefully remove the supernatant for analysis.

Final Analysis

Analysis is performed using gas chromatography-mass spectrometry with cold splitless PTV injection and selected ion monitoring.

Reagents

1. Methanol.
2. Ultra pure water.
3. Ethyl acetate.
4. Iso-octane (2,2,4-trimethylpentane).
5. Naphthalene-d8.

Additional Comments

It is essential that eluates are not evaporated to dryness at any stage, otherwise loss of geosmin and MIB will occur.

See below for typical recoveries using this method.

COMPOUND	RECOVERIES (%)			
	Raw Surface Water	Treated Surface Water	Treated Groundwater	Ultra Pure Water
Metaldehyde	98	101	101	102
Geosmin	99	99	100	101
2-Methylisoborneol	97	99	101	102



Notes

1. This method has been performance tested using the standard UK water industry protocol (“A Manual on Analytical Quality Control for the Water Industry”, Water Research Centre plc., Medmenham. Cheeseman RV and Wilson AL, Rev Gardner MJ, 1989 NS30).
2. Recoveries were derived using spiking concentrations of 100ng/l for metaldehyde and 25ng/l for geosmin and MIB.
3. The information presented in this application note was supplied courtesy of Natasha Keitch & Ian Townsend, South West Water Ltd, Bridge Road, Countess Wear, Exeter, Devon, UK, EX2 7AA.

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