AppBrief

GERSTEL
MAKING LABS WORK

Determination of Phenols in Water According to DIN 38407-27 using Stir Bar Sorptive Extraction

Highlights

- Stir Bar Sorptive Extraction (SBSE) GC-MS allows sensitive determination of phenolic compounds in water samples
- Sample preparation is performed according to DIN 38407-27
- Liquid-liquid extraction is replaced by SBSE, avoiding solvent consumption
- Another step towards green chemistry

Introduction

DIN 38407-27 is a German standard method for the examination of water, wastewater and sludge for the determination of selected phenols in groundwater and leachate, aqueous eluates and percolates [1].

After derivatization of the analytes with acetic anhydride to their corresponding acetates and subsequent liquid-liquid extraction, the determination is carried out using gas chromatography/mass spectrometry (GC-MS).

For this application liquid-liquid extraction has been replaced with stir bar sorptive extraction (SBSE) using the GERSTEL Twister.

Experimental

Instrumentation

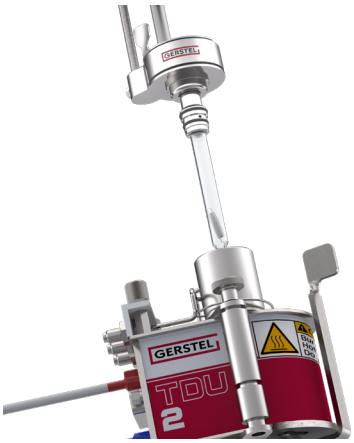
A GERSTEL LabWorks Platform on an Agilent 8890 GC with 5977B MS was used for the application. The GERSTEL LabWorks Platform is a universal system for sample introduction and offers unrivaled capabilities and flexibility to solve your critical challenges. Liquid, headspace and thermal desorption are all included without the need for additional bench space.

Sample Preparation

10 mL of each sample was pipetted into a 20 mL headspace vial, 100 mg ascorbic acid was added, the vial was shaken and left to stand for 15 min. Subsequently, 1.5 g sodium bicarbonate and 500 µL acetic anhydride were added and the samples were vortexed for 30 sec. Stir Bar Sorptive Extraction (SBSE) using the GERSTEL Twister was chosen as the technique for extracting the analytes from the solution. The Twister consists of a magnetic stir bar coated with glass and a sorptive phase (here PDMS). The Twister is placed in the sample like a stir bar and stirred for extraction. The sample analytes are distributed between the extraction and liquid sample phases, as in liquid-liquid extraction. The components to be determined accumulate in the PDMS phase.

Sample Introduction

The Twister is then placed in an empty thermal desorption tube and heated under carrier gas flow to release volatile and semi-volatile compounds. The analytes are re-focused in a cold injection system (CIS 4) for subsequent temperature-programmed transfer to the GC-MS system. There are no valves, transfer lines or active sites in the sample path, ensuring the best possible recovery of all analytes.



Twister introduction into GERSTEL TDU 2 thermal desorption unit

Non-volatile matrix residues are removed and discarded in the process, keeping the GC-MS system clean and stable.

GERSTEL
MAKING LABS WORK

Analysis

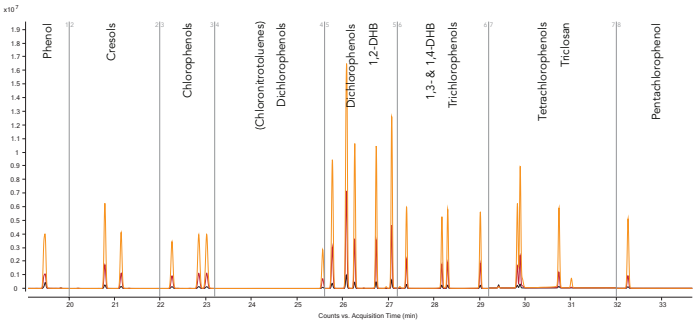
A calibration mixture containing phenol, cresols, mono-, di-, tri- and tetrachlorophenols, pentachlorophenol, dihydroxy benzenes, and triclosan was used to prepare a 3-point calibration curve.

Two real life samples were analyzed for these compound groups. The samples as well as the calibration mix was prepared according to DIN 38407-27.

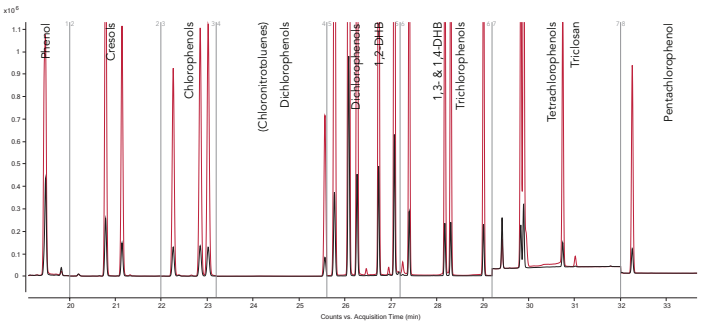
After removal from the sample the Twisters were thermally extracted at 280 °C. Detection was carried out using a mass selective detector from Agilent Technologies in SIM mode. All target analytes were detected.

Results and Discussion

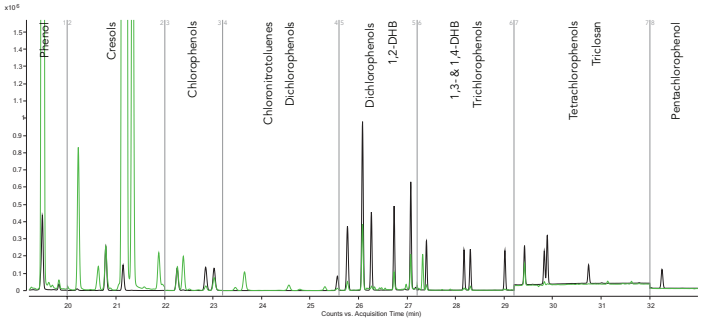
The following chromatograms show the resulting SIM traces for the calibration at 100, 20 and 2 µg/L. Note: chloronitrotoluenes were not included in the standard, but were identified at trace levels in the samples.



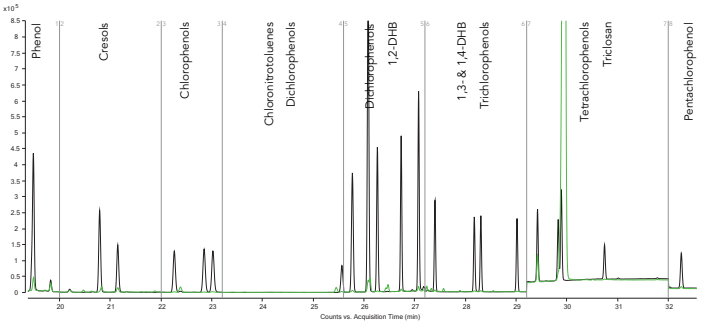
Standards overlay (100, 20 and 2 µg/L)



Standards overlay (zoom, 20 and 2 µg/L)



Overlay of 2 µg/L standard with sample 1



Overlay of 2 µg/L standard with sample 2

Conclusions

- SBSE is an excellent solventless extraction technique for the determination of phenols in water samples.
- Sample preparation method is identical to the procedure proposed in DIN 38407-27.
- The use of extraction solvents can be eliminated.
- Sensitivity complies with DIN 38407-27.

References

[1] DIN 38407-27:2012-10, German standard methods for the examination of water, waste water and sludge - Jointly determinable substances (group F) - Part 27: Determination of selected phenols in groundwater and leachate, aqueous eluates and percolates (F 27)