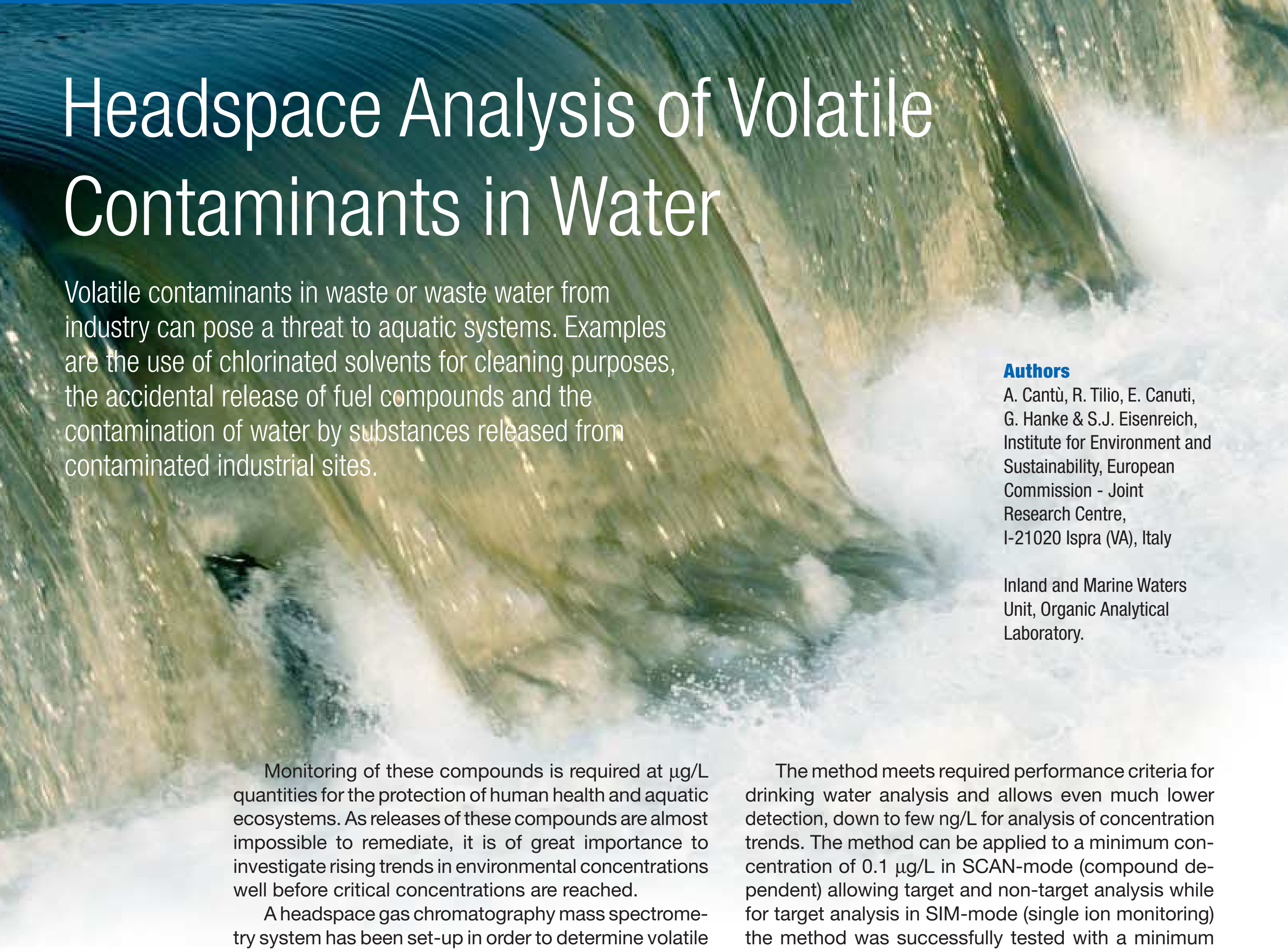

Headspace Analysis of Volatile Contaminants in Water

Volatile contaminants in waste or waste water from industry can pose a threat to aquatic systems. Examples are the use of chlorinated solvents for cleaning purposes, the accidental release of fuel compounds and the contamination of water by substances released from contaminated industrial sites.

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Monitoring of these compounds is required at µg/L quantities for the protection of human health and aquatic ecosystems. As releases of these compounds are almost impossible to remediate, it is of great importance to investigate rising trends in environmental concentrations well before critical concentrations are reached.

A headspace gas chromatography mass spectrometry system has been set-up in order to determine volatile compounds in water. After equilibration at elevated temperature a sample from the headspace is injected into a GC with Cooled Injection System (CIS). The mass spectrometric detection allows identification of contaminants and the isotope dilution quantification with deuterated or C_{13} internal standards. The system is highly automated and reduces the risk of cross-contamination to a minimum. The Headspace-GC/MS consists of an Agilent Technologies 6890 gas chromatograph with 5973N mass spectrometer, two GERSTEL CIS injection systems, a GERSTEL MultiPurpose Sampler (MPS 2) and a GERSTEL Thermal Desorption System (TDS A).

Table 1: Volatile compounds which were examinated with headspace gas chromatrography and detected with mass spectrometry.

Compounds	QS for In-Tr waters (WFD)*	LoD-HS-SIM	LoQ-HS-SIM
Dichloromethane	8.2 µg/L	0.010 µg/L	0.050 µg/L
Trichloromethane	3.85 µg/L	0.100 µg/L	0.250 µg/L
Carbon tetrachloride	7.2 µg/L	0.500 µg/L	1 µg/L
Benzene	16a µg/L	0.100 µg/L	0.200 µg/L
Trichloroethylene	10 µg/L	0.002 µg/L	0.005 µg/L
Tetrachloroethylene	10 µg/L	0.001 µg/L	0.005 µg/L
1,2,4 Trichlorobenzene	1.8 µg/L	0.005 µg/L	0.010 µg/L
1,2,3 Trichlorobenzene		0.005 µg/L	0.010 µg/L
Naphthalene	2.4 µg/L	0.001 µg/L	0.005 µg/L
Hexachlorobutadiene	0.003µg/L	0.002 µg/L	0.005 µg/L
1,2 Dichloroethane	1060 µg/L	0.500 µg/L	1 µg/L

** Quality standard referring to the annual average concentration for inland and transitional waters as proposed by the final outcome of the study: "Towards the Derivation of Quality Standards for Priority Substances in the Context of the Water Framework Directive", Lepper P. (2002), Fraunhofer-Institute Molecular Biology and Applied Ecology*

The method meets required performance criteria for drinking water analysis and allows even much lower detection, down to few ng/L for analysis of concentration trends. The method can be applied to a minimum concentration of 0.1 µg/L in SCAN-mode (compound dependent) allowing target and non-target analysis while for target analysis in SIM-mode (single ion monitoring) the method was successfully tested with a minimum concentration of 0.005 µg/L (compound dependent).

Figure 1: 50 ng/L hexachlorobutadiene measured in SCAN-mode. Ion 225 is used to quantitate also in sim mode (Figure 2).

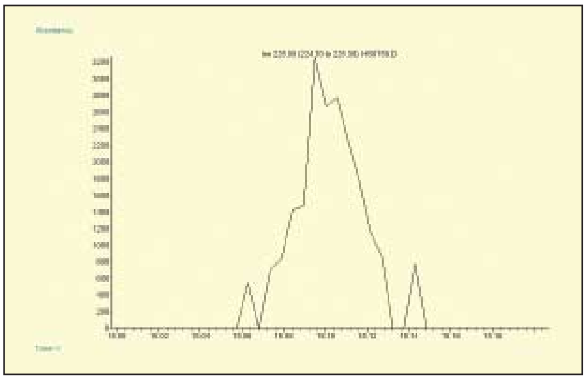
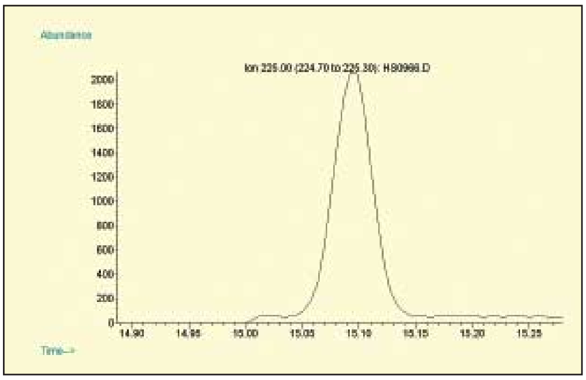
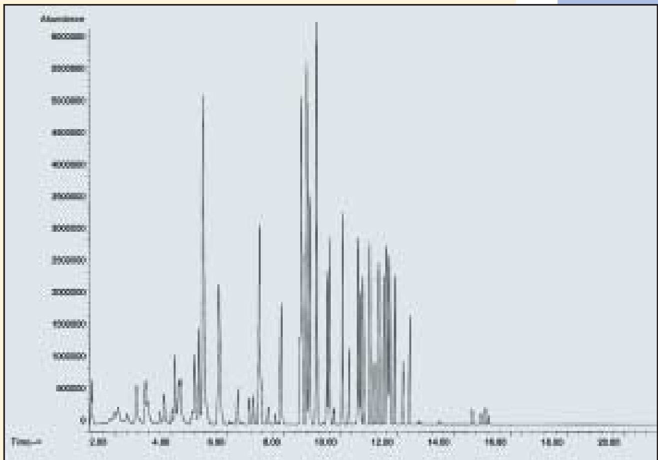


Figure 2: 50 ng/L hexachlorobutadiene measured in SIM-mode. It is evident that peaks are better defined and sharper.



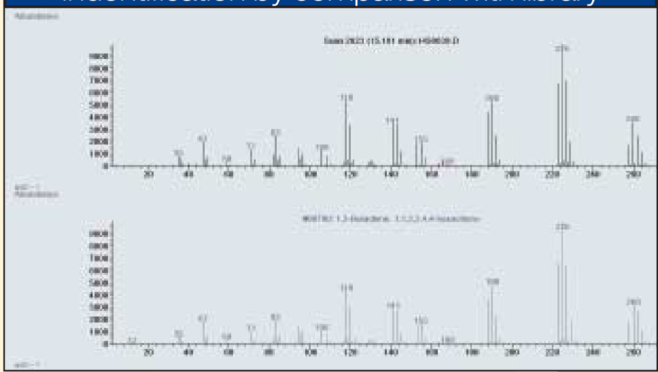
Analysis of 60 volatile compounds with headspace GC / MS

1) ?
2) Dichlorofluoromethane
3) Chloromethane
4) Vinyl chloride
5) Bromomethane
6) Chloroethane
7) Trichlorofluoromethane
8) Bromodichloromethane
9) cis - 1,2 - Dichloroethylene
10) t rans - 1,2 - Dichloroethylene
11) Methylene chloride
12) 1,1 - Dichloroethylene
13) tert -Butylmethylether
14) 1,1 - Dichloroethane
15) 2,2 - Dichloropropane
16) Chloroform
17) 1,1 - Dichloropropylene
18) 1,2 - Dichloroethane
19) Trichloroethylene
20) Bromochloromethane
21) 1,1,1 - Trichloroethane
22) Carbon tetrachloride
23) 1,2 - Dichloropropane
24) Dibromomethane
25) Benzene
26) Toluene
28) Tetrachloroethylene
29) Bromoform
30) Chlorobenzene
31) Styrene
32) p-Xylene
33) o-Xylene
35) 1,2 - Dibromonethane
36) 1,3 - Dichloropropane
37) 1,1,2 - Trichloroethane
38) t rans - 1,3 - Dichloropropylene
39) cis - 1,3 - Dichloropropylene
40) 1,1,1,2 - Tetrachlorethane
41) 1,1,2,2 - Tetrachloroethane
42) Ethylbenzene+m-Xylene
43) Dibromochloromethane
44) 1,2,3 - Trichloropropane
46) Isopropylbenzene
47) n-Propylbenzene
48) 2-Chlorotoluene
49) 4,-Chlorotoluene
50) tert -Butylbenzene
51) sec-Butylbenzene
52) 1,3-Dichlorobenzene
53) 1,4-Dichlorobenzene
54) 1,2-Dichlorobenzene
55) Bromobenzene
56) 1,3,5-Trimethylbenzene
57) 1,2,4-Trimethylbenzene
58) p-Isopropyltoluene
59) n-Butylbenzene
60) 1,2,4-Trichlorobenzene
61) Naphthalene
62) 1,2,3-Trichlorobenzene
63) 1,2-Dibromo-3-chloropropane
64) Hexachlorobutadiene



Currently 60 compounds are targeted

Indentification by comparison with library



Instrument parameters

MultiPurpose Sampler (MPS 2) (GERSTEL, autosampler)
Sample volume 1.5 mL
Incubation temperature 60 °C
Incubation time 20.00 min
Syringe temp. 70 °C

Cooled Injection System (CIS 4 PLUS) (GERSTEL)
Equilibration time 0.2 min
Initial temperature - 40 °C
Initial time 0.15 min
Rate 12 C / min
Final temperature 200 °C
Final time 3 min

5973 GC / MSD: HS / GC (GC 6890 Series, Agilent Technologies)
Iso time SB: 0 min
Iso temperature SB: 40 °C
Iso time 1: 10 min
Rate. 8 °C / min
Iso temperature 2: 200 °C
Iso time 2: 5 min
Zone temperature transfer line 230 °C
Capillary column: DB-VRX (J&W)
Column length: 30 m
Column i.d.: 0.32 mm
Film thickness: 1.8 µm
Glass vaporization tube filled with Tenax TA

HP 5973 GC / MSD: MSD 5973
Mass range 35 – 500
Solvent delay 1.00 min
Zone temperature MS Source 230 °C
Zone temperature Quad 150 °C

GERSTEL distribution news

GERSTEL is represented in more than 40 countries world-wide by our own sales and support organization and by a network of distributors. Our distributors are carefully selected according to their experience with analytical instrumentation and their ability to represent and support GERSTEL modules and solutions. Support is a critical factor in offering the best customer value as part of the overall solution. Our distributors can rely on the GERSTEL International Service Support at all times. Only recently, the list of countries with GERSTEL distributors has been extended to include **Egypt, Bahrain, Kuwait, Oman and Qatar.** For contact details, please see the distributor list in this magazine.

In order to provide the best possible support for our many customers in **The Netherlands**, GERSTEL products will be represented exclusively by *Da Vinci Europe BV* as of October 1, 2003. Centrally located in Schiedam, the Netherlands, Da Vinci Europe BV offers extensive experience in selling and supporting analytical instruments and in servicing Agilent products.

We are sorry to report the loss of our friend and long-time representative in **Poland**, Mr. Marek Daniewski Ph.D., from the company Chromtec. Our condolences and best wishes go out to Marek's family.

The new GERSTEL distributor in Poland is the company Perlan Technologies Polska Sp. z o.o. based in Warsaw. Perlan Technologies was formed from the former Agilent Technologies" organization in Poland and is an authorized Agilent Technologies distributor and service agent. We are certain that Perlan Technologies will provide outstanding support to the Polish GERSTEL customers.