Analysis of volatile halogen compounds in water

Application Note

Environmental

Authors

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Introduction

Gas chromatography using an Agilent CP-Sil 5 CB column separates nine volatile halogen compounds in a water sample.

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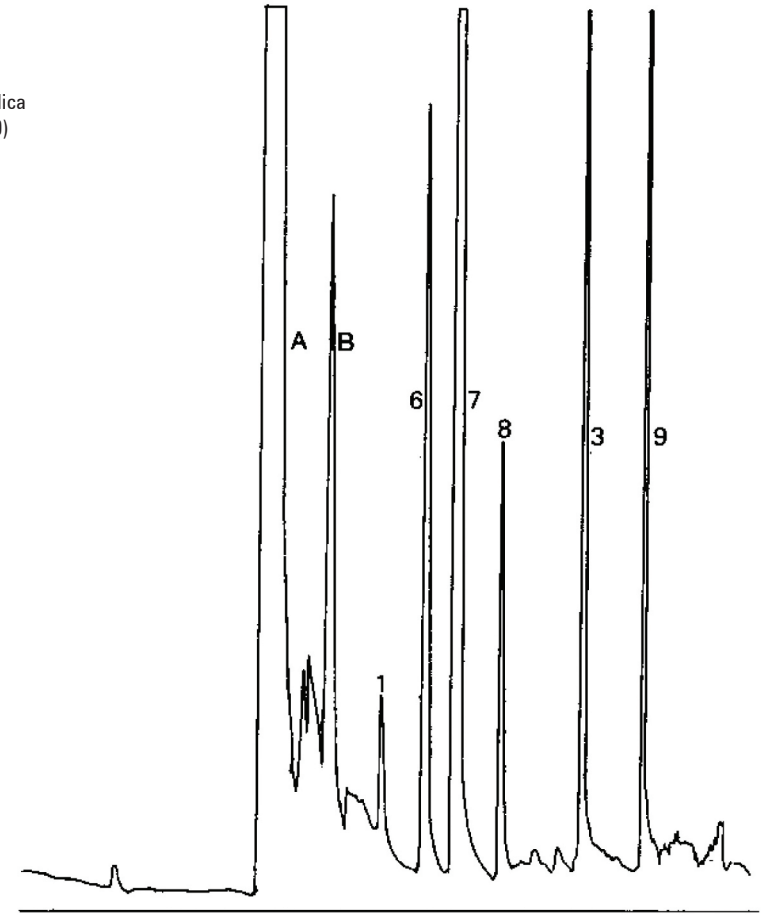
Conditions

Technique	:	GC-capillary
Column	:	Agilent CP-Sil 5 CB, 0.32 mm x 50 m fused silica WCOT CP-Sil 5 CB (1.2 µm) (Part no. CP7770)
Temperature	:	50 °C (11 min) 50 °C → 130 °C, 8 °C/min 130 °C, 1 min
Carrier Gas	:	He, 100 kPa, (1.0 bar, 14.5 psi)
Make-up Gas	:	N_2, 80 kPa, (0.8 bar, 11.6 psi)
Injector	:	Automatic headspace device; 1 mL, Split 1 : 5; 38 °C
Detector	:	ECD Ni^{63}, f = 5 kHz, 64 x
Peak Identification	:	see Table 1



Introduction

The analysis of volatile halogen compounds in water has been common practice in a number of laboratories for some time. A usual method of isolation is liquid/liquid extraction using petroleum ether (b.p. 40-60 °C). This method, however, is labour intensive and the risk of obtaining artifacts from the solvent is large. The analysis is carried out with a gas chromatograph using a Ni^{63} detector.

Another method described here is the static headspace method. However, a disadvantage of this method is the introduction of water vapour into the column. A column which is capable of separating volatile organic halogen compounds yet is not affected by water is a fused silica WCOT column with CP-Sil CB, film thickness 1.2 µm.

Instrumental

For the analysis a Carlo Erba 4130 gas chromatograph was used, equipped with an automatic headspace device and a Ni^{63} detector. A split injection (1 : 5) was applied to limit diffusion during injection (1 mL).

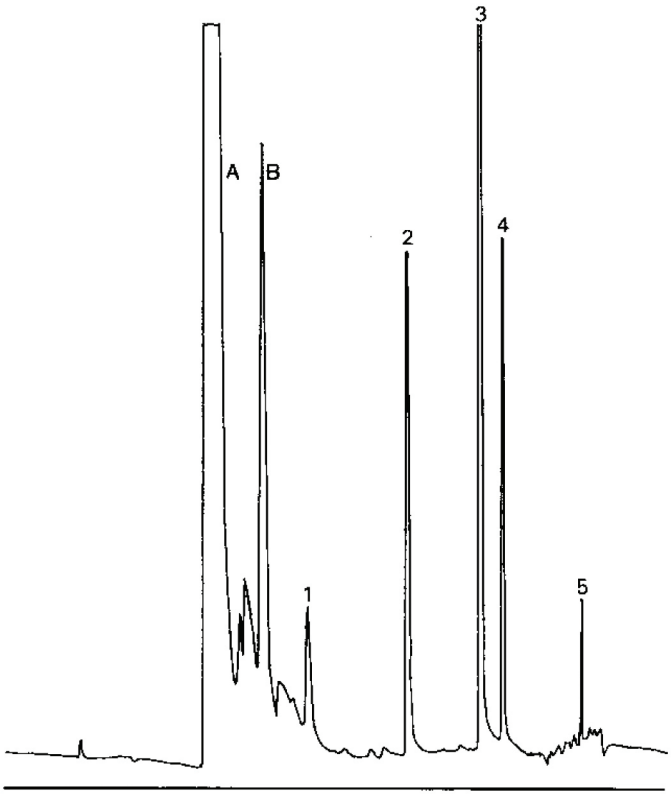
Results

The large water and air peaks were separated from the other components. Retention times were very reproducible (standard deviation < 0.02% for 20 analyses). The standard deviation of the signal from the determinated halogen compounds was 1-4%. The headspace temperature of 38 °C was selected to minimize the introduction of water vapor to prevent interference in the analysis. Detection level and reproducibility of a number of organic halogen compounds is given in Table 1.

Table 1

No. Peak	Conc. µg/L	Name	Dectection level (µg/L) 3x noise	Number of analyses	Standard deviation %
1	1.6	trichloromethane	0.1	30	1.0
2	2.0	dichlorobromomethane	0.05	37	2.0
3	2.0	trichlorobromomethane	0.05	30	3.1
4	2.5	dibromochloromethane	0.05	37	2.5
5	2.9	tribromomethane	0.1	37	2.0
6	1.3	1,1,1-trichloromethane	0.05	30	2.4
7	1.6	tetrachloromethane	0.01	30	2.0
8	1.5	trichloroethene	0.05	30	2.2
9	1.6	tetrachloroethene	0.01	30	4.1

A = water B = unknown (artifact from water)



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