



Determination of Organophosphates in Lake Water

Application Note

Authors

Julia Regnery
Goethe-Universität Frankfurt
Institut für Atmosphäre und Umwelt
AG-Umweltanalytik
60438 Frankfurt
Germany

Elisabeth Korte
Agilent Technologies, Inc.

Introduction

Chlorinated organophosphates are used as flame retardants in insulation foams, paints, coatings, plastics, and textiles while the non-chlorinated ones are mostly used as plasticizers. The compounds have been detected in air, sediment and soil, sewage sludge, streams and lakes. Particularly the chlorinated organophosphates are known to persist in the aquatic environment. Some of the compounds are supposedly neurotoxic or carcinogenic and have been reported to be toxic to aquatic organisms¹.

This application note shows the GC/MS analysis of chlorinated flame retardants as well as non-halogenated plasticizers extracted from lake water: tris(1-chloro-2-propyl)-phosphate (TCPP), tris(2-chloroethyl)-phosphate (TCEP) and tris(1,3-dichloro-2-propyl)-phosphate (TDCP) and the non-halogenated alkylphosphates tri-n-butylphosphate (TnBP), tri-isobutylphosphate (TiBP) and tris(2-butoxyethyl)-phosphate (TBEP).



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Sample Preparation

Due to the low concentration of the analytes in the water sample, an enrichment step is necessary. We chose a solid-phase-extraction (SPE) method which allows large volumes of water samples to be handled. In addition, SPE requires only a small amount of organic solvent for elution of the analytes from the cartridges. SPE is time-saving since several samples can be extracted simultaneously.

The filtered water sample (1.5 L up to 2.5 L) is extracted on Bond Elut PPL, a functionalized styrene-divinylbenzene polymer using the VacElut 20 cartridge processing station manifold. The high surface area of Bond Elut PPL is optimal for the extraction of polar and medium polar species from large volumes of water samples. Prior to the injection into the GC/MS system, 4 µg of the internal standard squalane is added to the extract.

SPE Conditions

Vacuum Manifold: VacElut 20
(part number 12234105)
Vacuum: 800 mbar
Cartridge: Bond Elut PPL,
100 mg sorbent
in 1 mL cartridge
(part number 12105003)

Condition cartridge with 1 mL methanol, 1 mL methanol/acetonitrile (1/1)
Apply 1.5-2.5 L water sample
Dry the cartridge using nitrogen
Elution with 3 x 333 µL methanol/acetonitrile (1/1)

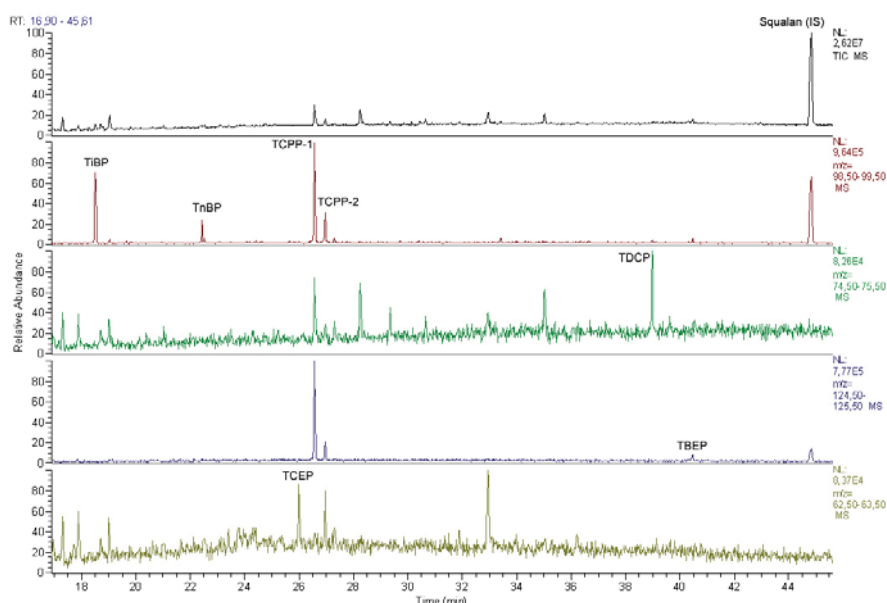


Figure 1. Total ion chromatogram and mass traces of organophosphates detected in lake water. The sample is extracted with Bond Elut PPL

GC Conditions

Capillary Column: 5% Phenyl, 0.25 µm,
30 m, ID 0.25 mm (similar
to Agilent FactorFour
VF-5ms; part number
CP 8944)
Injection: splitless injection of 1 µL
sample extract
Injector Temperature: 240 °C
Temperature Program: 1 min 80 °C, 4 °C/min
(linear) up to 300 °C
MS Conditions: EI-Modus, 70 eV
Mass Range: m/z 45-600, 1 scan/sec

Results

Figure 1 shows the total ion chromatogram (TIC) and the mass traces of an analyzed water sample taken from a lake. The quantification of the analytes is based on the mass trace m/z of a prominent ion of each compound using internal standard calibration. Limit of detection (LOD) is calculated to 1 ng/L-3 ng/L. The recoveries are in the range from 85-99%. Quantifying ions, LOD and recovery data of the organophosphates are listed in Table 1.

Table 1. Recoveries and LODs of organophosphates; extracted from the water sample with SPE

| Analyte | Recovery (%) | LOD (ng/L) | Quantifying ion (m/z) |
|--|--------------|------------|-----------------------|
| Tris(1-chloro-2-propyl)-phosphate (TCPP) | 91 | 1 | 99 |
| Tris(2-chloroethyl)-phosphate (TCEP) | 95 | 2 | 63 |
| Tris(1,3-dichloro-2-propyl)-phosphate (TDCP) | 99 | 1 | 75 |
| Tri-n-butylphosphate (TnBP) | 89 | 1 | 99 |
| Tri-isobutylphosphate (TiBP) | 85 | 2 | 99 |
| Tris(2-butoxyethyl)-phosphate (TBEP) | 93 | 3 | 125 |

Conclusion

The simple clean-up and enrichment with SPE has the advantage that up to 20 water samples can be extracted simultaneously without using complex apparatus. Bond Elut PPL has been proven to be a robust sorbent with high capacity for the extraction of polar and medium polar analytes.

References

¹ Online European Risk Assessment Tracking System

www.agilent.com/chem

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