Accelerated equilibrium sampling of hydrophobic organic chemicals in solid matrices: A proof of concept on how to reach equilibrium for PCBs within 1 day

Equilibrium sampling of hydrophobic organic chemicals (HOCs) is increasingly used to measure freely dissolved concentrations and chemical activities in sediments and soils. However, for the most hydrophobic chemicals (Log $K_{ow} > 6$) such equilibrium sampling requires often very long sampling times in the order of weeks to months. The aim of the present study was to explore two strategies for markedly increasing the HOC mass transfer from matrix to sampler with the overall goal to shorten equilibration times down to a few hours. Two Solid Phase Microextraction (SPME) approaches were thus developed and tested in sediment and soil contaminated by polychlorinated biphenyls (PCBs). In the first method, the SPME fiber was immersed directly in the aqueous suspension of the sample under vigorous agitation. In the second method equilibration took place via the headspace and was accelerated by elevating the temperature. Headspace-SPME at 80 °C provided fast equilibration within approximately 2 h without contacting the sample and thus avoiding fiber fouling. Both SPME methods were calibrated by passive dosing from preloaded silicone rods and yielded similar results, supporting the validity of HS-SPME at elevated temperatures on a proof of principle level. Finally, by using $^{13}$C labelled PCB standards, total concentrations were simultaneously measured, which in turn allowed calculation of matrix–water distribution coefficients.

General information
Publication status: Published
Organisations: Department of Environmental Engineering, Environmental Fate & Effect of Chemicals, Technical University of Denmark, University of Insubria
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Number of pages: 10
Publication date: 2019
Peer-reviewed: Yes

Publication information
Journal: Chemosphere
Volume: 237
Article number: 124537
ISSN (Print): 0045-6535
Ratings:
BFI (2019): BFI-level 2
Web of Science (2019): Indexed yes
Original language: English
Keywords: Direct immersion, Headspace, K determination, Mass transfer enhancement, Partitioning, Passive sampling
DOIs:
10.1016/j.chemosphere.2019.124537
Source: Scopus
Source ID: 85070521707
Research output: Contribution to journal › Journal article – Annual report year: 2019 › Research › peer-review