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# *In situ* benthic flow-through chambers to determine sediment-to-water fluxes of legacy hydrophobic organic contaminants<sup>☆</sup>



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## ABSTRACT

Contaminated sediment can release hydrophobic organic contaminants (HOCs) and thereby act as a secondary source of primarily legacy hazardous substances to the water column. There is therefore a need for assessments of the release of HOCs from contaminated sediment for prioritization of management actions. *In situ* assessment of HOC sediment-to-water flux is currently done with (closed) benthic flux chambers, which have a sampling time exceeding one month. During this time, the water inside the chamber is depleted of oxygen and the effect of bioturbation on the sediment-to-water release of HOCs is largely ignored. Here we present a novel benthic flux chamber, which measures sediment-to-water flux of legacy HOCs within days, and includes the effect of bioturbation since ambient oxygen levels inside the chamber are maintained by continuous pumping of water through the chamber. This chamber design allows for sediment-to-water flux measurements under more natural conditions. The chamber design was tested in a contaminated Baltic Sea bay. Measured fluxes were 62–2300 ng m<sup>-2</sup> d<sup>-1</sup> for individual polycyclic aromatic hydrocarbons (PAHs), and 5.5–150 ng m<sup>-2</sup> d<sup>-1</sup> for polychlorinated biphenyls (PCBs). These fluxes were 3–23 times (PAHs) and 12–74 times (PCBs) higher than fluxes measured with closed benthic chambers deployed in parallel at the same location. We hypothesize that the observed difference in HOC flux between the two chamber designs are partly an effect of bioturbation. This hypothesized effect of bioturbation was in accordance with literature data from experimental studies.

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## 1. Introduction

Legacy hydrophobic organic contaminants (HOCs) have over time reached aquatic environments through pathways such as direct emissions from point sources, atmospheric deposition or transport via run off from land. On a global scale, sediment acts as a sink for HOCs due to strong association of these hydrophobic substances to settling organic matter (Jönsson et al., 2003; Nizzetto et al., 2010). However, as primary emissions of legacy HOCs (such as

polychlorinated biphenyls; PCBs, polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans; PCDD/Fs) have been reduced, contaminated sediment may start acting as a secondary source of these regulated chemicals. Release of HOCs from sediment to water occurs via diffusive flux driven by higher chemical activity in the sediment compared to water, and mechanisms such as resuspension of particle-associated HOCs, advective pore water due to gas ebullition and bioirrigation (Meysman et al., 2006; Yuan et al., 2007; Cornelissen et al., 2008a; Armitage et al., 2009). For diffusive and advective processes to be significant, concentrations in the pore water need to be substantially different from the overlying bottom water.

The Baltic Sea was historically polluted by industrial activities along the coastlines, which caused high concentrations of e.g. PCBs, hexachlorobenzene and PCDD/Fs in sediments. Since the mid-1980s when concentrations peaked in coastal sediment, actions

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were taken and emissions strongly reduced (Assefa et al., 2014; Sobek et al., 2015). As sediments are still polluted, high chemical activity ratios between sediment and water may cause contaminated sediments to release legacy HOCs to water. For instance, high chemical activity ratios between pore water and bottom water for PCDD/Fs in coastal areas of the Baltic Sea were observed, demonstrating a potential release of PCDD/Fs from sediment to water (Sobek et al., 2014).

Several different methods exist and have been used for the assessment of flux of legacy pollutants from sediment to water, each with its advantages and disadvantages. In the end, the choice of method sets the limit for what is possible to determine, as there is an inherent trade-off between resembling the environment, on the one hand, and defining the boundaries and reducing uncertainties on the other. The diffusive flux of HOCs between sediment and water can indirectly be quantified by using Fick's first law of diffusion based on data on concentrations in bottom water and pore water (Eek et al., 2010; Liu et al., 2013; Lin et al., 2015), as chemical transport takes place along a concentration gradient. The same principle is frequently used in multimedia models describing sediment-water mass transfer of HOCs (Wania et al., 2000, 2006). Direct measurements of sediment-to-water flux have been done in micro- and mesocosm studies, where HOCs were collected by pumping the overlying water through a filter followed by a polyurethane foam (PUF) sorbent (Fisher et al., 1980; Larsson, 1985; Granberg et al., 2008; Hedman et al., 2009). The flux was thereafter calculated from the mass of substance collected on the sorbent, the area of the sediment surface, and time of incubation.

Benthic chambers offer a method for *in situ* determination of sediment-to-water fluxes of chemicals, and have recently been used to determine sediment-water fluxes of nitrogen and phosphorous in the Baltic Sea (Viktorsson et al., 2013; Ekeröth et al., 2016), where the flux was calculated based on the difference in concentration over time inside the chamber. Benthic chambers have also been used for direct measurements of HOC fluxes from sediment to water (Eek et al., 2010; Cornelissen et al., 2011, 2012). As it is difficult to measure HOC concentration changes over time in water inside the chamber, different systems for sediment-water flux measurements have been developed. *In situ* flux measurements of legacy HOCs have been performed by exposing an infinite sink (semi-permeable membrane device, SPMD, or a silicone polymer) to the water phase inside a closed chamber placed on the sediment bed (Eek et al., 2010; Cornelissen et al., 2011, 2012, 2016) and the HOCs are sorbed as they are diffusing from the seabed. This is a similar principle frequently used in *ex situ* measurements where water exposed to a contaminated sediment surface was pumped through a sorbent (Schaanning et al., 2006; Granberg et al., 2008; Josefsson et al., 2010). One drawback with the closed benthic chamber is that it does not allow for water flow through the chamber, which has the consequence that oxygen is depleted and anoxic conditions prevail during the main part of the deployment that often exceeds one month. Benthic organisms will therefore be severely disturbed and the natural effect of bioturbation on sediment-to-water flux is therefore not reflected in the measured flux.

Several studies have demonstrated that bioturbating organisms may increase the sediment-water flux of HOCs, with the observed flux being 0.25 (i.e. flux lowered due to bioturbation) to 25 times higher with bioturbation (Granberg et al., 2008; Hedman et al., 2009; Josefsson et al., 2010; Koelmans and Jonker, 2011). The effect of bioturbation on the sediment-water flux of HOCs depends on both benthic species composition and density, as organisms differ in their mode of reworking the sediment and in how deep they can penetrate in to the sediment (Granberg et al., 2008; Hedman et al., 2009; Josefsson et al., 2010; Kristensen et al., 2012). Two

bioturbation processes have been identified. i) moving of sediment particles vertically and horizontally, as organisms move within the sediment, and ii) moving of water within the sediment as organisms flush their burrows, termed bioirrigation (Kristensen et al., 2012). Both processes may increase the sediment-to-water flux of contaminants. Particle mixing may move contaminated sediment particles from deeper layers to the surface and thus enhance the chemical activity ratio between water and surface sediment (Josefsson et al., 2010). Bioirrigation can increase the transport of methyl mercury from pore water to bottom water (Benoit et al., 2009), and may accordingly increase the flux of dissolved HOCs. In areas where particle resuspension due to abiotic processes is negligible, bioturbation may be the most important process controlling the sediment-water flux of HOCs (Reible et al., 1996).

The aim of the present study was to improve the possibilities of measuring environmentally realistic fluxes of legacy HOCs from contaminated sediment to water. We therefore developed an *in situ* benthic flow-through flux chamber for the determination of sediment-to-water flux of HOCs under environmentally realistic, oxic conditions. The flow-through chamber has the advantage that the total effect of bioturbation on the flux may be assessed. Bioturbation may be of particular importance for the flux from sediment to water for legacy HOCs, as they in general occur at higher concentrations at sediment depths that may be reached by benthic biota (Sobek et al., 2015; Assefa et al., 2014). By constantly pumping bottom water through the chamber, the oxygen concentration inside the chamber is kept at ambient levels and the benthic organisms in the chamber remain undisturbed. The flux measurements by the flow-through chamber were compared with i) fluxes measured in parallel with closed *in situ* chambers, and ii) fluxes calculated based on chemical concentration gradients between pore water and bottom water. A dynamic mass balance model was developed and applied to estimate the effects of chamber design on the measured flux.

## 2. Materials and methods

Details on chemicals and materials, chemical analysis and instrumental set up and QA/QC are available as Supplementary material, Text S1, Text S2, and Text S3.

### 2.1. Flow-through chamber design

The benthic flow-through flux chamber (Fig. 1) was developed for assessments of sediment-to-water flux of legacy HOCs in areas with a strong concentration gradient between sediment and water. The principle of the chamber is to collect HOCs released from sediment to water on a sorbent at the chamber outlet, by pumping water through the chamber. This is the same principle as commonly used for *ex situ* flux measurements using retrieved sediment cores (Schaanning et al., 2006; Granberg et al., 2008; Josefsson et al., 2010). The experimental setup builds on the assumption that a steady state situation is quickly reached in the chamber where the net flux out of the sediment is equal to the flux out of the chamber, and the net flux from the sediment inside the chamber can therefore be estimated from the chemical mass captured on the infinite sink.

The chamber, made of stainless steel, is cylindrical with a diameter of 0.25 m, an area of 0.049 m<sup>2</sup> and a volume of 3.4 L. Water is pumped by suction from the outlet through the chamber with an approximate flow of 1 L h<sup>-1</sup> to retain aerobic conditions inside the chamber. First, water passes through a PUF sorbent at the chamber inlet to remove HOCs. At the chamber outlet, a second PUF sorbent captures HOCs released from sediment during deployment. A pre-combusted (450 °C, overnight) GF/F-filter (nominal pore size

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