

Measuring emerging organic micropollutants in the North Sea using high-resolution Orbitrap mass spectrometry: method validation and occurrence in harbour and open sea

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Abstract

The presence and long-term exposure of the aquatic environment to pharmaceuticals, pesticides and personal care products has awakened a growing concern amongst environmental policy makers and researchers. As many of these micropollutants are ubiquitously distributed, many researchers have focused on their occurrence in different matrices such as ground water, riverine water and wastewater. The marine environment has received noticeably less attention so far, given a.o. the challenges with respect to sampling and ultra-trace (\leq ppb-levels) concentrations of complex mixtures of micropollutants. New monitoring approaches are needed and the use of modern high resolution mass spectrometry offers promising potential for multi-residue ultra-trace analysis of unknown and quantification of known micropollutants.

The goal of this study was twofold. First, a novel full-scan SPE-UHPLC-Orbitrap-HRMS method was developed and validated. Second, a simultaneous monitoring of 89 target compounds – including pharmaceuticals, pesticides and personal care products – was performed in the Belgian Coastal Zone (BCZ). Triplicate samples were taken during spring 2016 from 2 different sites in the BCZ; i.e. the harbour of Zeebrugge and roughly 6 km offshore of Zeebrugge. The samples were extracted on an automated SPE-device using Oasis[®] HLB cartridges; separated through UHPLC on a Hypersil Gold column (50 mm x 2.1 mm, 1.9 μ m) prior to Q-Exactive[™] Orbitrap HRMS analysis.

Results of the validation and monitoring will be presented during the conference. Briefly, the method detection limits ranged from 0.01 ng L⁻¹ to 10 ng L⁻¹ for 68% of the target compounds. At a concentration of 15 ng L⁻¹, the method precision, expressed as relative standard deviation over triplicate injections, was less than 20% for 64 compounds. Next, a total of 14 and 18 compounds could be quantified offshore and in the harbour, respectively, with concentrations ranging from 0.35 ng L⁻¹ to 20 ng L⁻¹. A next sampling campaign is planned for the winter 2016-2017, from which the results will be included in the conference contribution.

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