Toxicity of coastal waters: use of a quick algal bioassay


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Optimization of the SPE step in the analysis of β-blockers and β-adenomimetics in natural water samples by SPE-GE technique

TU 081

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Environmental samples, especially sewage and marine water samples are complex and often contain interfering elements that can mask or interfere with the analysed pharmaceuticals. A direct ELSD analysis of these directly obtained samples may not be possible. An SPE is used to separate the desired components from the matrix of the sample. The choice of SPE is of critical importance as this will make sure that the SPE step is the most common sample preparation technique used in environmental analysis.

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Muststep fractionation based on normal phase SPE and reverse phase HPLC (RP-HPLC) for isolation of endocrine disrupting chemicals in environmental extracts

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"Directed Effect Analysis (EDA) approach aims to identify adverse pollutants by reducing the complexity of environmental matrices. Single hyperfractionation combined to bioassays is a useful tool to link direct chemical tolistings to the "classical" pollutants. However, although the emergence of promising chemical tools (e.g. Orcitrap), identification of unknown active chemicals is still time and cost consuming due to the complexity of each active fraction (e.g. mixture effect). Hence, further fractionation steps are often needed. The aim of this study was to develop and test the use of a first pre-fractionation step on SPE that will be followed by a RP-HPLC fractionation. First the separation of 12 EDC's have been evaluated with several elution conditions. Silica cartridges with 4 step elution - heptane, heptane/dichloromethane (50/50, v/v), ethyl-acetate and methanol/water (50/50, v/v) have been evaluated. For this purpose, three fractions (inside to outside) have been chosen for further investigations. For these conditions, recoveries were assessed for the mixture alone and for a blank sediment extract spiked with this mixture. Finally, a natural sediment known to exert estrogenic, PXR and AHR activity in vitro. Considering these conditions, Good mixture recoveries (74-110 %), were obtained. The fractionation F1 contained only the PCBs and the PAHs, whereas 4-tert-octylphenol, triphenyl phosphate and fenobrate were detected only in F2. Finally, steroids, benzo[a]anthracene and chlortetracyclines were found in F3 while F4 contained more polar chemicals.

Fractionation on natural sediment allows isolation of TCDD-like activity in F1 and F2, while PAH like activity was detected in F1, F2 and F3. Then estrogenic compounds were only detected in F2 and F3. Interestingly, the sum of the estrogenic activity found in these 2 fractions is higher than the activities found in the crude extract, which suggests an occurrence of anti-estrogenic chemicals. Finally, PXR-like activity was mainly detected in F3. This pre-fractionation protocol allows, in the present case study, the isolation of several biological active substances. Based on this first isolation directed hyperfractionation has then been undergone, RP-HPLC fractionation on 2 aminopyrine as a MCL substrate and a three-function sorbent (Stevenson Column for extraction of six β-blockers [acbutolol, atenolol, metoprolol, nadolol, propranolol, pindol), and two β-adenomimetics [terbutaline, salbutamol]) from natural water samples. Parameters such as pH of the loading samples, the amount and the kind of solvents used in conditioning, washing and eluting steps, were selected and optimized. The obtained extracts were evaporated to dryness, subjected to silylation using BSTFA, and finally analysed by GC-FID technique. The recovery of the analytes form natural water samples in the mentioned above SPE conditions will be discussed.

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Towards a common mass spectra database for the identification of unknowns in environmental samples

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"Screening or effect-directed analysis (EDA) is often a challenge on the way to the successful advancement of database search strategies and publishing of online databases has improved tenta-

The first step to unravel the complex interaction between algae and toxic pressure is to provide knowledge on chemical compounds causing phytotoxic effects. In this study we use passive sam-

Microcystins, highly toxic cyclic peptides, are a group of hepatotoxins produced by a number of aquatic species of cyanobacteria, such as Microcystis, Anabaena and Plankthotrix. Worldwide, concentrations in water have been measured in water bodies that are known to be contaminated with these agents and have no toxicological significance. Therefore, the fractionation of complex samples is required. One of the most commonly used methods of fractionation is liquid chromatography in combination with mass spectrometry (e.g. MS/MS, and LC-MS/MS) and open access mass spectra database including MS data from all instrument types and with so-

As predictable, endocellular toxin was 90-95% of the total microcystin content; the endocellular cystin extracellular concentration was never above the WHO limits for drinking waters (1 µg/L)