Toxicity of coastal waters: use of a quick algal bioassay


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abstract book
Optimization of the SPE step in the analysis of β-blockers and β-adrenomediators in natural water samples by SPE - GC technique

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Environmental water sample matrices, especially sewage and marine-water samples are complex and often contain interfering elements that can mask or interfere with the analysed pharmacals. In this study direct analysis these samples may not be possible. Additionally, the low concentrations in which the pharmacals are generally found cause that an initial stage of concentration and purification of the analytes prior to their analysis is necessary. The solid phase extraction (SPE) is the most common sample preparation technique used in environmental areas.

Choice of sorbent is a crucial in SPE because it can control such parameters as selectivity, affinity and capacity. This choice depends strongly not only on the target analytes and the interactions of the chosen sorbent through the functional groups of the analytes, but also on the kind of sample matrix and its interactions with both the sorbent and the analytes. This work describes the application of the different kinds of SPE sorbents: C18 bonded silica gel (Strata C18), copolymers (Oasis HLB, Strata X, and Lichrosorb EN), functionalised copolymers (Isolute ENV+), mixed-mode (mixed stationary phases such as MCX) and a safe drinking water SPE sorbent (Strata SCX). For extraction of six β-blockers (atenolol, atenolol, metoprolol, nadolol, propranolol, pindolol) and two β-adrenomediators (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 elution 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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TU 082

Mustard 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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3Effected Directed Analysis (EDA) approach aims to identify adverse pollutants by reducing the complexity of environmental matrices. Single hyperfractionation combined to biosay is failful to link direct active chemicals and to direct kinetic response to these "classical" pollutants. However, although the emergence of promising chemical tools (e.g. Or-bitrap²), 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 aims of this study was to develop and to 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 EDCs have been evaluated with several elution conditions. Silica cartridges with 4 step elution – heptane, hexane/dichloromethane (50/50, v/v), ethyl-acetate and methanol/water (50/50, v/v) – have been used. However, the isolation of the target compounds has 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 was used to test the fraction obtained from the pre-fractionation step. Good mixture recoveries (74–110 %), were obtained. The fractionation F1 contained only the PCBs and the PAHs, while 4-tert-octylphenol, triphenyl phosphate and fenobutrate were detected only in F2. Finally, steroids, benzophen A and clotrimazole 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 in 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 activity in the crude extract, which could be due to an occurrence of anti-estrogenic chemicals. Finally, PXR-like activity was mainly detected in F3. This first pre-fractionation protocol allows, in the present case study, the isolation of several biological activities. Based on this first isolation directed hyperfractionation has then been undergone, RP-HPLC fractionation and metabolomics. This process has already been chosen for further investigations.

TU 083

Towards a common mass spectrometry database for the identification of unknown environmental compounds

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5TU 084

The identification of unknown compounds in environmental samples isolated during non-target screening or effect-directed analysis (EDA) is often a challenge on the way to the successful outcome of the metabolomics study. Bioanalytical methods, based on liquid chromatography and mass spectrometry, are powerful tools in the identification of many compounds in recent years, but many chemicals and their transformation products are still not included in such databases. Improvements in the analytical technology and tools such as accurate and multidimensional mass spectrometry (e.g. Orbitrap - FTICR, Orbitrap) in combination with liquid chromatography and soft ionisation techniques such as electrospray ionisation (ESI) and atmospheric pressure chemical ionisation (APCI) allow the analysis of a broad range of compounds including polar one (or polar substances) and the restriction of the elemental composition in many cases to only a few fragment ions. However, most databases containing accurate mass spectra generally contain relatively few spectra and are not yet widely used, as many compounds relevant in environmental samples are still absent from these databases. One obstacle is the comparability of mass spectra generated with different settings, ionisation and spectrometric techniques due to increased instrument specificity, compared with the relatively reproducible ELMS spectra. Our aim is to improve the identification of unknown compounds in environmental samples using a common and open access mass spectra database including MS data from all instrument types and with so-called "phosphatized data evaluation tools. The web-based database MassBank [1] was developed within a metabolomics consortium [2] and is a possible tool to achieve this target. The database is free and allows the storage of a wide variety of spectra including ELMS, ESI-QToF-MSMS and ESI-FTI-CR-MS. Different tools are available to process the raw data and upload the data to MassBank including a spreadsheet based record editor for the addition of metadata.

References:

TU 085

Construction of a water toxicity sensor based on luminescent bacteria

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1 TU 086

Toxicity of coastal waters: use of a quick algal bioassay

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The last decades we have seen an increasing pressure on coastal waters by human activities. There are several chemical and biological detection methods, there is no suitable system yet for the real-time monitoring of toxicants in water, taking endpoints into account with human relevance. This gap may be partly bridged by a sensor that applies genetically modified bacteria that respond to specific groups of toxicants, called effector systems. The interactions of these effector systems with a promoter-gene that is known to be activated in case of exposure to certain types of toxicants, for example DNA damaging agents or heavy metals. This promoter gene is coupled to a luminescence gene-stage, so that luciferase is formed when the promoter is activated. The resulting production of light can then be detected and used as a measure of the toxic stress the bacteria were exposed to.

A new prototype of a flow-through sensor for on-line water monitoring based on these modified bacteria is being developed at KWR. The bacteria are fixed on an optic fiber or a glass slide and placed in a continuous water flow. The light generated by the bacteria is then measured by photomultiplier tubes. The current prototype is highly adjustable and allows of pH, temperature, flow, and pressure. Additionally, it is possible to add nutrients as well as to adjust compounds to the sample. This sensor prototype is being tested in both the laboratory and at monitoring stations along Dutch rivers. The ultimate aim is to develop a sensor that measures several types of toxicity and that can be applied continuously in the field, both at surface water inlets and at the distribution network.

TU 087

Dissolved and intracellular microcystins in lake waters during a Planktothrix rubescens algal bloom: HPLC quantification and crustacean acute toxicity test

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Microcystins, highly toxic cyclic peptides, are a group of hepatotoxins produced by a number of aquatic species of cyanobacteria, such as Microcystis, Anabaena and Plankothrix. Worldwide concentration in water has been measured and quantified for their identification and quantification. A massive seasonal development of Plankothrix rubescens in a reservoir destined for crop irrigation located in Southern Italy has lead to quantify algal toxin content in the water lake to verify the possible health risk. Microcystins dissolved into the water were separated from intracellular ones by filtering raw samples. Extracts were filtered by methanol/ water solutions after frozen/defrosten treatment over night. Water samples were concentrated and extracted by SPE-C18 cartridges. Toxicity content was determined and quantified using high performance liquid chromatography (HPLC-DAD). The only microcystin detected was [d-Asp3] microcystin-RR. It was identified by retention time and spectrum comparing with a certified performance liquid chromatography (HPLC-DAD). The only microcystin detected was [d-Asp3] microcystin-RR. It was identified by retention time and spectrum comparing with a certified