Toxicity of coastal waters: use of a quick algal bioassay
Sjollema, S.B.; Booij, P; van der Geest, H.G.; Laane, R.W.P.M.; Leonards, P.; Lamoree, M.;
Admiraal, W.; Vethaak, D.; de Voogt, W.P.

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Optimization of the SPE step in the analysis of β-blockers and β-adenornergic antagonists in natural water samples by SPE-EC technique

MF Cabau, A Michalak, N Migowska, M Kwiatkowski, P Šteposki, J Kumińska
Univ. of Silesia in Gdansk, GDANSK, Poland

Environmental sampling strategies, especially sewage and marine water samples are complex and often contain interfering elements that can mask or interfere with the analysed pharmacologial

TU 081

This work describes the application of the different kinds of SPE sorbents: C18 bonded silica gel (Strata C18), copolymers (Oasis HLB, Strata X, and Lichrolut EN), functionalized copolymers (Isolute ENV+), mixed-mode resins and bonded silica gel (MCMC) for the extraction of β-blockers (atenolol, atenolol, metoprolol, nadolol, propranolol, pindolol, and two β-adenornergic antagonists (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 styrlation 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

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

N Creuzet1, J-M Porcher1, H Budzinski2, S Aït-Aïssa1
1INERIS, VERNEUIL EN HALA TTE, France
2ISM, LPTC, T ALENCE, France

This work describes the outcome of these studies. Gas chromatography - electron ionisation mass spectrometry (GC-EI-MS) is useful to isolate known active chemicals and to direct chemical analyses to these "classical" pollutants. However, although the promise of emerging chemical tools (e.g. Orbitrap), identifcation 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 EDCs 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) led to a good separation of the tested compounds. However, the isolation of micronized compounds such as PCNs, PBDEs and PAHs 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 estrogentic, PXR and Ah receptor activity in vitro, was fractionated into activated compounds. Good mixture recoveries (74–110 %), were obtained. The fractionation F1 contained only the PCBs and the PAHs, while 4-tert-octylphenol, triphenyl phosphate and fenofibrate were detected only in F2. Finally, steroids, bisphenol A and clortalidone 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 activity in the crude extract, which indicates 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 activities. Based on this first isolation directed hyperfractionation has then been undertaken. RP-HPLC (pre-fractionated) and normal phase HPLC (NPH-C18) and a three-functional resins (PCA-MCM) were used for the separation of 35 EDCs.

TU 083

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

F Schuligoi, G Schymacki, S Neu mann, C Hug, C Gallampois, M Krauss, J Slobodnik, W Breck
1University of Amsterdam, AMSTERDAM, The Netherlands
2Ben Gurion University of the Negev, BEER SHEVA, Israel
3Biodetection Systems, AMSTERDAM, The Netherlands
4KWR Watercycle Research Institute, NIEUWEGEIN, The Netherlands

Long term exposure to persistent environmental contaminants pose a great threat to the health of ecosystems and the human population as a whole. 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 by emitting luminescence, linked to a sensor readout system with a promotor-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-set, 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 control of pH, temperature, flow, and pressure. Additionally, it is possible to add nutrients as well as test compounds to this 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 (pre-fractionated) and normal phase HPLC (NPH-C18) and a three-functional resins (PCA-MCM) were used for the separation of 35 EDCs.

TU 084

Construction of a water toxicity sensor based on luminescent bacteria

M Woutersen1, J Mink1, AP van Wesel1, A van de Geest1, RS Marks2, A Brouwer2, MB Herings1, JW R Watercycle Research Institute, NIEUWEGEIN, The Netherlands

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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 control of pH, temperature, flow, and pressure. Additionally, it is possible to add nutrients as well as test compounds to this 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 (pre-fractionated) and normal phase HPLC (NPH-C18) and a three-functional resins (PCA-MCM) were used for the separation of 35 EDCs.

TU 085

Toxicity of coastal waters: use of a quick algal bioassay

SB Sjollema1, P Booij2, H van der Geest1, R Laane1, P Leonards2, M Lamoree2, W Admiraal1, D Ten Kate3, Pde Voss1
1KWR Watercycle Research Institute, NIEUWEGEIN, The Netherlands
22M Engineering, VELTHOVEN, The Netherlands
3Ben Gurion University of the Negev, BEER SHEVA, Israel

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 control of pH, temperature, flow, and pressure. Additionally, it is possible to add nutrients as well as test compounds to this 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 (pre-fractionated) and normal phase HPLC (NPH-C18) and a three-functional resins (PCA-MCM) were used for the separation of 35 EDCs.

TU 086

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

V van den Hoogen, V Lijssens, E Janssen, GJ Hageman, L Giessner, JT Klok, A Persoons, A Goffin, PD Cotépi, CNR-IRSA, BRUGHERIO, Italy

Microcystis, highly toxic cyclic peptides, are a group of hepatotoxins produced by a number of aquatic species of cyanobacteria, such as Microcystis, Anabaena and Planktothrix. Worldwide, contamination in water has prompted the development of detection methods for their identification and quantification. A massive seasonal development of Planktothrix rubescens in a reservoir destined for crop irrigation located in Southern Italy has lead to quantify algal toxin content in lake water to verify the possible health risk. Microcystins dissolved into the water were extracted from water samples which were tested by HPLC and extracted by SPE-C18 cartridges. Toxin content was detected and quantified using high performance liquid chromatography (HPLC-DAD). The only microcystin detected was d-(Aep)-microcystin-RR. It was identified by retention time and spectrum comparing with a certified standard. Quantification was made by means of a calibration curve obtained at 238 nm. Microcystins extracellular concentration was never above the WHO limits for drinking waters (1 µg/L). The highest endocellular concentration (30.8 µg/L) of d-(Aep)-microcystin-RR was measured. As predictable, endocellular toxin was 90.95% of the total microcystin content; the endocellular