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A novel sample preparation procedure for effect-directed analysis of micro-contaminants of emerging concern in surface waters.

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Abstract

A novel sample preparation procedure relying on Solid Phase Extraction (SPE) combining different sorbent materials on a sequential-based cartridge was optimized and validated for the enrichment of 117 widely diverse contaminants of emerging concern (CECs) from surface waters (SW) and further combined chemical and biological analysis on subsequent extracts. A liquid chromatography coupled to high resolution tandem mass spectrometry LC-(HR)MS/MS protocol was optimized and validated for the quantitative analysis of organic CECs in SW extracts. A battery of *in vitro* CALUX bioassays for the assessment of endocrine, metabolic and genotoxic interference and oxidative stress were performed on the same SW extracts. Satisfactory recoveries ([70-130] %) and precision (<30%) were obtained for the majority of compounds tested. Internal standard calibration curves used for quantification of CECs, achieved the linearity criteria ($r^2 > 0.99$) over three orders of magnitude. Instrumental limits of detection and method limits of quantification were of [1-96] pg injected and [0.1-58] ng/L, respectively; while corresponding intra-day and inter-day precision did not exceed 11% and 20%. The developed procedure was successfully applied for the combined chemical and toxicological assessment of SW intended for drinking water supply. Levels of compounds varied from <10 ng/L to <500 ng/L. Endocrine (i.e. estrogenic and anti-androgenic) and metabolic interference responses were observed. Given the demonstrated reliability of the validated sample preparation method, the authors propose its integration in an effect-directed analysis procedure for a proper evaluation of SW quality and hazard assessment of CECs.

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