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A MULTIRESIDUE METHOD TO EVALUATE EMERGING MICRO-POLLUTANTS LEVELS IN WATERS

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ABSTRACT

During the last decade, the interest on the fate of emerging organic micropollutants, including pharmaceuticals and pesticides, in rivers and waste waters has constantly grown. Analysis of these pollutants at trace level in complex matrices is an analytical challenge that requires the development of reliable analytical methods. Based on the conclusions of national research programs on wastewater and sludge treatments and on expert opinions, indicative compounds typical of urban domestic pollution were selected according to i) their frequency of quantification in untreated wastewaters, secondary and tertiary wastewater effluents and in receiving rivers, ii) their toxic effect in aquatic and terrestrial ecosystems, iii) their large range of physico-chemical properties (log K_{ow}, pK_a) and chemical classes. Finally, 49 targeted compounds were selected, including 8 pesticides, 35 pharmaceuticals compounds (antibiotics, antidepressants, betablockers, anti-inflammatories, hypolipemiant and anticarcinogens) and 6 metabolites, to be part of a multiresidue method. This method also includes 4 priority substances (simazine, atrazine, diuron and isoproturon) of the Water Framework Directive (WFD, 2000/60/CE) and one watch-list substance (diclofenac) of the WFD daughter directive (2013/39/UE).

After collection in field, water samples were filtered and acidified. Samples were concentrated and purified by solid phase extraction (SPE). Twenty-two stable isotope labelled compounds were used as surrogates to overcome matrix effects generated during whole analytical procedure. Ultra high performance liquid chromatography (UHPLC) coupled to electrospray (both positive and negative ionization) tandem mass spectrometry was performed in the multiple reaction monitoring (MRM) mode, so molecule identity could be confirmed by two quantitation and confirmation transitions according to the EC Commission Decision (2002/657/CE), except for diclofenac.

We present accuracy, linearity and limits of quantification (LOQ) in intermediate fidelity conditions (intra-laboratory reproducibility), evaluated in real waters. As examples of application, we present concentration ranges for these compounds measured in rivers and wastewaters.



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