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Determination of seven pyrethroids and six pyrethrins in water by liquid chromatography/mass spectrometry

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Pyrethroids are the synthetic analogues of pyrethrins which were developed as pesticides from the extracts of dried and powdered flower heads of Chrysanthemum cinerariaefolium. They are increasingly used in agriculture due to their broad biological activity and slow development of pest resistance. Contamination of fresh-water ecosystems appears either because of the direct discharge of industrial and agricultural effluents or as a result of effluents from sewage treatment works; residues can thus accumulate in the surrounding biosphere [1, 2].
These substances, mostly determined by gas chromatography mass spectrometry (GC-MS) can be difficult to analyse due to their volatility and degradability. The purpose of this study is, as an alternative, to develop a fast and sensitive multi-residue method for the target analysis of 7 pyrethroids and the 6 natural pyrethrins currently used in water samples by liquid chromatography tandem mass spectrometry (LC-MS/MS). The compounds included in the study were acrinathrin, etofenprox, cyfluthrin, esfenvalerate, cyhalothrin, cypermethrin and flumethrin as pyrethroids and a commercial mix of pyrethrins containing Cinerin I, Jasmolin I, pyrethrin I, cinerin II, jasmolin II, pyrethrins II in different percentages.
As a preliminary step, the ionization and fragmentation of the compounds were optimized injecting individual solutions of each analyte at 10 ppm in the system, using a gradient elution profile of water-methanol both with 10 mM ammonium formate. The ESI conditions were: capillary voltage 4000 V, nebulizer15 psi, source temperature 300[U+25E6]C and gas flow 10 L min^{-1}. [M+H]+, [M+Na]+ ,[M+NH3]+ ,[M+NH4+]+ were tested as precursor ions. The most intense signal was for ammonium adduct for all compounds. The optimal fragmentor range for product ions were between 20 to 80 ev and the collision energy ranged between 5 to 86 ev.
The efficiency of the method was tested in water samples from Turia River without any known exposure to pyrethroids. At least three of the seven pyrethroids were detected.