

Development of a Stir Bar Sorptive Extraction/Liquid Desorption/Large Volume Injection-Capillary Gas Chromatography-Mass Spectrometry Method for Pyrethroid Pesticides in Water Samples

P. Serôdio¹, J.M.F. Nogueira^{1,2}

¹Chemistry and Biochemistry Department and ²Center for Molecular Sciences and Materials, Faculty of Sciences of the University of Lisbon, Campo Grande Ed. C8, 1749-016 Lisboa, Portugal; nogueira@fc.ul.pt

Stir bar sorptive extraction followed liquid desorption and large volume injection capillary gas chromatography with mass spectrometry detection (SBSE/LD/LVI-GC-MS), had been applied for the determination of ultra-traces of eight-pyrethroid pesticides (acrinathrin, cypermethrin, deltamethrin, esfenvalerate, fenpropathrin, fenvalerate, permethrin *cis* and *trans* isomers) in water samples. Instrumental calibration under selected-ion monitoring acquisition and parameters that could affect the SBSE/LD efficiency are fully discussed.

By performing systematic assays on 30 mL water samples spiked at the 0.10 µg/L level, it had been established that stir bars coated with 47 µL of polydimethylsiloxane, an equilibrium time of 60 min (750 rpm), 5% of methanol as organic modifier, and acetonitrile as back extraction solvent, provided the best analytical performance to monitor pyrethroid pesticides in water matrices. Good accuracy (81.8-105.0 %) and a remarkable reproducibility (< 11.7 %) were obtained, and the experimental recovery data allowed good agreement with theoretical equilibrium described by the octanol-water partition coefficients ($\log K_{O/W}$), with the exception of acrinathrin for which lower yields was measured. Additionally, excellent linear dynamic ranges between 25 and 400 ng/L ($r^2 > 0.994$), low quantification (3.0-7.5 ng/L) and detection (1.0-2.5 ng/L) limits were also achieved for the eight-pyrethroid pesticides studied.

The feasibility of the actual methodology had been successfully applied to tap and ground water matrices spiked at the 0.10 µg/L, which revealing the suitability for the determination of pyrethroid pesticides in real samples. The present method showed to be reliable, sensitive and with a low sample volume requirement to monitor pyrethroids at the ultra-trace level, in compliance with

international regulatory directives on water quality.

(SFRH/BD/9108/2002)

References

P. Serôdio, J.M.F. Nogueira, *Anal. Bioanal. Chem.*, 2005, 382, 1141-1151.