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Title :

Stir bar sorptive extraction coupled to liquid chromatography-tandem mass spectrometry for the determination of pesticides in water samples: method validation and measurement uncertainty

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Abstract

Stir bar sorptive extraction followed by liquid desorption and high performance liquid chromatography with tandem mass spectrometry (SBSE-LD-LC-MSMS) has been developed for the determination of 15 pesticides or selected metabolites from different families (herbicides, insecticides, fungicides) in surface water samples. The optimization of parameters that could influence SBSE-LD efficiency was carried out by means of experimental design. Optimized conditions were established as follows concerning extraction time, stirring speed, aqueous medium characteristics (ionic strength and polarity) and back desorption solvent and time, respectively: 3 h (800 rpm), addition of 10% of sodium chloride, no addition of methanol as organic modifier, and 15 min ultrasonic desorption in equivolume mixtures of acetonitrile-methanol. A specific and thorough cleanup procedure was developed and applied to each stir bar to avoid possible carry-over between consecutive extractions with the same stir bar. Pesticide quantification in water was achieved thanks to matrix matched calibration. Mean recoveries ranged from 93 to 101% (RSD < 17%, n=30). Validated limits of quantification in matrix were between 0.02 and 1 µg L^{-1}, depending on the compound. A specific experimental design was conducted to evaluate the measurement uncertainty, which was comprised between 13 and 51%, whatever the pesticide and the concentration level. The applicability of the SBSE-LD-LCMSMS method was evaluated by analyzing surface water samples and by comparing with conventional solid phase extraction-LC-MSMS procedure.

Keywords: stir bar sorptive extraction (SBSE), liquid chromatography-tandem mass spectrometry (LC-MSMS), pesticides, surface water, validation, measurement uncertainty