

Evaluation of a multiresidue method for measuring fourteen chemical groups of pesticides in water by use of LC-MS-MS

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Abstract European Council Directive 98/83/EC on the quality of water intended for human consumption brought a new challenge for water-quality control routine laboratories, mainly on pesticides analysis. Under the guidelines of ISO/IEC 17025:2005, a multiresidue method was developed, validated, implemented in routine, and studied with real samples during a one-year period. The proposed method enables routine laboratories to handle a large number of samples, since 28 pesticides of 14 different chemical groups can be quantitated in a single procedure. The method comprises a solid-phase extraction step and subsequent analysis by liquid chromatography-mass spectrometry (LC-MS-MS). The accuracy was established on the basis of

participation in interlaboratory proficiency tests, with encouraging results (majority $|z\text{-score}| < 2$), and the precision was consistently analysed over one year. The limits of quantitation (below $0.050 \mu\text{g L}^{-1}$) are in agreement with the enforced threshold value for pesticides of $0.10 \mu\text{g L}^{-1}$. Overall method performance is suitable for routine use according to accreditation rules, taking into account the data collected over one year.

Keywords Pesticides · Water · Multiresidue · Solid-phase extraction · Liquid chromatography-mass spectrometry · Uncertainties

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Notes: IAREN is an accredited laboratory under the ISO/IEC 17025:2005 for 165 analytical determinations and part of the NORMAN Network of Reference laboratories for monitoring of emerging environmental pollutants in the European Union.

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Introduction

The monitoring of pesticides in water samples has been an intense field of research in the past 10–20 years. Several methods have been proposed covering diverse chemical families and involving different analytical approaches in sample preparation [1–6], separation [7–11], and detection [1, 5, 6, 9–14]. Among these techniques, gas chromatography [2, 3, 8, 11] and liquid chromatography [9, 15–17] coupled to mass spectrometric detection have lately become the first choice tools in environmental analysis. Regarding sample preparation/concentration, solid-phase extraction (SPE) [4–6, 9, 15, 17] and solid-phase microextraction (SPME) [2, 3, 8, 11, 18] are the most widely described techniques in published works. The implementation of Directive 98/83/EC [19] brought a new challenge to laboratories in charge of water-quality control for human consumption in the European Union (EU). Besides establishing a maximum value of $0.10 \mu\text{g L}^{-1}$ for individual pesticides, the aforementioned directive demands $0.025 \mu\text{g L}^{-1}$ as detection limit for each individual compound. Additionally, the directive