A multi-residue method for trace analysis of pesticides in soil with special emphasis on rigorous quality control

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Pesticides are applied on agricultural fields to fight or prevent crop pests, diseases, and weeds in order to maintain crop yield. Whereas knowledge on chemical pollution in surface water bodies located in agriculturally influenced areas is constantly improving, information on soil contamination with agrochemicals is relatively scarce. Therefore, within the Swiss Action Plan for Risk Minimization and Sustainable Use of Plant Protection Products (AP PPP) one measure focuses on developing a long-term monitoring of pesticide residues in agricultural soils. As part of this measure, a multi-residue trace analytical method was developed to accurately quantify ~150 pesticides in (agricultural) soils with varying soil properties. An optimized quick, easy, cheap, effective, rugged, and safe (QuEChERS) approach was selected to extract pesticides from soil. Chemical analysis was carried out by liquid chromatography coupled to tandem mass spectrometry using electrospray ionization. Accurate quantification was guaranteed by the use of ~100 isotopically labelled internal standards to compensate soil specific matrix effects. In contrast to the common approach of method validation using soil samples spiked with pesticides shortly before the extraction, our method was additionally validated via an in-house prepared partly aged soil that contained all target analytes and via agricultural field soils with native pesticide residues. In this way, a highly sensitive (median method limit of quantification 0.2 ng/g), precise (median intra-day and inter-day precision both 4% based on field soils), and true ((i) quantified pesticide concentrations of the partly-aged soil remained stable during 6 months and were close to the initially spiked nominal concentration; (ii) median freshly spiked relative recovery: 103%; and (iii) participation in a ring trial: median z-scores close to one (good to satisfactory result)) analytical method is presented, which is already in use for routine monitoring as part of the AP PPP to evaluate soil quality.

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