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LC-MS/MS determination of 106 pesticides in nuts by microwave-assisted solvent extraction followed by a freezing-out combined with dispersive solid-phase extraction purification

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Sample pretreatment is still a major challenge to distinguish the low concentration of multiple pesticide residues from the complicated matrix interference with high accuracy and reliability of the results in dried and fat sample. A simple, selective and sensitive method to determine 106 pesticides in the complicated nuts sample by ultra-high pressure liquid chromatography coupled to a tandem mass spectrometer (UHPLC-MS/MS). After extracted by the high efficiency of microwave-assisted solvent extraction (MAE), the sample was purified by a two-step cleanup method combining freeze-out with dispersive solid-phase extraction. In our study, there are some considerable superiorities over other works: (1) 106 pesticides were determined simultaneously in nut, whereas other published works involved much less pesticides; (2) In the extraction procedure, MAE was used to improve the extraction efficiency; (3) Two-step cleanup procedure was used and eliminated efficiently the interference of matrix compounds and; (4) The analysis and pretreatment method was time shorten and labor intensity lighten. Guaranteed by the successful pretreatments to the dried and fat nut samples, average recoveries for 106 analytes ranged from 75.3% to 119.3%, (relative standard deviations <13%) at three concentrations levels. The limits of detection and quantification were in the ranges of 0.3-3.0 µg kg-1 and 1.0-10.0 µg kg-1 respectively. By analyzing 180 tree nut samples, this method was validated suitable for the determination of pesticides in nuts. We expect that it will provide a new technique for those who are interesting in solving the problems of extraction of pesticides from the low water and high fat complicated matrices.

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