


TOPIC: Investigation of Pesticide Metabolites in Food Matrices by LC/MS

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BIOGRAPHY

Dr. Felix Hernandez is Professor of Analytical Chemistry at the University Jaume I, Castellón, Spain. Director of the Research Institute for Pesticides and Water, he leads a 25-people research group in the field of analytical chemistry. His work is mainly focused on the development of analytical methodology for pesticide residue analysis in environmental, toxicological and food matrices. He is also Director of the Laboratory of Pesticide Residue Analysis (LARP) at this University, a GLP-certified laboratory performing the analytical phase of studies for registration purposes and setting up the maximum Residue Limits (MRLs) for pesticides in food samples. LARP is reference laboratory of the Ministry of Agricultural at Spain for GLPs studies related to pesticides.

Dr. Hernandez is the author of around 160 publications in specialized international journals and director of 12 doctoral theses, with most of his work dealing with analytical and environmental aspects of pesticides and metabolites. Most recent research lines are focused on the applications of hyphenated chromatography/mass spectrometry using both GC-MS and LC-MS with a variety of mass analyzers, as triple quadrupole, ion trap, TOF and hybrid QTOF. It is worth mentioning the development of analytical strategies for rapid screening of organic micro-pollutants in water by combined use of GC-TOF and LC-QTOF MS. Special attention has been paid in the last two years to the determination of pharmaceuticals (mainly antibiotics), pesticide transformation products and illicit drugs in surface and urban waste water. In the food safety field, the last work has been focused on mycotoxins and pesticide metabolites. Besides, rapid methodology has been developed to investigate the presence of melamine in food and diethylene glycol in toothpaste. Another research line is devoted to the investigation of anthropogenic contaminants in human breast adipose tissues by GC-MS with triple quadrupole and TOF MS.

ABSTRACT

Investigation of Pesticide Metabolites in Food Matrices by LC/MS

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Pesticide residue research in food is an important topic of modern analytical chemistry. Because of the toxicological properties of some pesticides could remain in their metabolites, designing efficient strategies for pesticide metabolite investigation in food is necessary. There are thousands of potential metabolites resulting from the metabolism of hundreds of parent pesticides in plants, in animals, and from different transformation processes in the environment. Some pesticide metabolites are specifically included in the residue definition and they are commonly determined in routine analysis, as they are included in analytical methods as target analytes. However, there are still differences in the international regulations when considering a metabolite as relevant. At present, the analyst has to face to many potential unknown compounds with little information available. In addition, many reference standards are commercially unavailable (especially if the metabolite is unregulated), being their discovering in samples still more difficult.

LC-TOF MS has shown a great potential for searching pesticide metabolites in food [1], especially when these metabolites are unexpected, due to the combined characteristics of high full-spectral sensitivity and increased mass accuracy and mass resolution, which facilitate the reliable identification of the compounds detected in samples. Moreover, hybrid QTOF MS provides additional features for confirmation [2], because of the useful information given by accurate mass product ion spectra after performing MS/MS experiments.

Investigation of parent-positive samples is an interesting option when searching for pesticide metabolites in food. In this lecture I will show the use of two different strategies that facilitate this task, avoiding the tedious manual selection of “relevant” peaks in the Total Ion Chromatogram given by TOF MS. The work has been performed using UHPLC coupled to (Q)TOF MS. One strategy is based on considering a common fragmentation pathway between the parent pesticide and their potential metabolites. After an enhanced fragmentation in the collision cell when acquiring in TOF MS mode (it was feasible thanks to the use of a QTOF instrument), this approach was applied to several fruit samples collected in the market. Parent positive samples were analyzed in this way, and it allowed the discovering of (non-target) post-harvest fungicide metabolites in lemon and insecticide metabolites in grape without previous selection of the compounds to be searched.

The other strategy is based on using specific software (MetaboLynx, Waters) and applying it to treated and untreated samples from field residue trials. This has led to the discovering of insecticide metabolites in treated samples. In both strategies, every metabolite detected after a first acquisition in TOF MS has been confirmed using QTOF and/or triple quadrupole (QqQ) instruments. Accurate masses given by TOF MS together with the valuable information on product ions given by QTOF MS/MS experiments were crucial for the safe identification of metabolites.

[1] F. Hernández, J.V. Sancho, M. Ibañez, S. Grimalt, *Trends Anal. Chem.* 2008, 27, 862.

[2] S. Grimalt, O.J. Pozo, J.V. Sancho, F. Hernandez, *Anal. Chem.* 2007, 79, 2833.