Accepted Manuscript

Title: EVALUATION OF SUPERCIRTICAL FLUID CHROMATOGRAPHY COUPLED TO TANDEM MASS SPECTROMETRY FOR PESTICIDE RESIDUES IN FOOD

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PII: S0021-9673(18)30232-2

DOI: https://doi.org/10.1016/j.chroma.2018.02.048

Reference: CHROMA 359227

To appear in: Journal of Chromatography A

 Received date:
 22-12-2017

 Revised date:
 17-2-2018

 Accepted date:
 22-2-2018

Please cite this article as: Víctor Cutillas, María Martínez Galera, Łukasz Rajski, Amadeo R.Fernández-Alba, EVALUATION OF SUPERCIRTICAL FLUID CHROMATOGRAPHY COUPLED TO TANDEM MASS SPECTROMETRY FOR PESTICIDE RESIDUES IN FOOD, Journal of Chromatography A https://doi.org/10.1016/j.chroma.2018.02.048

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ACCEPTED MANUSCRIPT

EVALUATION OF SUPERCIRTICAL FLUID CHROMATOGRAPHY COUPLED TO TANDEM MASS SPECTROMETRY FOR PESTICIDE RESIDUES IN FOOD

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Highlights

- Analysis of pesticide residues in fruits and vegetables with Supercritical fluid chromatography.
- Reduction of matrix effects in complex matrices.
- Improvement of sensitivity of polar/acidic pesticides.
- Short run times.

Abstract

Supercritical fluid chromatography coupled to triple quadrupole mass spectrometry has been evaluated for pesticide residues in food. In order to check its advantages and limitations it was developed a method to identify and quantify 164 pesticides in three different matrices (tomato, orange and leek). A carbon dioxide gradient with methanol (containing 1mM ammonium formate) was used allowing a flow rate of 1.5 mL/min that made the total run time of 12 min without any problem of overpressure. Addition of a post column flow $150\mu L/min$ of Methanol with ammonium formate/ formic acid was necessary to improve the ionization.

The matrix effect study revealed that the percentages of pesticides with irrelevant matrix effect (suppression lower than 20%) was 99% in tomato, 87% in orange and 62% in leek, whereas significant suppression (higher than 50%) was not found in tomato and only 1% of the compounds in orange and 3% in leek. These results compare favorably with that typically obtained in LC-MS/MS. The absence of water in the mobile phase, also provided some important advantages regarding LC-MS/MS as (i) higher retention of polar compounds in the column, which elute with high sensitivity and good peak shape and (ii) a general increase of the sensitivity of the analysis, consequence of the high ionization and ion extraction efficiency.

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