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Title: Validation and application of analytical method for glyphosate and glufosinate in foods by liquid chromatography-tandem mass spectrometry

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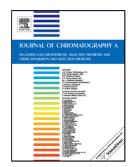
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## ACCEPTED MANUSCRIPT

Validation and application of analytical method for glyphosate and glufosinate in foods by liquid chromatography-tandem mass spectrometry

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#### **Highlights:**

- A combination of several steps makes the method more sensitive and selective.
- Validation with the accuracy profile was done for glyphosate and glufosinate.
- Method was applied to the analysis of 136 food samples and to routine analysis.

Abstract: A reliable and sensitive method was developed for simultaneous determination of glyphosate and glufosinate in various food products by liquid chromatography-tandem mass spectrometry. Based on extraction, derivatization with 9-fluorenylmethylchloroformate and purification on solid phase extraction column, quantification was done by using isotopic-labeled analytes as internal standard and calibration in matrix. Good selectivity and sensitivity were achieved with a limit of quantification of 5 µg/kg. The recoveries of these two pesticides ranged from 91 % to 114 % with inter-day and relative standard deviation of 3.8 to 6.1 % in five matrices of cereal group spiked at 5, 10, and 20 µg/kg. An accuracy profile was performed for method validation, demonstrating the accuracy and precision of the method for the studied food groups. The verification results in expanded food groups indicated extensive applicability for the analysis of glyphosate and glufosinate. Finally, the developed method was applied to analyze 136 food samples including milk-based baby foods from the French Agency for Food, Environmental and Occupational Health & Safety. Glyphosate residues were detected in two breakfast cereal samples (6.0 and 34 µg/kg). Glufosinate residues were found in a sample of boiled potatoes (9.8 µg/kg). No residues were detected in the other samples, including milk-based baby foods with limits of detection ranging from 1 to 2 µg/kg. The method has been applied for routine national monitoring of glyphosate and glufosinate in various foods.

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