

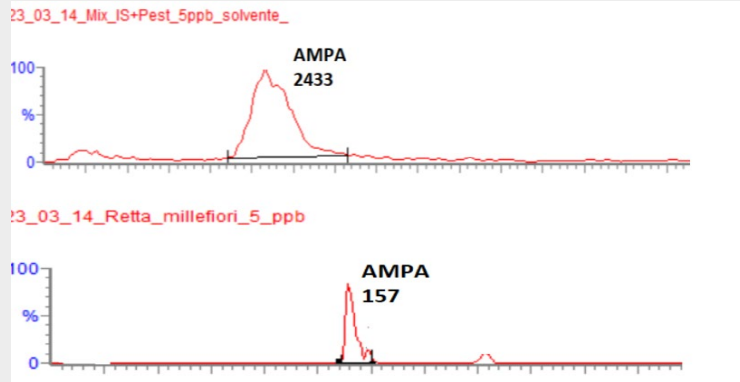


Development of a LC-MS/MS multi-residues method for the analysis of glyphosate, glufosinate and their metabolites in honey

Background and Objective: In recent years, concern about the effects of glyphosate and glufosinate on both human health and the environment has increased worldwide; the results of scientific studies to date and the opinions of regulatory agencies on health effects are controversial. Furthermore, the data on contamination levels of honey and hive products are still scarce. Due to their physico-chemical characteristics, LC-MS/MS analysis of polar pesticides still represents an analytical challenge. The aim of one of our latest ongoing activities is to develop and validate a multi-residual method for the detection and quantification of glyphosate and glufosinate residues and their metabolites in honey.

Materials and Methods: QuPPE-PO-Method proposed by the EU reference laboratories for residue of polar pesticides (EURL-SRM) was tested as first approach, other methods were tested for the determination and quantification of glyphosate, glufosinate, AMPA, N-acetyl AMPA, N-acetyl glyphosate, N-acetyl glufosinate, MPPA by LC-MS/MS in three types of honey (mixedflowers, acacia and honeydew).

Results: The method currently being developed and optimized can extract a wide range of polar analytes, the extracts may therefore contain high concentrations of non-target substances that can cause the suppression of target ions, affecting the sensitivity of the method. However, good analytical performance in terms of linearity, recovery, precision and accuracy in all three matrices for 5 of the 7 analytes were achieved. AMPA and glufosinate were hardly detectable at lower concentrations of the established range, showing 50-99% ion suppression due to the matrix effect. In mixed-flowers and acacia honey, the ionic suppression was slightly lower for glufosinate than for honeydew, which proved to be the more complex matrix.



Conclusions: At this preliminary stage, the method showed good analytical performance in line with EU Reg. 808/2021 for 5 of the 7 analytes. AMPA and glufosinate were found to be hardly detectable in the concentration range examined due to a strong matrix effect.

Future Proposal: Further tests are in progress to improve sample purification in order to increase the recovery of AMPA and glufosinate in the examined range. Moreover, the method will also be validated using Ion Chromatography coupled with High-Resolution Mass Spectrometry (IC-HRMS) in collaboration with the Department of Veterinary Medicine and Animal Sciences at UNIMI, in order to compare the two analytical instruments.

In addition, it is intended to extend the method to other matrices.

References: Rampazzo, G., Gazzotti, T., Zironi, E., & Pagliuca, G. (2023). Glyphosate and Glufosinate Residues in Honey and Other Hive Products. *Foods*, 12(6), 1155.