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Determination of glyphosate in honey by high-performance liquid chromatography

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Abstract

The article presents the results of the developed highly sensitive and easily implemented method of sample preparation and determination of the content of residual amounts of glyphosate – one of the most widely used non-selective herbicides of systemic action for the destruction of weeds, in flower honey samples by high-performance liquid chromatography (HPLC) with a fluorescent detector (FD) in the framework of ensuring the safety of beekeeping products.

The HPLC stage with FD was preceded by sample preparation, which included the following steps: extraction of glyphosate from an artificially contaminated honey sample with a mixture of water/methanol (1:1) with subsequent addition of a four-fold excess of acetonitrile to the resulting solution to separate a complex multicomponent matrix of honey by centrifugation; filtration of the supernatant and evaporation of the filtrate on a rotary evaporator; dissolution of the resulting residue in a borate buffer solution in order to achieve an alkaline pH value of the medium equal to 8.5-9.5; pre-column derivatization of glyphosate in an alkaline medium with an acetonitrile solution of FMOC-Cl for 30 minutes to obtain a fluorescent herbicide derivative – *N*-alpha-(9-fluorenylmethyloxycarbonyl)-*N*-alpha-(phosphonomethyl)-glycine; adding at the end of the derivatization process to the reaction mixture of formic acid in order to transition from alkaline to acidic conditions and stabilize the glyphosate derivative; adding dichloromethane to remove the unreacted excess of the derivatizing agent.

Subsequent chromatographic analysis of the fluorescent derivative glyphosate was performed on a reverse-phase packed column *"Luna 100 C18-2.5 μ"*, characterized by versatility and stability in the range of pH values from 1.5 to 11.0, in the isocratic elution mode of mobile phase composition acetonitrile/an aqueous buffer solution of potassium phosphoric acid with a pH value equal to 4.5-5.5 (30:70).

The developed method was successfully tested on real samples of flower honey.

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