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APPLICATION OF ULTRA-HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY /TANDEM QUADRUPOLE MASS SPECTROMETRY FOR MULTI-CLASS MONITORING OF PESTICIDES IN HONEY SAMPLES FROM MACEDONIA

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The possible presence of pesticide residues in honey has impelled the need for setting up monitoring programs to determine the proper assessment of human exposure to pesticides. Nevertheless, there are two forms of contamination of honey: during pollen and nectar collection by bees, characterizing cross-contamination, and through treatment of hives in which pesticides can migrate into honey as direct contamination. Established maximum residue limits (MRLs) of pesticides in honey by the European Union are generally at the limit of quantification of the analytical methods. This paper describes an effective multi-class method using a modified QuEChERS sample preparation for detection and quantification of 18 pesticides with Ultra-high-performance liquid chromatography (UHPLC) – tandem quadrupole mass spectrometry.

The method was validated according to the requirements laid down in DG SANCO 12571/2013 document. Levels of detection and quantification were lower than the established MRLs, the obtained precision was better than 20 %, and the recovery values were between 74.4 and 104.1 %. Fifty honey samples within the national monitoring program were collected from August 2014 until November 2014 and tested for presence of carbaryl, carbofuran, fenvalerate, cypermethrin, deltamethrin, permethrin, bifenthrin, amitraz, coumaphos, bromopropylate, dichlorvos, diazinone, malathion, parathion, dimethoate, omethoate, methomyl and thiametoxam. Trace levels of methomyl, diazinone and fenvalerate were detected somewhat above the reporting level for these pesticides (0.010 mg/kg), indicating a possible cross-contamination during pollen and nectar collection by bees.

UHPLC coupled with tandem mass spectrometry has demonstrated to be valuable technique for the detection and quantification of pesticides in monitoring programs, designed to cover a wide range of pesticides in honey samples. The monitoring of pesticide residues is important because honey is a route of human intake of pesticides, since it is a food and an alternative medicine, and serves as a way to evaluate the dispersion of pesticides in the environment and can be associated with the global phenomenon of disappearance of hives. This work presents the first insight on the presence of pesticide residues in honey samples from Macedonia.

Key words: tandem quadrupole, liquid chromatography, pesticides, QuEChERS, honey