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V.6. APPLICATION OF LIQUID CHROMATOGRAPHY WITH DIODE-ARRAY DETECTOR FOR DETERMINATION OF ACETAMIPRID AND 6-CHLORONICOTINIC ACID RESIDUES IN SWEET CHERRY SAMPLES

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Acetamiprid is a broad-spectrum neonicotinoids insecticide used on a wide range crops, especially fruits. Besides its positive effects, acetamiprid also has been posing various health risks to consumers. Due to the growing use of insecticides from the family of neonicotinoids, their increased presence in the environment is evident. For this reason, the concentration of acetamiprid residues, including its metabolite 6-chloronicotinic acid, in agricultural products should be monitored.

A rapid and simple method for the confirmation, simultaneous analysis and quantification of acetamiprid and 6-chloronicotinic acid as intermediate of acetamiprid decomposition in sweet cherry samples has been developed. This residue analysis method is based on the reversed phase separation on C18 column with gradient elution. Analytes' determination and quantification were performed by high performance liquid chromatography (HPLC) with photodiodes detection and chromatograms were extracted at 230nm. Extraction efficiency experiments demonstrated the ability of this method to extract neonicotinoids from sweet cherry samples. These insecticides were extracted with mixture acetonitril/0.1N ammonium-chloride (8/2, v/v). The extract was filtered through layer of celit and evaporated, the residue dissolved with acetone and then analyzed by liquid chromatography. The standard addition method was used for acetamiprid and 6-chloronicotinic acid determination in order to eliminate the matrix effect. Sweet cherry samples spiked with the concentration levels of 0.5mg/kg and 1mg/kg was used to ensure method accuracy (recovery) and data precision. The repeatability of the retention times and peak areas were checked by injecting the standard mixture of acetamiprid and 6-chloronicotinic acid solution five times. Average recoveries of acetamiprid and 6-chlornicotinic acid from sweet cherry samples were in the range of 95-101% and 73-83%, respectively with the associated relative standard deviations (RSDs) < 5%. Limit of detection (LOD) for the analyzed acetamiprid and 6-chloronicotinic acid was estimated from fortified samples. The limit of quantification (LOQ) was 10 and 30µg/kg for acetamiprid and 6-chloronicotinic acid, respectively. Thus, it can be concluded that the developed HPLC-DAD method represents a useful tool for a sensitive and rapid determination of acetamiprid and 6-chloronicotinic acid. Hence, the method may find further application in the analysis of real sweet cherry samples contaminated with these insecticides at a ppb level.

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