

METHOD FOR PESTICIDES ANALYSIS IN WATER USING C18 SPE DISK EXTRACTION AND GC/MS/MS TRIPLE QUADRUPOLE ANALYSIS WITH LARGE VOLUME INJECTION*

Viviane E Nakano, Tereza A Kussumi, Sonia B Rocha, Vera RR Lemes, Iracema A Kimura, Maria Celeste Cardeal de Oliveira, Kumi S Ozawa**

Núcleo de Contaminantes Orgânicos, Centro de Contaminantes. Instituto Adolfo Lutz. Coordenadoria de Controle de Doenças. Secretaria da Saúde do Estado São Paulo, Brasil. Center for Organic Contaminants, Contaminants Center, Instituto Adolfo Lutz. Coordination for Diseases Control, State Health Department of São Paulo, Brazil.

SUMMARY

Brazil is one of the largest pesticide consumers in the world and is the most responsible for consumption in Latin America. Due to the possible risks from pesticide use, water needs to be monitored for pesticides. This study describes a method to analyze pesticide residues in water using C18 SPE disk extraction and GC/MS/MS triple quadrupole with Large Volume Injection (LVI) system. Thirty pesticides were analyzed in water samples, including isomer and metabolite compounds. The methodology was based on an Environmental Protection Agency method, with some adaptations. The extraction, under vacuum, consisted of slowly percolating 1L of water sample acidified, through a pretreated solid membrane disc (C18 SPE), elution using solvents: methanol, ethyl acetate and dichloromethane, concentration to 1 ml and injection in GC / MS / MS. Calibration curves for each pesticide were prepared at 7 levels (3 replicates) to quantify pesticide residues, after the optimal analytical conditions were established. The recovery rates for 6 replicates, at the limit of quantification (LOQ), for LVI and for 2 μ L-injection, ranged from 70.40 to 88.03% and the relative standard deviations were 2.2 to 18%, which comply with parameters established by the European Commission (SANCO). The LOQ values for the majority of the 30 pesticides studied were 0.05 μ g/L with exception for Alachlor (0.02 μ g/L), for Carbaryl (0.10 μ g/L) and Endosulfan Sulfate

(0.10µg/L). The limits of detection (LOD) values ranged from 0.01µg/L to 0.097µg/L. The analysis of water using SPE disk extraction is a technique that reduces solvent consumption making it, economically and environmentally, a better choice. Additionally, to the large volume injection technique, analyte protectant was used for chromatographic resolution improvement. The 30 analyzed pesticides showed good recoveries with this procedure. This study demonstrated the suitability of this method for the analysis of selected pesticides in water, the importance of using analyte protectants as well as, the possibility of large volume injection replacing the traditional concentration step.

*This work is part of a project with Agilent Thecnologies.

**Agilent Thecnologies