

Method validation for the simultaneous analysis of organophosphate pesticides in blood by GC/MS

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AIMS: The purpose of this study was to provide a standard method for the analysis of organophosphate pesticides such as chlorpyrifos, diazinon, malathion and parathion in blood.

METHODS: We performed method validation for these pesticides in blood according to the EURACHEM (A Focus for Analytical Chemistry in Europe) guide. For the analysis of the pesticides, we used a solid-phase extraction column (Waters Oasis HLB®). After the extraction, the supernatants were evaporated to dryness under the nitrogen stream. They were analyzed by gas chromatography/mass spectrometry (GC/MS) after reconstituting with ethanol. Terbufos was used as an internal standard. To validate this method, we performed verification procedures with the following parameters: *selectivity, linearity of calibration, accuracy, precision, recovery, limit of detection and quantification.*

RESULTS: Analysis of the blank blood sample showed that there was no interference of endogenous substances with chlorpyrifos, diazinon, malathion and parathion. Straight standard curves were obtained in the wide range of concentrations (135-1500 ng/mL, $R^2 > 0.99$). The LODs and LOQs of chlorpyrifos, diazinon, malathion and parathion were 0.08, 0.04, 0.09, 0.09 mg/L and 0.15, 0.15, 0.17, 0.13 mg/L, respectively. From the results of the determination of intra- and inter-day precisions, the coefficients of variation (CVs) were less than 10% in all experiments. The recoveries of 4 organophosphate pesticides ranged from 91.4 to 104.0%.

CONCLUSIONS: Our validation data according to the Eurachem guide were adequate for the purpose for the analysis of chlorpyrifos, diazinon, malathion and parathion in blood.

KEYWORDS: *Organophosphate pesticides, Method validation, Solid-phase extraction, GC/MS*

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