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V.12. METHOD VALIDATION AND UNCERTAINTY EVALUATION OF THE INSECTICIDE CHLORPYRIFOS IN WATER SAMPLES BY USING HPLC ANALYSIS

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Chlorpyrifos is one of organophosphorus (OPPs) insecticides, which are the most important and widely used classes of agricultural pesticides. Because of its intensive use of ecosystems are contaminated by chlorpyrifos and public concerns on safety have been increased.

The method has been developed for confirmation and quantitation of the chlorpyrifos in water samples reverse phase high performance liquid chromatography (HPLC) with photodiodes detection and chromatograms were extracted at 300 nm. This method was single-lab validate in order to demonstrate its performance for monitoring of the insecticide chlorpyrifos in water.

Method accuracy was quantified through measurement uncertainty estimate based on method validation data. Tap and groundwater samples were used to demonstrate linearity and accuracy. Linearity for chlorpyrifos was evaluated by calibration curves in the range of 0.01–10 µg/ml. The accuracy of the proposed method was evaluated as the mean recovery. The analytical method allowed us to quantify chlorpyrifos in tap water and groundwater in concentration level between 0.01–10 µg/ml with recovery values between of 80.72-109.57 % for tap water and 89.99-97.45 % for groundwater and repeatability lower or equal than 0.6% for both the matrices. The experiments showed that there were no interference peaks from the water matrix (tap and ground) on the elution region of the chlorpyrifos. The RSD values were satisfactory being in the ranges of 0.283-1.237 % (for tap water) and 0.681-3.887 % (for groundwater). The variation of the retention time was less than 0.05 % during these experimental series. The combined relative uncertainty for water samples was 0.481 % (for tap water) and 0.654 % (for groundwater). The expanded uncertainty (Uc) was calculated as $Uc = k \cdot u_c$, where k is the coverage factor with level of confidence of approximately 95% considering a coverage factor of 2 (EURACHEM/CITAC, 2000).

We proved that the method was specific for determination of chlorpyrifos in the relevant matrices. After validation and measurement uncertainty evaluation steps, results obtained showed that the method can be applied to efficiently for monitoring of chlorpyrifos in water samples.

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