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DETERMINATION OF TEMBOTRIONE IN URINE BY SOLID-PHASE EXTRACTION AND LC-MS/MS

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Tembotrion is one of the most recently introduced herbicides in the triketone group and has been in application in Europe since 2007. Due to its relatively short agricultural use, studies of its impact on human health and the environment are rare.

The aim of this study was to develop and validate a method for extraction and determination of tembotrione from rat urine.

As already established, after oral exposure, more than 90% of initial tembotrion doses are absorbed, and more than 93% of the absorbed dose is excreted through urine and feces within 96 hours.

The extraction procedure was based on forcing the 5 mL acidified urine samples (pH 2) through the Oasis HLB sorbent, rinsing the sorbent with water in order to eliminate urinary interferences and elution of the retained compound with methanol. The eluate was evaporated under a stream of nitrogen and the dry residue was dissolved in water.

The extracts were analysed using high performance liquid chromatography - tandem mass spectrometry (LC-MS QQQ system). The sensitivity of the LC-MS/MS analysis was 1 pg/ml in standard solutions and 0.1 ng/ml based on treatment of 5 ml urine samples.

For recovery experiments, urine samples were spiked with aqueous solution of tembotrione at 80 ng/mL (n=5). The extraction recovery was 70 % (RSD 7 %). The method was applied for the determination of tembotrione in 24-hour urine samples of the treated animals. Adult male Wistar rats received tembotrione at 0.0007 mg/kg b.w./day and 0.7 mg/kg b.w./day, respectively, for 28 consecutive days by oral gavage. Tembotrione was determined in urine samples collected on the 1st and 28th day of treatment.

Tembotrione was not detected in the urine samples collected on the first day of treatment with 0.0007 mg/kg b.w./day. However, for the highest dose, the mass of tembotrione recovered in urine ranged from 7 ng to 17 ng. Mass of urinary tembotrione on the 28th day of treatment ranged from 99 ng to 566 ng and from 746 ng to 5862 ng for the lowest and highest doses, respectively.