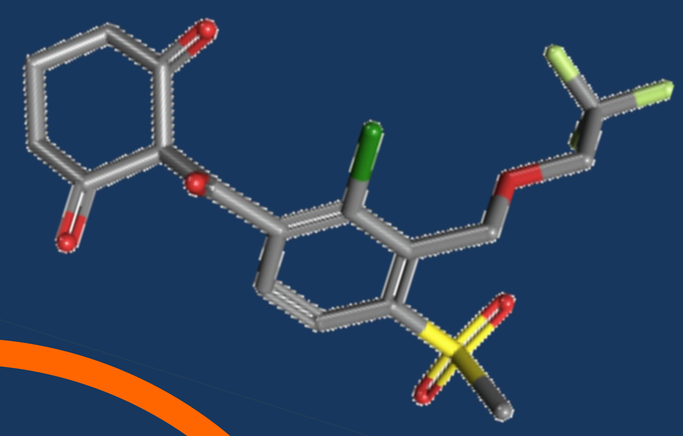
INSTITUTE FOR MEDICAL RESEARCH
AND OCCUPATIONAL HEALTH
ZAGREB, CROATIA

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

G. Mendaš, S. Stipičević, M. Dvoršćak, S. Fingler

OBJECTIVES

- To investigate long term exposure to low tembotrione concentrations based on acceptable operator exposure level (AOEL)
- To learn more about tembotrione excretion in rats
- To develop a solid phase extraction (SPE) procedure for determining the accumulation of tembotrione in rat urine and optimise it for LC-MS/MS



Treatment
- Two groups of male Wistar rats + control
- Tembotrione via oral gavage for 28 days:
  0.0007 mg/kg b.w./day (AOEL)
  0.7 mg/kg b.w./day (1000 x AOEL)

Sample
- 24-hour urine samples collected on the 1st and 28th day of treatment

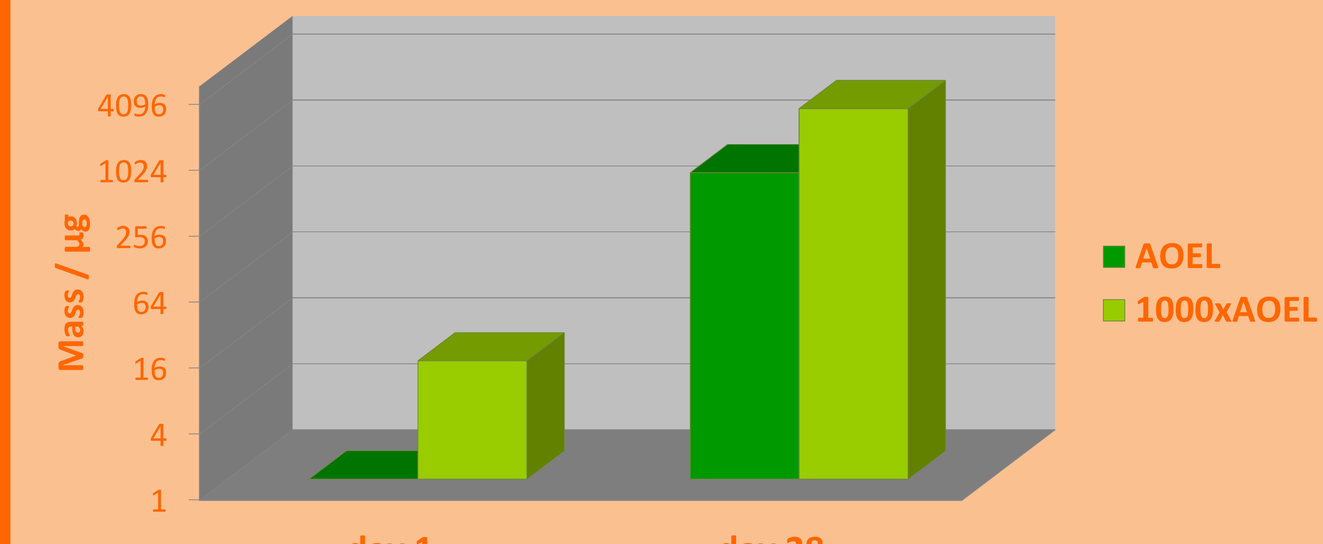
Analysis
- 5 mL of urine (pH 2)
- SPE: Oasis HLB
- LC-MS QQQ

RESULTS

Extraction efficiency of tembotrione from spiked urine samples

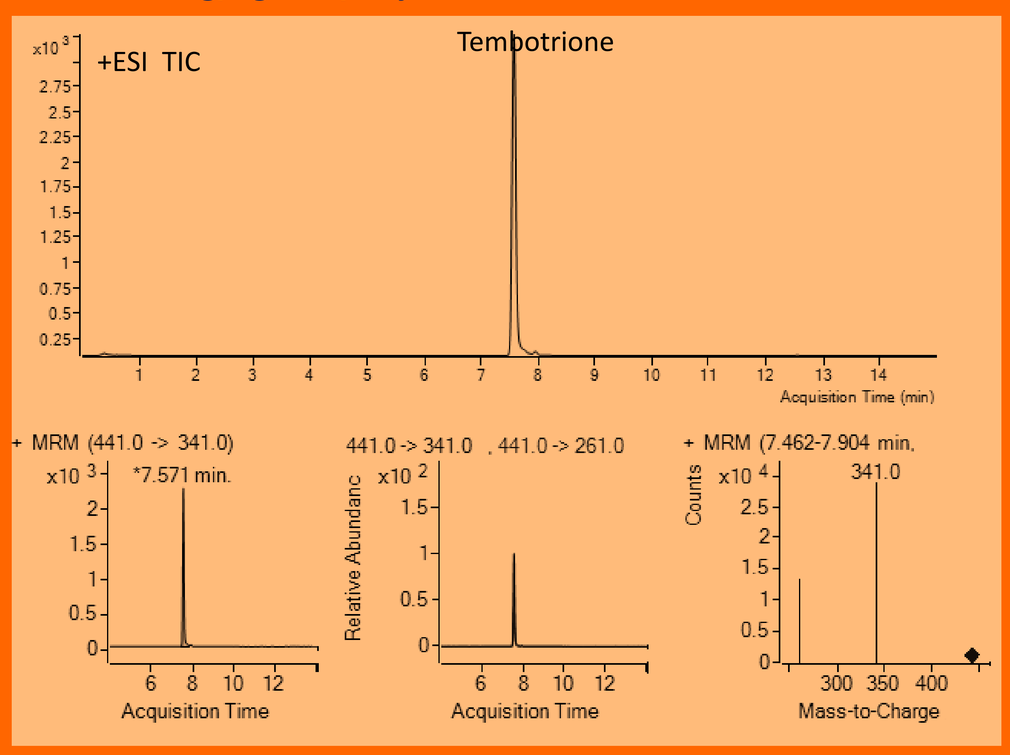
Concentration 80 ng/mL | Recovery = 70 % S.D. = ± 7 (*n* = 5) | Detection limit 0.1 ng/mL

Average masses (µg) of the tembotrione in 24-hour urine samples collected on the 1st and 28th day of treatment with doses 0.0007 mg/kg b.w./day (AOEL) and 0.7 mg/kg b.w./day (1000 × AOEL)



LC-MS/MS analysis

Urine sample collected on the 28th day of treatment; Dose: 0.7 mg/kg b.w./day



SPE with Oasis HLB proved suitable for accumulation of tembotrione from spiked urine samples.
Recovery: 70 % (RSD < 7)

Due to mass selective detection, LC-MS/MS analysis of urine extracts enables highly reliable and sensitive determination.

Tembotrione was not detected in the urine samples collected in the first 24 hours of treatment with 0.0007 mg/kg b.w./day.

Increasing the dosage administered to the rats resulted in higher recoveries of tembotrione in urine samples.

The fraction of tembotrione detected in urine was less than 8 % of the exposure doses.

Further studies are necessary to investigate the excretion mechanism of tembotrione (e.g. more frequent sampling, analysis of metabolites).

HRZZ Hrvatska zaklada

This work has been supported in full by the Croatian Science Foundation under project 8366 (OPENTOX).

Opentox