

## Poster Session

Thursday, 20<sup>th</sup> September

Event Hall

Core Time : 11:10 - 12:20 (Odd number), 13:30 - 14:40 (Even number)

Session 35: Environment I

**PTTh-042**

13:30 – 14:40

### SBSE probe desorption GC-IT-MS analysis of degradation products of esfenvalerate obtained by chemical oxidation process

Renata Colombo, Marcos Roberto V Lanza, Janete H Yariwake

University of Sao Paulo/IQSC, Sao Carlos, Brazil

#### Keywords:

SBSE-GC-IT-MS ; esfenvalerate ; chemical oxidative degradation

#### Novel aspects:

Study of degradation of esfenvalerate by using a chemical oxidative process. Utilization of SBSE-GC-IT-MS for extraction of degradation products of esfenvalerate and their identification.

#### Abstract:

A growing number of investigations have been reported recently showing the widespread occurrence of agrotoxics in the environment, notably in the aquatic compartment. The treatment of these pollutants by oxidative process using hydroxyl radicals ( $\cdot\text{OH}$ ) have been highlighted because of its high efficiency in the degradation of numerous organic compounds and low operating cost. To evaluate the efficiency of this process and monitoring of intermediates and final products, hyphenated chromatographic techniques are indispensable. However, due to the low concentration of pollutants and their degradation products in aqueous medium associated to the incompatibility of the aqueous matrix with conventional GC-MS techniques, some steps of preparation and pre-concentration of the samples are necessary. The SBSE (stir bar sorption extraction) technique combined with hyphenated chromatographic techniques such as GC-MS resulted in rapid analysis, low solvent consumption, higher analytical precision and sensitivity, and has been successfully employed in the extraction of agrotoxics in water at the concentration range of sub-ng L<sup>-1</sup>. However, SBSE-GC-MS analysis requires thermic desorption of the analytes, and nowadays only one commercial SBSE thermal desorption system is available. On the other hand, an approach called "SBSE probe desorption" is being investigated by us as an alternative to integrate the advantages of SBSE and the power of GC-IT-MS (gas chromatography-ion trap-mass spectrometry) as the detection technique. In this procedure, the SBSE bar, containing the analytes sorbed (extracted) is placed into an appropriated probe and thermally desorbed, without using the commercial SBSE thermal desorption system. In this work the degradation of esfenvalerate, a pyrethroid insecticide, was studied by using a chemical oxidation process and the degradation products were monitored by using SBSE probe desorption GC-IT-MS. Degradation was performed by using 50% hydromethanolic solution containing 45 mg L<sup>-1</sup> esfenvalerate, pH 11.25, 25 mg L<sup>-1</sup> of hydrogen peroxide, within a 4 hour reaction period. After degradation process, the products were extracted by SBSE by using stir bars (10mm × 0.5mm, 24  $\mu\text{L}$  PDMS coating, Twister, Gerstel) at room temperature (25°C), for 120 min, with 15% methanol, 12% NaCl and stirring at 1000 rpm. After extraction, the stir bar was placed into the probe of a GC CP 3800 Varian, coupled to an ion trap MS Saturn 2000 (Varian). Thermal desorption of the analytes were done at 250°C, and the probe heating was held for 18 min. GC analysis were performed on a DB-5 ms fused silica capillary column (30 m x 0.25 mm i.d., 0.5  $\mu\text{m}$  film thickness, Agilent). The oven temperature was programmed from 70°C (held for 0.5 min) to 300°C/min (held for 6 min), at 20°C/min. Helium was used as carrier gas at flow rate of 1.2 mL min<sup>-1</sup>. The MS analyses were done in the scan mode ( $m/z$  40 to 450) using electron impact ionization (70 eV). The temperature of transfer line, ion trap and manifold were set at 300°C, 220°C and 40°C, respectively. By using the SBSE probe desorption-GC-IT-MS method, it was possible to fully identify two products of chemical oxidation of esfenvalerate, 3-phenobenzoic acid and 3-phenoxbenzaldehyde. These compounds were previously described as being metabolites of esfenvalerate, with small estrogenic (endocrine-disrupting) activity and possibly with small environmental impact (McCarthy *et al.*, *J. Environ. Monit.* 2006, **8**,197). The structural elucidation of the other oxidation products of esfenvalerate is still in progress.

**Acknowledgements:** FAPESP, CNPq