MEASUREMENT OF DIOXINS AND WHO PCBs IN FOODSTUFFS USING GCxGC-IDTOFMS

Jean-François Focant, Catherine Pirard, Anne Cécile Massart, Gauthier Eppe and Edwin De Pauw

Center for Analysis of Trace Residues (CART), Mass Spectrometry Laboratory, Dioxin Laboratory, University of Liège, B6c Sart-Tilman, B-4000 Liège, Belgium.
E-mail : JF.Focant@ulg.ac.be

The use of comprehensive two–dimensional gas chromatography coupled to isotope dilution time-of-flight mass spectrometry (GCxGC-IDTOFMS) is investigated for the measurement of 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), as well as selected polychlorinated biphenyls (PCBs), in foodstuff matrices. Sample pre-treatment is based on the use of commercially available automated high throughput instruments [PLE (ASE200^{TM}, Dionex, Sunnyvale, CA, USA) and Power-Prep^{TM} system (FMS Inc., Waltham, MA, USA)] capable to extract samples sequentially and/or in parallel. Among the several column sets that have been tested, a set made of a RTX-500 40m x 0.18mm ID x 0.11µm df (Restek) as ^{1}D and a BPX-50 1.5m x 0.10mm ID x 0.10µm df. (SGE, Austin, TX, USA) as ^{2}D offered a good chromatographic separation, which is backed up by the deconvolution capability of the TOFMS. The temperature of the modulator had an offset of 40°C compared to the temperature of ^{1}D oven. The modulator period was 4 s. The MS The data acquisition rate was set at 60 scans/s for a mass range of 100 to 550 *m/z*. The selectivity of the unit mass resolution MS was ensured by isotope ratio check on two of the most intense ions for both ^{12}C- native and ^{13}C-labelled analytes. Instrumental LODs were estimated to be between 0.3 and 3 pg/µL injected. A comparison between GCxGC-IDTOFMS [Pegasus 4D (Leco Corp., St Joseph, MI)] and GC-IDHRMS [Finnigan MAT95XL HR mass spectrometer (Finnigan, Bremen, Germany) and Autospec Ultima HR mass spectrometer (Micromass, Manchester, UK)] is presented.