ON-LINE AUTOMATED PRESSURIZED LIQUID EXTRATION-MULTI STEP CLEAN-UP AND FRACTIONATION FOR THE MEASUREMENT OF DIOXINS AND PCBS IN FOOD AND FEED

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Sample preparation-fractionation for the measurement of dioxins and PCBs in biological matrices is a complex field of investigation[1]. Several different approaches are possible, but the common point is to aim for coupling and automation to reduce and simplify the inputs. Three years ago, we started a new project based on the direct coupling of pressurized liquid extraction (PLE) with automated multi-sorbent clean-up and fractionation[2]. The design of the early prototype system has evolved through several generations of changes dictated by a long term testing exercise. This paper reports on the latest data that were obtained using this system for food and feed samples dedicated to dioxin and PCB analyses.

For method development, lyophilized yolk quality control (QC) samples and animal feedingstuff QC samples at regulation level (low pg/g) are used. Sodium sulfate is used to estimate method blank levels (BCs). Isotope dilution (ID) gas chromatography (GC) high resolution mass spectrometry (HRMS) is used for identification and quantification (ISO 17025). PLE cells are filled with the sample that is extracted at 125°C and 1500 psi. Pressure and temperature are continuously recorded for QA/QC purposes. The extraction solvent is directly purged on a set of low pressure LC columns made of a multi-layer silica column, a basic alumina column, and a Celite-dispersed carbon column[3]. The extract is purified and subsequently fractionated depending on molecule geometries and polarity. The total processing time is 1.5h for 3 samples prepared in parallel. Dioxins and furans (2,3,7,8-substituted congeners) are separated from mono-*ortho*-PCBs and analyzed separately. Recovery rates range from 70% to 100%. The precision was good with RSD values between 5% and 20%. Both repeatability and reproducibility were studied. The accuracy versus reference values was included in a 95% confident interval based on 2 times the SD of the reference mean for most analytes.

1. Focant J.-F., Pirard C. and De Pauw E. (2004) *Talanta* 63: 1101-1113.

2. Focant J.-F., Shirkhan H. and De Pauw E. (2002) *Organohalogen Comp.* 55: 33-36.

3 Focant J.-F., Eppe G., Pirard C. and De Pauw E. (2001) *J. Chromatogr. A* 925: 207.