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POPs and perfluorinated alkylated substances (PFAS) in fish feed

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The main aim of the European project CONffIDENCE "Contaminants in food and feed: Inexpensive detection for control of exposure" was to develop and validate simple and fast methods which enable determination of various contaminant classes in food and feed. For this purpose, two analytical procedures were developed and further validated: (i) simultaneous analysis of polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs) and polycyclic aromatic hydrocarbons (PAHs), and (ii) analysis of 3 major representatives of perfluorinated alkylated substances (PFAS) in fish feed and further validated.

The first procedure used gas chromatography coupled to tandem mass spectrometry (GC–MS/MS), or, alternatively, two-dimensional gas chromatography coupled with time of flight mass spectrometry (GC?GC-TOFMS) for the final instrumental determination of 18 PCBs, 7 PBDEs, and 16 PAHs within one single run. A substantial simplification of sample processing procedure prior to quantitation step was achieved: after addition of water to homogenized sample, transfer of hydrophobic analytes into ethyl acetate was supported by added inorganic salts. Bulk fat, contained in crude organic extract obtained by partition, was removed on a silica minicolumn. This approach enabled to process six samples in less than 1 hr. The recoveries of target analytes were in the range of 73–120% even at the lowest spiking level (1 µg/kg), repeatabilities (relative standard deviations, RSDs) ranged from 1 to 20%. The LOQs achieved using LV–PTV–GC×GC–TOFMS were as follows (µg/kg): 0.01–0.25, 0.025–5, and 0.025–0.5 for PCBs, PBDEs and PAHs, respectively; even lower levels could be controlled by GC-MS/MS. The second method enabled to determine perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA) and perfluorooctane sulfonamide (FOSA) in fish feed using methanol extraction and subsequent clean-up of crude extract by addition of carbon powder (activated charcoal). After centrifugation, the aliquot of supernatant was analyzed by liquid chromatography tandem mass spectrometry (LC–MS/MS). In comparison with other traditionally used, very laborious methods for PFAS determination, this sample preparation procedure needed only ca 1 hr for 10 samples. Recoveries of target PFAS were in the range 85–110%, repeatabilities range from 2 to 15%. LOQs of PFOS, PFOA and FOSA were (µg/kg): 0.15, 0.3 and 0.3, respectively; they fulfilled the request of the European Commission (EC) for LOQ at level 1 µg/kg (2010/161/EU).

Within the presentation, approaches for development of analytical procedures for determination of a range of environmental contaminants in complex matrices will be discussed.

Keywords fish feed;POPs;PFAS;sample prep;GC-MS/MS;LC-MS/MS

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