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Critical Assessment of Clean-up Techniques Employed in Analysis of Organochlorine Pesticides and Persistent Organic Pollutants in Fatty Samples

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Purification is a very important and in many cases a critical step in analysis of organochlorine pesticide residues (OCPs) and other persistent organic pollutants (POPs). Removing of matrix co-extracts from the primary extracts improving results in terms of both selectivity and sensitivity. The common techniques which are typically applied for the clean-up of the crude extract are gel permeation chromatography (GPC) and solid phase extraction (SPE). Currently the dispersive SPE (d-SPE) is more and more widely used especially in the QuEChERS based method.

A main focus of this study was to evaluate purification potential of Enhanced Matrix Removal—Lipid (EMR – Lipids), a new sorbent for selective removing of lipids in complex biotic matrices, for fatty foods such as fish. This contribution is focused on the simultaneous determination of 23 organochlorine pesticides (OCPs such as dieldrin, heptachlor, chlordane and DDTs), 17 polychlorinated biphenyls (PCBs), 22 polybrominated diphenyl ethers (PBDEs) and 16 carcinogenic polycyclic aromatic hydrocarbons (PAHs) in fish and seafood. Two sample preparation procedures for isolation above mentioned groups of compounds from the smoked trout sample (fat content 15 % w/w) were tested: (i) the QuEChERS method followed by purification of acetonitrile phase using d-SPE and (ii) the routinely used ethylacetate based extraction method followed by clean-up using SPE on silica column for the complex comparison of the clean-up efficiency of a new sorbent. For the determination of target OCPs and other POPs in purified extract gas chromatography coupled with tandem mass spectrometry (GC-MS/MS) was employed.

Employing rapid inspection of purified extracts by direct analysis in real time coupled with time-of-flight-mass spectrometry (DART-TOFMS) fingerprinting, significant decrease of a triacylglycerols (TAGs) content in samples cleaned using tested d-SPE was achieved.

The obtained results show that the new clean-up strategy is applicable for the determination of all target groups of POPs in high fatty matrices such as fish. The QuEChERS extraction method with a clean-up step using the new sorbent EMR-lipid was successfully validated with recoveries 70-120 % and RSD ˂ 20 %.

This fast method enabling high laboratory throughput was used for the determination of OCPs and POPs in fatty fish and seafood samples.

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