

Validation of extraction and clean-up methods for compound-specific carbon stable isotope analysis of organochlorine pesticides in complex matrices

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Compound-specific stable isotope analysis (CSIA) has demonstrated to be an efficient tool to understand sources and transformation processes of various pollutants in the environment. However, complex matrices such as soils and biota have been barely investigated by CSIA yet due to challenges for sample preparation and chromatographic separation of target compounds from interfering substances.

Therefore, efficient extraction and clean-up procedures, which do not impart changes on the isotope ratio of the target compounds, need to be applied for accurate and precise isotope analysis. We performed a comprehensive evaluation of pre-treatment methods for CSIA of Hexachlorocyclohexanes (HCHs), *p,p'*-Dichlorodiphenyl-trichloroethane (DDT) and their chlorinated metabolites in soil and in fish oil samples.

As regards to soil samples, we evaluated user- and environmentally friendly extraction methods including the Quick, Easy, Cheap, Effective, Rugged and Safe procedure (QuEChERS), Ultrasonic Assisted Extraction (USE) and Focused Ultrasonic Extraction (FUSE), as well as clean-up methods including sulfuric acid and Florisil® clean-up procedures. All optimized methods showed to be compatible with CSIA, with good recoveries and negligible changes in carbon isotope ratios of the target compounds.

In the case of fish oil samples, we tested a lipid oxidation procedure using sulfuric acid, followed by solid phase extraction (SPE) clean-up with silica, and pre-concentration of the organic extract by solvent evaporation. The recoveries of the target compounds were higher than 60 % and the induced isotope effects are under evaluation.

This study will provide the analytical pre-requisite to apply CSIA for identification of sources, assessment of degradation of HCHs, DDT and their chlorinated metabolites in contaminated soils, and for evaluation of bioaccumulation in the food chain.