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OPTIMIZATION AND VALIDATION OF A PARALLEL PLE PROCEDURE FOR THE EXTRACTION OF ULTRA-TRACE LEVELS OF PCDD/Fs, and DL-PCBs IN BIOLOGICAL MATRICES, SEPIOLITE, AND GUAR GUM

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Preparing biological samples for dioxin and PCB measurement is a task that is challenging many research centers and routine laboratories. For either human or food-related matrices, the implementation of proper procedures requires time, know-how, and money. More recently, food-feed additives such as sepiolite and guar gum have also attracted lots of attention due to their great complexity in terms of efficient and reliable extraction of target analytes. Among other techniques, pressurized liquid extraction (PLE) has been used for several years to save time and to reduce solvent consumption, while maintaining good extraction efficiency. Samples are normally sequentially extracted and extraction sequences can be performed unattended overnight. The unavoidable clean-up procedure that follows the extraction step has also evolved over the last 15 years to offer automated parallel sample processing capabilities, based on multi-column set-ups and computer piloted solvent deliveries. Recently, a novel PLE instrumental design has been proposed to allow parallel extraction of several samples in order to better fit with sample throughput requirements of the parallel clean-up procedure. We investigated the use of both parallel PLE and clean-up procedures for sample preparation of reference and quality control (QC) food and serum samples. A Placket-Burman experimental design was used as a screening test to study the influence of the major parameters within a pre-defined working range for sepiolite and guar gum. The parameters studied were the following: a mixture of toluene/ethanol, temperature, pressure, extraction time, number of extraction cycles, sepiolite mixed (or not mixed) with sodium sulphate, ^{13}C labelled compounds spiked below or above the sample in the extraction cell.

Recovery rates, extraction and transfer efficiency, accuracy, precision, robustness, and usability of the system have been estimated. For BCR607 (powder milk), RSDs were below 20% for all congeners. Setting the certified value as 100%, all measured concentrations were between 100% and 116% (all overestimations). For all analytes, the recovery rates ranged between from 60-110%. A full procedure using GC-IDHRMS has been validated for food and feed. For guar gum, the limit of quantification (LOQ) for most of the toxic congeners is 0.01 ng/kg. The precision assessed by repeatability tests provided RSDs between 1 and 20% for PCDD/Fs and DL-PCBs. Within-laboratory reproducibility study provided RSDs between 3 to 17% for the same congeners. Recoveries are between 54 to 93% and the bias was calculated with spiked samples. The bias met the EU directive[2] requirement (i.e. ± 20%) for results expressed in toxic equivalent units.

The extraction stainless steel cell costs 15 EUR and each cap costs 8 EUR. Both are reusable, if needed. The extraction cell volumes range from 10 to 250mL. The modular system allows the extraction of 6 samples in 1.5h. The decontamination of the system between runs is easily carried out by flushing and pressurising a low volume of solvent inside the lines and in the pressure relief line. So far, no cross-contaminations have been observed. Compared to the ASE, one can expect less tubing clogging as the inner diameters are significantly larger. The system is characterized by a good sample throughput-cost effective ratio, even for limited size laboratories because a single line design is available. Finally, an interesting feature of the PLE system is to be able to accommodate a sulphuric acid impregnated silica column right after the extraction column, allowing 'in-line' partial clean-up without the disadvantage of producing highly corrosive fluids under high pressure, as is the case when adding such sorbent inside the ASE cell.

Reference:

1. Commission Regulation 1883/2006 Official journal of the European Union L364/32-43