A 'COOK BOOK' METHOD FOR DIOXIN ANALYSIS

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Preparing either human or food samples for dioxin and PCB measurement is a task that is challenging many research centres and routine laboratories. Accurate measurement of dioxins and related compounds requires high standard analytical strategies, time, extensive know-how, and money. One of the major reasons is that PCDDs, PCDFs and PCBs are found at levels as low as pico- or femtogram per gram of matrix, depending on the investigated biological samples. Additionally, matrix-related interferences are present in concentrations at orders of magnitude higher than the analytes of interest. For those reasons, a complex multi-step approach is required to 1) extract the analytes from the matrix core, 2) separate undesirable interferences and, 3) finally isolate, separate and quantify analytes of interest. Those complex multistep strategies include sample extraction, sample cleanup, sample fractionation, several steps of solvent reduction, and, finally, analyte measurements by GC-MS under strict quality assurance/quality control (QA/QC) criteria. Accredited laboratories often require a week or more for reporting, due to the tedious manual multi-step procedures that are mandatory for this ultra-trace analysis (ppt, ppq). In terms of cost per sample and sample throughput, it is not only the final measurement of the analyte concentration, but – maybe even more importantly - the complex sample preparation procedure, which makes this measurement possible. In the past few years, efforts focused on the development of alternative procedures to speed up and simplify the process while maintaining a high level of QA/QC.

In order to move towards simplification of the entire sample preparation procedure, automation, coupling, and hyphenation of the various analytical steps are required. In that context, an integrated strategy is proposed. It rests on the use of PLE coupled to an automated solvent reduction-exchange device that produce sample extracts that can automatically be further cleaned-up via a multi-step LC setup. The LC setup includes a multi-layer silica column (acid, neutral, basic), a basic alumina column, and a column containing carbon dispersed on celite. The fractionated extracts are further evaporated using the hyphenated solvent reduction-exchange device to satisfy to the required concentration factor and, then, transferred to the GC injection device for GC-MS measurements.

The automated semi-integrated sample preparation system is modular and expandable from one to six sample configurations. Batches of samples (n=6) can then be processed in parallel so that the sample throughput is significantly improved. A 'same-day testing' situation can then be achieved for large series of samples (18 samples a day). The system is capable of utilizing a wide range of extraction cell sizes, clean up columns, and multiple solvent selection valves. It is well suited for new method development and for experimenting with different sample sizes, solvents, clean up packing materials, extraction pressures and temperatures. Each channel operates independently of other channels, if one channel malfunctions the rest will still work. The large bore plumbing of the extraction module makes it virtually clog free. The exposed construction makes parts replacement extremely easy. On a practical point of view, a set of 5 unknown samples plus 1 QC sample can be processed in a working day. The development of such a fast 'cook book' procedure has large interest in the food processing industry where testing has to be performed as quickly as possible to avoid down time in production lines. The level of automation and coupling is such that it is now possible to imagine performing the dioxin screening on site in a regular industrial laboratory that does not have specific 'dioxin skills'.

The presentation will first describe the global procedure and will then illustrate performances on real biological samples.