STRATEGIES FOR RAPID SAMPLE PREPARATION AND ANALYSIS OF DIOXINS IN BIOLOGICAL SAMPLES

Jef Focant, Gauthier Eppe and Edwin De Pauw

Mass Spectrometry Laboratory, University of Liege, Belgium.

Abstract

Sample preparation is a critical step inside the analytical procedure to isolate and quantify persistent organic pollutants (POPs), such as dioxins, furans and polychlorinated biphenyls (PCBs), that are present at ppt level in biological matrices. Many methods for extraction and clean-up have been described since last 20 years but most of them require significant amount of time to be carried out and are often only suitable to prepare a single sample at a time. In order to increase the sample throughput, simplification and automation of procedures have to be completed. After several years of investigations of various sample preparation techniques, we ended up with rapid procedures including pressurized liquid extraction (PLE) for freeze-dried solid samples and octadecyl bounded solid phase extraction (SPE) for liquid samples; both process being completely automated and able to accommodate up to 10 samples at a time. Clean-up can automatically be carried out online using disposable liquid chromatography columns and computer controlled valve system. Extraction and clean-up time can, for example, be reduced down to 3 hours for 10 samples of milk. These procedures are applicable to a broad range of even high lipid content matrices such as meat products, fish, dairy products, ... Since concentrated purified extracts present very good level of cleanness, they can automatically be injected on bench type GC/MS system using large volume injection (LVI), additionally reducing the duration of the sample concentration step. The global methodology appears to be a possible alternative to classical sample preparation coupled to GC/HRMS

