Validation of a pressurised liquid extraction (PLE) multi-columns low pressure LC coupled to GC-HRMS for PCDD/Fs and dioxin-like PCBs in guar gum

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Abstract

During the last decade a number of dioxin related incidents occurred in the food and feed chain. As a result, monitoring programs in European countries were intensified. The most recent incident occurred during the last summer period with contaminated guar gum. The contamination with polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and pentachlorophenol (PCP) was discovered in Switzerland in additive samples with levels far above the maximum EU limits. Guar gum, produced only in India and Pakistan, is extracted from the green vegetable guar bean. It is used as an additive in a wide range of foods as well as dairy products. Its role is that of an emulsifier, thickener and stabilizer. The present paper describes the validation of an automated parallel pressurized liquid extraction (PLE) coupled to preparative multi-column low pressure liquid chromatography for sample preparation. This new approach matches the high sample throughput demand of routine laboratories while reducing human input at affordable cost. The optimisation of the extraction parameters is toluene/ethanol 90:10, temperature 150°C, pressure 1500 psi, extraction time 20 min and minimum 2 cycles. The extract was further cleaned and fractionated with a Power-Prep system using acid silica, alumina and carbon columns. PCDD/Fs fraction was injected into a gas chromatography - high resolution mass spectrometer (GC-HRMS, autospec ultima, Waters, UK). All details regarding the analytical procedure for Power-Prep and GC-HRMS can be found elsewhere[1]. The limit of quantification (LOQ) for most of the toxic congeners is 0.01 ng/kg. The precision assessed by repeatability tests provided relative standard deviation (RSDs) between 1 to 20% for PCDDs, PCDFs and dioxin-like 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.

References:

1. J.-F. Focant, G. Eppe, C. Pirard, E. De Pauw, J. Chromatogr.A 925 (2001) 207.
2. Commission Regulation 1883/2006 Official journal of the European Union L364/32-43