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FINAL RESULTS OF THE EUROPEAN RESEARCH PROJECT DIFFERENCE: NEW ALTERNATIVE METHODS FOR DIOXIN ANALYSIS

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The European research project DIFFERENCE ("Dioxins in Food and Feed – Reference methods and New Certified Reference Materials") was focussed on the development of alternative methods for the analysis of polychlorinated dibenzodioxins (PCDDs), dibenzofurans (PCDFs) and dioxin-like polychlorinated biphenyls (dl-PCBs) using comprehensive multi-dimensional gas chromatography (GC×GC), gas chromatography combined with low resolution ion-trap mass spectrometry (GC-LRMS/MS), the CALUX bioassay and an Ah-PCR technique. GC combined with high resolution mass spectrometry (HRMS) was used as a reference method in all comparisons. Given the need for a regular monitoring of PCDD/Fs and PCBs in Europe, which was further enhanced by the implementation of the maximum residue levels (MRLs) for PCDD/Fs in food and animal feed per 1 July 2002, and the relatively high costs of GC-HRMS and the limited capacity for HRMS analyses in European laboratories, cheaper, faster, but reliable methods were badly needed. The method development part included a developmental phase, a validation phase and a standardisation phase. In addition to the method development, attention was also paid to alternative extraction and clean-up methods, in particularly on accelerated solvent extraction. A feasibility study on the preparation and certification of reference materials was also included.

Three out of the four methods have successfully been developed and validated. CALUX may be considered as a valuable screening method that can be used in times of crisis for a large number of samples to indicate if those samples are below, around of above the EU MRLs. Quantitative total-TEQ values may also be produced by this method, but due to the variability of those data, in particular for the mono-ortho substituted PCBs, the use of CALUX in that way is not recommended. The application of recovery correction appeared to be essential in any case. GC-LRMS/MS may be used as a reliable routine method that will produce congener-specific data. GCxGC-ECD or GCxGC-ToF-MS emerged as viable routine methods for dl-PCBs and PCDD/F measurement. No doubt for environmental samples, and for food and feed materials in which dioxins and dl-PCBs occur at higher concentrations (around the MRLs in fatty fish, fish oil), GCxGC is able to serve as an excellent routine method and alternative for HRMS. As soon as dioxin and dl-PCB concentrations drop to low levels, i.e. lower than the MRLs and action levels, a lot of time is needed to produce congener-specific data, to integrate the chromatograms, to adjust the baseline, to compare relative retention times, etc. However, even then GCxGC may still serve as a screening method. GCxGC-ToF-MS is an improvement compared to GCxGC-ECD, but obviously the instrument is considerably more expensive. The fourth method that was studied, an Ah-PCR analysis, is still under development. It is not excluded that that technique may finally also result in a suitable alternative screening method.