

# DIFFERENCE/DIAC NEWSLETTER

A Newsletter of the European Research projects 'Dioxins in Food and Feed – Reference Methods and New Certified Reference Materials' (DIFFERENCE) and 'Dioxin Analysis by Comprehensive Multi-dimensional Gas Chromatography' (DIAC).

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## Progress

DIAC and DIFFERENCE, two European research projects on the development of alternative methods for the determination of dioxins in food and feed, have made good progress over the last half year. The different methods are optimized prior to the validation in the interlaboratory study. The first round of the interlaboratory study, comparing GC-HRMS with GCxGC, GC-LRMS and CALUX was conducted under DIFFERENCE, with a second one currently being organised. The first meeting with the panel of experts was held in February in Brussels.

## Extraction and clean-up

The ultimate goal is to come up with a strong alternative method to the currently existing extraction methods. Microwave Assisted Extraction (MAE) can be used to meet this goal and has been tested. However, Accelerated Solvent Extraction (ASE) is most likely the most potent technique as it combines extraction and clean-up. Two different ASE extraction strategies have been tested but both of them still require

further development. The first method is a non-selective extraction of PCBs and dioxins, in which the fat and the contaminants are extracted. The extracts then need to undergo external clean-up just as in classical extraction methods. The second method is a selective extraction of PCBs and dioxins, in which the contaminants are extracted while the fat is left behind in the extraction cell by means of a fat retainer. This concept was first presented in 1996 but was only recently investigated in more detail. The influence of the fat-fat

retainer ratio (FFR) was tested as well as different extraction solvents and temperatures. Preliminary results show that nearly all the fat is retained in the extraction cell at a FFR of 0.50 (Figure 1). This means that with the application of a fat retainer clean and fat-free extracts can be obtained for fractionation and detection. Other strategies for combined extraction and clean-up are currently being investigated.

## 1st Interlaboratory Study

The goal of the inter-

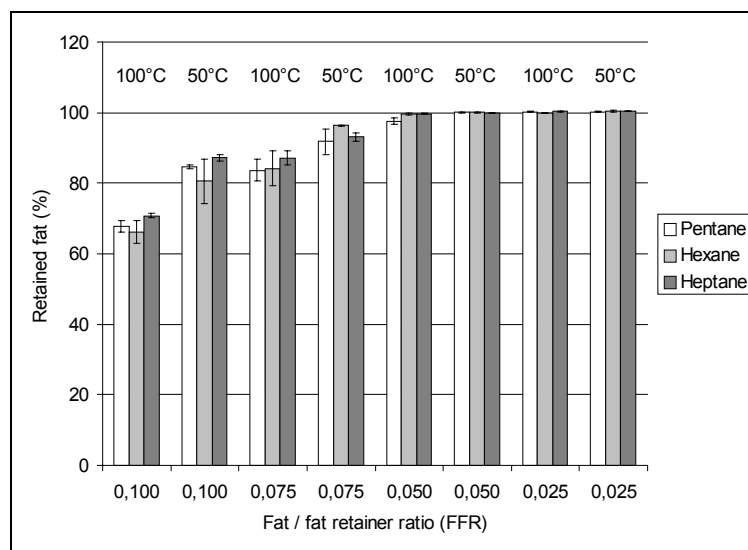


Figure 1. Preparation of cleaned ASE extracts by retention of fat in the extraction cell at 50 and 100°C using different solvents. The retention of fat rather depends on the fat-fat retainer ratio (FFR) than on the solvent used.

laboratory studies is to validate the alternative methods for the determination of dioxins and dioxin-like PCBs in food. The validation scheme consists of three rounds assessing the detection capability, robustness, accuracy, selectivity and linearity of the different techniques.

In the first round, a spiked vegetable oil, a cleaned fish extract, a fish oil and a milk sample were tested.

The results of the first round showed generally good agreement among the different screening techniques and with GC-HRMS. The Z-scores for the spiked vegetable oil sample were almost all within the satisfactory range of -2 to 2 for the spiked vegetable oil sample (Figure 2). The GCxGC-ECD technique was somewhat outlying due to the combination of reporting upperbound levels and high detection limits. Attention will be paid to lowering the detection limits so that they will meet the strict criteria for analysis of food and feed samples. Round 2 and 3 are currently ongoing and will include tests on interferences and different food and feed matrices.

### Meeting with Expert Panel

At 13 February the DIFFERENCE and DIAC teams met with the panel of experts to present the results of the first project year. The constructive meeting has resulted in a report of the

experts in which they express their appreciation and positive expectations of these projects, and also give a number of suggestions for improvement. They stressed in particular the need to obtain enough sensitivity with GCxGC and to prepare candidate certified reference materials with concentrations around the European actions limits, rather than around the tolerance limits. The panel was pleased to see the good results obtained with GC-MS/MS. The next meeting with the experts is scheduled for February 2004.

### Presentations at Dioxin 2003 conference

Several presentations of the results obtained during the project will be presented at Dioxin 2003 in Boston, USA. S. van Leeuwen (RIVO) will present a general outline of the project with some results of the first round of the validation of the screening assays. P. Korytar (RIVO) will give a presentation on

progress in GCxGC (DIAC). S. Sparring (Lund University) and P. Haglund (Umea University) will both present results on fast, and selective ASE extraction of the target contaminants from various matrices. More presentations are expected on CALUX and GC-LRMS.

### Web site

The address of the DIFFERENCE/DIAC website is [www.dioxins.nl](http://www.dioxins.nl).

### Questions?

For specific questions, comments, suggestions, etc., please email to: [Stefan.vanleeuwen@wur.nl](mailto:Stefan.vanleeuwen@wur.nl) (DIFFERENCE), and [Pim.Leonards@wur.nl](mailto:Pim.Leonards@wur.nl) (for DIAC).

This newsletter is scheduled to appear once per half year

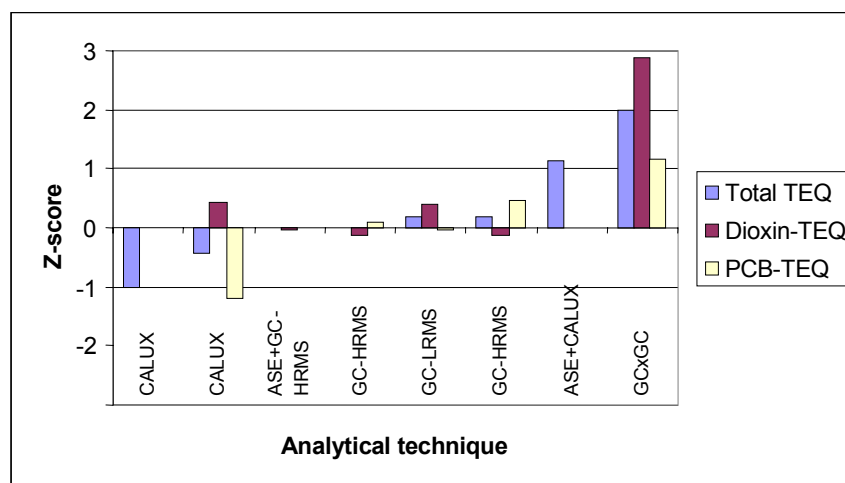


Figure 2. Results of the 1<sup>st</sup> validation round on a spiked vegetable oil. Z-scores of the different screening techniques are compared with the reference technique GC-HRMS. Nearly all techniques show good z-scores of <2.