

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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The two European research projects DIAC and DIFFERENCE are approaching their finalization. The DIAC project ends at 1 October 2004, the DIFFERENCE project at 31 January 2005. This newsletter gives a brief overview of the achievements so far.

Experts support New Methods - Jacob de Boer

An important meeting for the DIFFERENCE and DIAC projects took place at 14 April of this year in Antwerp. At that day the project partners met for the second time with the expert panel: dr. Frans Verstraete (EU DG SANCO), dr. Peter Behnisch (Eurofins, Münster, Germany), dr. Rainer Malisch (CVUA Freiburg, Germany), and dr. Bert van Bavel (Örebro University, Sweden). The results so far obtained in the two projects

were presented by the various partners. The results of the interlaboratory tests and validation studies, coordinated by Joris van Loco (Scientific Institute for Public Health, Brussels) shown below, were well received by the experts. Although a number of useful comments were given by the experts, it may be expected that the two methods GC-LRMS/MS and GCx GC-ECD will be good enough to be used as routine methods or even, in the case of GC-LRMS/MS as a reference method. For each method, a realistic calculation of time and costs

needed for the analysis (laboratory work until an extract is ready for detection and time for detection of the substances of interest including evaluation of the acquired data) should be provided. Also, further evidence is needed on the sensitivity of the methods to be used at a level below the European action levels. Finally, the integration of the GCxGC chromatograms is still time-consuming but good progress has been made in new software packages for this technique.

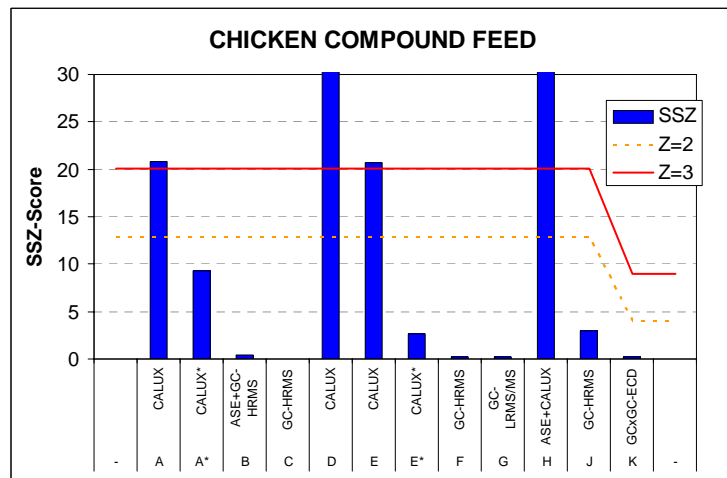


Figure 1: Sum of the squared Z- scores (SSZ) for the total TEQ Chicken Compound Feed results (evaluated with a chi-square test. The full and dotted line represents the SSZ-score which corresponds with the same probability of Z = 3 and 2, respectively)

Table 1: Chicken compound feed validation data

Lab	Method	n	Mean conc. (ng Σ TEQ/kg)	Bias %	CV _r %	CV _w %	CV _R %
A	CALUX	6	0.730	-55.6	12.0	13.0	50.3
D	CALUX	6	0.262	-84.1	9.6	9.6	
E	CALUX	6	0.740	-55.0	17.4	22.9	
A*	CALUX*	6	1.043	-36.6	12.0	12.8	42.9
E*	CALUX*	6	1.895	15.2	13.7	13.7	
C	GC-HRMS	6	1.642	-0.2	1.1	1.1	14.5
F	GC-HRMS	6	1.716	4.3	3.4	3.6	
J	GC-HRMS	6	1.302	-20.9	1.0	4.2	
K	GCxGC-ECD	1	1.415	-14.0	-	-	-
G	GC-LRMS/MS	6	1.741	5.8	4.9	5.3	-
H	ASE+CALUX	6	0.475	-70.7	6.9	23.4	-
B	ASE+GC-HRMS	6	1.733	5.4	2.8	19.3	-

CV_r: repeatability CV, CV_w: within lab reproducibility, CV_R: reproducibility.

*: Corrected for recovery

Results of Interlab Studies – Joris van Loco

The DIFFERENCE project focuses on the development, optimisation and validation of screening methods for dioxin analysis, including bio-analytical and chemical screening techniques (CALUX, GC-LRMS/MS, GCxGC-ECD) and on the optimisation and validation of new extraction and clean-up procedures. The performance of these techniques is assessed in an international validation study and the results are compared with the reference technique GC-HRMS.

The first round of the validation study focussed on the goodness-of-fit of the calibration curve and on the accuracy of the methods. In round 2 the detection capability and selectivity were assessed. In round 3, the accuracy and robustness of the methods were evaluated on several samples of different origin (pork tissue, chicken, compound

feed, sepiolithic clay, egg, chicken tissue and vegetable oil). The results of round 3 are summarized in Table 1. The CV_w is below 30% for all the labs, which is the EU tolerance for screening method results. (no precision data are available for GCxGC-ECD). The uncorrected CALUX results underestimate the total TEQ concentration of the feed sample. The results of the CALUX labs (= lab A, D, E and H) are more than 50% lower than the median of the GC-HRMS results. The SSZ-scores of the CALUX labs are unsatisfactory (Figure 1). Applying recovery correction improves the total TEQ results of these labs, since the SSZ-scores for CALUX* are satisfactory. The results of the GC-LRMS/MS and GCxGC-ECD are all satisfactory, however only 1 result for GCxGC-ECD was reported. The results of the ASE + GC-HRMS show a larger variation than those of GC-HRMS with conventional extraction, but

their SSZ-score is still satisfactory.

Workshop

At 13 January 2005 the final workshop of the two projects will be held in Brussels. The workshop is open for everybody interested in dioxin analysis. More information can be found at the website.

Film

In the week of 27 September shooting of the film "A World of DIFFERENCE" has taken place at RIKILT, RIVO and various other locations in The Netherlands. The film that will be sent to authorities and dioxin laboratories in Europe will be presented by Kate Sanderson, anchorwoman of the 07.00 News of the English television..

Website

The address of the DIFFERENCE/DIAC website, now including the refs. of DIFFERENCE/ DIAC presentations made at the recent Dioxin2004 Symposium in Berlin, and the special issue of Talanta including a number of papers on DIFFERENCE/DIAC work is www.dioxins.nl.

Questions?

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The next newsletter is scheduled to appear in December 2004.