

IV REUNIÓN NACIONAL DE DIOXINAS, FURANOS Y COMPUESTOS ORGÁNICOS PERSISTENTES RELACIONADOS

Alicante, 26-28 Junio 2013



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**IV Reunión Nacional de Dioxinas,
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ANALYTICAL METHODOLOGY SET UP FOR THE ANALYSIS OF DIOXINS AND FURANS IN ANIMAL FEED AND FEED ADDITIVES

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Introduction

Dioxins (PCDD) and furans (PCDF) are environmentally persistent substances that have been associated with human health effects. The major source of human background exposure to dioxins is food, representing more than 90%, with food of animal origin being the predominant source. Since the dioxin contamination of animal feed in Belgium in 1999, public concern about PCDD and PCDF levels in animals and food has been raised and a number of measures to protect and improve the quality of human health have been enforced in the European Union, mainly regarding the methods of sampling and analysis for the official control of feed¹ and regarding the maximum levels for dioxins, furans and polychlorinated biphenyls (PCBs)². Although a general decrease in dietary exposure of dioxins has been observed between the periods 2002-2004 and 2008-2008 as reported by the European Food Safety Agency³, several studies have found high levels of PCDDs and PCDFs in animals and food resulting from the use of contaminated animal feed⁴, and since feed can contribute considerably to the contamination of food, it is crucial to monitor the dioxin contamination of feeds and feed additives.

The main objective of this work is to set up an analytical scheme for the simultaneous analysis of PCDD/Fs and dioxin-like PCBs in animal feed and feed additives by high resolution gas chromatography coupled to high resolution mass spectrometry (HRGC-HRMS) according to EPA Method 1613, EPA Method 1668 and following the minimum requirements described in Commission Regulation (EU) N° 278/2012.

Materials and Methods

Samples

Different feed matrices were analyzed like calcium pidolate, calcium bicarbonate, silages and fish meals.

Instrumental analyses

At the laboratory samples were homogenized and grounded when necessary as pretreatment steps. Samples were extracted in Soxhlet for about 24h with toluene and anhydrous sodium sulfate after having been spiked with known amounts of mixtures of 15 ¹³C₁₂-PCDD/Fs (EPA-1613LCS, Wellington Lab., Guelph, Canada) and 12 ¹³C₁₂-DL-PCBs (WP-LCS, Wellington Lab., Guelph, Canada) to check the recoveries. Next, the extracts were rotary evaporated and with nitrogen until dryness in order to eliminate the solvents prior to gravimetric fat determination. After that, fat residues were redissolved in n-hexane and treated with sulphuric acid to eliminate fat, organic compounds and other interfering substances. The fractionation and further sample purification was carried out in an automated Power-PrepTM System (FMS, Inc, MA, USA) through a multilayer silica column, then a basic alumina and finally a PX-21 active carbon column. In some cases an additional Jumbo column was necessary to get a better purification of samples. Instrumental analysis was performed according to EPA method 1613 by HRGC-HRMS by means of a TRACE GC UltraTM gas chromatograph fitted with a 60m x 0,25mm x 0,25 µm film thickness DB-5ms fused silica column (J&W Scientific, CA, USA) coupled to a high-resolution magnetic sector mass spectrometer (HRMS) DFS model (Thermo Fisher Scientific, Bremen, Germany) set at a resolution of 10000 and controlled by a Xcalibur data system. Positive electron ionization (EI+) with an electron energy of 45 eV operating in the MID mode was used. For all compounds, the two most abundant signals of the molecular ion clusters were monitored for each group of PCDD/Fs or PCBs with the same degree of chlorination. Quantification was done according to the isotope dilution technique. Final results were expressed in WHO2005-TEQ.

Results and Discussion

Acceptable recoveries higher than 60% was obtained for 15 $^{13}\text{C}_{12}$ -labelled 2,3,7,8-PCDD/Fs congeners and 12 $^{13}\text{C}_{12}$ -labelled dioxin-like PCBs. Blanks covering the whole analytical methodology were included. Some difficulties were found for DL-PCBs analyses of congeners PCB77, PCB 81, PCB 123 and PCB 114, for which significant errors were found in calculate concentrations.

Total WHO2005-TEQ Levels found in the analyzed samples in this preliminary study were in all cases below the maximum levels established by Commission Regulation (EU) N° 277/2012, being the highest for the fish meals.

Acknowledgements

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References

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