PCDDs/PCDFs AND DIOXIN-LIKE PCBs IN FEEDINGSTUFFS FOR FISH

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Introduction

The range of contributions to the average daily intake of dioxins in ten countries of the EU was assessed in the EU SCOOP report (1). The main contributors are milk and dairy products (contributions ranged from 16-39 %), meat and meat products (6-32 %) and fish and fish products (2-63 %). The Scientific Committee on Food (SCF) summarized the national figures of the SCOOP database with the following results for food of animal origin (in pg I-TEQ/g lipid basis): eggs around 1, wild fish and farmed freshwater fish in the order of 10 for dioxins (and 30 pg PCB-TEQ/g fat), meat 0.5 – 0.7, milk about 0.6 to 1.0 (2). As about 95 % of the dioxin intake comes from food and here about 90 % from food of animal origin, feedingstuffs are a decisive parameter to control the dioxin intake via the food chain. The Scientific Committee on Animal Nutrition concluded that fish meal and fish oil are the most heavily contaminated feed materials with products of European fish stocks more heavily contaminated than those from South Pacific stock. The contribution of individual feed materials to the dioxin content of the whole diet for farmed animals depends on the degree of the contamination and the proportion used in the diet. Greatest concern arises from the use of fish meal and fish oil of European origin. These are most critical when used in diets for farmed fish and where fish meal is incorporated in diets or other food producing animals (3). Therefore, feedingstuffs were analysed to determine the extent of contamination as most important factor for the dioxin contamination of farmed fish.

Materials and methods

The authorities for control of feedingstuffs of the German State of Baden-Württemberg collected 28 samples of feedingstuffs for fish between October and December 2000 and 24 samples between January and March 2001. The samples were analysed according to methods which were presented for different sorts of food (4, 5, 6). The feedingstuffs were extracted with cyclohexane/toluene (1:1) in a hot extraction device for 8 hrs. After evaporation of the solvent, the amount of residue was determined (fat content). Then, the usual clean up procedure was applied as described in the above mentioned references (gel chromatography on Bio Beads S-X3, eluent ethyl acetate/cyclohexane; sulphuric acid impregnated silica column; florisil column; Carbopack C column). GC/MS determination was performed on a VG Autospec at 10,000 resolution on a 60 m DB5-MS-column; for confirmation a DB-Dioxin column was used.

Results and discussion

As reaction to the SCOOP, SCF and SCAN report, 28 samples of feedingstuffs for fish were collected between October and December 2000 and analysed for their dioxin content. Additionally, 9 of these samples were analysed for their content of dioxin-like PCBs. Table 1 summarizes the

results. The median of all samples was 1.56 ng WHO-TEQ/kg product (only PCDDs/PCDFs included), 3.72 ng PCB-TEQ/kg product and 5.66 ng WHO-TEQ/kg product for the sum of PCDDs/PCDFs and dioxin-like PCBs. As a result of this particular aspect, the WHO-TEQ content of feedingstuffs for fish increases by a factor of 3.6 when dioxin-like PCBs are determined in addition to PCDDs/PCDFs, only.

Table 1: Results of feedingstuffs for fish, samples collected between October and December 2000

	WHO-TeQ PCDD/F ng/kg product	WHO-TeQ PCB ng/kg product	WHO-TeQ PCDD/F+PCB ng/kg product	WHO-TeQ PCDD/F pg/g fat	fat content (%)
No. of samples	28	9	9	28	28
Min	0.12	0.91	1.43	1.19	10.3
Median	1.56	3.72	5.66	7.50	21.5
Mean	1.60	3.50	5.75	7.51	20.6
90 % Percentile	2.25	5.04	8.84	10.79	24.4
95 % Percentile	3.38	5.82	9.67	12.34	24.9
Max	3.89	6.60	10.49	18.85	29.9

The German Federal Institute for Health Protection of Consumers and Veterinary Medicine (BgVV) recommended that an action level of 1 ng WHO-TEQ/kg (only PCDDs/PCDFs included) for feedingstuffs for fish should not be exceeded in the short run and of 0.5 ng WHO-TEQ/kg product not in the future (containing about 10 – 15 % fish oil). (7). The German authorities for control of feedingstuffs supported this approach, and it was concluded that these action levels should be applied for legal evaluation. The producers of farmed fish and of feedingstuffs for fish were informed of this policy. About 85 % of all samples collected in the last quarter of 2000 exceeded the action level as proposed in October 2000, with a maximum of about four times the proposed immediately applicable action level.

In order to check whether measures were taken quickly to reduce the dioxin content in feedingstuffs, 24 samples were collected from January to March 2001. Table 2 presents the results.

Table 2: Results of feedingstuffs for fish, samples collected between January and March 2001

	ng WHO-TEQ/kg produkt	pg WHO-TEQ/g fat	fat %
No. of samples	24	24	24
Min	0.08	0.70	8.6
Median	0.46	2.39	12.0
Mean	0.44	3.25	14.1
90 % Percentile	0.78	6.55	21.3
95 % Percentile	0.83	7.91	21.8
Max	1.00	10.61	22.1

It was surprising to see that all samples of the first quarter of 2001 were below the proposed immediately applicable action level of 1 ng WHO-TEQ/kg product. 75 % of the samples were even below the action level of 0.5 ng WHO-TEQ/kg product (only PCDD/PCDF included) as proposed by BgVV for the future.

Immediately, the results were confirmed by repeated analysis. All results of the year 2000 had been analysed in double, already. Thus, the third repeated analyses of 4 selected samples were performed together with 4 selected samples from 2001. As a result, the fat content was confirmed with a variation of about 0.1 to 0.2 %. In comparison to the fat content as declared on the packaging of the samples of 2000, the median of "recovery" of the fat determinations was 98.2 % of the declared fat amount, for the 2001 samples 94 %. The dioxin content was confirmed with a variation between 0.3 and 3.6 % in comparison to the previous analyses. Therefore, the reduction was clearly a result of changes in the product.

The significant reduction of the dioxin content on product basis was the result of two parameters:

1) The dioxin content on fat basis was reduced: Whereas the samples of 2000 had a mean and median dioxin content of about 7.5 pg WHO-TEQ/g fat (only PCDDs/PCDFs included), the samples of 2001 had a mean dioxin content of 3.25 and a median dioxin content of 2.39 pg WHO-TEQ/g fat. This could have been achieved by use of lower contaminated fish meal (see SCAN report: fish meal of European waters is about 8times higher contaminated than fish meal from Pacific waters [Chile, Peru]) and/or by use of refined fish oil: According to our own results of samples collected at the same time, 3 samples of refined fish oil had a dioxin content between 0.4 and 1.5 ng WHO-TEQ/kg fat, whereas a sample of not refined fish oil was highly contaminated (10.4 ng WHO-TEQ/kg fat).
2) The fat content was reduced by about one third: Whereas the samples collected in 2000 had a fat content of about 21 % on average (range 10.3 to 29.9 %), the samples from 2001 had a fat content of about 14 % on average (range 8.6 to 22.1 %). This is in the range of 10 – 15 % which was mentioned in the BgVV recommendations as typical for feedingstuffs for fish.

As a result, the dioxin content of feedingstuffs for fish could be reduced to levels below the proposed immediately applicable action level of 1 ng WHO-TEQ/kg product (inclusion only of

PCDDs/PCDFs) within short time. 75 % of the samples fell below the action level of 0.5 ng WHO-TEQ/kg product which should be applied in the future. This progress is even more important as dioxin-like PCBs increase the previously high WHO-TEQ results of feedingstuffs for fish by a factor of about 3.6. The effects on the development of dioxin levels of fish will be followed in separate studies.

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