# **BACKGROUND CONTAMINATION OF FEEDSTUFF WITH DIOXINS**

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## Introduction

The major source of human background exposure is food (more than 90 %) with food of animal origin being the predominant source. The recent cases of contamination of feedingstuffs (citrus pulp pellets, oils and fats and kaolinitic clay) highlighted the impact of contaminated feedingstuffs as a source of contamination of food of animal origin with dioxins. In 1998, a WHO consultation group for assessment of risks for human health agreed on a tolerable daily intake (TDI) of PCDD/Fs and dioxin-like PCBs in the range of 1 - 4 pg WHO-TEQ/kg bw/day, stressing that the upper range of the TDI of 4 pg WHO-TEQ/kg should be considered as a maximum tolerable intake on a provisional basis and that the ultimate goal is to reduce human intake levels below 1 pg WHO-TEQ/kg bw/day. The actual daily intake usually exceeds this ultimate goal. Therefore, it is mandatory to reduce the uptake of dioxins through the food chain. An important tool in this approach is the definition of the the actual background contamination of feedstuff as tool to identify and to withdraw possibly elevated feedstuff.

#### Methods and materials

The Chemisches und Veterinäruntersuchungsamt Freiburg analyzed 245 samples of feedstuff for their dioxin content. The analytical method is very similar to the method for analyses of vegetable food which was tested succesfully in a collaborative study for kale (1).

### **Results and Discussion**

Numerous reports or publications show a wide range of dioxin contents in feedstuff. However, most publications do not contain information on two important points:

- the upper bound limit of determination of the method
- whether possibly contaminated feedstuff is included

For comparison of analytical results, the limits of detection or limits of determination have to be taken into account. Some laboratories used to calculate the contribution of not detectable congeners to the TEQ as "0". As result, low dioxin contents could have been the result of really low levels of the sample or of insufficient detection / determination limits without considering these detection / determination limits in the final TEQ calculation. To make sure that low dioxin levels are really the result of low levels in the sample, the concept of tolerances as upperbound detection limit" or "upperbound determination limit" was developed. This concept should be applied generally, with a clear preference of "upper bound determination limits". When the determination limits are high for the decisive congeners, high numbers of TEQ result. This effect has to be considered for the definition of background contamination. Especially the use of low resolution mass spectrometers in feedstuff analysis or a low weight-in quantity of a sample (for a quick and easy analysis) can cause relatively high "values" of dioxin contents as upper bound determination limits. This cannot be seen from a reported TEQ level without knowledge about the results of the individual congeners. Thus, for definition of a background contamination, published data must be reviewed critically to avoid that relatively high values are included which are only the result of insufficient detection limits.

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# **POPS IN FOOD**

It is difficult to conclude the range of the normal background contamination from published results because also elevated levels from contaminated samples could be included. (For reasons of space, a summary of available data has to be presented separately.) Therefore, the results of dioxin determinations in 245 feedstuff samples which were analysed between 1993 and 1999 in the Chemisches und Veterinäruntersuchungsamt Freiburg give an orientation in this discussion. Almost 80 % of these samples were analyzed in 1998 and 1999, due to severe problems with dioxin contamination of feedstuff (Brazilian citrus pulp, Belgian crisis and kaolinitic clay).

From all 245 analyzed samples, 72 samples were related to a specific dioxin contamination (Brazilian citrus pulp pellets; kaolinitic clay; hay from PCP-treated storage rooms; grass fertilized with contaminated Thomas phosphate). The remaining 173 samples were considered to reflect the background contamination and evaluated statistically. The samples were differentiated into four groups according to systematic division used in feedstuff science (2). The following table summarizes the statistical evaluation. All data were recalculated for the WHO-TEQ content as upper bound determination limit (with inclusion only of PCDD/F).

The usual background contamination of products of the groups A2, A3 and A4 is far below 0.2 ng (= 200 pg) WHO-TEQ/kg (air-)dried matter. Feedstuff of the group A1 has a tendency to slightly higher levels. This is the result of numerous grass samples which are here included. Grass has a big surface area with wax layers which is able to absorb PCDD/F. In comparison to grass and leaves, tubers, roots, cereals, seeds or other by-products from plant origin have much less surface layer for absorption. The time for growth (season of the year) could have an influence on the dioxin content as the growth rate is different. To take into account as many parameters as possible, the grass, hay and grass silage samples of this study were taken at different times and reflect rural and highly populated areas, however without known dioxin emitters in the surrounding area. Most compound feeds (group D) are below 0.1 ng (= 100 pg) WHO-TEQ/kg (air-)dried matter, with decreasing numbers of samples having a dioxin content up to 0.25 ng (= 250 pg) WHO-TEQ/kg.

As a result, for feedstuff of the groups A2 to A4 or D a cut off value of 0.5 ng (= 500 pg) WHO-TEQ/kg (air-)dried matter (upper bound determination level; only PCDD/F included) could be used as a criterion to distinguish background contamination from elevated levels. The usual background contamination is in the range < 0.1 to < 0.3 ng (= < 100 to < 300 pg) WHO-TEQ/kg (air-dried) matter. Grass, hay and grass silage have a tendency to slightly higher dioxin levels. Many samples from the group A1 from rural and highly populated areas are below this cut off value, as well, however more data from other areas in Europe could help to broaden this overview.

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7	Table: Dioxin results of feedstuff samples (WHO-TEQ as uppe	r bound determination limit, with inclusion only of PCDD/F)
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	TeQ/	TeQ/ kg (air-)	pg WHO- TeQ/ kg (air-) dried matter	TeQ/	TeQ/ kg (air-)	TEQ/	TeQ/ kg fat	TeQ/	pg WHO- TeQ/ kg (air-) dried matter
COMPOLINDS	A1: forages and conservates	2	A3: cereals and seeds	A4: by- products of plant origin	Summary: all samples of group A without specific contami- nation		B2: pure fat or oil (definitely or probably of animal origin)	C: mineral feed	D: compound feed
no. of samples	67	2	8	19	96	3	19	22	33
mean	188.6	36.4	37.1	71.8	149.7	108.3	1040	126.0	67.7
median	148.0	36.4	10.3	62.4	120.5	103.8	680	71.3	52.1
minimum	36.7	30.1	6.6	16.0	6.7	61.0	260	15.1	11.4
maximum	509.3	42.7	184.3	132.3	509.3	160.1	6640	483.0	246.0
90 %-percentile	335.6	41.4	92.4	124.3		148.8	1420	297.7	136.1
95 %-percentile	380.1	42.1	138.3	131.9		154.4	2420	355.0	154.5

Also for mineral feeds, the majority of samples has a dioxin content below 0.1 ng (= 100 pg) WHO-TEQ/kg. Most samples were analyzed shortly after the first reports about the new detected dioxin source (kaolinitic clay). At that time, some manufacturers of mineral feed had different stocks of raw material. Thus, mineral feeds from the market from the same producer could have a wide range of contamination. Samples below 0.5 ng (= 500 pg) WHO-TEQ/kg were considered "not contaminated". However, it cannot be excluded that after elimination of the dioxin source, the frequency distribution in the range between 0.1 and 0.5 ng (= 100 and 500 pg) WHO-TEQ/kg would change to lower dioxin values.

Except one sample, feedstuff of animal origin had a dioxin contamination below 2000 pg WHO-TEQ/kg fat (upper bound determination level; only PCDD/F included). Most samples were below 1000 pg WHO-TEQ/kg fat. One sample had a dioxin content of 6640 pg WHO-TEQ/kg fat. For a broad overview, however, it is necessary to include the contribution of fish oils, as well. The figure here can represent only feedstuff of animal origin which was sampled randomly at certain occasions. It is possible that more samples of pure fish oil or fat with only a part of fish oil is on the market.

As a result, the average background contamination of vegetable feed and mineral feed is in the range  $_{,,<} 0.1$  to < 0.3 ng (= < 100 to < 300 pg) WHO-TEQ/kg d.m. (upper bound determination level; only PCDD/F included)". Numerous samples exceeded this range, partly considerably, however with hints on or clear identification of dioxin sources. Thus, these elevated samples cannot be taken into account if the background range should be defined. As conclusion, it would be a wrong approach to summarize simply any reported data and to try to define the background contamination from these data if there is no information given whether possibly contaminated samples are included. Feedstuff of animal origin besides fish has a background contamination below 1000 pg WHO-TEQ/kg fat. Published data show that the only feedstuff with generally elevated dioxin levels is fish meal and oil, with fish from the northern hemisphere being contaminated much more than fish from the southern hemisphere.

Tolerances for feedstuff could be based on two components:

- a value for cut-off of elevated levels, e.g. above 0.5 ng WHO-TEQ (only PCDD/F included)/kg d.m. for vegetable (except grass) and mineral feed
- an ultimate target value below the present background contamination, in order to reduce the average daily intake according to the WHO recommendation

For grass, a specific evaluation with inclusion of data from other areas is recommendable which has to include the concept of upper bound determination levels and the indication of distances to possible dioxin sources.

<sup>&</sup>lt;sup>1</sup> Malisch R., Bruns-Weller E., Knoll A., Thoma H. and Peichl L. (1997) Results of a quality control study of different analytical methods for the determination of PCDD/PCDF in kale samples

<sup>&</sup>lt;sup>2</sup> Jeroch H., Flachowsky G. and Weißbach F. (1993) Futtermittelkunde. Gustav Fischer Verlag Jena, Stuttgart