

PCDD/PCDF and Dioxin-like PCBs in Animal Fat Samples from Switzerland: A Monitoring Program for Revising Swiss Food Limits

Horst Rottler¹, Claude Wüthrich², Arnold Kuchen³

¹Eurofins Oekometric, Bayreuth

²Swiss Federal Office of Public Health, Schwarzenburgstrasse 165, CH-3097 Liebefeld, Switzerland claud.wuethrich@bag.admin.ch

³Swiss Federal Office of Public Health, Schwarzenburgstrasse 165, CH-3097 Liebefeld, Switzerland arnold.kuchen@bag.admin.ch

Introduction

The European Commission (EC) stipulated maximum levels for dioxins in food in 2001¹ as an outcome result of their strategy to reduce dioxin and PCB levels². At that time the data base for dioxin-like PCBs was regarded as insufficient to include maximum levels for PCBs. Consequently, a study was initiated by the EC on a European-wide scale to collect information on dioxin-like PCBs in feed and food³. The decision on European limits for dioxin-like PCBs is announced for end of 2004.

As Switzerland is not a member of the European Union, European Commission limits are not valid per se and must be reassessed. As laid down in the Swiss Food Law it is the task of the Swiss Federal Office of Public Health (SFOPH) to collect the basic information necessary for initiating appropriate measures to protect consumers from foodstuff that could adversely affect their health. In this context information about total dioxin intake of the Swiss population is necessary.

To this point, some monitoring programs have been carried out in the past. In 1984 and 1990/1991, investigations on Swiss cow milk from various locations were initiated by the Swiss Agency of Environment, Forest and Landscape (SAEFL) and the SFOPH⁴. A similar study was conducted by SAEFL in 2002 to reveal temporal and local trends of PCDD/PCDF⁵. As a consequence of the "Belgium dioxin scandal" in 1999 a dioxin control program was started to reveal if any contaminated food or feed entered the Swiss market⁶. Since then the SFOPH conducted two further analysis programs for eggs and fish from Swiss lakes⁷.

In view of the upcoming revision of the Swiss Ordinance on Foreign and Toxic Components in Foodstuffs (FIV) which should integrate limits for dioxins in food⁸, it is now important to know whether the maximum contaminant limit for dioxins as fixed by the European Commission is also appropriate for Swiss consumer protection purposes. To answer this question and to fill existing data gaps in view of a more accurate calculation of the total dioxin intake of the Swiss population, an additional dioxin monitoring program started end of 2003 especially focusing on animal fat. Dioxin-like PCBs were included to complete the Swiss data base and to assess the Swiss intake situation for these compounds just in time before the corresponding European activities on dioxin-like PCBs.

Methods and Materials

Monitoring Design and Sampling: The monitoring program targeted principally the animal fat samples (cattle and pig). Additionally selected butter and fish samples were included in the program (no representative sample numbers; not presented here).

Monitoring design was intended to provide a country-wide survey, covering both, regional and production differences (e.g. in-door/outdoor, conventional/organic). Cattle samples included a differentiation according to age and sex:

- calf: < 9 month, male and female
- heifer beef: 9 – 18 month, female, no first calf
- steer beef: 9 – 18 month, male
- cow: > 36 month, female, at least one calf born

106 individual samples of cattle and pig have been collected, 60 samples of them were taken for preparing eight pool samples (each containing 3 – 10 individual samples) resulting in a total of 54 samples for analysis.

Samples were taken in slaughterhouses. About 100 g per sample were taken from animal depot fat (kidney) – packed in glass vessels, sealed with aluminium foil, refrigerated and sent to the laboratory where they were kept frozen until analysis.

Analysis: Analyses of dioxins and dioxin-like PCBs were executed in the Oekometric laboratory accredited according to ISO 17025 standard using isotope dilution method with $^{13}\text{C}_{12}$ -labeled dioxin and PCB standards.

Fat samples were mixed with an equal amount of silica and about 3.5 times that amount of Na_2SO_4 . Cold extraction was executed using a 1:1 (v:v) mixture of hexane and dichloromethane.

Lipid content was determined by weight after evaporation of the solvent. Cleanup was based on mixed silica column and aluminium oxide column methods. For analysis, non-ortho- and mono-ortho-PCBs were separated in two different fractions on a further aluminium oxide column.

Measurement of dioxin, non-ortho and mono-ortho PCBs were performed using high resolution capillary gas chromatography with a DB5-MS, DB5 and a DB-Dioxin GC-column (HRGC) and high resolution mass spectrometry (HRMS) on a Finnigan MAT 90 and MAT 95 system, respectively. The mass resolution was in the range of 6'000 – 10'000. For 30 g sample input usual achievable limits of quantification (LOQ) were 0.07 pg PCDD/F-WHO-TEQ/g lipid for the dioxins and 0.03 pg WHO-PCB-TEQ/g lipid for the dioxin-like PCBs.

Analysis met QA/QC requirements for official dioxin and dioxin-like PCBs control as laid down by the European Commission⁹. Results are reported as upper bound concentrations.

Results and Discussion

PCDD/PCDF: Considering the European maximum levels for animal fat intended to be included into the Swiss Ordinance on Foreign and Toxic Components in Foodstuffs (cattle fat = 3 pg WHO-PCDD/F-TEQ/g lipid; pig fat = 1 pg WHO-PCDD/F-TEQ/g lipid^{1,8}) results given in Table 1 show no exceeding of these levels, for both pig and cattle samples. With one exception, the pig samples are below 0.3 pg WHO-PCDD/F-TEQ/g lipid and are therefore significantly below the European action level of 0.6 pg WHO-PCDD/F-TEQ/g lipid intended to represent an upper background level in Europe¹⁰. The same observation is made for cattle (European action level = 2 pg WHO-PCDD/F-TEQ/g lipid) with most results below 1.0 pg WHO-PCDD/F-TEQ/g lipid.

Results are in good agreement with data from Germany^{11,12} indicating that there is no significant difference in the dioxin load of animals fat from neighbor regions. The significant difference between pig and cattle fat levels of dioxins (roughly a factor 4) is also in agreement with other data¹².

PCB: Up to a certain point the results from Switzerland are comparable with the results from a European-wide survey³. The median dioxin-like PCB content for pig fat is lower for the Swiss samples (0.18 compared to 0.28 pg WHO-PCB-TEQ/g lipid). The European survey showed no levels below 0.2 pg WHO-PCB-TEQ/g lipid while more than half of the Swiss samples are below. The opposed tendency is apparent for the cattle samples, with a higher median for the Swiss samples (2.5 compared to 1.4 pg WHO-PCB-TEQ/g lipid).

The importance of the dioxin-like PCBs to the total WHO-TEQ intake from these foodstuffs is obvious for both cattle and pig. For cattle the TEQ-ratio of dioxins:PCBs is by far dominated by the PCBs (~ 1: 4) while for pig fat there is roughly a 1:1 situation. In contrast to dioxins, the variance for PCBs is significantly larger in cattle while it is in similar range for pig samples. Furthermore, the increase ratio from pig to cattle is different for dioxins and PCBs. While the average increase for dioxins is about a factor of three and therefore is in good agreement with the ratio of the European levels for pig and cattle samples, the average increase for PCBs reaches a factor of 14 in the analyzed Swiss samples.

Table 1: Levels of dioxins and dioxin-like PCBs (pg WHO-TEQ/g lipid; upper bound) in Swiss animal fat samples (minimum, maximum, median, 90 percentile and variance)

		pg WHO-TEQ/ g lipid				
	n	Min	Max	Median	P90	Variance
Pig						
PCDD/PCDF	16	0.12	0.66	0.15	0.28	0.02
WHO-PCB	16	0.09	0.40	0.18	0.26	0.01
Total TEQ	16	0.22	0.87	0.36	0.57	0.03
Cattle Total (Calf + Beef + Cow)						
PCDD/PCDF	38	0.15	1.42	0.53	0.97	0.09
WHO-PCB	38	0.78	7.79	2.46	5.25	2.59
Total TEQ	38	0.93	9.21	3.10	5.80	3.22
Calf						
PCDD/PCDF	14	0.15	0.82	0.52	0.71	0.03
WHO-PCB	14	0.78	5.57	2.61	5.40	2.51
Total TEQ	14	0.93	6.25	3.16	6.05	3.01
Beef total (Heifer + Steer)						
PCDD/PCDF	12	0.26	1.42	0.56	1.29	0.13
WHO-PCB	12	1.08	7.79	2.81	5.37	3.23
Total TEQ	12	1.59	9.21	3.78	5.70	3.95
Heifer						
PCDD/PCDF	6	0.42	1.33	0.76	1.15	0.11
WHO-PCB	6	1.45	4.48	2.63	3.73	0.91
Total TEQ	6	1.90	5.43	3.54	4.72	1.37
Steer						
PCDD/PCDF	6	0.26	1.42	0.54	1.09	0.14
WHO-PCB	6	1.08	7.79	3.17	6.63	4.91
Total TEQ	6	1.59	9.21	3.78	7.47	6.02
Cow (total)						
PCDD/PCDF	12	0.24	1.28	0.37	0.97	0.10
WHO-PCB	12	0.91	4.04	1.50	3.18	0.93
Total TEQ	12	1.22	5.04	1.80	3.83	1.39
Cow (3 - 4 a)						
PCDD/PCDF	6	0.28	0.74	0.52	0.70	0.03
WHO-PCB	6	0.91	3.24	1.89	2.92	0.69
Total TEQ	6	1.22	3.89	2.23	3.61	0.88
Cow (7 - 9 a)						
PCDD/PCDF	6	0.24	1.28	0.32	1.14	0.17
WHO-PCB	6	1.03	4.04	1.22	2.95	1.14
Total TEQ	6	1.29	5.04	1.54	4.09	1.90

Conclusion: Dioxin and dioxin-like PCB results from Swiss animal fat samples are in good agreement with other data from European countries. No need has been identified for setting Swiss maximum residue limits for dioxins different from the ones given in the EU regulation. Differences of the dioxin:PCB ratios for pig and cattle indicate a differences in feed and/or environmental contamination and should be carefully considered in view of upcoming regulations for dioxin-like PCBs.

Acknowledgements

We wish to thank Mr. J. Hosseinpour for his kind assistance in this study.

References

1. COMMISSION REGULATION (EC) No 2375/2001 of 29 November 2001 amending Commission Regulation (EC) No 466/2001 setting maximum levels for certain contaminants in foodstuffs.
2. European Commission (2001) Community Strategy for Dioxins, Furans and Polychlorinated Biphenyls, COM (2001) 593 final, 24 October 2001.
3. Hosseinpour J., Rottler H., Joas R., Potrykus A., Schott R. (2002) *Organohalogen Compounds* 57, 77.
4. Schmid P. and Schlatter C. (1992) *Chemosphere* 24, 1013.
5. Schmid P., Gujer E., Zennegg M. and Studer C. (2002) *Chemosphere* 53, 129.
6. Schmid P., Gujer E., Degen S., Zennegg M., Kuchen A. and Wüthrich C. (2001) *Mitt. Lebens. Hyg.* 92, 483.
7. Swiss Federal Office of Public Health, unpublished results.
8. Swiss Federal Office of Public Health (2002) Informationsschreiben Nr. 79 über Höchstkonzentrationen für Dioxine in Lebensmitteln.
9. European Commission (2002) Commission Directive 2002/69/EC, OJ L 209, 5.
10. COMMISSION RECOMMENDATION of 4 March 2002 on the reduction of the presence of dioxins, furans and PCBs in feedingstuffs and foodstuffs. OJ L 67, 69.
11. Malisch R., Gleadle A. and Wright C. (1999) *Organohalogen Compounds* 43, 265.
12. Hecht H. and Blüthgen A. (1998) *Organohalogen Compounds* 38, 117.