



Feeding Fats Safety

FEEDING FATS SAFETY

One-Year Meeting

9-10 February 2006 – Barcelona

WP-1: Characterization and classification of feeding fats
(preliminary results)

Analysis of PCDD/Fs and DL-PCBs

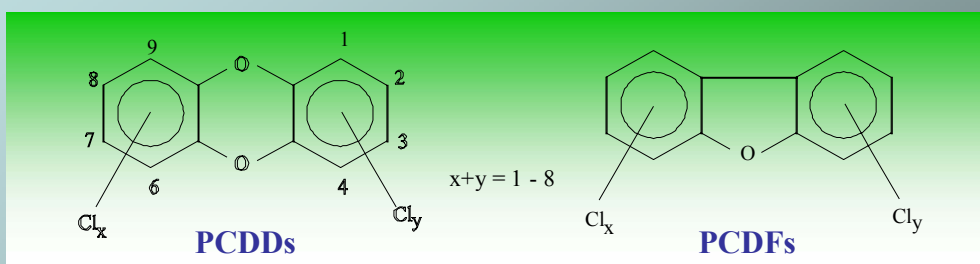
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Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) - 210 different congeners



- Only congeners substituted in the 2,3,7,8-positions are considered to be toxic:

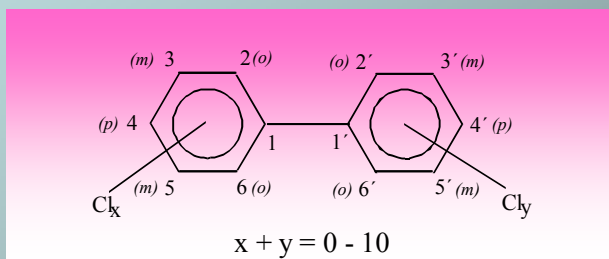
7 PCDDs and 10 PCDFs

- 2,3,7,8-TCDD is the most toxic congener. Toxic Equivalent Factor (TEF) = 1
- The rest of congeners have relative TEFs to 2,3,7,8-TCDD



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Polychlorinated biphenyls (PCBs) - 209 different congeners



12 DIOXIN-LIKE PCBs (DL-PCBs)



4 non-ortho PCBs and 8 mono-ortho PCBs

•DL-PCBs congeners have also relative TEFs to 2,3,7,8-TCDD



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Toxic equivalent factors assigned by The World Health Organisation (WHO-TEFs) to PCDD/Fs and DL-PCBs

Compound	WHO-TEF	Compound	WHO-TEF
Dioxins		Non-ortho PCBs	
2378-TCDD	1	33'44'-TeCB (# 77)	0.0001
12378-PeCDD	1	344'5'-TeCB (# 81)	0.0001
123478-, 123678-, 123789-HxCDD	0.1	33'44'5'-PeCB (#126)	0.1
1234678-HpCDD	0.01	33'44'55'-HxCB (# 169)	0.01
OCDD	0.0001	Mono-ortho PCBs	
Furans		233'44'-PeCB (# 105)	0.0001
2378-TCDF	0.1	2344'5'-PeCB (# 114)	0.0005
12378-PeCDF	0.05	23'44'5-, 2344'5'- PeCB (#118, 123)	0.0001
23478-PeCDF	0.5	233'44'5-, 233'44'5'-HxCB (# 156, 157)	0.0005
123478-, 123678-, 123789-, 234678-HxCDF	0.1	23'44'55'-HxCB (# 167)	0.00001
1234678-, 1234789-HpCDF	0.01	233'44'55'-HpCB (# 189)	0.0001
OCDF	0.0001		

Expression of the results:

$$TEQ = \sum_{i=1}^N C_i \times TEF_i$$



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Analytical Procedure

EXTRACTION

Sample dissolved in n-hexane

Treatment with H_2SO_4 or modified silica, 44% H_2SO_4

PURIFICATION AND FRACTIONATION

Solid-liquid adsorption chromatography

INSTRUMENTAL ANALYSIS

HRGC-HRMS

QUANTIFICATION

Isotopic dilution method

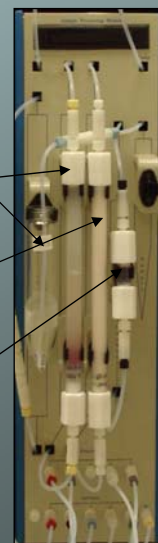


Sample

Silica

Alumina

Carbon



Automated clean-up system Power Prep (FMS)



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Analytical Procedure

EXTRACTION

Sample dissolved in n-hexane

Treatment with H_2SO_4 or modified silica, 44% H_2SO_4

PURIFICATION AND FRACTIONATION

Solid-liquid adsorption chromatography

INSTRUMENTAL ANALYSIS

HRGC-HRMS

QUANTIFICATION

Isotopic dilution method

GC capillary column: DB-5 ms
Positive Electron Ionisation (EI+)
SIM Mode
Power Resolution at 10.000
Labelled Standards



HRGC-HRMS (EI+) SIM



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Sampling

Category	N° of samples	Samples for PCDD/Fs and DL-PCBs analysis
ANFA	36	22
AOCHE	25	14 (2 animal origin + 7 vegetal origin + 2 tropical origin + 3 olive oil)
AOPHY	16	10 (1 animal origin + 3 vegetal origin + 4 tropical origin + 2 olive oil)
MIX	10	1 POOL of the total 10 MIX samples
FISH	9	9
LECI	8	8
RECY	8	8
HYBY	6	4
FACS	3	1 POOL of the total 3 FACS samples
EBE	2	2
Total	124	88



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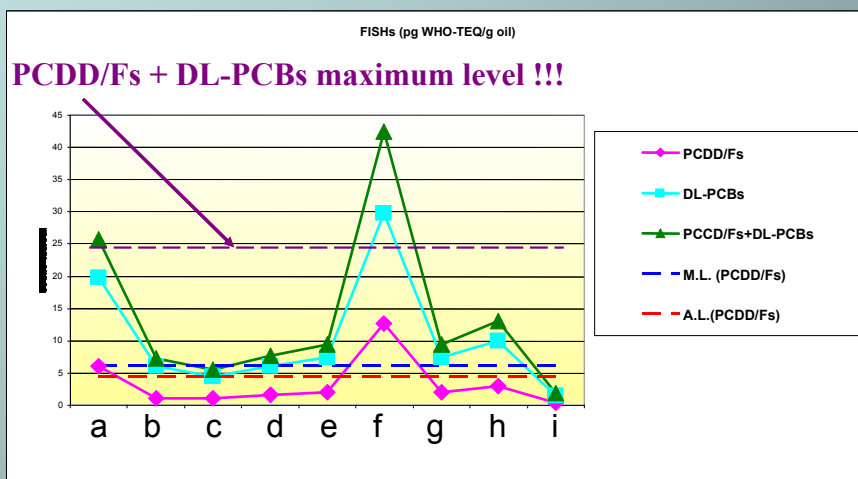
EU Regulatory Framework for Feedstuffs

	COUNCIL DIRECTIVE 2001/102/EC of 27 November 2001	COMMISSION RECOMMENDATION of 4 March 2002	COMMISSION DIRECTIVE 2006/13/EC of 3 February 2006
	PCDD/Fs Maximum levels	PCDD/Fs Action levels	PCDD/Fs and PCDD/Fs+DL-PCBs!!! Maximum levels
(a) All feed materials of plant origin including vegetable oils and by-products .	0.75 ng WHO-TEQ/ kg .	0.5 ng WHO-TEQ/ kg .	1.25 ng WHO-TEQ/ kg (feed materials of plant origin) 1.5 ng WHO-TEQ/ kg (vegetable oils and by-products)
(d) Animal fat, includ milk fat and egg fat .	2.0 ng WHO-TEQ/kg .	1.2 ng WHO-TEQ/kg .	
(f) Fish oil	6 ng WHO-TEQ/kg	4.5 ng WHO-TEQ/kg	24 ng WHO-TEQ/kg



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Results Fish Oil Samples (FISH)

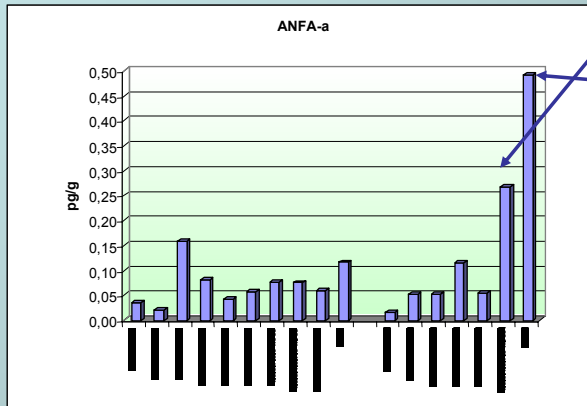


PCDD/F and DL-PCB levels in analysed FISH samples



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Results Animal Fat Samples (ANFA)

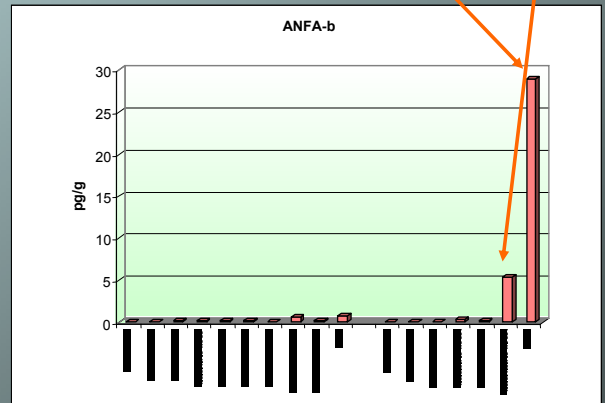


15% 1,2,3,4,6,7,8 HpCDD

28% OCDD

14% 1,2,3,4,6,7,8 HpCDD

79% OCDD

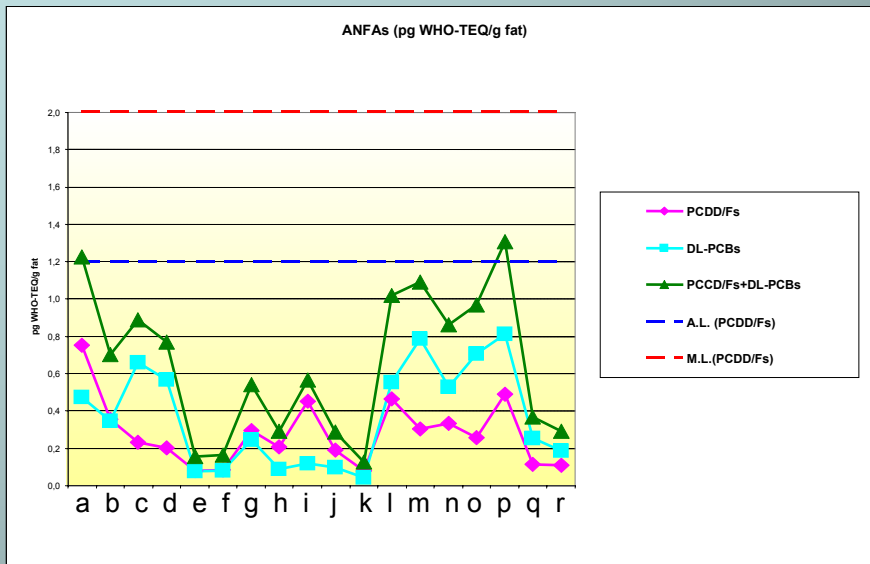


PCDD/F congener distribution in two representative ANFA samples



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Results Animal Fat Samples (ANFA)



PCDD/F and DL-PCB levels in analysed ANFA samples



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Other categories of fats and oils

	PCDDs/PCDFs (pg WHO-TEQ/g)	DL-PCBs (pg WHO-TEQ/g)	PCDDs/PCDFs and DL-PCBs (pg WHO-TEQ/g)
AOCHE			
a)	0.67	0.21	0.88
b)	0.24	0.21	0.45
c)	0.15	0.14	0.29
d)	1.03	4.31	5.35
AOPHY			
a)	2.15	0.11	2.26
b)	0.13	0.22	0.35
LECI			
a)	0.13	0.04	0.17
EBE			
a)	1.12	0.63	1.75
b)	0.44	0.57	1.01
RECY			
a)	0.21	0.32	0.53
b)	0.11	0.05	0.16



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