# DETERMINATION OF PCB CONGENERS AND ORGANOCHLORINE PESTICIDES IN FISH OIL FOOD SUPPLEMENTS

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#### Introduction

Levels of organochlorine pesticides and PCB congeners were determined in dietary supplements containing fish oil. The fish oils are sold for dietary purpose because of their content of omega-3 fatty acids, known to reduce the risk of cardiovascular disease. No complete picture of the Danish consumption of dietary supplements containing fish oil exists.

In this survey results from analysis of 30 dietary supplements containing fish oil from the Danish retail market are reported. Fish is known to accumulate persistent organic pollutants, including PCB and organochlorine pesticides in the oil <sup>1,2</sup>. Results from the survey are compared with results previously reported in other studies, and confirm earlier data for these compounds in fish oil, including cod liver oil <sup>3,4,5,6,7,8</sup>.

### Methods and Materials

Compounds: The samples were analysed for their content of the following organochlorine pesticides:  $\alpha$ -HCH (hexachlorocyclohexane),  $\beta$ -HCH, lindane ( $\gamma$ -HCH), HCB (hexachlorobenzene), heptachlor, heptachlorepoxid (cis), aldrin, dieldrin, endrin, isodrin,  $\alpha$ -chlordan,  $\gamma$ -chlordan, oxychlordan, trans-nonachlor,  $\alpha$ -endosulfan, p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT. The following PCB congeners (IUPAC no.) were analysed PCB-28, PCB-52, PCB-101, PCB-105, PCB-118, PCB-138, PCB-153, PCB-156, PCB-170 and PCB-180.

Fish oil samples: 30 Samples of fish oil dietary supplements were taken from the Danish retail market. The samples were chosen to cover as wide a range as possible e.g. different brand names or batch numbers. The samples should therefore be representative of fish oil dietary supplements from the Danish market. Dietary supplements with either fish liver oil or fish body oil or a blend of both, are sold either as liquid oil or microencapsulated.

Sample clean-up: Florisil was standardised by activating it for 18 hours at 450°C and deactivated by adding 4% of water. 0.6 g fish oil was added to the standardised Florisil column and eluted with dichloromethane:n-pentane (1:4). The eluate was carefully evaporated and the sample dissolved in isooctane. The final samples were analysed by gas chromatography using two different columns and electron capture detectors.

GC-ECD parameters: Perkin Elmer autosystem gas chromatograph. Column: 50 m CP-Sil-5CB (Chrompack) and 60 m DB-17 (J&W), 0.25 mm i.d., 0.25μm film thickness. Carrier gas: Helium, 15 psi (CP-Sil-5CB) or 37 psi (DB-17). 2 μl injected splitless, splitless time 2.5 min. Injector held **ORGANOHALOGEN COMPOUNDS** 

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at 220°C. Temperature programme: 90°C for 1 min., 30°C/min. to 180°C in 10 min., 2°C/min. to 240°C, 10°C/min. to 280°C in 20 min. (CP-Sil-5CB) or 30 min. (DB-17). Detector temperature 320°C. PCB congeners and organochlorine pesticides were quantified by comparing responses with those of standard mixtures. Limits of quantification for organochlorine pesticides and PCB congeners were 4 to 20  $\mu$ g/kg and 2 to 10  $\mu$ g/kg, respectively.

#### Results and discussion

As the fish oil samples were from the retail market, no information is available, neither concerning the origin of the fish and fish species used for the oil production, nor on any possible refining procedure. It is known, from previous studies, that fish from different waters may have varying content of persistent organic pollutants and also that the fish species and fish age influence the content <sup>9</sup>. Refining of fish oil can also influence the levels of organochlorine contaminants, as some refining procedures can reduce the levels of organochlorine contaminants in the oil <sup>10</sup>.

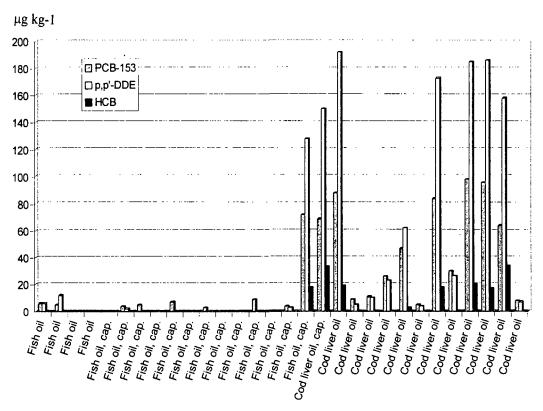


Figure 1. Levels of PCB-153, p,p'-DDE and HCB in fish oil dietary supplements. (cap.: Capsules).

From the results of the survey it can be concluded, that the content of organochlorine pesticides and the content of PCB congeners vary considerable in the products. Figure 1 show the levels of

some selected compounds (PCB-153, p,p'-DDE and HCB) in the samples. Results are reported for the fish oil either from the capsules or the liquid product i.e. irrespective of the fat content.

Highest levels of the organochlorine contaminants are found in cod liver oil whereas low levels are usually found in products of body oils. The levels for organochlorine contaminants in the cod liver oil samples differ, with half of the samples having high levels and the other half having lower levels. The low levels are similar to the levels found in the fish oil samples. The levels of the organochlorine contaminants found in this study are in fairly good agreement with previously reported results.

The manufacturers recommended daily intake of the cod liver products are 5 ml (adults). The calculated daily intake of  $\Sigma$ PCB,  $\Sigma$ DDT and HCB are given in table 1, with calculations based on the results obtained in this study and an intake of 5 ml per day and an oil density of 0.9 g/ml.

Table 1: Daily intake of ΣPCB, ΣDDT and HCB for cod liver oil samples

	Intake ΣPCB [μg]	Intake ΣDDT [μg]	Intake HCB [μg]
Average	0.82	0.81	0.05
Min.	0.12	0.02	n.d.
Max.	1.79	1.62	0.15

Calculations based on 5 ml and an oil density of 0.9 g/ml.

n.d.: Not detected.

Since the consumer will typically buy a bottle or package of capsules of fish oil and use the entire content the calculated intake of the compounds may deviate considerably from the calculated average of cod liver oil.

#### Conclusion

The level of organochlorine pesticides and PCB congeners present in dietary supplements containing fish oil varies considerably with the highest levels found in cod liver oil. The levels found in the fish oil samples are in agreement with previously reported data for the same kind of samples. The average intakes of  $\Sigma$ PCB and  $\Sigma$ DDT for cod liver oil are calculated to 0.82 and 0.81  $\mu$ g/day, respectively. However, since the consumer will typically buy one bottle of fish oil and use the entire content the average intake of the compounds may be somewhat misleading.

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