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PCB in Norwegian minke whale

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1. Introduction

1.1 Background

The work described in this report was carried out following the request from the Norwegian Ministry of Trade, Industry and Fisheries. The aim was to provide data and scientific basis for a possible future harmonization of analytical methodologies for determination of polychlorinated biphenyls (PCBs) and mercury between Norway/Iceland and Japan. As the project has progressed, the harmonization of analytical methodologies for mercury has been settled, since it has become evident that similar methods for mercury determination are already used in Norway/Iceland and Japan. The focus of this report is therefore on PCB only.

1.2 PCB

Theoretically, PCB is a mixture of 209 possible congeners which have varying number and position of the chlorine atoms attached to the biphenyl moiety. The seven ICES (International Council for the Exploration of the Sea) PCB congeners (PCB 28, 52, 101, 118, 138, 153 and 180) were recommended for monitoring by the European Union Community Bureau of Reference (BCR) (now Institute of Reference Materials and Measurement (IRMM)) as a proxy to avoid analyses of all 209 congeners. These seven ICES PCBs were selected from the 209 theoretically possible congeners since they had relatively high concentrations (about 50% of the total PCB) and had varying chlorination range from three to seven chlorine atoms. Later, EU has included PCB_6 instead of PCB_7 as the measurement of non-dioxin like PCBs in food as the omitted PCB118 was included in the measurement of dioxins and dioxin-like PCBs.

2. Materials and methods

2.1 Samples

Three types of blubber (back, belly and underjaw), together with meat samples, were collected from 46 minke whales (*Balaenoptera acutorostrata)*The sampling was done during the 2015 hunting season by the boats MF Kato (40 whales from the Barents Sea) and MF Fiskebank 1 (six whales from the Norwegian Sea). Sampling positions in the Barents Sea with whale ID are shown in Figure 1. The samples were sealed in individual plastic boxes with journal numbers. Individual characteristics of each whale was registered, such as capturing position, whale number, meat weight, length, sex etc at the harvesting boat. The three individual samples from each whale were then put in a separate plastic bag for each animal along with the accompanying information on the whale. When the boat reached mainland Norway the samples were sent to NIFES. At NIFES the samples were received and the accompanying information was entered into the laboratory information management system (LIMS). Meat samples where lyophilized before they were sent to the laboratory for mercury analysis. Mercury data are not part of this report. Samples of back blubber were delivered at the NIFES laboratory for PCB determinations and 35 samples of blubber (29 from the Barents Sea and six from the Norwegian sea) were sent to Eurofins for PCB_{209} determinations.

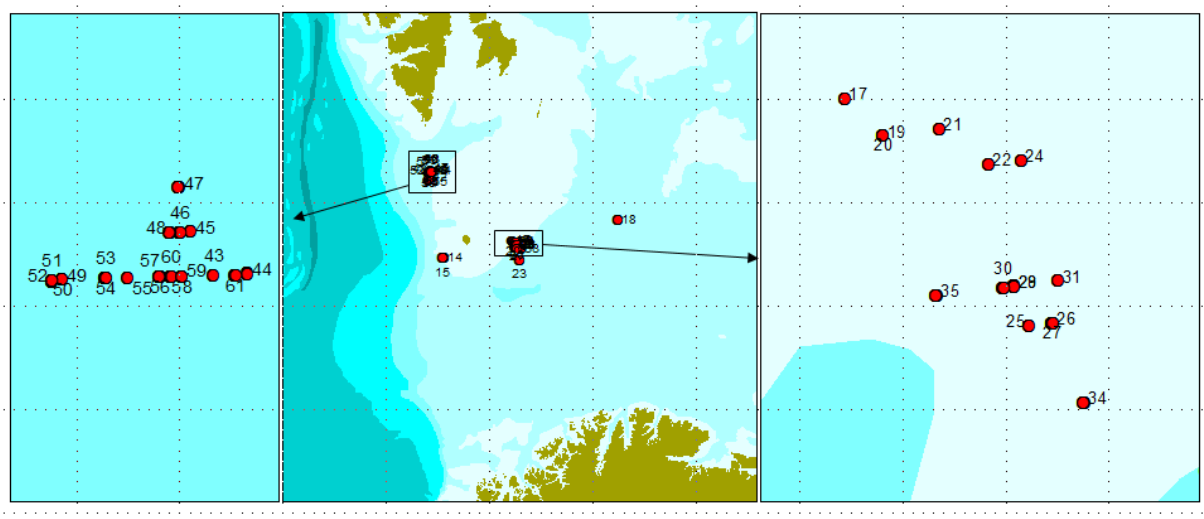

Figure 1: Sampling postions and whale IDs for the whales sampled in the Barents Sea by MF Kato

2.2 Methods

Both methods used in the determinations of PCB are NS-EN ISO/IEC 17025 accredited.

2.2.1 Determination of PCB_7 at NIFES

Analyses of PCB_7 as a sum of PCB 28, 52, 101, 118, 138, 153 and 180. PCB were carried out by adding ^{13}C enriched PCB internal standards to the sample and extracting the sample with hexane at elevated temperature and pressure. Fat was removed by using sulphuric acid and the samples were determined by GC/MS EI in SIM-mode. Quantification was performed using the internal standards and calibration curves. The expanded measurement uncertainty of PCB_7 was 15 %.

2.2.2 Determination of PCB_{209} at Eurofins

Thirty five samples of back blubber were further shipped to Eurofins laboratory; 29 samples from MF Kato's harvest in the Barents sea and six samples from MF Fiskebank 1 in the Norwegian sea. Determinations at Eurofins were carried out using about 10 g of material. If more than 10 g of sample material was received, the whale blubber was divided into several pieces and a number of pieces was chosen randomly for extraction to give in total about 10 g. The samples were pre-dried with sodium sulfate and homogenized. The pre-dried samples were the extracted by cold extraction, optimized with regard to complete extraction of PCBs. The extraction method is not a standard method for fat determination. The fat content in this crude extract was determined gravimetrically. After the gravimetrically determination of the extractable lipids, about 0.3 g of the extracted fat was used for PCB_{209} analysis. Eurofins regarded this fat as “fish oil” and proceeded with the samples as described in Appendix I: Details regarding Eurofins method for PCB209 determination. Results using this method were given as pg PCB congener/g fat. Fat percent for the 35 samples ranged from 5.8% to 49.2%. By NIFES request, the results on fat basis were later back calculated to fresh weight by Eurofins. The expanded measurement uncertainty of PCB_{209} was estimated to be in the range 40-50%.

2.2.3 Overlapping congeners

Overlapping congeners is a well known source of bias and uncertainty in analysis of multi-congener mixtures like PCB. The method at NIFES and Eurofins have some differences in overlapping congeners for PCB_7 as shown in Table 1. The overlapping congeners in the Japanese PCB_7 data presented by Japan in the expert meeting regarding trade in whale products between Iceland, Norway and Japan 30/06/2015 are not known (see Figure 4).

Table 1: Overview of overlapping congers in the different methods

PCB_7 congener	NIFES overlapping congeners	Eurofins overlapping congeners
28	31 (possible to separate by integration)	-
52	Non known	69 and 73
101	Non known	89 and 90
118	106	106
138	163 and 164	-
153	Non known	168
180	Non known	-

2.2.4 Uncertainty

The Codex Alimentarius Commission's - Procedural Manual states that an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification, except when a direct health hazard such as pathogens are concerned. More relevant text regarding equivalence, analytical results, uncertainty and decisions with regard to compliance assessment from international documents are given in Appendix II: International documents and references.

3. Results and discussion

3.1 Data from sampling

Overview of data collected by the crew on the boats for each of the 46 whales are shown in Table 2.

Table 2: Overview of whales

ID	Jno	Boat	Sex	Age	Coordinates	Meat Weight, kg	Whale Length, m
41*	2015-982	Fiskebank 1	Male	Mature	6338542N 00628919E	1400	7.4
42*	2015-983	Fiskebank 1	Male	Mature	6340213N 00608291E	1300	7.2
43*	2015-984	Fiskebank 1	Male	Unknown	6401115N 00618707E	1000	6.5
44*	2015-985	Fiskebank 1	Male	Mature	6431225N 00817727E	1350	7.1
45*	2015-986	Fiskebank 1	Female	Young	6400717N 00826920E	500	5.0
46*	2015-987	Fiskebank 1	Male	Unknown	6339205N 00741655E	1000	6.8
14*	2015-914	Kato	Female	Mature	7411N 1731E	1500	8.0
15	2015-917	Kato	Female	Young	7411N 1729E	500	6.0
17*	2015-921	Kato	Female	Mature	7423N 2202E	1800	7.9
18*	2015-922	Kato	Male	Mature	7425N 2902E	1800	7.5
19	2015-924	Kato	Female	Mature	7422N 2205E	2000	8.5
20	2015-926	Kato	Female	Mature	7422N 2205E	1800	8.2
21*	2015-929	Kato	Female	Mature	7422N 2210E	2200	9.0
22*	2015-930	Kato	Female	Mature	7421N 2214E	1800	8.9
23*	2015-931	Kato	Female	Mature	7402N 2223E	1800	8.1
24*	2015-933	Kato	Female	Mature	7421N 2217E	1900	8.4
25*	2015-935	Kato	Female	Mature	7417N 2216E	1200	7.0
26*	2015-937	Kato	Female	Mature	7417N 2218E	1200	6.8
27*	2015-938	Kato	Female	Mature	7417N 2218E	1000	7.3
28*	2015-940	Kato	Male	Mature	7418N 2215E	1200	6.5
29*	2015-941	Kato	Female	Mature	7418N 2215E	1500	8.0
30*	2015-943	Kato	Female	Young	7418N 2214E	800	6.7
31*	2015-945	Kato	Female	Mature	7418N 2219E	1800	8.5
33*	2015-946	Kato	Female	Mature	7413N 2230E	1500	8.1
34*	2015-947	Kato	Male	Mature	7415N 2220E	1000	7.1
35*	2015-948	Kato	Female	Mature	7418N 2208E	2200	8.5
42*	2015-949	Kato	Female	Mature	7535N 1642E	1600	8.1
43	2015-950	Kato	Male	Young	7542N 1657E	1000	6.3
44*	2015-951	Kato	Female	Mature	7542N 1700E	1800	8.2
45	2015-952	Kato	Female	Mature	7543N 1655E	1500	8.1
46	2015-953	Kato	Female	Mature	7543N 1654E	1200	6.9

ID	Jno	Boat	Sex	Age	Coordinates	Meat Weight, kg	Whale Length, m
47*	2015-954	Kato	Female	Mature	7544N 1654E	2000	8.5
48	2015-955	Kato	Female	Mature	7543N 1653E	1500	8.4
49	2015-956	Kato	Female	Mature	7542N 1643E	1800	8.0
50*	2015-957	Kato	Female	Mature	7542N 1643E	1200	7.8
51*	2015-958	Kato	Female	Mature	7542N 1642E	1200	7.8
52	2015-959	Kato	Female	Mature	7535N 1642E	1500	8.5
53*	2015-960	Kato	Female	Mature	7542N 1647E	1200	7.5
54*	2015-961	Kato	Female	Mature	7542N 1647E	2000	7.9
55*	2015-963	Kato	Female	Mature	7542N 1649E	1300	7.7
56*	2015-965	Kato	Female	Mature	7542N 1652E	1200	7.9
57*	2015-966	Kato	Female	Mature	7544N 1654E	1800	7.9
58	2015-967	Kato	Female	Mature	7542N 1654E	1700	8.4
59*	2015-968	Kato	Female	Mature	7542N 1654E	1400	8.5
60*	2015-969	Kato	Female	Mature	7542N 1653E	2000	8.4
61	2015-970	Kato	Female	Mature	7542N 1659E	2000	8.5

* = Back blubber from these 35 whales were sent to Eurofins for PCB_{209} determination

An overview of sex and age of the animals is shown in Table 3.

Table 3: Sex and age of the animals

Sex	Female		Male			Total
Age	Young	Mature	Young	Mature	Unknown	
Norwegian Sea	1	-	-	3	2	6
Barents Sea	2	34	1	3	-	40
Total	3	34	1	6	2	46

Length of the whales are summarized in Table 4.

Table 4: Length of animals

Sex		Female		Male			Total
Age		Young	Mature	Young	Mature	Unknown	
Norwegian Sea	Min	5.0			7.1	6.5	5.0
	Max	5.0	-	-	7.4	6.8	7.4
	Average	5.0			7.2	6.7	6.7
Barents Sea	Min	6.0	6.8	6.3	6.5		6.0
	Max	6.7	9.0	6.3	7.5	-	9.0
	Average	6.4	8.1	6.3	7.0		7.9
Total	Min	5.0	6.8	6.3	6.5	6.5	5.0
	Max	6.7	9.0	6.3	7.5	6.8	9.0
	Average	5.9	8.1	6.3	7.1	6.7	7.7

3.2 PCB_7 in different types of minke whale blubber samples determined by NIFES

An overview of the data from each type of blubber from each area, gender and age with min, max, average and N, is given in Table 5 to Table 7.

Table 5: PCB_7 (UB) in belly blubber

Sex		Female		Male			Total
Age		Young	Mature	Young	Mature	Unknown	
Norwegian Sea	Min	170			140	350	140
	Max	170	-	-	490	440	490
	Average	170			363	395	342
	N	1			3	2	6
Barents Sea	Min	96	8	170	290	-	8
	Max	410	480	170	460		480
	Average	253	120	170	380		148
	N	2	34	1	3		40
Total	Min	96	8	170	140	350	8
	Max	410	480	170	490	440	490
	Average	225	120	170	372	395	173
	N	3	34	1	6	2	46

Table 6: PCB_7 (UB) in back blubber

Sex		Female		Male			Total
Age		Young	Mature	Young	Mature	Unknown	
Norwegian Sea	Min	190			160	210	160
	Max	190	-	-	550	960	960
	Average	190			403	585	428
	N	1			3	2	6
Barents Sea	Min	59	9	160	120	-	9
	Max	220	780	160	410		780
	Average	139.5	141	160	260		150
	N	2	34	1	3		40
Total	Min	59	9	160	120	210	9
	Max	220	780	160	550	960	960
	Average	156	141	160	332	585	186
	N	3	34	1	6	2	46

Table 7: PCB_7 (UB) in underjaw blubber

Sex		Female		Male			Total
Age		Young	Mature	Young	Mature	Unknown	
Norwegian Sea	Min	140			85	70	70
	Max	140	-	-	280	450	450
	Average	140			192	260	206
	N	1			3	2	6
Barents Sea	Min	77	12	33	110	-	12
	Max	140	490	33	370		490
	Average	109	105	33	203		111
	N	2	34	1	3		40
Total	Min	77	12	33	85	70	12
	Max	140	490	33	370	450	490
	Average	119	105	33	198	260	123
	N	3	34	1	6	2	46

Complete results from the PCB_7 determinations at NIFES can be found in

Appendix III: Raw data from PCB7 determinations at NIFES
. The results of PCB_7 (Upper Bound) for all samples from MF Kato is shown in Figure 2.

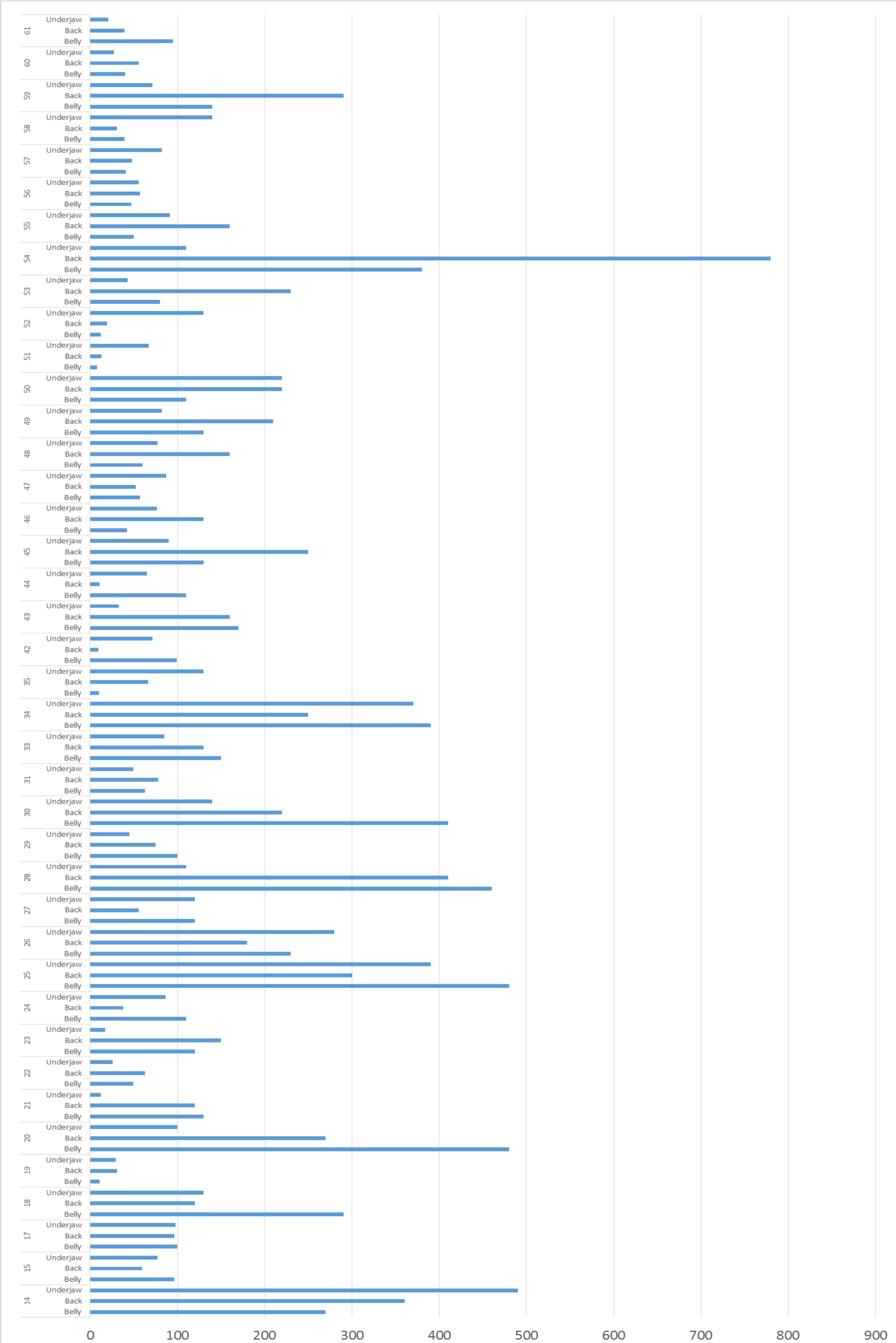

Figure 2: Overview of PCB_7 (Upper Bound) results in ng/g fresh weight for samples from Kato with whale ID

3.3 Comparison of PCB_7 levels in different types of minke whale blubber from Kato

T-tests for dependent samples was carried out to look for possible differences between PCB_7 levels in different types of minke whale blubber from MF Kato (Figure 3). As Table 8 and Table 9 shows, no difference between different types of blubber was found ($p > 0.05$), se also Appendix IV: Comparison between PCB7 in different types of blubber from Kato.

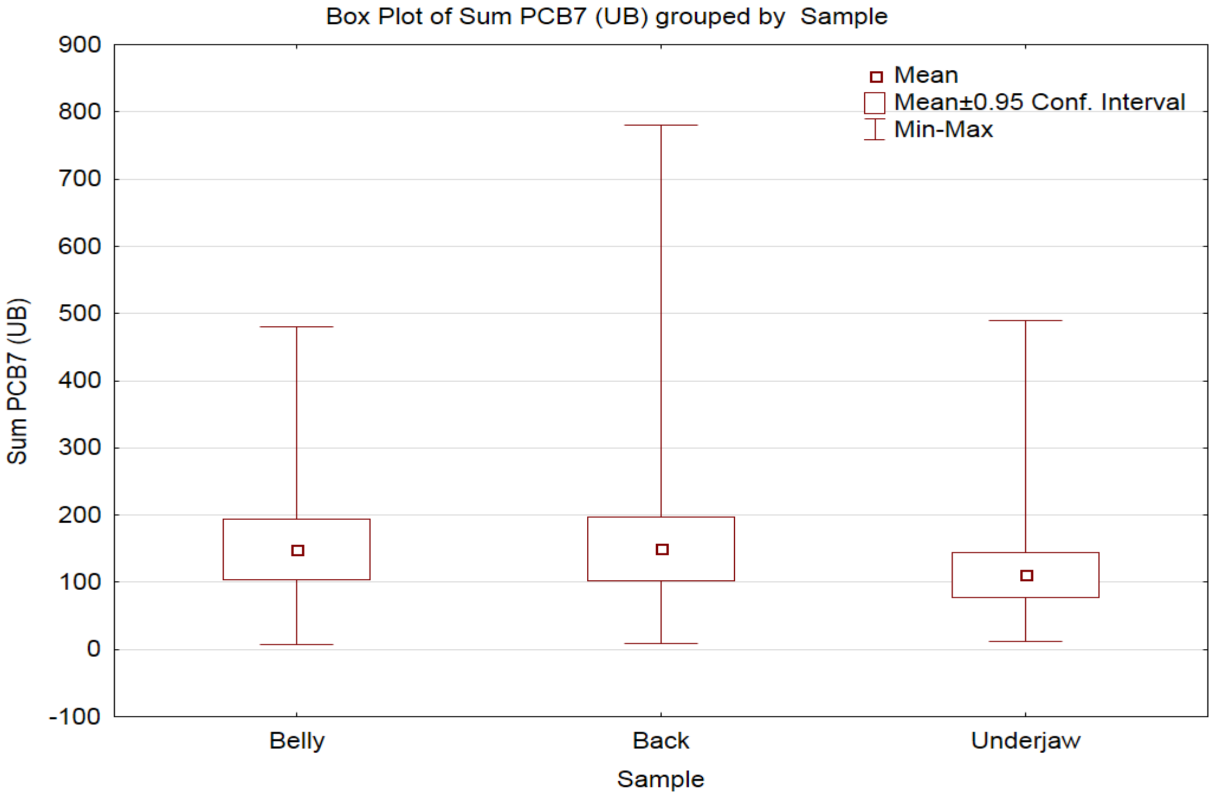

Figure 3: No difference between sum PCB_7 in different types of minke whale blubber from MF Kato (ng/g fresh weight)

Table 8: p-values from t-test for dependent samples of PCB_7 (upper bound) in different types of blubber

Upper bound p-values	Sum PCB_7 (UB) Belly, ppb	Sum PCB_7 (UB) Back, ppb	Sum PCB_7 (UB) Underjaw, ppb
Sum PCB_7 (UB) Belly, ppb	-	0.900846	0.059926
Sum PCB_7 (UB) Back, ppb	0.900846	-	0.090187
Sum PCB_7 (UB) Underjaw, ppb	0.090187	0.090187	-

Table 9: p-values from t-test for dependent samples of PCB_7 (lower bound) in different types of blubber

Lower bound p-values	Sum PCB_7 (UB) Belly, ppb	Sum PCB_7 (UB) Back, ppb	Sum PCB_7 (UB) Underjaw, ppb
Sum PCB_7 (LB) Belly, ppb	-	0.904564	0.05812
Sum PCB_7 (LB) Back, ppb	0.904564	-	0.087153
Sum PCB_7 (LB) Underjaw, ppb	0.05812	0.087153	-

3.4 PCB_{209} and PCB_7 in back blubber samples determined by Eurofins

PCB_{209} determinations were carried out on the 35 samples mentioned in Table 2 designated with an asterix (*). Due to the size of the dataset all results from the PCB_{209} determination, both on fat and fresh weight basis, can be found in the attached excel file exactly as it was provided to us by Eurofins. LOQ of each congener is also attached as a separate excel sheet also exactly as it was provided to us by Eurofins.

3.5 Correlations between PCB_7 and total PCB (PCB_{209})

Previous results provided by Japan in the expert meeting regarding trade in whale products between Iceland, Norway and Japan 30/06/2015 showed a good correlation (R^2 = 0.9974) between PCB_7 and total PCBs as shown in Figure 4. A factor of 2.0468 was found between PCB_7 and total PCBs on wet weight basis.

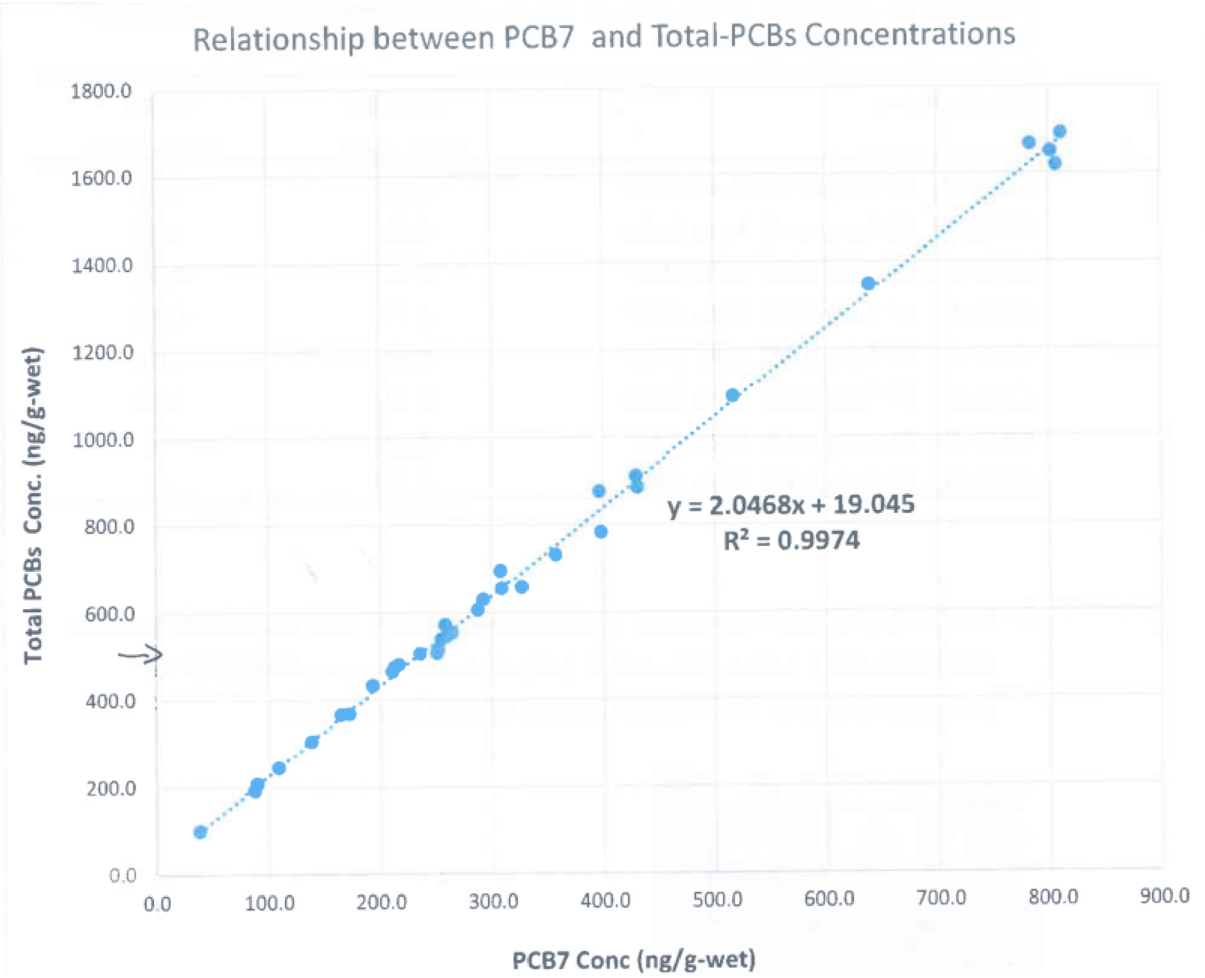

Figure 4: Relationship between PCB_7 and total PCB as presented by Japan (taken from the memorandum regarding PCB analyses in whale products regarding trade)

Results from all the correlations of PCB_{209} and PCB_7 both on fresh weight and fat weight basis can be found in Appendix V: Regressions, correlations and scatterplots. Correlation results from the determinations carried out at Eurofins on fat weight upper bound concentrations are shown in Figure 5, and the results on fresh weight upper bound concentrations are displayed in Figure 6 as examples. Results on both fat and fresh weight for upper bound, medium bound and lower bound is summarized in Table 10.

Good correlations were found for both fresh and fat weight concentrations with R^2 values close to 1. Intercept for the relationship on fat basis was somewhat larger than the intercept on fresh weight basis and all intercept were significantly different from zero ($p < 0.005$). The intercept of about 27 on fresh weight basis was of similar magnitude as 19, which has previously been demonstrated by Japan on wet weight basis (Figure 4). A significant linear relationship (p-values << 0.00005) was found between PCB_7 and PCB_{209}. The slope of the curve was slightly larger for fat weight concentrations (1.84) compared to fresh weight (1.80). This slope is similar to the slope of 2.05 previously found by Japan.

Table 10: Summary of relationship between PCB_{209} and PCB_7

	PCB_{209}	R	R^2	intercept	p-value	slope	p-value
Fat weight	Upper bound	0.99279961	0.98565106	130.9562	0.000142	1.8351	0.000000
	Medium bound	0.99256086	0.98517707	128.3124	0.000224	1.8358	0.000000
	Lower bound	0.99266975	0.98539324	123.9643	0.000309	1.8374	0.000000
Fresh weight	Upper bound	0.99254612	0.9851478	27.65203	0.000427	1.80614	0.000000
	Medium bound	0.99234693	0.98475243	27.56011	0.000502	1.80261	0.000000
	Lower bound	0.99237339	0.98480495	26.99703	0.000612	1.80171	0.000000

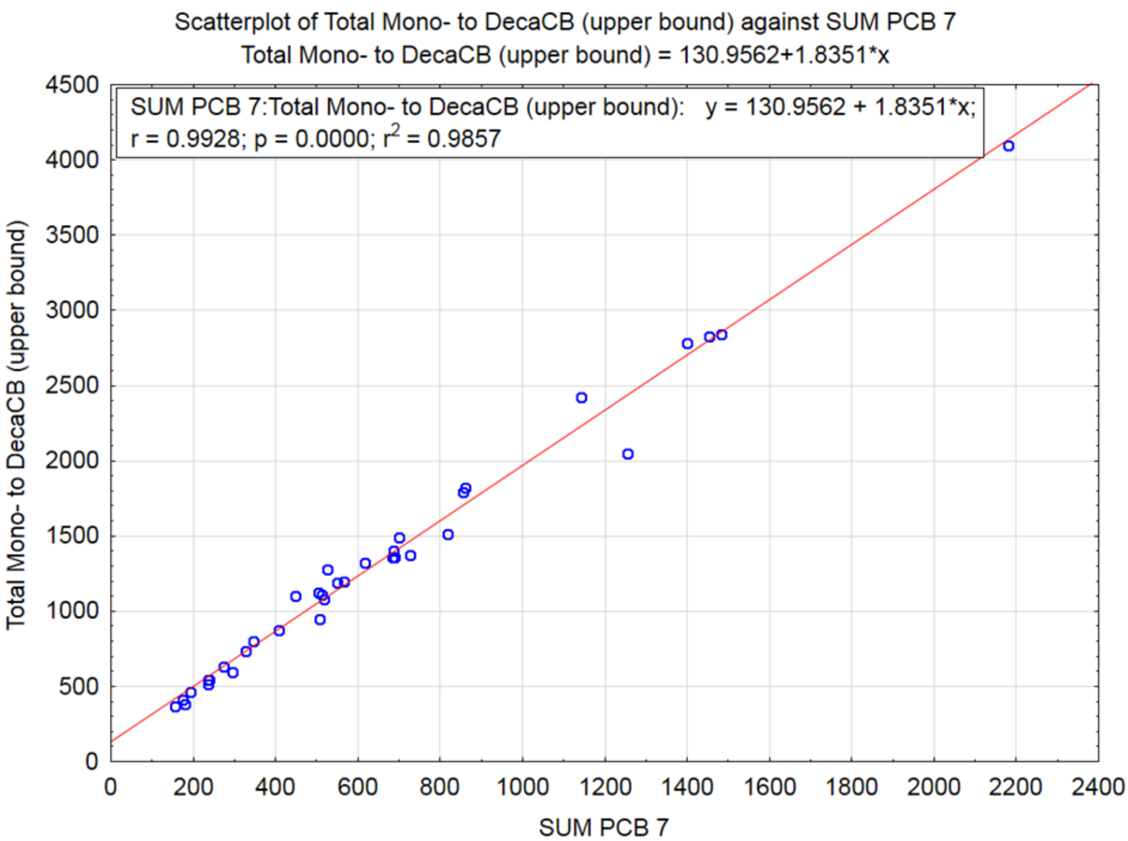

Figure 5: Relationship between PCB_7 and PCB_{209} (ng/g upper bound) from Eurofins determinations (fat weight)

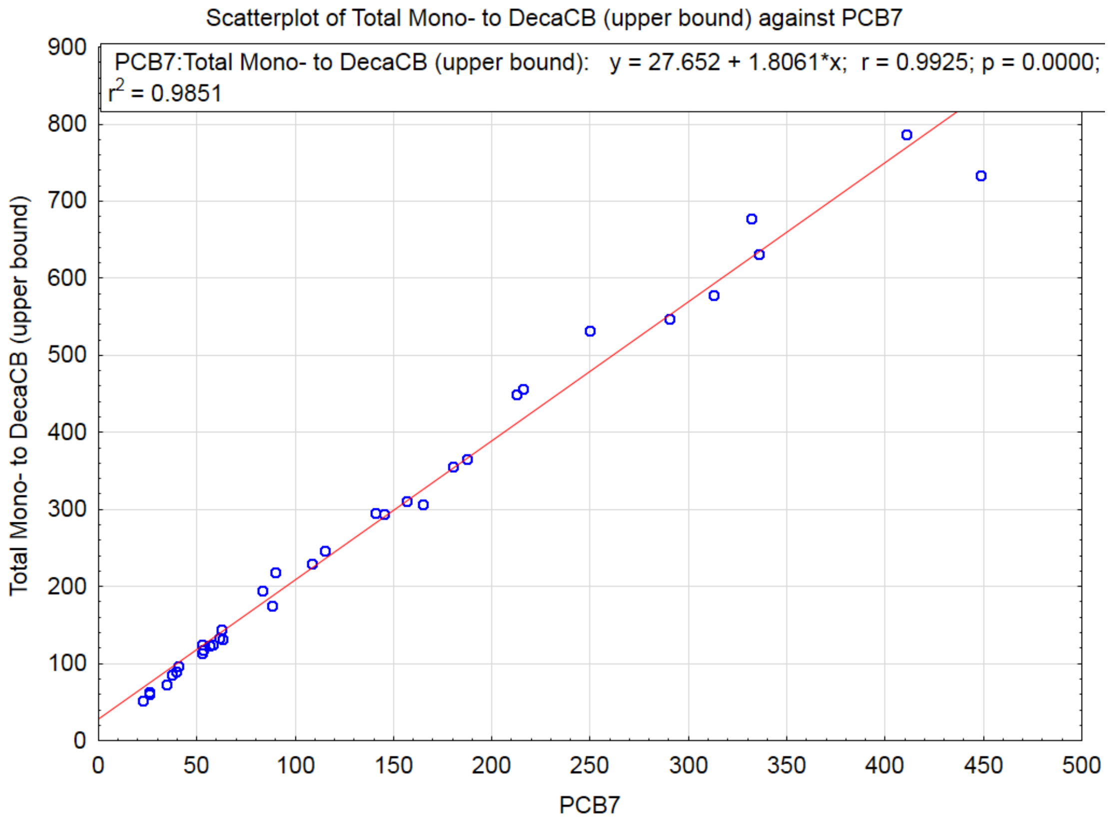

Figure 6: Relationship between PCB_7 and PCB_{209} (ng/g upper bound) from Eurofins determinations (fresh weight)

3.6 Comparison between NIFES PCB_7 and Eurofins PCB_7

All 35 back blubber samples sent to Eurofins for PCB_{209} determination were also determined for PCB_7 at NIFES. The results for each whale ID is compared graphically in Figure 7.

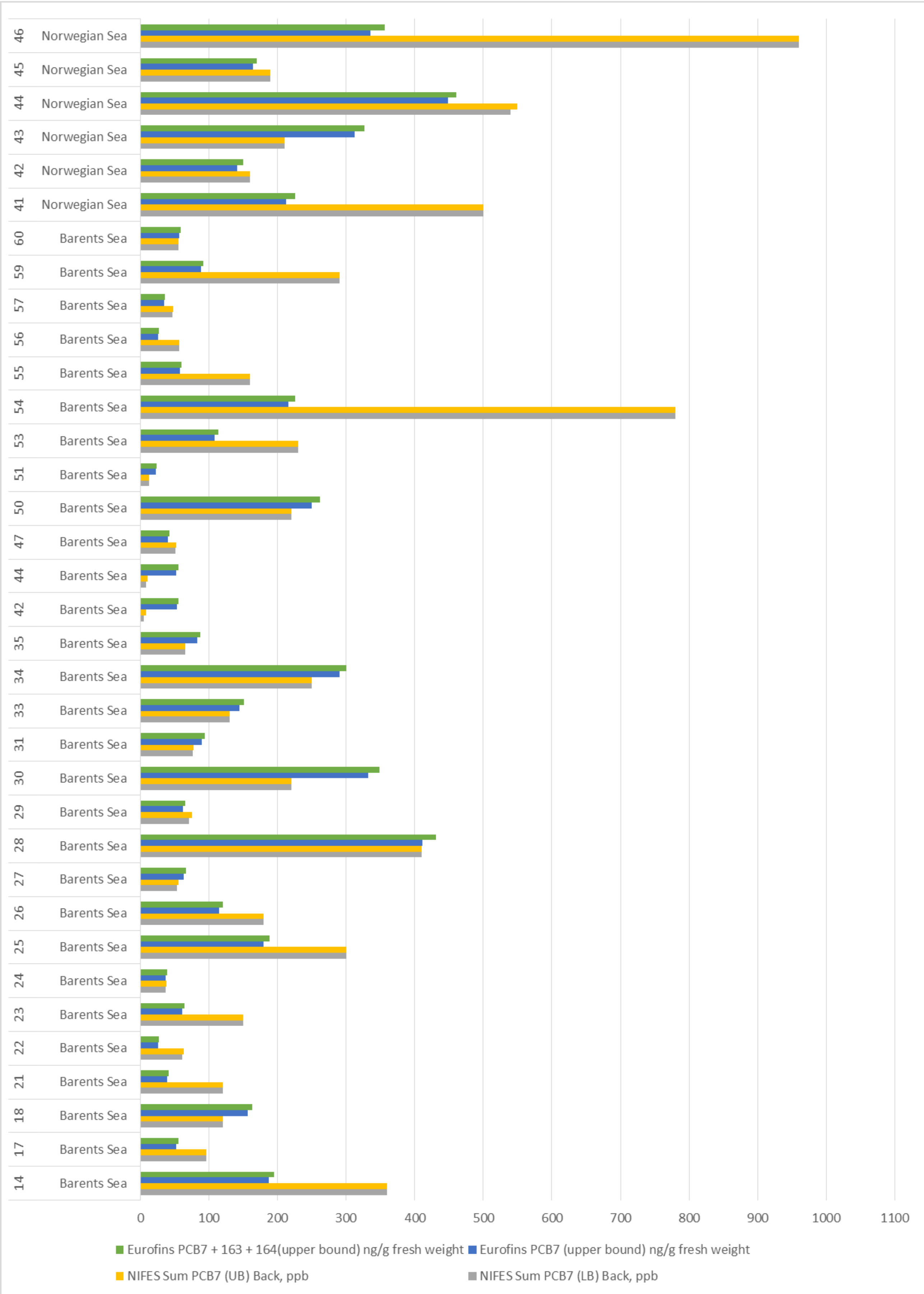

Figure 7: Comparison between NIFES PCB$_7$ and Eurofins PCB$_7$

T-tests for dependent samples were carried out to investigate for statistical differences between the PCB_7 results from NIFES compared to Eurofins. An overview of the results are shown in Table 11 and further details are given in Appendix VI: Comparison between NIFES PCB7 and Eurofins PCB7.

Table 11: Overview of results from t-tests

NIFES PCB_7	Eurofins PCB	NIFES Mean	Eurofins Mean	NIFES Std.Dv.	Eurofins Std.Dv.	p-value
Lower bound	PCB_7	205.0857	141.5596	214.158	117.5673	0.021257
Lower bound	PCB_7+PCB163 +PCB164	205.0857	148.0705	214.158	122.7271	0.035419
Upper bound	PCB_7	205.9429	141.5596	214.1782	117.5673	0.019534
Upper bound	PCB_7+PCB163 +PCB164	205.9429	148.0705	214.1782	122.7271	0.032679

The t-tests revealed that PCB_7 determinations at NIFES were higher ($p < 0.05$) than PCB_7 from Eurofins, also when including PCB-163 and PCB-164 in the Eurofins PCB_7 sum.

3.7 Correlations between NIFES PCB_7 and Eurofins PCB_7

The linear relationship between PCB_7 determined at NIFES and PCB_7 determined by Eurofins was investigated and an overview of the results are shown in Table 12: Overview of relationship between NIFES PCB7 and Eurofins PCB7. Further details are given in Appendix VII: Regressions, correlations and scatterplots of PCB7.

Table 12: Overview of relationship between NIFES PCB_7 and Eurofins PCB_7.

NIFES PCB_7	Eurofins PCB	R	R2	intercept	p-value	slope	p-value
Lower bound	PCB_7	0.70437433	0.49614319	23.45439	0.572801	1.28307	0.000002
Lower bound	PCB_7+PCB163 +PCB164	0.70806714	0.50135907	22.13382	0.593035	1.23557	0.000002
Upper bound	PCB_7	0.70585415	0.49823008	23.91283	0.564593	1.28589	0.000002
Upper bound	PCB_7+PCB163 +PCB164	0.70946303	0.50333779	22.61305	0.584384	1.23813	0.000002

The results showed that a significant ($p<0.05$) linear relationship was found between Eurofins PCB_7 and NIFES PCB_7. NIFES PCB_7 was higher than Eurofins PCB_7 by factor of 1.24-1.28. This difference could be due to a number of factors including sample inhomogeneity since different analytical samples from the bulk sample was determined. Differences in analytical methodology between NIFES and Eurofins results such as differences in overlapping congeners, sample workup, extraction and quantification between the two methods may also contribute.

4. Conclusion

Investigations carried out by both Japan, Iceland and Norway has shown significant linear correlations ($p << 0.05$) between PCB_7 and total PCB (PCB_{209}) with R^2/R-values close to 1. A factor of 2.0 between PCB_7 and total PCB has previously been demonstrated by Japan. The factor found in this survey based on 35 samples of minke whale was 1.8.

NIFES PCB_7 showed a significant linear correlation ($p<< 0.05$) with Eurofins PCB_7, although the NIFES PCB_7 on average was somewhat higher than Eurofins PCB_7. This difference is probably due to a number of factors including sample inhomogeneity and differences in overlapping congeners, sample workup, extraction and quantification between the two methods.

Based on the data presented by both Japan, Iceland and Norway there is scientific basis for harmonization of methodologies of total PCB (PCB_{209}) and PCB_7 by using an appropriate conversion factor between PCB_7 and total PCB (PCB_{209}). The results of this survey in minke whale suggest a factor of 1.8.

5 Appendix I: Details regarding Eurofins method for PCB_{209} determination

CONGENER GROUP DISTRIBUTION AND OVERALL PATTERN OF PCB IN FAT OF DIFFERENT FISH AND MARINE ORGANISMS

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Introduction

Polychlorinated Biphenyls (PCBs) are known for long as being toxic. There are different toxicological action modes of PCBs, comprising dioxin-like (dl-) and non-dioxin-like (ndl-) effects, depending on the congener specific chlorination patterns. Derived from these facts, the Californian government issued a "safe harbour level" for PCBs in legislative Proposition 65[1], describing a maximum daily intake of "Total PCBs" considered as being harmless for human health. Furthermore, PCBs in general have recently been recognised as being carcinogenic to humans by the IARC[1]. The legislative demand as well as the overall recognition of toxicity result in the question about the PCB content of e.g. certain fish oil products as food additives. It implies the ability of precisely analysing the Total PCB content in terms of determining all 209 possible congeners.

There have been several approaches for analysing all PCB congeners and calculating the total PCB amount, reaching from quantification against technical PCB mixtures over fractionation of PCBs to complete generic templates as e.g. US-EPA method 1668C[3]. With all these approaches showing difficulties to a different extent, we developed a method for marine biota samples using a specialised multistep-clean-up in combination with a modern HRGC column and HRMS detection, which enables a comprehensive analysis of all PCB congeners with around 170 peak separations. Thus, a single compound analysis beyond the regularly analysed 12 dl-PCB and 6 ndl-PCB can be achieved. The use of fish oil and other marine oils can be considered to be representative for fish as a biota matrix category since PCBs will solely be present in the lipid fraction. It has been applied on crude and refined marine oils and fats from different origins and species which are intended for dietary supplement use directly associating environmental aspects with human nutrition.

Materials and methods

In total, 38 biota oil/fat samples have been analysed for PCBs during 2012 and 2013 at the Eurofins GfA Lab Service lab in Hamburg. These samples consisted of several groups of crude or refined marine oils and fats:
Cod (Gadida spec.), crude oil (n=5) and treated oil (n=10), 2012-2013, North Atlantic;
Tuna (Thunnus spec.) (Scombridae spec.), crude oil (n=4) and treated oil (n=6), South America;
Shark liver. crude oil (n=1), 2011, Faeroer Islands;
Mink Whale (Balaenoptera acutorostrata) crude fat (n=2), 2012, North Atlantic;
krill crude oil (n=4) and treated oil (n=6), 2012, Arctic Sea/North Atlantic.
The analytical method consisted of an adapted EPA1668C[3] protocol, modified for a comprehensive analysis of all 209 PCB-congeners. Samples have been diluted with n-hexane for sample preparation and then cleaned with an adapted multiple column chromatography clean-up. The following HRGC-analysis was performed on an HT8-PCB 60m * 0.25 mm * 0.25 µm GC-column using a Thermo DFS and Waters Autospec high resolution mass spectrometer at mass resolution $R \geq 10.000$. With the chosen setup, a maximum separation of 180 signals is possible. Quantification was narrowed down to 170 separations for reasons of constant data quality.
Isotope dilution quantification has been performed using a set of 35 $^{13}C_{12}$-quantification standards (Mono- to DecaCB) added prior to extraction, and internal standard quantification for all congeners not covered by their own isotope-labelled analogue. Process quality was monitored with 7 $^{13}C_{12}$-injection standards (Di- to NonaCB, except HeptaCB). QA/QC measures consisted e.g. in monitoring the quantification standard recovery rates (acceptance 40-120%), as well as batch blanks and control samples. The limit of quantification was established based on an approach according to EN1948-4 xxx using averaged blank values plus 5-fold standard deviation. Calibration was established preparing an initial multipoint calibration curve for reference purposes, and daily single-point calibrations checked against the multipoint curve. This has been performed individually for all reported congeners/groups. Further details of the method and quality criteria are described elsewhere[4].

All analytical data are reported in pg/g product, all TEQ values are given as upperbound TEQ using WHO-TEF(2005).

Results and discussion

The presented method enables us to get sufficiently detailed insight into the characteristics of PCB distributions in marine samples, amongst others regarding toxicological and regulatory aspects, e.g. totals of dioxin-like PCBs, different sets of marker PCBs, as well as congener group totals. The main results for the PCB congener groups are shown in *table 1* and *figure 1*.

	Krill oil, crude		Krill oil, refined		Whale fat, crude	Cod liver oil, crude		Cod liver oil, refined		Tuna oil, crude		Tuna oil, refined		Shark liver oil, crude
	(n=4)		(n=6)		(n=2)	(n=5)		(n=10)		(n=4)		(n=6)		(n=1)
	mean pg/g	SD %	mean pg/g	SD %	mean pg/g	mean pg/g	SD %	mean pg/g	SD %	mean pg/g	SD %	mean pg/g	SD %	mean pg/g
total MonoCB	178	54%	4	78%	49	152	157%	5	257%	5	118%	12	100%	10
total DiCB	1794	64%	36	75%	222	1509	166%	36	176%	54	123%	32	137%	744
total TriCB	1776	68%	98	71%	4465	14160	143%	107	179%	82	112%	86	203%	32099
total TetraCB	559	63%	201	97%	25198	27429	86%	943	190%	698	98%	292	236%	551589
total PentaCB	325	55%	264	99%	51704	39549	89%	3459	120%	3378	76%	733	237%	2300305
total HexaCB	465	87%	214	93%	86359	52353	114%	9282	104%	10139	56%	1214	200%	5099460
total HeptaCB	195	86%	55	105%	36515	15404	128%	3952	97%	5823	39%	424	160%	2388476
total OctaCB	63	78%	6	177%	6120	2246	130%	769	93%	1146	33%	149	100%	503238
total NonaCB	8	74%	1	155%	788	221	165%	116	67%	182	35%	28	83%	43597
DecaCB	2	67%	1	168%	377	98	127%	80	53%	73	62%	19	49%	11758
total PCB (Mo-Dc)	5366	63%	879	88%	211796	153123	89%	18749	105%	21582	55%	2989	198%	10931276
WHOTEQ05, lowerbound	0,23	77%	0,03	153%	7,53	1,20	112%	0,03	70%	0,03	148%	0,82	172%	26,82
WHOTEQ05, upperbound	0,28	69%	0,13	54%	7,53	1,56	70%	0,34	58%	0,09	78%	0,96	138%	29,41

Table 1: PCB homologue groups in fish oil samples

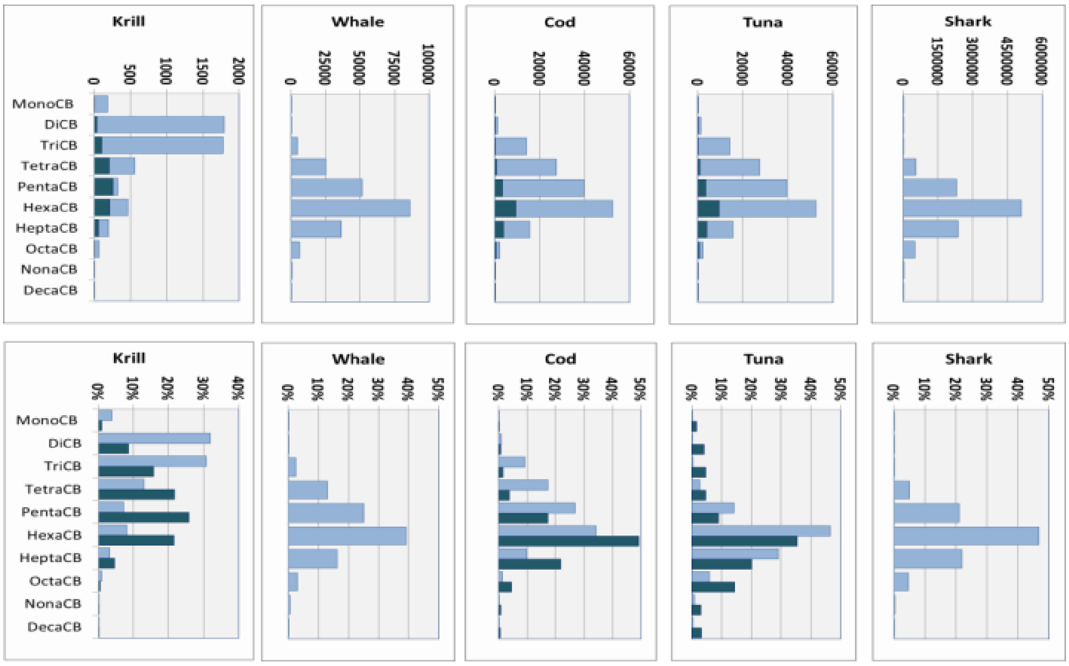

Figure 1: PCB homologue groups in fish oil samples. (All values are expressed in pg/g for the upper row and % for the lower row; light blue =crude oils; dark blue = refined oils)

The analysed concentrations for PCB are generally lower than levels published previously for fish[5,6], even for shark oil. A comparison is difficult due to a lack of analytical data for congener groups or total PCBs and can only be made on basis of those few single congeners which have been reported in literature. Also, many values reported in literature are related to coastal areas of industrialised countries as opposed against open seas. Therefore, the low concentrations observed here seem to be plausible for proveniences as e.g. the North Atlantic Ocean around the Faeroer islands (see shark oil). Regarding TEQ values, only the whale and the shark samples are in the range or above the EU limit value for use as human food[7]. Though the samples can only be considered as being random, they do reflect a part of the real ranges of PCB concentrations in fish and marine animals.

The results from the evaluated samples provide several insights into PCB behavior. For example, the similar congener- and total PCB distributions (*figure 2*) with a usual maximum at the Hexa- to HeptaCB, show a relatively uniform pattern even in between different marine (fish or mammal) species. This reflects mainly biological persistance in terms of similar end points of metabolism within the trophic chain linked to the ecological situation but also physicochemical stability of the higher chlorinated compounds and also the use of higher chlorinated PCB mixtures, e.g. Arochlor 1248 and 1260. This is best illustrated by looking onto different PCB distributions for the different species habitats. Several fish species are carnivorous and might feed on benthic organisms prone to sediment contact. These fish (of fair or higher trophic levels) show the most common PCB distribution centered around the hexachlorobiphenyl maximum.

This is opposed by the PCB distribution in krill as a category of pelagic organisms, being in a low position within the trophic chain. This in combination with their daily vertical migration through the water column could explain the higher concentrations of better water soluble mono- to trichlorobiphenyls within these samples. Another part of the explanation might be the predominant use of lower chlorinated PCB formulations as e.g. Arochlor 1242 in the later PCB production time[8], being released into the environment later than the higher chlorinated ones. To a certain extent this shift in chlorination degree can also be found in the whale and cod oil pattern. Considering all presented species, whale and cod have a relatively high contribution of tri- to tetrachlorobiphenyls to the total PCB sum, reflecting their feeding on crustaceans as e.g. krill.
Even throughout processing crude marine oils or fats enabling the use for human consumption, e.g. as nutrition supplements rich in ω–3-fatty acids, the chlorination pattern is somewhat maintained.

One difference points towards the apparent use of mainly physical treatment methods. This is indicated by the relative decrease in lower chlorinated congeners comparing processed with crude oils. Apart from that, there is little or no difference in single congener composition within the chlorination degrees. These facts in turn could easily be explained by distillative processes. The exact distribution and fate for the analysed PCB congeners cannot be discussed in total due to lack of data for the whole aquatic system, beginning with concentrations in water, phytoplankton, and suspended matter. A complete mass balance would be necessary to completely understand distribution effects and metabolic effects, especially for the lower chlorinated PCBs.

References

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2. *B. Lauby-Secretan et.al.: The Lancet Oncology, Volume 14, Issue 4, Pages 287 - 288, April 2013;*
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4. *Neugebauer F., Ast C., Paepke O.: Organohalogen Compounds 74, 28-31 (2012)*
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6. *PS Rossa,G.M Ellis, M.G Ikonomoua, L.G Barrett-Lennard, R.F Addisona: Marine Pollution Bulletin Vol.40,(6),504–515 (2000)*
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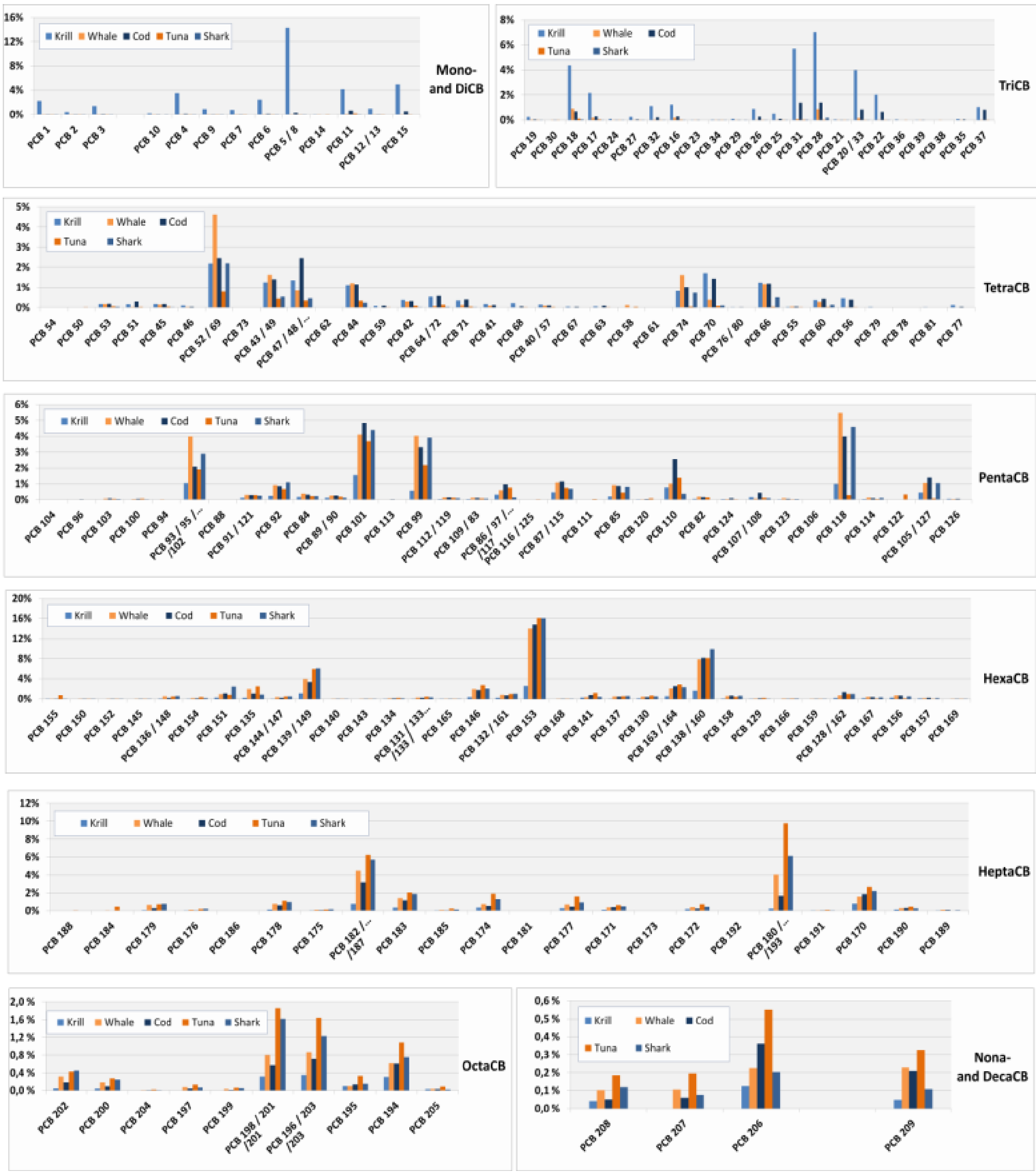


Figure 2: PCB Single congener distribution pattern for selected crude fish oils in percent of the total PCB

6 Appendix II: International documents and references

Article 4 – "Equivalence" of the World Trade Organizations' Agreement on Sanitary and Phytosanitary measures (SPS Agreement) states that "*Members shall accept the sanitary or phytosanitary measures of other Members as equivalent, even if these measures differ from their own or from those used by other Members trading in the same product, if the exporting Member objectively demonstrates to the importing Member that its measures achieve the importing Member's appropriate level of sanitary or phytosanitary protection. For this purpose, reasonable access shall be given, upon request, to the importing Member for inspection, testing and other relevant procedures.*"

Reference to Codex food safety standards is made in the World Trade Organizations' SPS Agreement. The Codex Alimentarius develop international food standards, guidelines and codes of practice in order to contribute to the safety, quality and fairness of international food trade.

The Codex Procedural Manual (23rd edition page 85) on "THE USE OF ANALYTICAL RESULTS: SAMPLING PLANS,RELATIONSHIP BETWEEN THE ANALYTICAL RESULTS, THEMEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND PROVISIONS IN CODEX STANDARDS" point 2 on Measurement Uncertainty states that "*An allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens*."

CAC/GL 70-2009 "GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS" section 2: "Prerequisits/assumptuions" states that "*laboratories report quantitative analytical results in the form of "a ± 2u" or "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty and "U" (equal to 2u) is the expanded uncertainty. The range "a ± 2u" represents a 95% level of confidence where the true value would be found. The value of "U" or "2u" is the value which is normally used and reported by analysts and is referred to as the "measurement uncertainty"; it may be estimated in a number of different ways...."*

CAC/GL 54-2004 "GUIDELINES ON MEASUREMENT UNCERTAINTY" section 8.1 shows an example of several situations when decisions are made based on a single test sample where an analytical result with analytical measurement uncertainty is compared against a maximum level.

CAC/GL 59-2006 “GUIDELINES ON ESTIMATION OF UNCERTAINTY OF RESULTS” section 5.1 explains the different relationships of measured value with associated uncertainty and MRL in compliance assessment. Different decision environments are discussed in section 5.2 of the guideline.

CAC/GL 83-2013 “PRINCIPLES FOR THE USE OF SAMPLING AND TESTING IN INTERNATIONAL FOOD TRADE” principle 5 states that “*The selection of the product assessment procedure should take into account analytical measurement uncertainty and its implications.” The explanatory notes further states that “The exporting country and the importing country should agree on how the analytical measurement uncertainty is taken into account when assessing the conformity of a measurement against a legal limit. This agreement should cover all situations where a limit or specification level is to be met, including limits for potential health hazards if such characteristics are to be assessed under the agreement.*”

A “DISCUSSION PAPER ON SAMPLING IN CODEX STANDARDS”” was prepared for the thirty-fifth Session of CCMAS (CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING) by an electronic working group chaired by the Inter-Agancy Meeting. Pages 17-18 of this document (CX/MAS 14/35/7) elaborates on the allowance for measurement uncertainty, enforcement situation and action to be taken by Authority Setting the Specification Level.
On enforcement situation the following is stated: “*The significance of this section in the Procedural Manual is that the laboratory at importation will deduct the measurement uncertainty. If the value after deduction is still greater than the specification, then it may be stated, beyond reasonable doubt, that the sample is not compliant with the specification. If sampling uncertainty is taken into account then without an alteration to a (maximum) control level, more samples will be deemed to be compliant with the control level.*
It is important for the exporter to realize that in order to be sure that the exported product meets the specification the “certificated value” obtained by the producer/exported must have the uncertainty of the result added to it, and for that value to be below the specification.”

7 Appendix III: Raw data from PCB_7 determinations at NIFES

Table 13: Complete results for PCB_7 in belly samples

ID	JNR	Boat	PCB-28 Belly, ng/g fresh weight	PCB-52 Belly, ng/g fresh weight	PCB-101 Belly, ng/g fresh weight	PCB-118 Belly, ng/g fresh weight	PCB-138 Belly, ng/g fresh weight	PCB-153 Belly, ng/g fresh weight	PCB-180 Belly, ng/g fresh weight	Sum PCB_7 (Lower Bound) Belly, ng/g fresh weight	Sum PCB_7 (Upper Bound) Belly, ng/g fresh weight
14	2015-914	Kato	1	15	20	30	79	97	32	270	270
15	2015-917	Kato	1	13	11	15	26	27	3.5	95	96
17	2015-921	Kato	2.2	11	7	12	27	29	11	100	100
18	2015-922	Kato	2.3	41	25	47	76	82	13	290	290
19	2015-924	Kato	1	1	1	1.4	2.8	3	1.2	8	11
20	2015-926	Kato	3.3	35	47	72	130	150	35	480	480
21	2015-929	Kato	2	15	12	20	35	39	12	130	130
22	2015-930	Kato	2	6	4.9	6	12	14	4.5	47	49
23	2015-931	Kato	2	13	14	17	32	34	10	120	120
24	2015-933	Kato	1.5	12	9	11	30	34	12	110	110
25	2015-935	Kato	1.9	75	43	73	130	140	24	480	480
26	2015-937	Kato	1.9	33	25	29	60	65	13	230	230
27	2015-938	Kato	2	13	9	19	34	37	8	120	120
28	2015-940	Kato	2	34	46	61	130	150	37	460	460
29	2015-941	Kato	4	11	8	13	27	29	9	96	100
30	2015-943	Kato	2	44	52	54	110	120	23	410	410
31	2015-945	Kato	1	5	6	7	16	19	9	62	63
33	2015-946	Kato	1.2	19	14	21	42	47	9	150	150
34	2015-947	Kato	1.6	44	39	56	110	120	21	390	390
35	2015-948	Kato	1	1	1	1	2.1	2.6	1	4.7	10
42	2015-949	Kato	1	10	11	12	26	30	10	98	99
43	2015-950	Kato	1	26	19	23	43	48	11	170	170
44	2015-951	Kato	1	12	12	14	28	31	9	110	110
45	2015-952	Kato	1	18	12	22	34	38	8	130	130
46	2015-953	Kato	1	4.3	4.5	6	11	13	3.6	41	42
47	2015-954	Kato	1	8	7	7	14	15	4.4	56	57
48	2015-955	Kato	1	5	7	8	16	18	4.4	59	60
49	2015-956	Kato	1	12	16	17	34	38	10	130	130
50	2015-957	Kato	1	12	15	17	28	30	7	110	110
51	2015-958	Kato	1	1.3	1	1	1.3	1.7	1	4.3	8
52	2015-959	Kato	1	1.3	1	1.3	2.8	3.2	1.7	10	12
53	2015-960	Kato	1	8	9	11	20	25	7	79	80
54	2015-961	Kato	1.5	40	46	51	100	110	32	380	380
55	2015-963	Kato	1	7	6	8	13	13	2.7	49	50
56	2015-965	Kato	1	6	5	6	11	13	5	46	47
57	2015-966	Kato	1	4.3	3.9	6	11	12	3.7	40	41
58	2015-967	Kato	1	3.4	3.7	5	10	12	3.7	38	39

ID	JNR	Boat	PCB-28 Belly, ng/g fresh weight	PCB-52 Belly, ng/g fresh weight	PCB-101 Belly, ng/g fresh weight	PCB-118 Belly, ng/g fresh weight	PCB-138 Belly, ng/g fresh weight	PCB-153 Belly, ng/g fresh weight	PCB-180 Belly, ng/g fresh weight	Sum PCB_7 (Lower Bound) Belly, ng/g fresh weight	Sum PCB_7 (Upper Bound) Belly, ng/g fresh weight
59	2015-968	Kato	1	14	12	19	38	42	12	140	140
60	2015-969	Kato	1	5	4.6	4.6	10	11	3.9	39	40
61	2015-970	Kato	1.2	11	10	11	24	28	10	95	95
41	2015-982	Fiskebank 1	1.2	29	52	52	130	160	36	460	460
42	2015-983	Fiskebank 1	1	10	14	15	40	50	14	140	140
43	2015-984	Fiskebank 1	1	27	35	48	100	110	19	350	350
44	2015-985	Fiskebank 1	1	23	37	84	150	170	35	490	490
45	2015-986	Fiskebank 1	1.3	15	18	26	48	55	11	170	170
46	2015-987	Fiskebank 1	1	20	31	47	140	160	43	440	440

Table 14: Complete results for PCB_7 in back samples

ID	JNR	Boat	PCB-28 Back, ng/g fresh weight	PCB-52 Back, ng/g fresh weight	PCB-101 Back, ng/g fresh weight	PCB-118 Back, ng/g fresh weight	PCB-138 Back, ng/g fresh weight	PCB-153 Back, ng/g fresh weight	PCB-180 Back, ng/g fresh weight	Sum PCB_7 (Lower Bound) Back, ng/g fresh weight	Sum PCB_7 (Upper Bound) Back, ng/g fresh weight
14	2015-914	Kato	1.4	21	28	41	110	120	36	360	360
15	2015-917	Kato	1	6	5	10	16	18	2.9	58	59
17	2015-921	Kato	2.3	12	9	12	25	27	9	96	96
18	2015-922	Kato	1	18	10	21	30	33	5	120	120
19	2015-924	Kato	1	3.5	2.2	4.6	8	9	2.8	30	31
20	2015-926	Kato	1.9	23	26	44	73	84	19	270	270
21	2015-929	Kato	2	12	10	16	32	35	10	120	120
22	2015-930	Kato	2	8	7	8	16	17	6	61	63
23	2015-931	Kato	2	16	15	22	41	43	15	150	150
24	2015-933	Kato	1	4.5	2.8	3.9	10	12	3.8	37	38
25	2015-935	Kato	1	47	29	47	80	85	14	300	300
26	2015-937	Kato	2	30	20	26	46	50	10	180	180
27	2015-938	Kato	2	7	4.3	8	16	16	3.6	54	56
28	2015-940	Kato	2	30	40	51	120	140	34	410	410
29	2015-941	Kato	4	9	5	10	19	21	7	71	75
30	2015-943	Kato	2	24	25	33	59	63	13	220	220
31	2015-945	Kato	1	6	8	8	20	24	11	77	78
33	2015-946	Kato	1.3	16	12	17	36	40	8	130	130
34	2015-947	Kato	1	27	23	36	68	78	16	250	250
35	2015-948	Kato	1	5	5	6	18	22	9	65	66
42	2015-949	Kato	1	1	1	1.1	1.9	2.1	1	5	9
43	2015-950	Kato	1	25	18	24	38	41	9	150	160

ID	JNR	Boat	PCB-28 Back, ng/g fresh weight	PCB-52 Back, ng/g fresh weight	PCB-101 Back, ng/g fresh weight	PCB-118 Back, ng/g fresh weight	PCB-138 Back, ng/g fresh weight	PCB-153 Back, ng/g fresh weight	PCB-180 Back, ng/g fresh weight	Sum PCB_7 (Lower Bound) Back, ng/g fresh weight	Sum PCB_7 (Upper Bound) Back, ng/g fresh weight
44	2015-951	Kato	1	1	1	1.5	2.6	3.2	1.1	8	11
45	2015-952	Kato	1	31	21	41	65	73	14	250	250
46	2015-953	Kato	1	17	16	19	32	34	7	130	130
47	2015-954	Kato	1	8	6	7	13	13	3.9	51	52
48	2015-955	Kato	1	13	18	21	43	52	14	160	160
49	2015-956	Kato	1	22	27	25	56	62	15	210	210
50	2015-957	Kato	1	22	30	30	60	65	15	220	220
51	2015-958	Kato	1.6	1.6	1.3	1.5	2.7	3.4	1.4	13	13
52	2015-959	Kato	1	1.8	1.3	2	4.5	6	2.5	18	19
53	2015-960	Kato	1.2	33	30	35	58	61	12	230	230
54	2015-961	Kato	1.2	70	90	110	210	240	68	780	780
55	2015-963	Kato	2.1	24	19	25	40	41	8	160	160
56	2015-965	Kato	1.3	7	6	7	14	16	6	57	57
57	2015-966	Kato	1	5	4.6	7	12	14	4.4	47	48
58	2015-967	Kato	1	3.6	3	3.8	8	9	2.9	30	31
59	2015-968	Kato	1	29	27	38	82	89	24	290	290
60	2015-969	Kato	1.1	7	6	7	14	16	5	56	56
61	2015-970	Kato	1	3.5	4.2	4.9	10	12	3.6	38	39
41	2015-982	Fiskebank 1	1.2	26	50	51	150	180	42	500	500
42	2015-983	Fiskebank 1	1	13	18	21	44	50	10	160	160
43	2015-984	Fiskebank 1	1	19	21	32	58	64	14	210	210
44	2015-985	Fiskebank 1	2	26	43	75	170	190	39	540	550
45	2015-986	Fiskebank 1	2.1	18	20	29	48	59	12	190	190
46	2015-987	Fiskebank 1	1.2	37	70	100	320	360	73	960	960

Table 15: Complete results for PCB_7 in underjaw samples

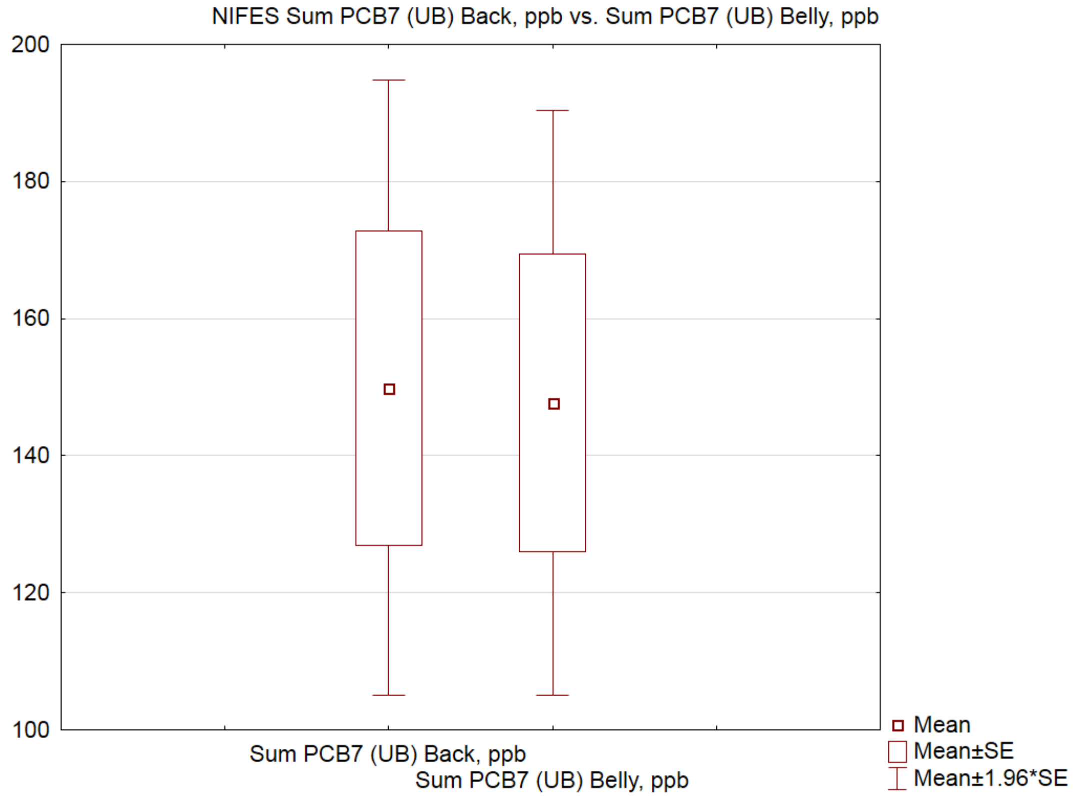
ID	JNR	Boat	PCB-28 Under-jaw, ng/g fresh weight	PCB-52 Under-jaw, ng/g fresh weight	PCB-101 Under-jaw, ng/g fresh weight	PCB-118 Under-jaw, ng/g fresh weight	PCB-138 Under-jaw, ng/g fresh weight	PCB-153 Under-jaw, ng/g fresh weight	PCB-180 Under-jaw, ng/g fresh weight	Sum PCB_7 (Lower Bound) Under-jaw, ng/g fresh weight	Sum PCB_7 (Upper Bound) Under-jaw, ng/g fresh weight
14	2015-914	Kato	1.9	27	37	51	150	170	51	490	490
15	2015-917	Kato	1	9	7	15	20	22	3.2	76	77
17	2015-921	Kato	2.2	13	8	13	25	28	9	98	98
18	2015-922	Kato	1	17	10	22	33	36	6	120	130
19	2015-924	Kato	1	3.3	1.9	3.8	7	8	2.6	28	29

ID	JNR	Boat	PCB-28 Under-jaw, ng/g fresh weight	PCB-52 Under-jaw, ng/g fresh weight	PCB-101 Under-jaw, ng/g fresh weight	PCB-118 Under-jaw, ng/g fresh weight	PCB-138 Under-jaw, ng/g fresh weight	PCB-153 Under-jaw, ng/g fresh weight	PCB-180 Under-jaw, ng/g fresh weight	Sum PCB_7 (Lower Bound) Under-jaw, ng/g fresh weight	Sum PCB_7 (Upper Bound) Under-jaw, ng/g fresh weight
20	2015-926	Kato	2	9	10	13	28	32	8	100	100
21	2015-929	Kato	1.9	1	1	1.2	2.6	3.8	1	9	12
22	2015-930	Kato	2	2.7	2.3	2.7	6	7	2.6	24	26
23	2015-931	Kato	2	2	1.2	2.2	3.8	4.2	1.5	13	17
24	2015-933	Kato	1	7	6	9	24	29	10	85	86
25	2015-935	Kato	1.8	67	37	60	100	100	17	390	390
26	2015-937	Kato	2.9	44	31	41	71	77	15	280	280
27	2015-938	Kato	2	16	11	20	33	35	7	120	120
28	2015-940	Kato	2	6	9	12	31	36	9	100	110
29	2015-941	Kato	2	5	3.3	6	12	13	4.1	43	45
30	2015-943	Kato	2	16	18	20	38	41	8	140	140
31	2015-945	Kato	1	3.2	4.2	4.6	12	15	9	48	49
33	2015-946	Kato	2	10	7	12	23	26	5	83	85
34	2015-947	Kato	1.6	40	34	54	100	120	24	370	370
35	2015-948	Kato	1	13	12	14	34	41	16	130	130
42	2015-949	Kato	1	8	8	9	18	20	7	71	71
43	2015-950	Kato	1	3.9	2.8	4.5	8	10	3	32	33
44	2015-951	Kato	1	7	7	9	16	19	5	64	65
45	2015-952	Kato	1	13	8	15	23	25	5	89	90
46	2015-953	Kato	1	11	9	12	19	21	3	75	76
47	2015-954	Kato	1.4	12	11	11	21	24	7	87	87
48	2015-955	Kato	1	6	8	11	20	24	7	76	77
49	2015-956	Kato	1	7	10	12	22	24	6	81	82
50	2015-957	Kato	1	21	31	30	56	61	15	210	220
51	2015-958	Kato	1.5	8	8	7	16	20	7	67	67
52	2015-959	Kato	1	12	12	16	35	40	12	130	130
53	2015-960	Kato	1	4.9	5	6	11	12	3	42	43
54	2015-961	Kato	1	10	13	15	30	34	10	110	110
55	2015-963	Kato	1	14	11	15	22	24	4.9	90	91
56	2015-965	Kato	1.7	8	6	7	14	15	5	56	56
57	2015-966	Kato	1.2	9	8	12	21	24	7	82	82
58	2015-967	Kato	1	13	14	15	37	44	13	140	140
59	2015-968	Kato	1	7	6	9	20	22	6	70	71
60	2015-969	Kato	1	3.9	3.1	3.7	6	7	2.4	26	27
61	2015-970	Kato	1	2.1	2.3	2.7	5	6	1.7	20	21
41	2015-982	Fiskebank 1	1	4	8	9	24	32	8	84	85
42	2015-983	Fiskebank 1	1	23	33	37	79	88	16	280	280
43	2015-984	Fiskebank 1	1	4.5	5	10	20	24	5	69	70

ID	JNR	Boat	PCB-28 Under-jaw, ng/g fresh weight	PCB-52 Under-jaw, ng/g fresh weight	PCB-101 Under-jaw, ng/g fresh weight	PCB-118 Under-jaw, ng/g fresh weight	PCB-138 Under-jaw, ng/g fresh weight	PCB-153 Under-jaw, ng/g fresh weight	PCB-180 Under-jaw, ng/g fresh weight	Sum PCB_7 (Lower Bound) Under-jaw, ng/g fresh weight	Sum PCB_7 (Upper Bound) Under-jaw, ng/g fresh weight
44	2015-985	Fiskebank 1	1	9	14	32	63	72	15	200	210
45	2015-986	Fiskebank 1	1	10	13	21	37	44	10	130	140
46	2015-987	Fiskebank 1	1	16	30	50	150	170	34	450	450

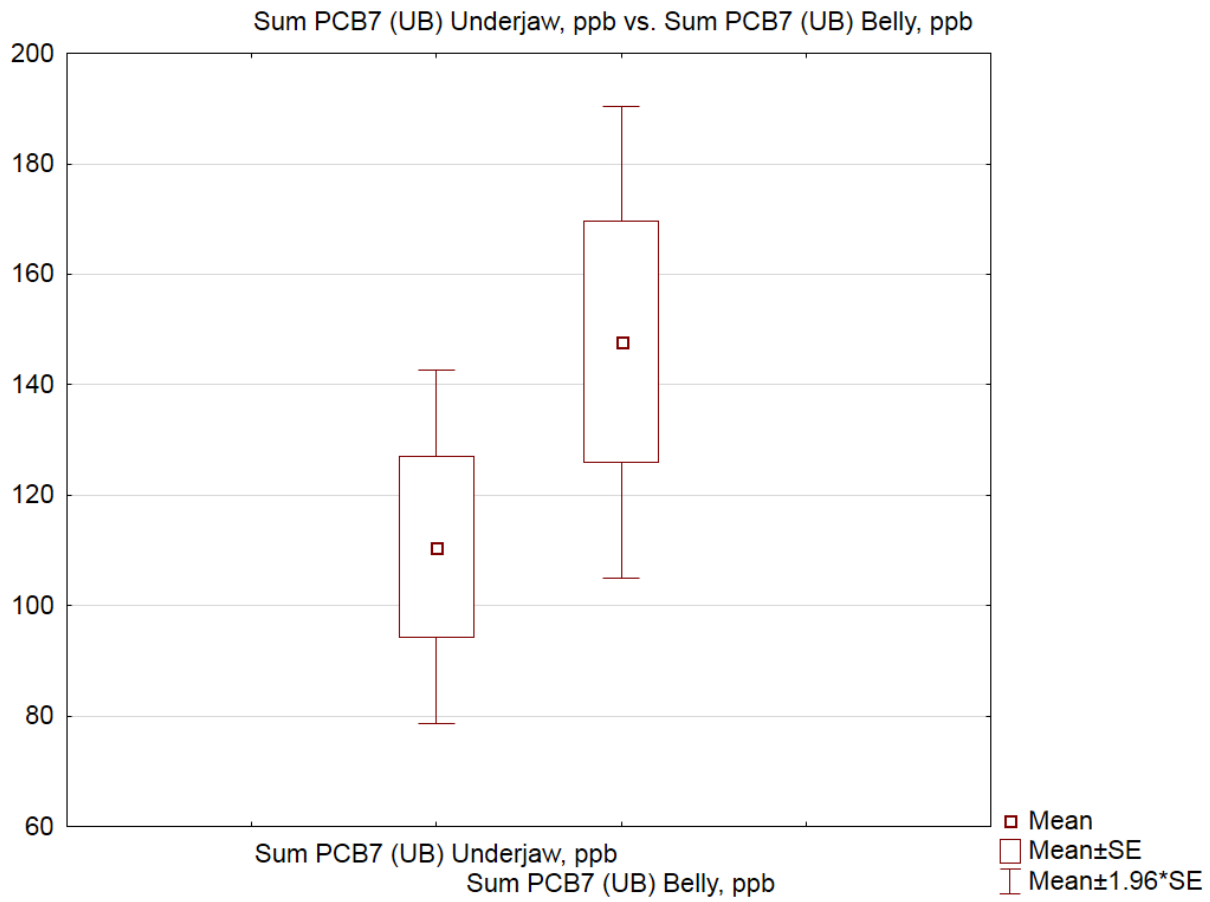
8 Appendix IV: Comparison between PCB_7 in different types of blubber from Kato

8.1 PCB_7 (UB) in blubber from belly and back



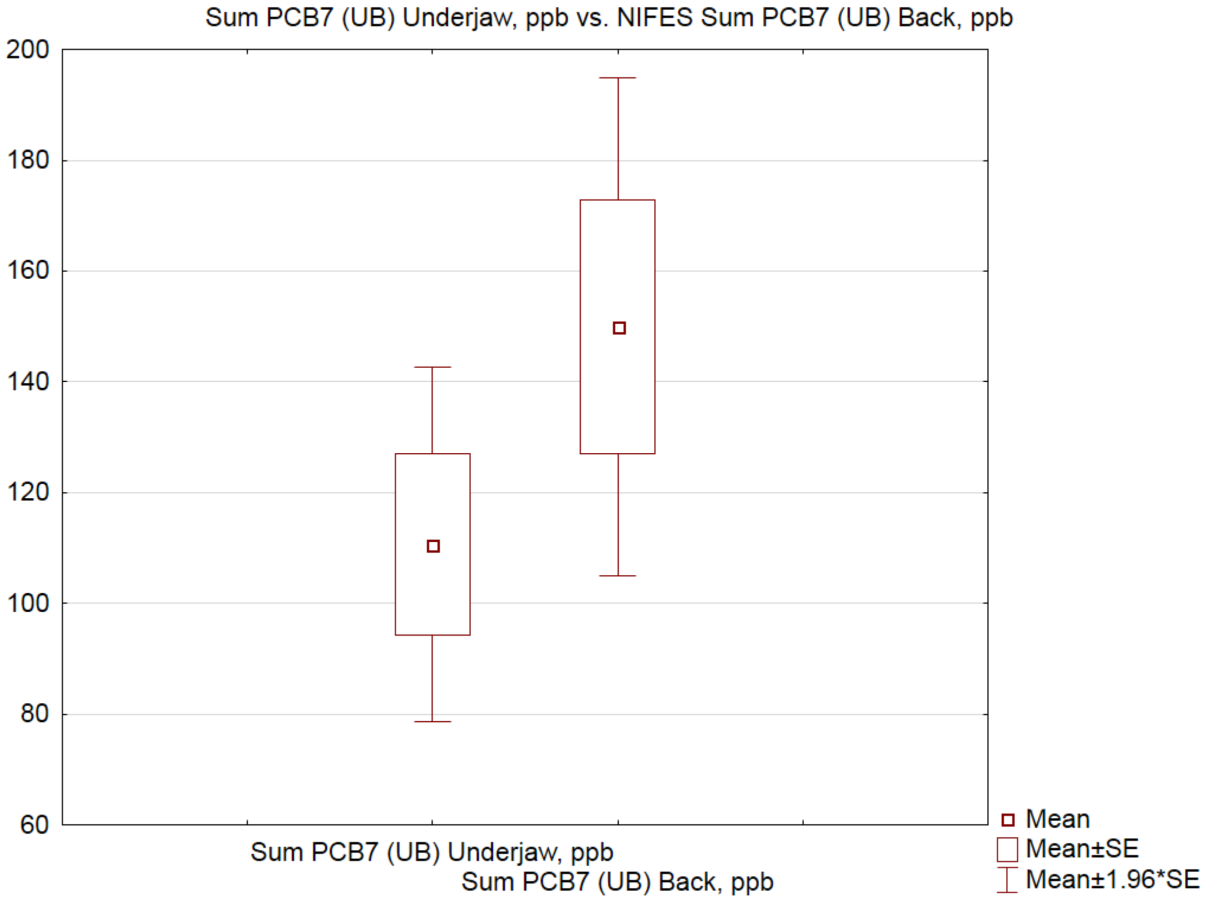
Variable	Sum PCB_7 (UB) Belly, ppb	Sum PCB_7 (UB) Back, ppb
Mean	147.725	149.925
Std.Dv.	137.7621	145.0003
N		40
Diff.		-2.2
Std.Dv. Diff.		110.9519
t		-0.12541
df		39
p		0.900846
Confidence -95.00%		-33.2841
Confidence 95.00%		37.68413

8.2 PCB$_7$ (UB) in blubber from belly and underjaw



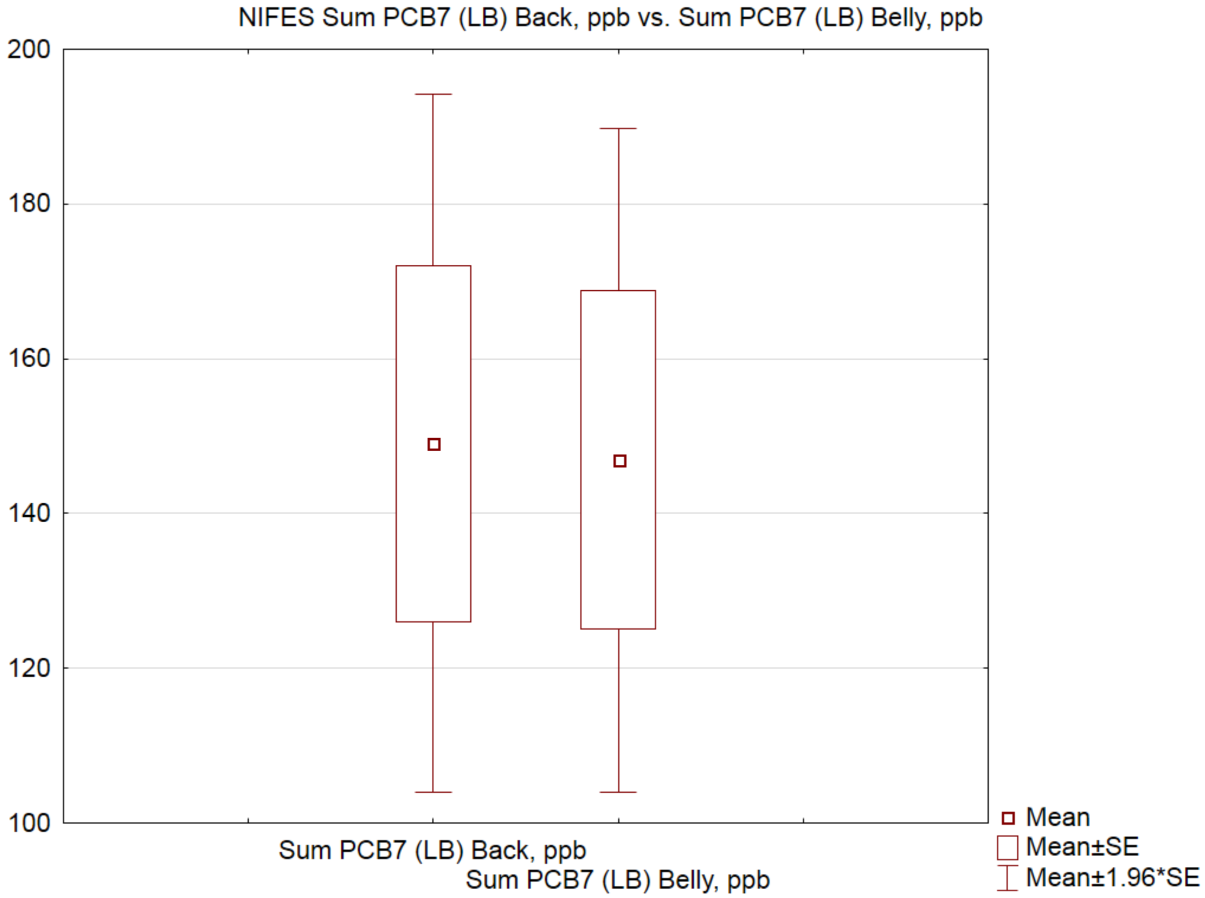
Variable	Sum PCB$_7$ (UB) Belly, ppb	Sum PCB$_7$ (UB) Underjaw, ppb
Mean	147.725	110.575
Std.Dv.	137.7621	103.4036
N		40
Diff.		37.15
Std.Dv. Diff.		121.2561
t		1.93769
df		39
p		0.059926
Confidence -95.00%		-75.9296
Confidence 95.00%		1.6296

8.3 PCB_7 (UB) in blubber from underjaw and back



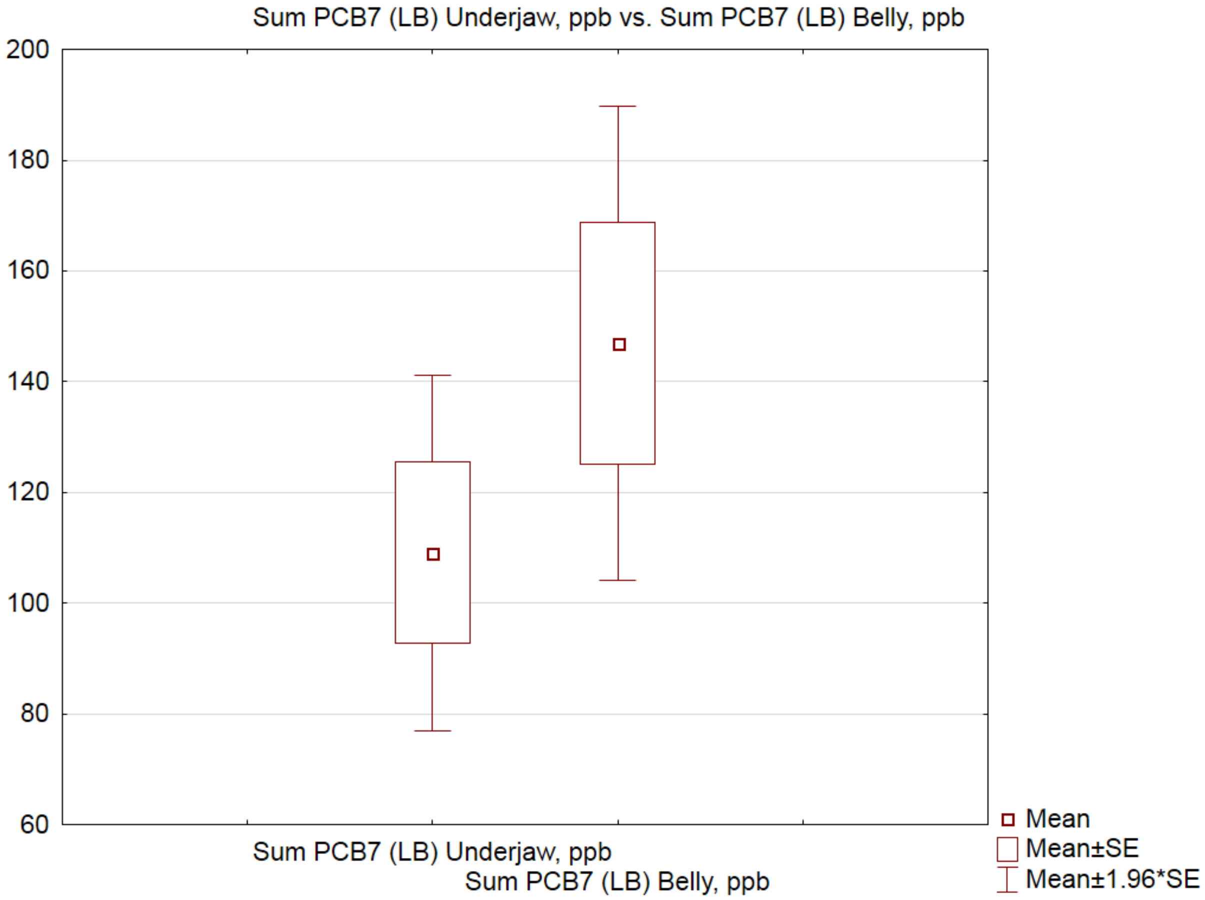
Variable	Sum PCB_7 (UB) Underjaw, ppb	Sum PCB_7 (UB) Back, ppb
Mean	110.575	149.925
Std.Dv.	103.4036	145.0003
N		40
Diff.		-39.35
Std.Dv. Diff.		143.2331
t		-1.73753
df		39
p		0.090187
Confidence -95.00%		-85.1582
Confidence 95.00%		6.45817

8.4 PCB_7 (LB) in blubber from belly and back



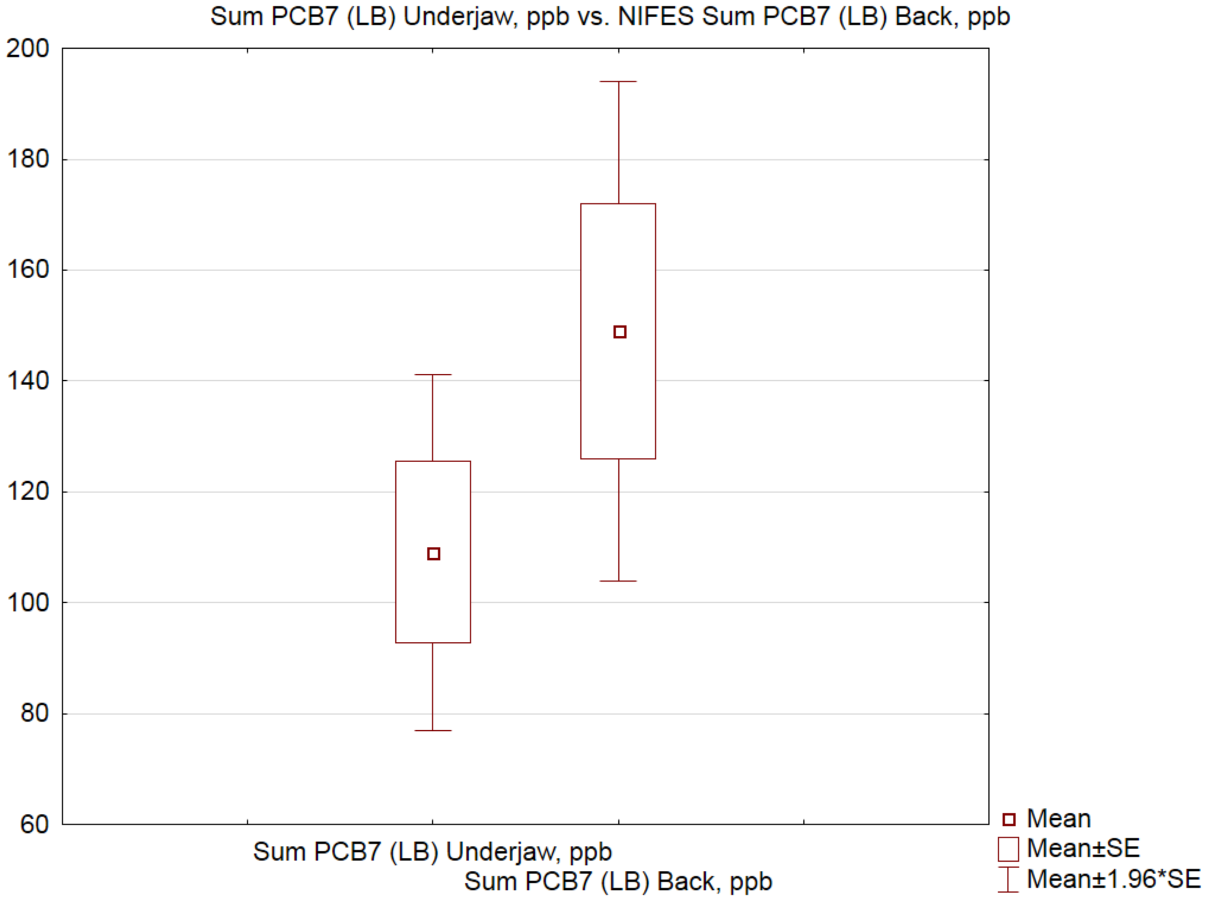
Variable	Sum PCB_7 (LB) Belly, ppb	Sum PCB_7 (LB) Back, ppb
Mean	146.925	149.05
Std.Dv.	138.3947	145.4671
N		40
Diff.		-2.125
Std.Dv. Diff.		111.3656
t		-0.12068
df		39
p		0.904564
Confidence -95.00%		-33.4914
Confidence 95.00%		37.74143

8.5 PCB_7 (LB) in blubber from belly and underjaw



Variable	Sum PCB_7 (LB) Belly, ppb	Sum PCB_7 (LB) Underjaw, ppb
Mean	146.925	109.125
Std.Dv.	138.3947	103.5558
N		40
Diff.		37.8
Std.Dv. Diff.		122.4609
t		1.9522
df		39
p		0.05812
Confidence -95.00%		-76.9649
Confidence 95.00%		1.36488

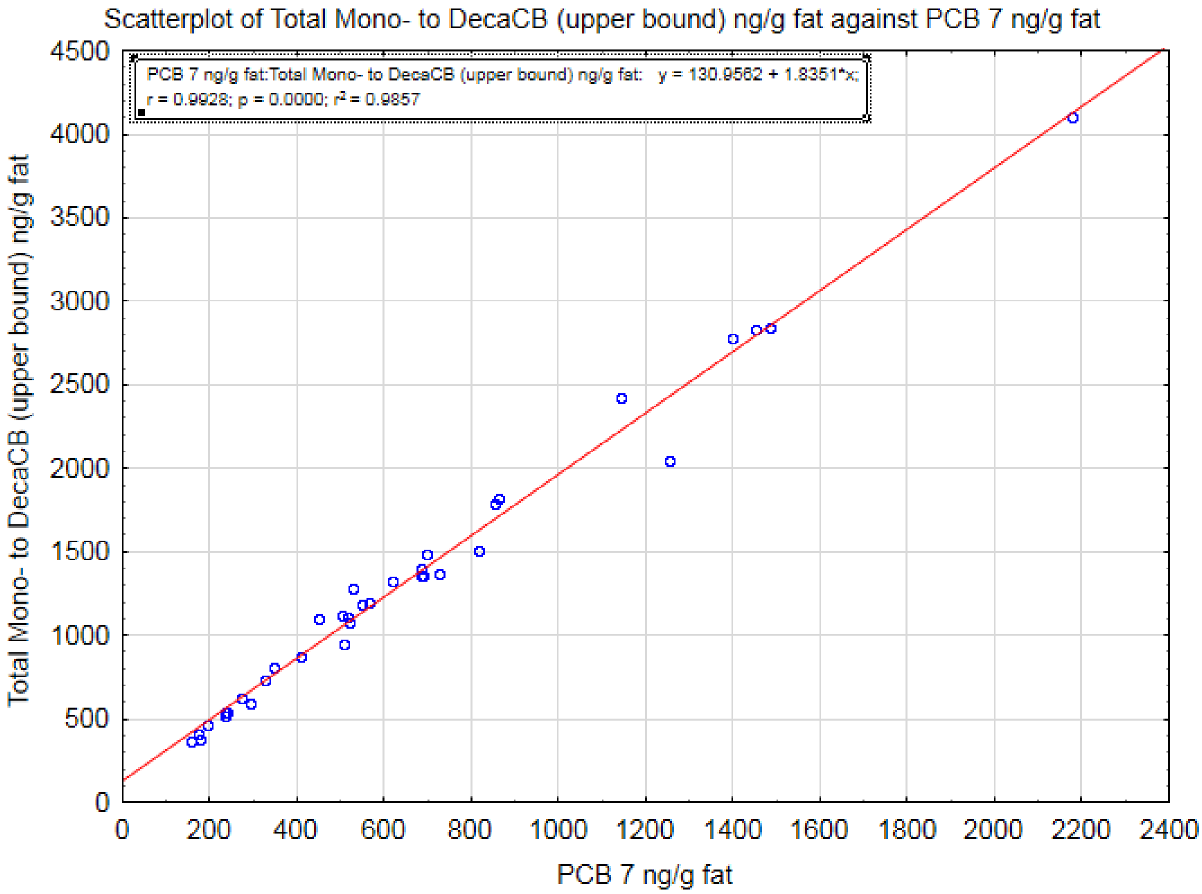
8.6 PCB_7 (LB) in blubber from underjaw and back



Variable	Sum PCB_7 (LB) Underjaw, ppb	NIFES Sum PCB_7 (LB) Back, ppb
Mean	109.125	149.05
Std.Dv.	103.5558	145.4671
N		40
Diff.		-39.925
Std.Dv. Diff.		143.898
t		-1.75477
df		39
p		0.087153
Confidence -95.00%		-85.9458
Confidence 95.00%		6.09581

9 Appendix V: Regressions, correlations and scatterplots between PCB_7 and PCB_{209} determined by Eurofins

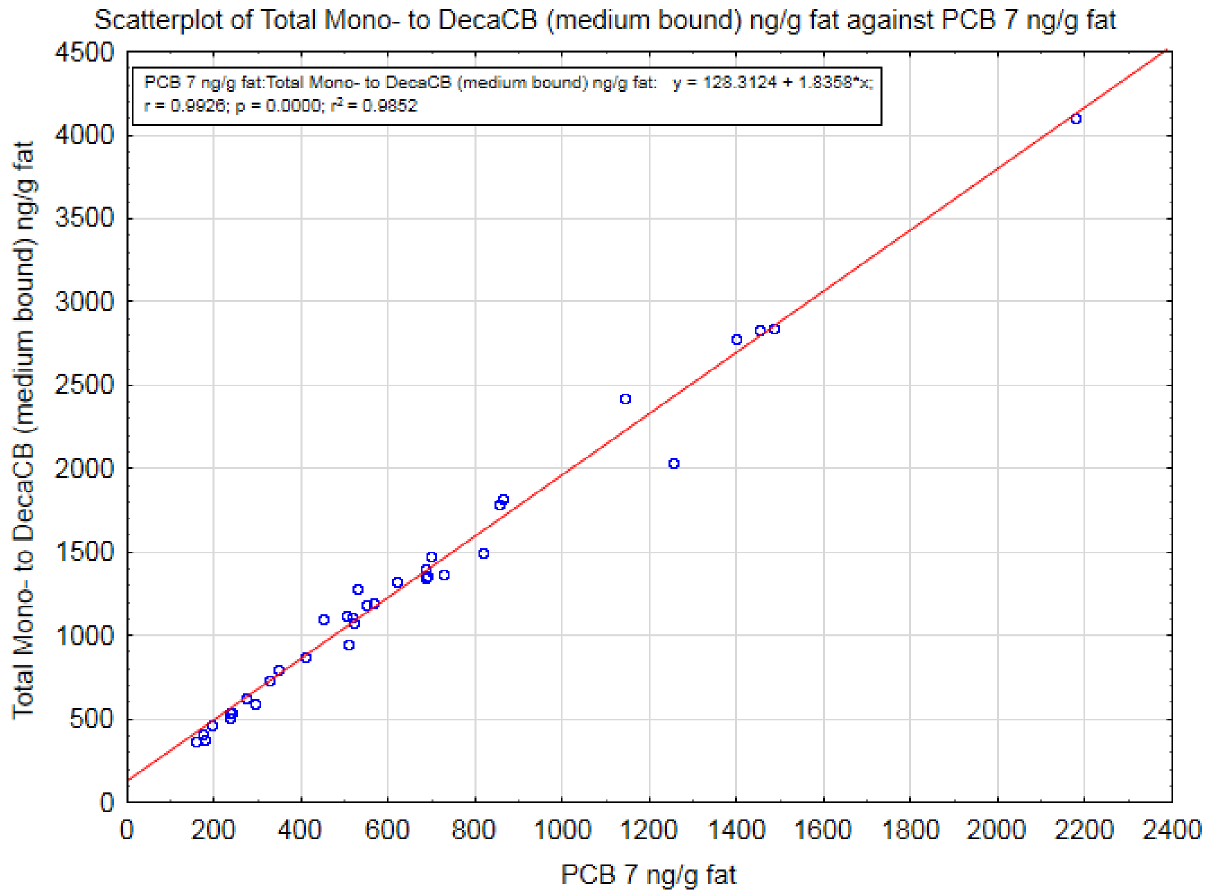
9.1 PCB_{209} (Upper Bound) vs PCB_7 (ng/g fat)



N=35	Regression Summary for Dependent Variable: Total Mono- to DecaCB (upper bound) ng/g fat R= .99279961 R²= .98565106 Adjusted R²= .98521624 F(1,33)=2266.8 p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			130.9562	30.44892	4.30085	0.000142
PCB_7 ng/g fat	0.992800	0.020852	1.8351	0.03854	47.61115	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (upper bound) ng/g fat
	Value
Multiple R	0.992799607
Multiple R²	0.985651061
Adjusted R²	0.985216244
F(1,33)	2266.82155
p	5.36881184E-32
Std.Err. of Estimate	102.420057

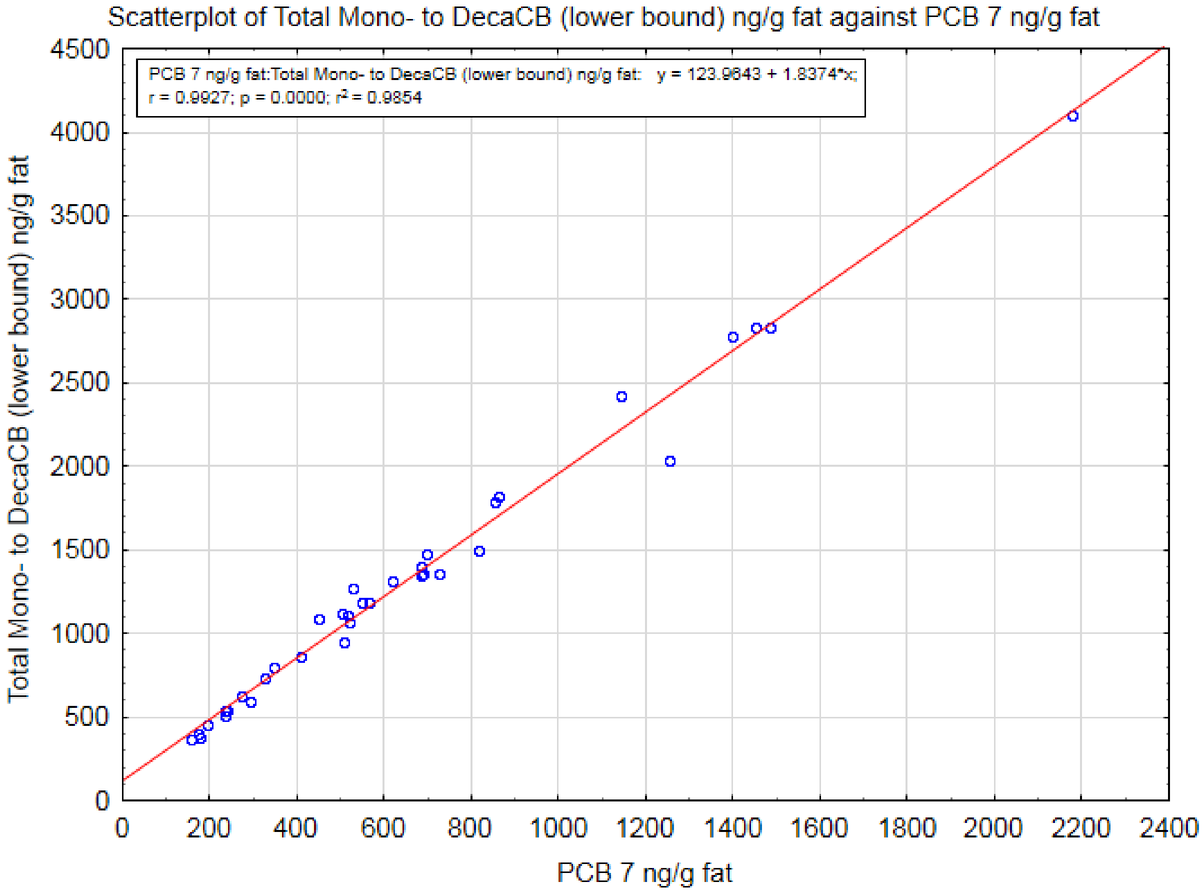
9.2 PCB_{209} (Medium Bound) vs PCB_7 (ng/g fat)



	Regression Summary for Dependent Variable: Total Mono- to DecaCB (medium bound) ng/g fat R= .99256086 R²= .98517706 Adjusted R²= .98472788 F(1,33)=2193.3 p					
N=35	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			128.3124	30.96746	4.14346	0.000224
PCB_7 ng/g fat	0.992561	0.021194	1.8358	0.03920	46.83246	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (medium bound) ng/g fat
	Value
Multiple R	0.992560862
Multiple R²	0.985177065
Adjusted R²	0.984727885
F(1,33)	2193.2797
p	9.18045219E-32
Std.Err. of Estimate	104.164246

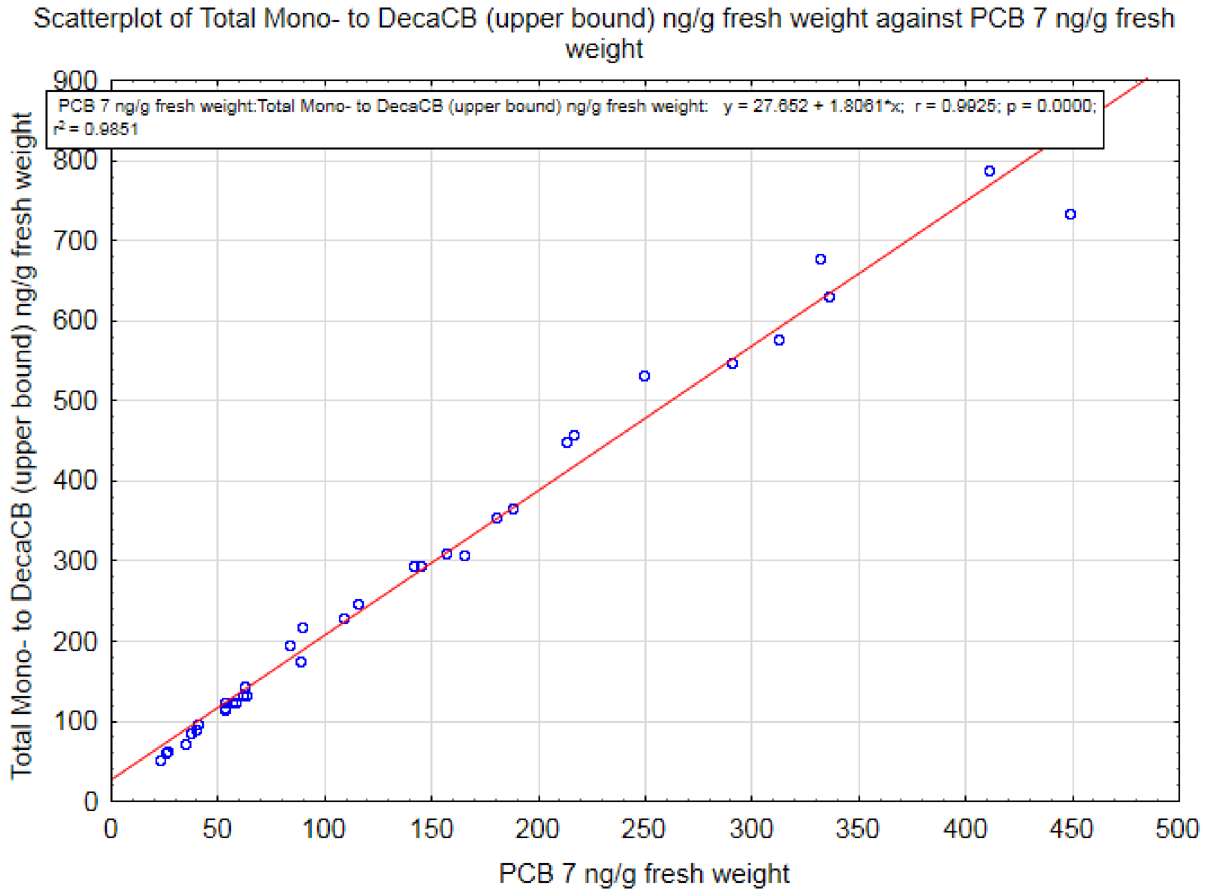
9.3 PCB209 (Lower Bound) vs PCB7 (ng/g fat)



	Regression Summary for Dependent Variable: Total Mono- to DecaCB (lower bound) ng/g fat R= .99266975 R^2= .98539323 Adjusted R^2= .98495061 F(1,33)=2226.2 p					
N=35	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			123.9643	30.76408	4.02951	0.000309
PCB_7 ng/g fat	0.992670	0.021039	1.8374	0.03894	47.18291	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (lower bound) ng/g fat
	Value
Multiple R	0.992669751
Multiple R^2	0.985393235
Adjusted R^2	0.984950606
F(1,33)	2226.22711
p	7.20355634E-32
Std.Err. of Estimate	103.480164

9.4 PCB_{209} (Upper Bound) vs PCB_7 (ng/g fresh weight)

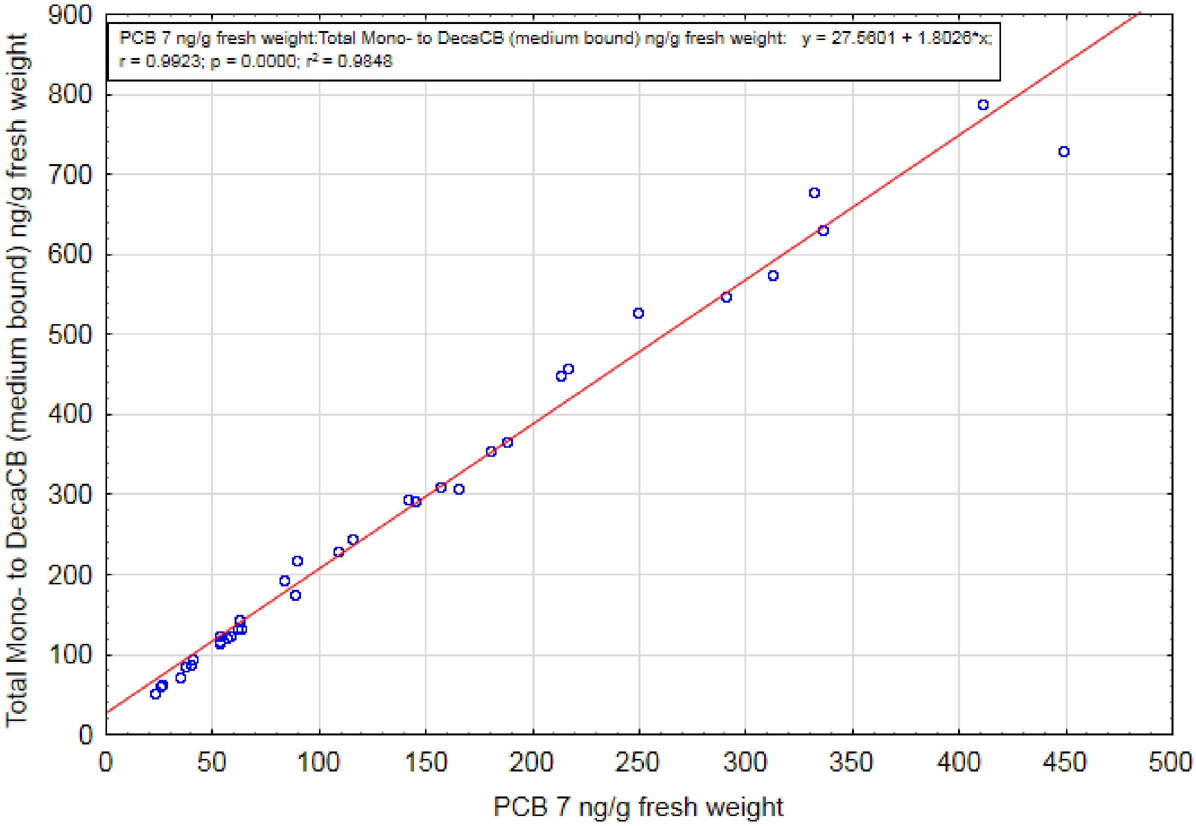


N=35	Regression Summary for Dependent Variable: Total Mono- to DecaCB (upper bound) ng/g fresh weight R= .99254612 R²= .98514780 Adjusted R²= .98469773 F(1,33)=2188.9 p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			27.65203	7.062247	3.91547	0.000427
PCB_7 ng/g fresh weight	0.992546	0.021215	1.80614	0.038605	46.78561	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (upper bound) ng/g fresh weight
	Value
Multiple R	0.99254612
Multiple R²	0.985147801
Adjusted R²	0.984697734
F(1,33)	2188.89318
p	9.48426928E-32
Std.Err. of Estimate	26.4645929

9.5 PCB_{209} (Medium Bound) vs PCB_7 (ng/g fresh weight)

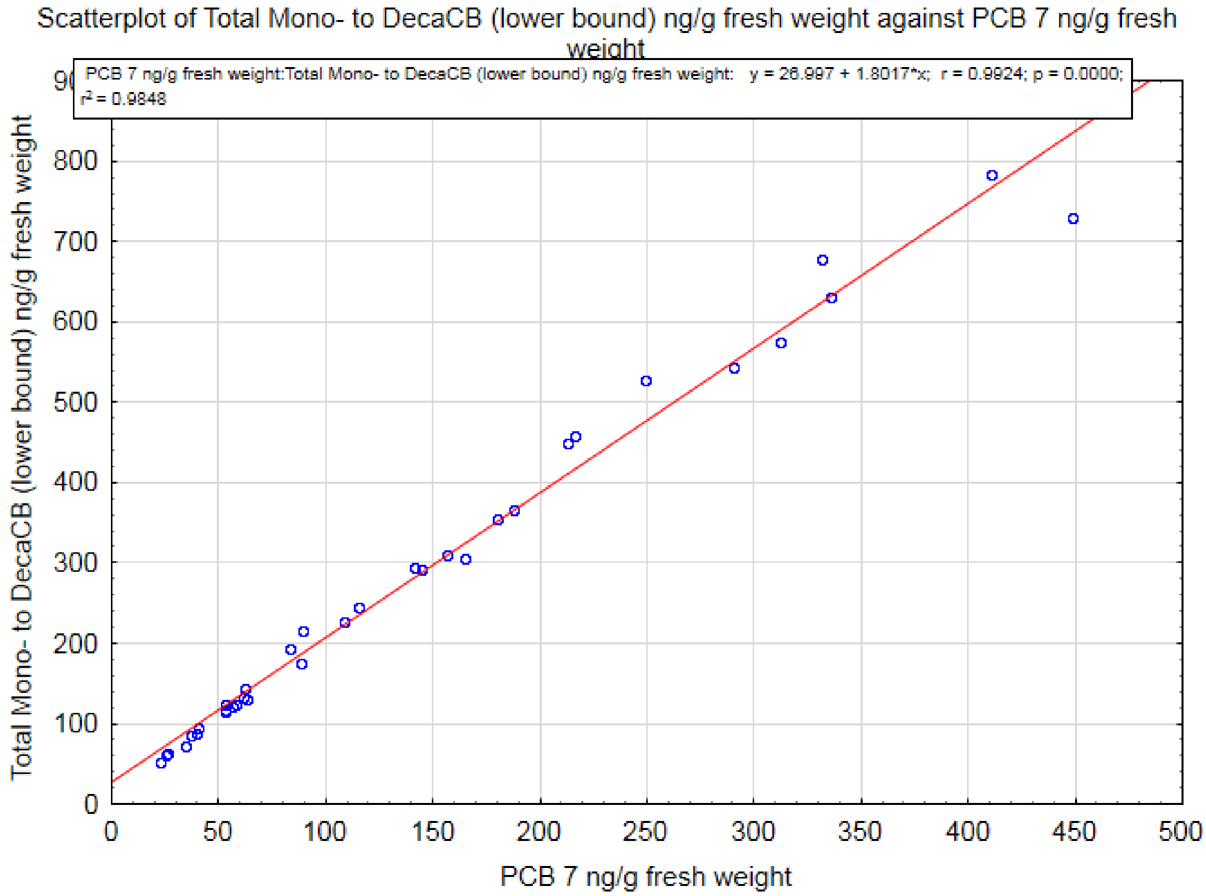
Scatterplot of Total Mono- to DecaCB (medium bound) ng/g fresh weight against PCB 7 ng/g fresh weight



N=35	Regression Summary for Dependent Variable: Total Mono- to DecaCB (medium bound) ng/g fresh weight R= .99234693 R²= .98475242 Adjusted R²= .98429038 F(1,33)=2131.3 p				
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			27.56011	7.143085	3.85829	0.000502
PCB_7 ng/g fresh weight	0.992347	0.021495	1.80261	0.039047	46.16577	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (medium bound) ng/g fresh weight
	Value
Multiple R	0.992346928
Multiple R²	0.984752425
Adjusted R²	0.984290377
F(1,33)	2131.27853
p	1.46335916E-31
Std.Err. of Estimate	26.7675181

9.6 PCB_{209} (Lower Bound) vs PCB_7 (ng/g fresh weight)

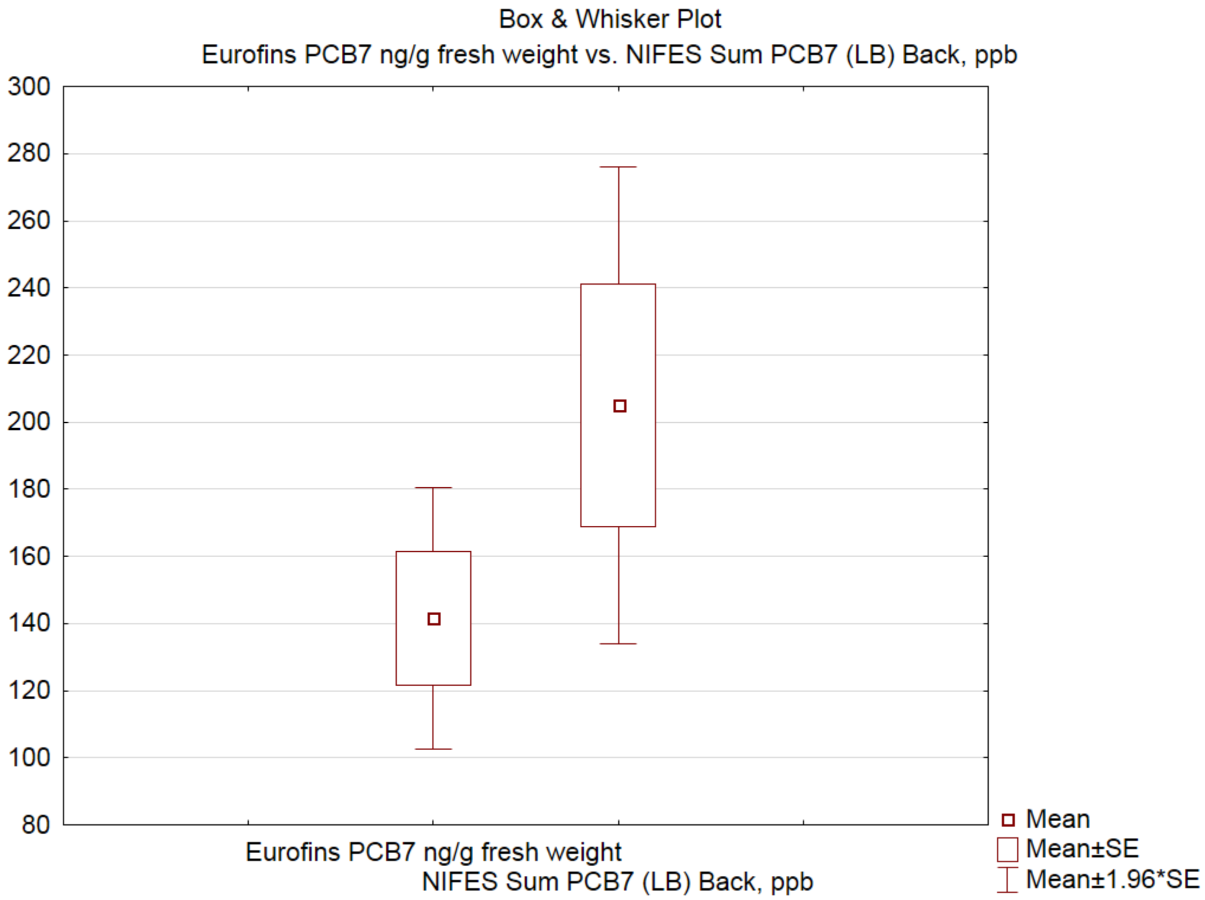


N=35	Regression Summary for Dependent Variable: Total Mono- to DecaCB (lower bound) ng/g fresh weight R= .99237339 R²= .98480495 Adjusted R²= .98434449 F(1,33)=2138.8 p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			26.99703	7.126994	3.78800	0.000612
PCB_7 ng/g fresh weight	0.992373	0.021458	1.80171	0.038959	46.24673	0.000000

Statistic	Summary Statistics; DV: Total Mono- to DecaCB (lower bound) ng/g fresh weight
	Value
Multiple R	0.992373392
Multiple R²	0.98480495
Adjusted R²	0.984344494
F(1,33)	2138.75985
p	1.38233304E-31
Std.Err. of Estimate	26.7072209

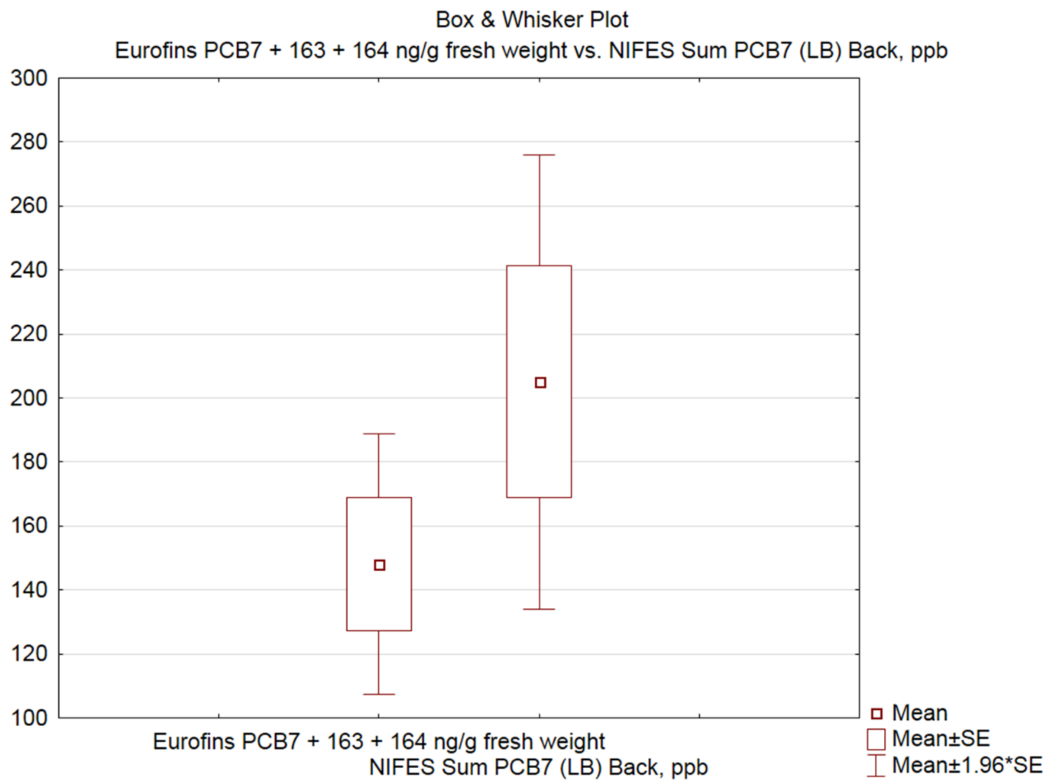
10 Appendix VI: Comparison between NIFES PCB_7 and Eurofins PCB_7

10.1 Eurofins PCB_7 (ng/g fresh weight) vs NIFES PCB_7 (Lower Bound)



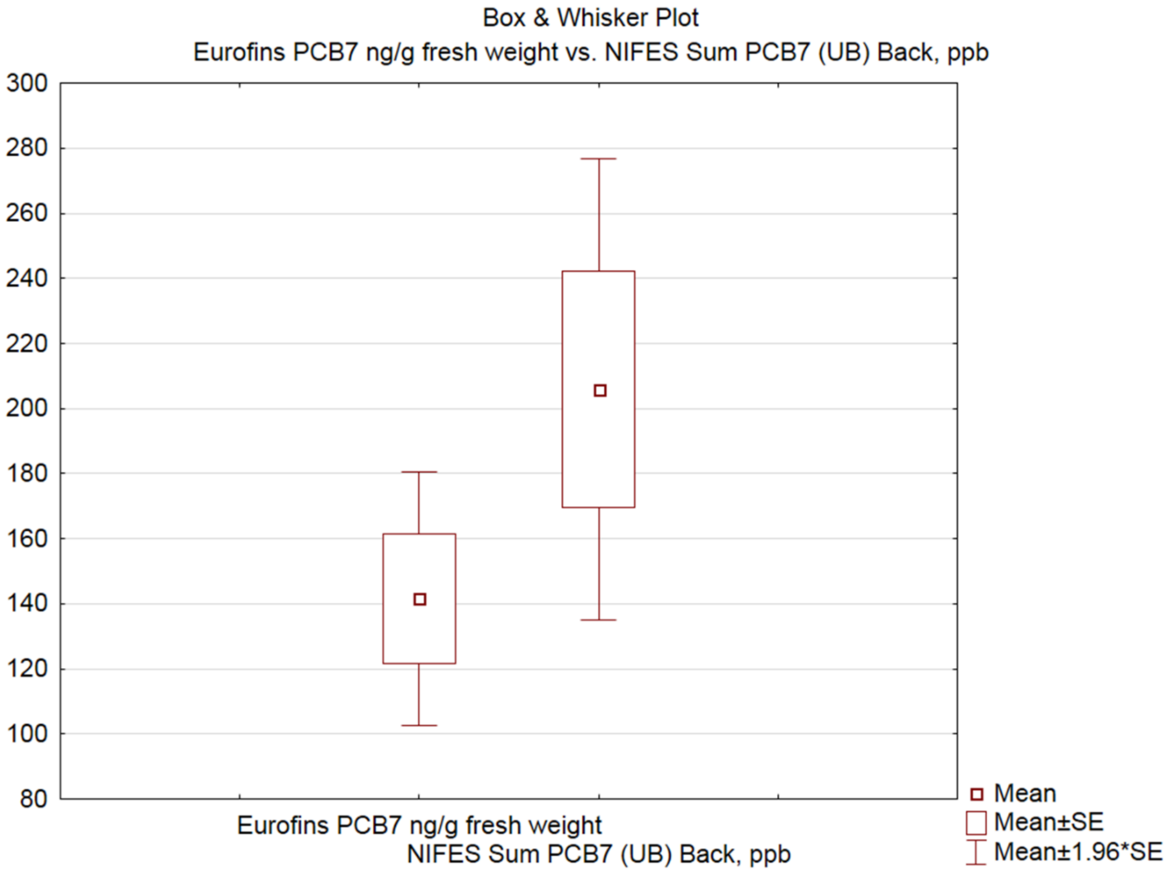
T-test for Dependent Samples are significant at $p < .05000$		
Variable	Eurofins PCB_7 ng/g fresh weight	NIFES Sum PCB_7 (LB) Back, ppb
Mean	141.5596	205.0857
Std.Dv.	117.5673	214.158
N		35
Diff.		-63.5261
Std.Dv. Diff.		155.6158
t		-2.41509
df		34
p		0.021257
Confidence -95.00%		-116.982
Confidence 95.00%		-10.0702

10.2 Eurofins PCB_7 + PCB 163 + PCB 164 (ng/g fresh weight) vs NIFES PCB_7 (Lower Bound)



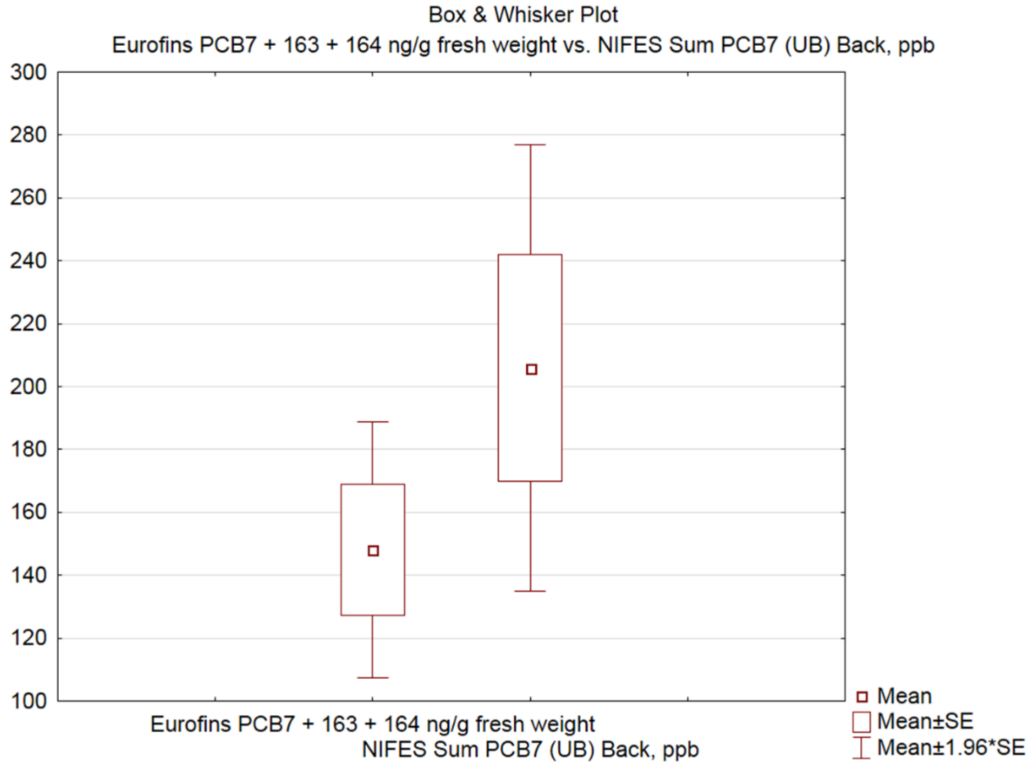
T-test for Dependent Samples Marked differences are significant at $p < .05000$		
Variable	Eurofins PCB_7 + 163 + 164 ng/g fresh weight	NIFES Sum PCB_7 (LB) Back, ppb
Mean	148.0705	205.0857
Std.Dv.	122.7271	214.158
N		35
Diff.		-57.0153
Std.Dv. Diff.		153.9655
t		-2.1908
df		34
p		0.035419
Confidence -95.00%		-109.904
Confidence 95.00%		-4.12627

10.3 Eurofins PCB_7 (ng/g fresh weight) vs NIFES PCB_7 (Upper Bound)



T-test for Dependent Samples are significant at $p < .05000$		
Variable	Eurofins PCB_7 ng/g fresh weight	NIFES Sum PCB_7 (UB) Back, ppb
Mean	141.5596	205.9429
Std.Dv.	117.5673	214.1782
N		35
Diff.		-64.3833
Std.Dv. Diff.		155.3932
t		-2.45118
df		34
p		0.019534
Confidence -95.00%		-117.763
Confidence 95.00%		-11.0038

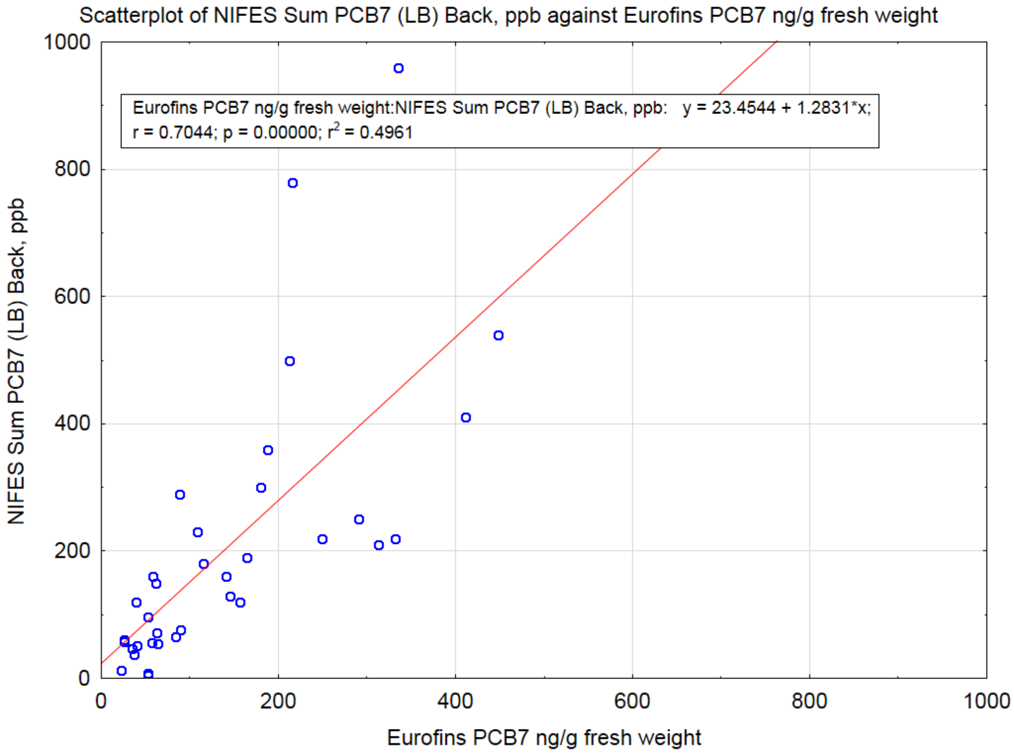
10.4 Eurofins PCB_7 + PCB 163 + PCB 164 (ng/g fresh weight) vs NIFES PCB_7 (Upper Bound)



T-test for Dependent Samples are significant at p < .05000		
Variable	Eurofins PCB_7 + 163 + 164 ng/g fresh weight	NIFES Sum PCB_7 (UB) Back, ppb
Mean	148.0705	205.9429
Std.Dv.	122.7271	214.1782
N		35
Diff.		-57.8724
Std.Dv. Diff.		153.7436
t		-2.22694
df		34
p		0.032679
Confidence -95.00%		-110.685
Confidence 95.00%		-5.05962

11 Appendix VII: Regressions, correlations and scatterplots of PCB_7

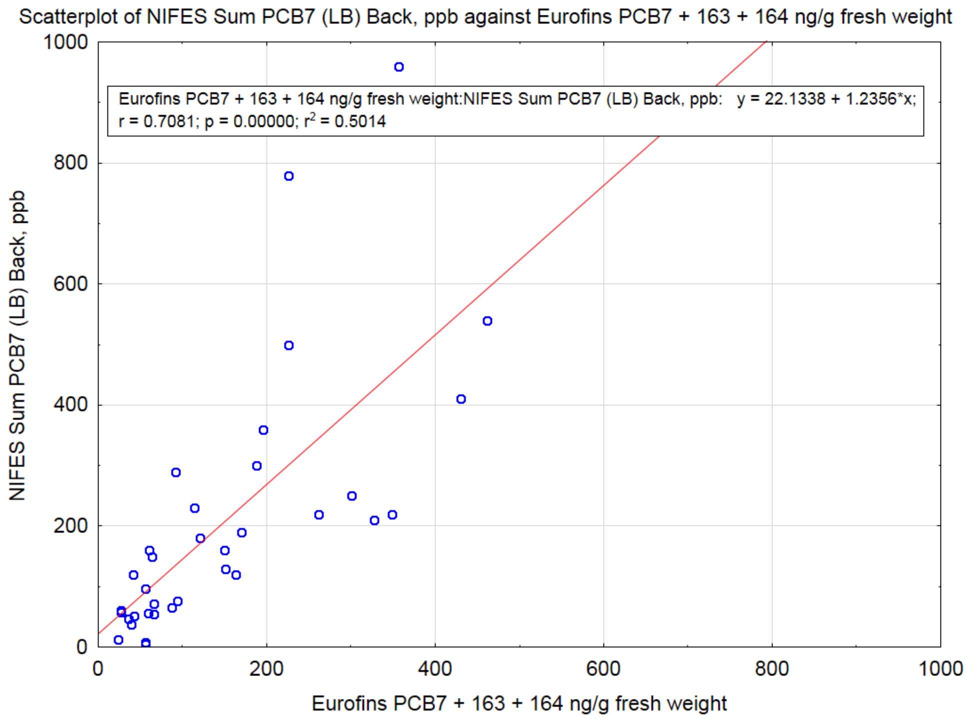
11.1 NIFES PCB_7 (Lower Bound) vs Eurofins PCB_7



N=35	Regression Summary for Dependent Variable: NIFES Sum PCB_7 (LB) Back, ppb R= .70437433 R^2= .49614319 Adjusted R^2= .48087480 F(1,33)=32.495 p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			23.45439	41.17638	0.569608	0.572801
Eurofins PCB_7 ng/g fresh weight	0.704374	0.123565	1.28307	0.22508	5.700421	0.000002

Statistic	Summary Statistics; DV: NIFES Sum PCB_7 (LB) Back, ppb
	Value
Multiple R	0.704374327
Multiple R^2	0.496143193
Adjusted R^2	0.480874805
F(1,33)	32.4947984
p	0.00000233248306
Std.Err. of Estimate	154.301607

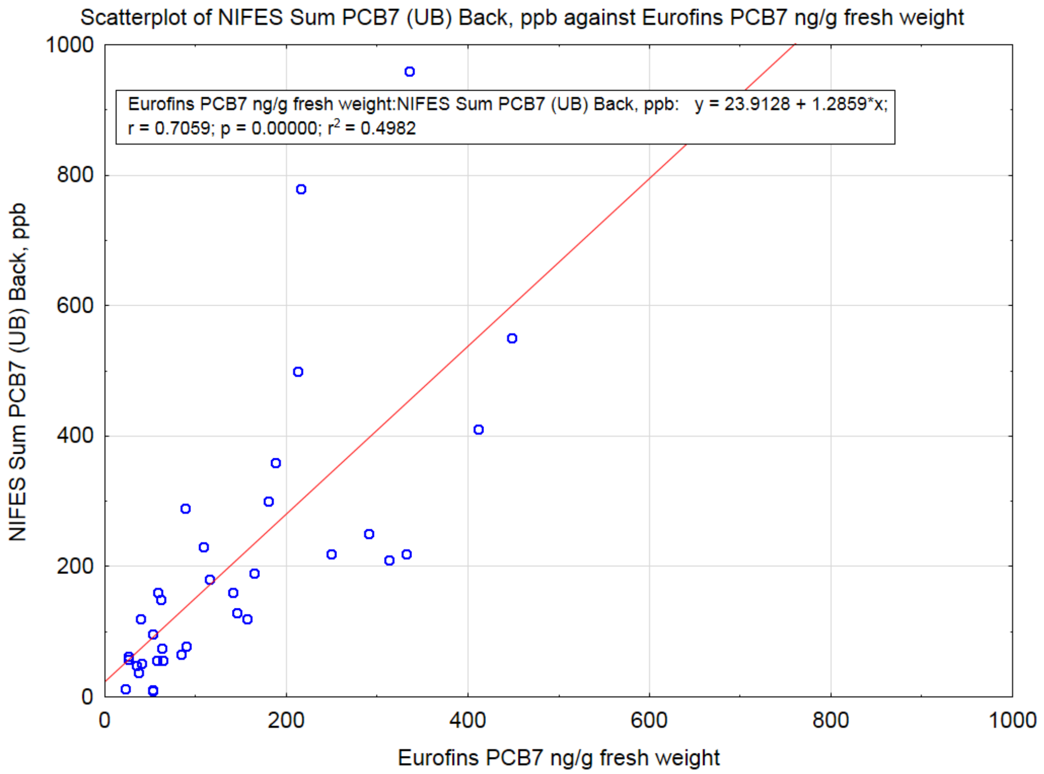
11.2 NIFES PCB_7 (Lower Bound) vs Eurofins PCB_7 + PCB163+ PCB164



N=35	Regression Summary for Dependent Variable: NIFES Sum PCB_7 (LB) Back, ppb $R= .70806714$ $R^2= .50135907$ Adjusted $R^2= .48624874$ $F(1,33)=33.180$ p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			22.13382	41.01220	0.539689	0.593035
Eurofins PCB_7 + 163 + 164 ng/g fresh weight	0.708067	0.122924	1.23557	0.21450	5.760198	0.000002

Statistic	Summary Statistics; DV: NIFES Sum PCB_7 (LB) Back, ppb
	Value
Multiple R	0.708067136
Multiple R^2	0.501359068
Adjusted R^2	0.486248737
F(1,33)	33.179886
p	0.00000195528992
Std.Err. of Estimate	153.500872

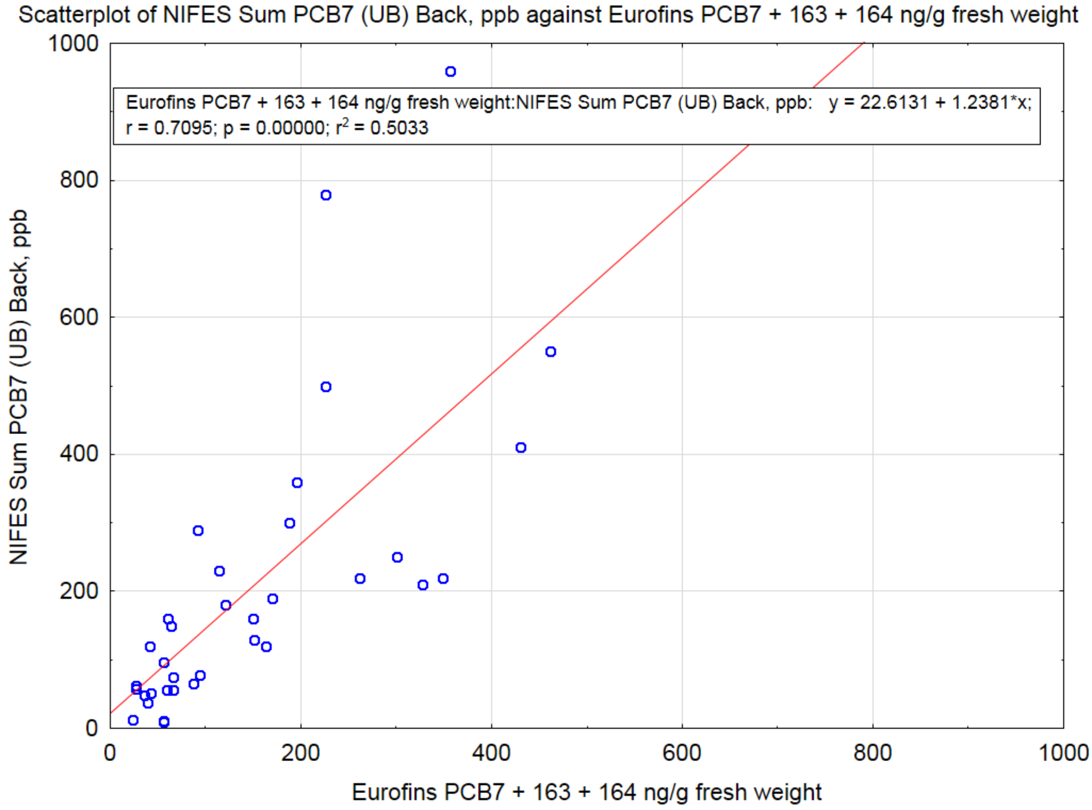
11.3 NIFES PCB_7 (Upper Bound) vs Eurofins PCB_7



	Regression Summary for Dependent Variable: NIFES Sum PCB_7 (UB) Back, ppb R= .70585415 R²= .49823008 Adjusted R²= .48302493 F(1,33)=32.767 p					
N=35	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			23.91283	41.09488	0.581893	0.564593
Eurofins PCB_7 ng/g fresh weight	0.705854	0.123309	1.28589	0.22464	5.724264	0.000002

	Summary Statistics; DV: NIFES Sum PCB_7 (UB) Back, ppb
Statistic	Value
Multiple R	0.705854151
Multiple R²	0.498230082
Adjusted R²	0.483024933
F(1,33)	32.7671949
p	0.00000217399247
Std.Err. of Estimate	153.996203

11.4 NIFES PCB_7 (Upper Bound) vs Eurofins PCB_7 + PCB163+ PCB164



N=35	Regression Summary for Dependent Variable: NIFES Sum PCB_7 (UB) Back, ppb R= .70946303 R²= .50333779 Adjusted R²= .48828742 F(1,33)=33.444 p					
	b*	Std.Err.of b*	b	Std.Err.of b	t(33)	p-value
Intercept			22.61305	40.93459	0.552419	0.584384
Eurofins PCB_7 + 163 + 164 ng/g fresh weight	0.709463	0.122680	1.23813	0.21410	5.783040	0.000002

Statistic	Summary Statistics; DV: NIFES Sum PCB_7 (UB) Back, ppb
	Value
Multiple R	0.709463032
Multiple R²	0.503337794
Adjusted R²	0.488287424
F(1,33)	33.4435498
p	0.00000182788858
Std.Err. of Estimate	153.210405