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Relationship between PCB_7 and total PCB in Norwegian minke whale (*Balaenoptera acutorostrata*)

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Summary

Data on minke whale and fin whale from Japan, Norway, and Iceland all show very good correlations between PCB_7 and total PCBs. From these relationships it is possible to infer a maximum limit for PCB_7 that is equivalent to the total PCB maximum limit of 0.5 mg/kg fresh weight in Japan.

The maximum limit for PCB_7 should take into account all the information and data available for PCB_7 and total PCB for Icelandic fin whale, Norwegian minke whale, and Japanese minke whale and be based on international agreements and harmonized international standards developed to protect consumers health and promote fair practices in food trade.

1. Introduction

This report summarizes the work carried out following decisions at the trilateral expert meeting held between Norway, Iceland and Japan on 18th march, 2016 [1]. Based on the previous reports and data provided by Norway and Iceland and as a result of the discussions, it was agreed that Norway and Iceland would perform further statistical analysis on already undertaken PCB analyses including further elaborations on fat base/wet base[1] –upper/lower as specified in the Record of Discussions (ROD). This includes:

1) Statistical analysis data of ratio of PCB_7 and total-PCB (%PCB_7) including its basic statistic data.

2) Statistical analysis data of prediction interval (95%, 99%)

3) Norway also agreed to arrange a collaborative examination among Norway (NIFES), Iceland (Innovation Center Iceland) and Eurofins Europe to compare the test results of PCB_7.

2. Materials and methods

The materials and methods have previously been described in the reports of Norway and Iceland provided prior to the expert meeting in Japan on 18th march, 2016 [2]. Samples of back blubber from 20 whales were used for the interlaboratory comparison. The samples were homogenized in Iceland at the Innovation Center Iceland using the microtome method previously developed for whale blubber [3]. Results were evaluated according to ISO 13528:2005 “Statistical methods for use in proficiency testing by interlaboratory comparison” [4]. The assigned value was determined using the consensus value from participants. Due to the low number of participants in the interlaboratory comparison the standard uncertainty was determined from the general model for the reproducibility of analytical methods given by Horwitz [5-7]. Calculation of performance statistics were done using z-scores. Statistical analyses were done using Microsoft Excel 2013 and Statsoft Statistica Version 12.

3. Results and discussion

[1] The wording wet base, wet weight (ww) and fresh weight is used interchangeably in this report, and their meaning is equivalent.

3.1 Statistical analysis data of ratio PCB_7 and total-PCB including its basic statistic data

An overview of the data on fat base and wet base are given below in table 1 and table 2, respectively. Both upperbound (UB), mediumbound (MB) and lowerbound (LB) results are shown.

Table 1: Overview of data on fat base

			Fat base, ng/g				%PCB_7 of T-PCB		
ID	JNR	Boat	T-PCB, UB	T-PCB, MB	T-PCB, LB	PCB_7, UB	UB	MB	LB
14	2015-914	Kato	2830	2830	2830	1453	51.4	51.4	51.4
17	2015-921	Kato	409	406	403	174	42.5	42.8	43.1
18	2015-922	Kato	1360	1350	1350	685	50.4	50.7	50.7
21	2015-929	Kato	736	733	730	328	44.5	44.7	44.9
22	2015-930	Kato	1100	1100	1090	449	40.8	40.8	41.2
23	2015-931	Kato	1190	1190	1180	549	46.1	46.1	46.5
24	2015-933	Kato	629	627	624	273	43.4	43.5	43.7
25	2015-935	Kato	1360	1360	1360	690	50.8	50.8	50.8
26	2015-937	Kato	871	868	865	408	46.9	47.1	47.2
27	2015-938	Kato	1080	1080	1070	518	48.0	48.0	48.4
28	2015-940	Kato	2840	2840	2830	1483	52.2	52.2	52.4
29	2015-941	Kato	543	541	538	235	43.3	43.5	43.7
30	2015-943	Kato	1400	1400	1400	686	49.0	49.0	49.0
31	2015-945	Kato	1280	1280	1270	527	41.2	41.2	41.5
33	2015-946	Kato	598	595	592	295	49.3	49.5	49.8
34	2015-947	Kato	1370	1370	1360	727	53.0	53.0	53.4
35	2015-948	Kato	804	801	798	345	42.9	43.1	43.2
42	2015-949	Kato	1120	1120	1120	503	44.9	44.9	44.9
44	2015-951	Kato	1110	1110	1110	514	46.3	46.3	46.3
47	2015-954	Kato	462	459	456	195	42.1	42.4	42.7
50	2015-957	Kato	1490	1480	1480	699	46.9	47.2	47.2
51	2015-958	Kato	540	537	535	239	44.2	44.4	44.6
53	2015-960	Kato	1200	1200	1190	565	47.1	47.1	47.5
54	2015-961	Kato	2420	2420	2420	1143	47.2	47.2	47.2
55	2015-963	Kato	1320	1320	1310	617	46.7	46.7	47.1
56	2015-965	Kato	371	368	366	157	42.3	42.6	42.9
57	2015-966	Kato	379	376	373	179	47.2	47.6	48.0
59	2015-968	Kato	2780	2780	2780	1399	50.3	50.3	50.3
60	2015-969	Kato	513	510	507	237	46.2	46.5	46.8
41	2015-982	Fiskebank 1	1820	1820	1820	861	47.3	47.3	47.3
42	2015-983	Fiskebank 1	1790	1790	1790	855	47.8	47.8	47.8
43	2015-984	Fiskebank 1	1510	1500	1500	817	54.1	54.5	54.5
44*	2015-985	Fiskebank 1	2050	2040	2040	1255	61.2	61.5	61.5
45	2015-986	Fiskebank 1	949	947	945	508	53.6	53.7	53.8
46†	2015-987	Fiskebank 1	4100	4100	4100	2179	53.2	53.2	53.2

* = Grubb's test outlier with respect to %PCB_7, † = Grubb's test outlier with respect to PCB concentration

Table 2: Overview of data on wet base

			Wet base, ng/g				%PCB7 of T-PCB		
ID	**JNR**	**Boat**	**T-PCB, UB**	**T-PCB, MB**	**T-PCB, LB**	**PCB_7, UB**	**UB**	**MB**	**LB**
14	2015-914	Kato	365	365	365	188	51.4	51.4	51.4
17	2015-921	Kato	124	123	123	53	42.5	42.9	42.9
18	2015-922	Kato	311	309	309	157	50.4	50.8	50.8
21	2015-929	Kato	89	89	88	40	44.5	44.7	44.9
22	2015-930	Kato	64	64	63	26	40.8	40.8	41.2
23	2015-931	Kato	133	133	132	61	46.2	46.2	46.5
24	2015-933	Kato	86	86	86	37	43.3	43.5	43.7
25	2015-935	Kato	355	355	355	180	50.7	50.7	50.7
26	2015-937	Kato	246	245	244	115	46.9	47.1	47.3
27	2015-938	Kato	132	132	131	63	47.9	47.9	48.3
28	2015-940	Kato	787	787	784	411	52.2	52.2	52.4
29	2015-941	Kato	144	144	143	63	43.5	43.5	43.8
30	2015-943	Kato	678	678	678	332	48.9	48.9	48.9
31	2015-945	Kato	218	218	216	90	41.1	41.1	41.5
33	2015-946	Kato	294	293	291	145	49.3	49.5	49.8
34	2015-947	Kato	547	547	543	290	53.0	53.0	53.4
35	2015-948	Kato	195	194	193	83	42.8	43.0	43.3
42	2015-949	Kato	118	118	118	53	44.9	44.9	44.9
44	2015-951	Kato	114	114	114	53	46.4	46.4	46.4
47	2015-954	Kato	96	96	95	40	42.0	42.3	42.6
50	2015-957	Kato	532	528	528	250	46.9	47.3	47.3
51	2015-958	Kato	51	51	51	23	44.2	44.5	44.7
53	2015-960	Kato	230	230	228	108	47.1	47.1	47.5
54	2015-961	Kato	457	457	457	216	47.3	47.3	47.3
55	2015-963	Kato	124	124	123	58	46.8	46.8	47.1
56	2015-965	Kato	61	60	60	26	42.3	42.6	42.8
57	2015-966	Kato	73	72	72	34	47.2	47.6	48.0
59	2015-968	Kato	175	175	175	88	50.4	50.4	50.4
60	2015-969	Kato	123	122	121	57	46.1	46.5	46.8
41	2015-982	Fiskebank 1	450	450	450	213	47.3	47.3	47.3
42	2015-983	Fiskebank 1	295	295	295	141	47.8	47.8	47.8
43	2015-984	Fiskebank 1	578	575	575	312	54.1	54.3	54.3
44*	2015-985	Fiskebank 1	734	730	730	449	61.1	61.5	61.5
45	2015-986	Fiskebank 1	307	307	306	165	53.6	53.6	53.8
46	2015-987	Fiskebank 1	631	631	631	336	53.2	53.2	53.2

* = Grubb's test outlier with respect to %PCB_7

3.1.1 Outliers

Outliers, also called anomalous, contaminated, rogue, spurious, unusual or wild observations, may be due to a number of different reasons such as *e.g.* chance, biological diversity, mistakes or wrong assumptions [8, 9]. Identifying single outliers with basic statistics can be done using *e.g.* Grubb's outlier test [10]. However, there is not a simple answer to whether or not identified outliers should be included or excluded in a data evaluation, and all exclusions of data should ideally be based on rules and methods established before the data was collected. The following statistics have therefore been carried out both with and without outlier(s).

3.1.2 Basic statistics of PCB and %PCB data with outlier detection

Basic statistic evaluation of the dataset is shown in table 3. Outlier detection using Grubb's test identified sample 2015-987 as an outlier for the PCB concentrations on fat base (table 1) due to much higher PCB concentrations (total PCB 4100 ng/g fat and PCB_7 2179 ng/g fat) than the rest of the samples. No outlier was detected for the PCB concentrations on wet base. Sample 2015-985 was identified as an outlier for $\%PCB_7$, with 61%, on both fat and wet base.

Table 3: Overview of basic statistic for the dataset (N = 35)

	Variable	Mean	Std.Dev.	Minimum	Maximum	Grubbs Test (G)	p-value	Identified outlier (JNR)
Fat base	T-PCB, LB	1318	844	366	4100	3.30	0.011	2015-987
	T-PCB, MB	1321	843	368	4100	3.30	0.011	2015-987
	T-PCB, UB	1324	842	371	4100	3.30	0.011	2015-987
	PCB_7, UB	650	456	157	2179	3.36	0.008	2015-987
	$\%PCB_7$ of T-PCB209 UB	47.5	4.4	40.8	61.2	3.11	0.028	2015-985
	$\%PCB_7$ of T-PCB209 MB	47.7	4.4	40.8	61.5	3.15	0.023	2015-985
	$\%PCB_7$ of T-PCB209 LB	47.8	4.3	41.2	61.5	3.16	0.022	2015-985
Wet base	T-PCB, LB	282	213	50.8	784	2.35	0.513	-
	T-PCB, MB	283	214	51.0	787	2.36	0.498	-
	T-PCB, UB	283	214	51.3	787	2.35	0.509	-
	PCB_7, UB	142	118	22.7	449	2.61	0.213	-
	$\%PCB_7$ of total PCB UB	47.5	4.4	40.8	61.1	3.09	0.030	2015-985
	$\%PCB_7$ of total PCB MB	47.7	4.4	40.8	61.5	3.14	0.023	2015-985
	$\%PCB_7$ of total PCB LB	47.8	4.3	41.2	61.5	3.15	0.023	2015-985

For the variables where outliers were detected the outlier was removed and the data analysis was repeated. The results of the repeated basic statistic evaluation is shown in table 4.

Table 4: Overview of basic statistic for the dataset after removal of identified outliers

	Variable	N	Mean	Minimum	Maximum	Std.Dev.	Grubbs Test (G)	p-value
Fat base	T-PCB, LB	34	1236	366	2830	701	2.27	0.63
	T-PCB, MB	34	1240	368	2840	701	2.28	0.61
	T-PCB, UB	34	1242	371	2840	700	2.28	0.61
	PCB_7, UB	34	605	157	1483	376	2.34	0.51
	%PCB_7 of T-PCB209 UB	34	47.1	40.8	54.1	3.7	1.86	1.00
	%PCB_7 of T-PCB209 MB	34	47.3	40.8	54.5	3.7	1.93	1.00
	%PCB_7 of T-PCB209 LB	34	47.4	41.2	54.5	3.7	1.92	1.00
Wet base	T-PCB, LB	35	282	51	784	213	2.35	0.51
	T-PCB, MB	35	283	51	787	214	2.36	0.50
	T-PCB, UB	35	283	51	787	214	2.35	0.51
	PCB_7, UB	35	142	23	449	118	2.61	0.21
	%PCB_7 of total PCB UB	34	47.1	40.8	54.1	3.8	1.84	1.00
	%PCB_7 of total PCB MB	34	47.3	40.8	54.3	3.7	1.90	1.00
	%PCB_7 of total PCB LB	34	47.4	41.2	54.3	3.7	1.88	1.00

The results showed a mean %PCB_7 of 47% with a standard deviation just below 4%. The range of individual %PCB_7 was from 41-55%.

3.2 Statistical analysis data of prediction interval (95%, 99%)

The following sections displays different linear regressions on both fat and wet base of PCB_7 UB concentrations versus total PCB on both UB, MB and LB basis. For PCB_7 only UB concentrations were used in the statistical analyses since UB is always used when comparing indicator PCB concentrations to maximum limits in Europe [11]. Regressions were first done using all data points and repeated after removal of identified outliers for %PCB_7. For the data on fat base only the linear regression plots are given. For the data on wet base a comparison with Japanese maximum limit on wet base is also shown with rounded numbers, rounded to two significant figures. Both the linear regression equations, calculated PCB_7 conversion factors which can be used to calculate the total PCB and calculated proxy PCB_7 limits at the Japanese maximum limit for total PCB (0.5 ppm (mg/kg)) are shown [12].

3.2.1 Fat weight – UB total PCB

The linear regression of PCB_7 and total PCB with prediction intervals using all data points is shown in figure 1. In figure 2 the sample outside the 99% prediction interval (JNR 2015-985), previously identified as an outlier based on %PCB_7, was removed before the linear regression analysis was repeated.

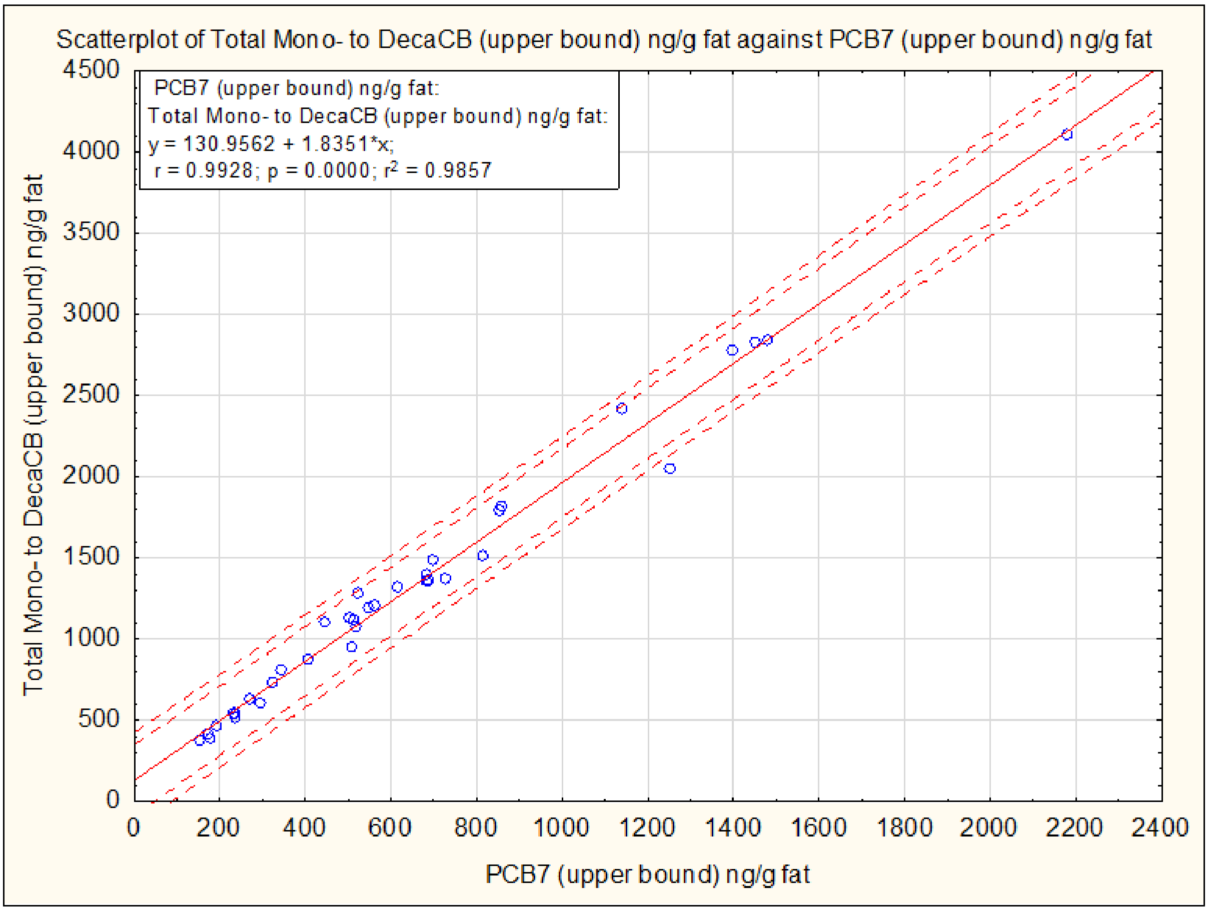

Figure 1: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

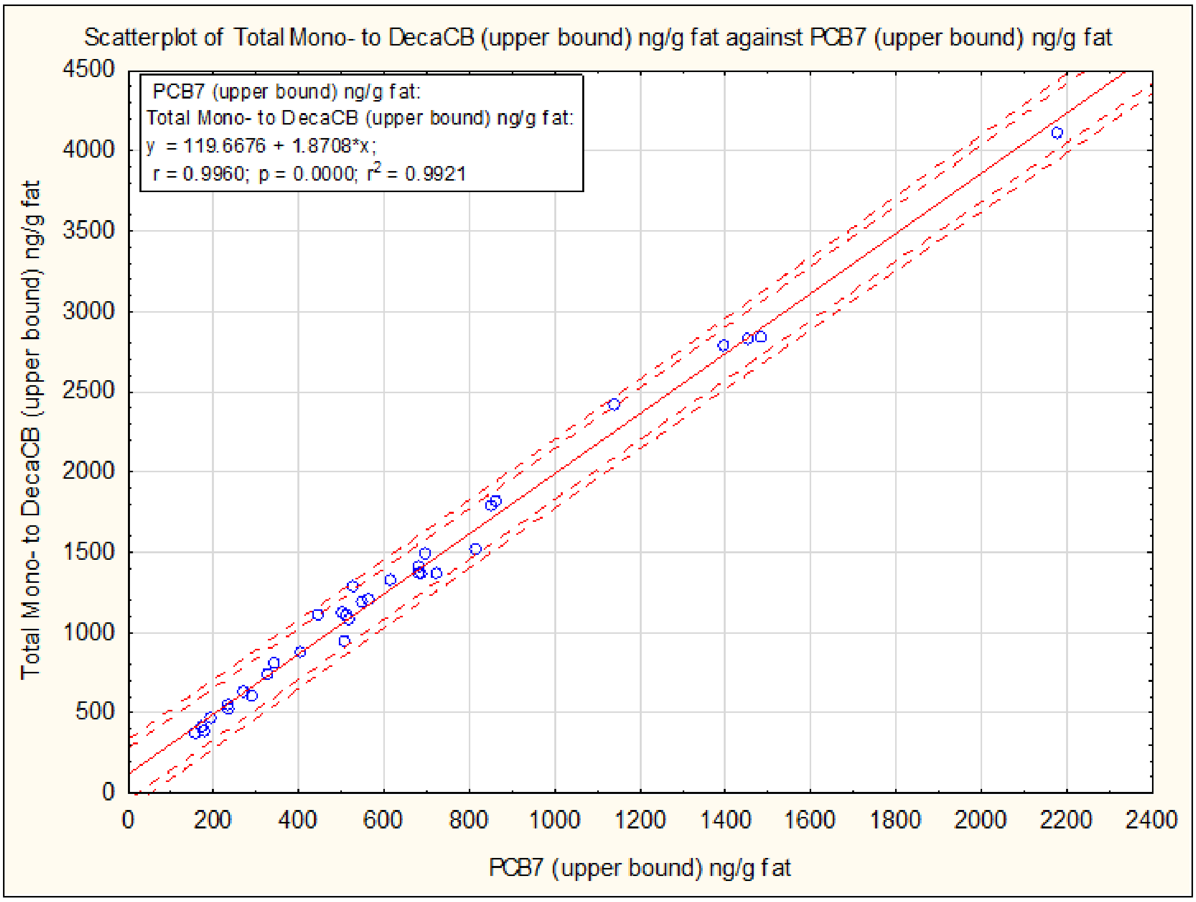

Figure 2: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

3.2.2 Fat weight – MB total PCB

The linear regression of PCB_7 and total PCB with prediction intervals using all data points is shown in figure 3. In figure 4 the sample outside the 99% prediction interval (JNR 2015-985), previously identified as an outlier based on $\%PCB_7$, was removed before the linear regression analysis was repeated.

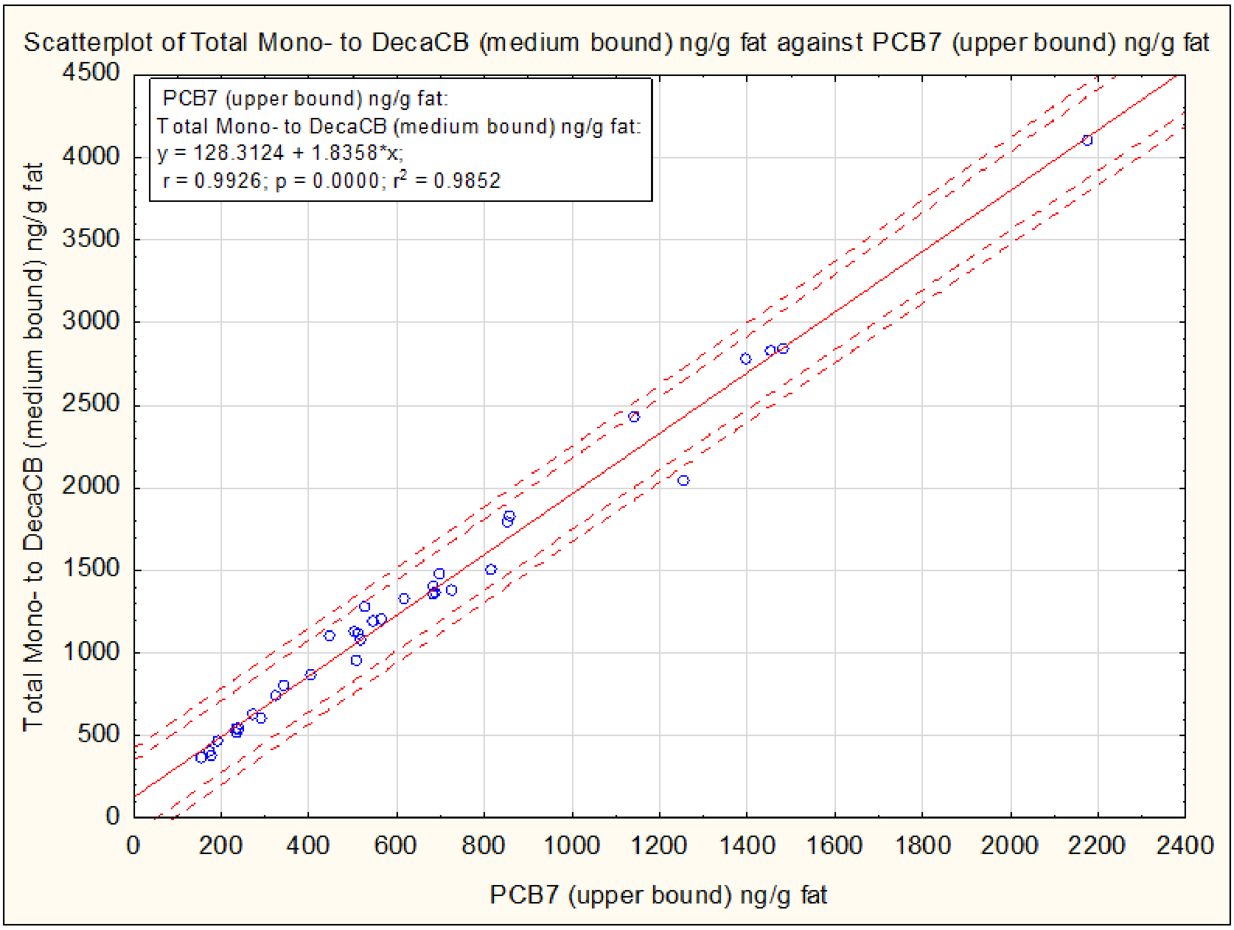

Figure 3: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

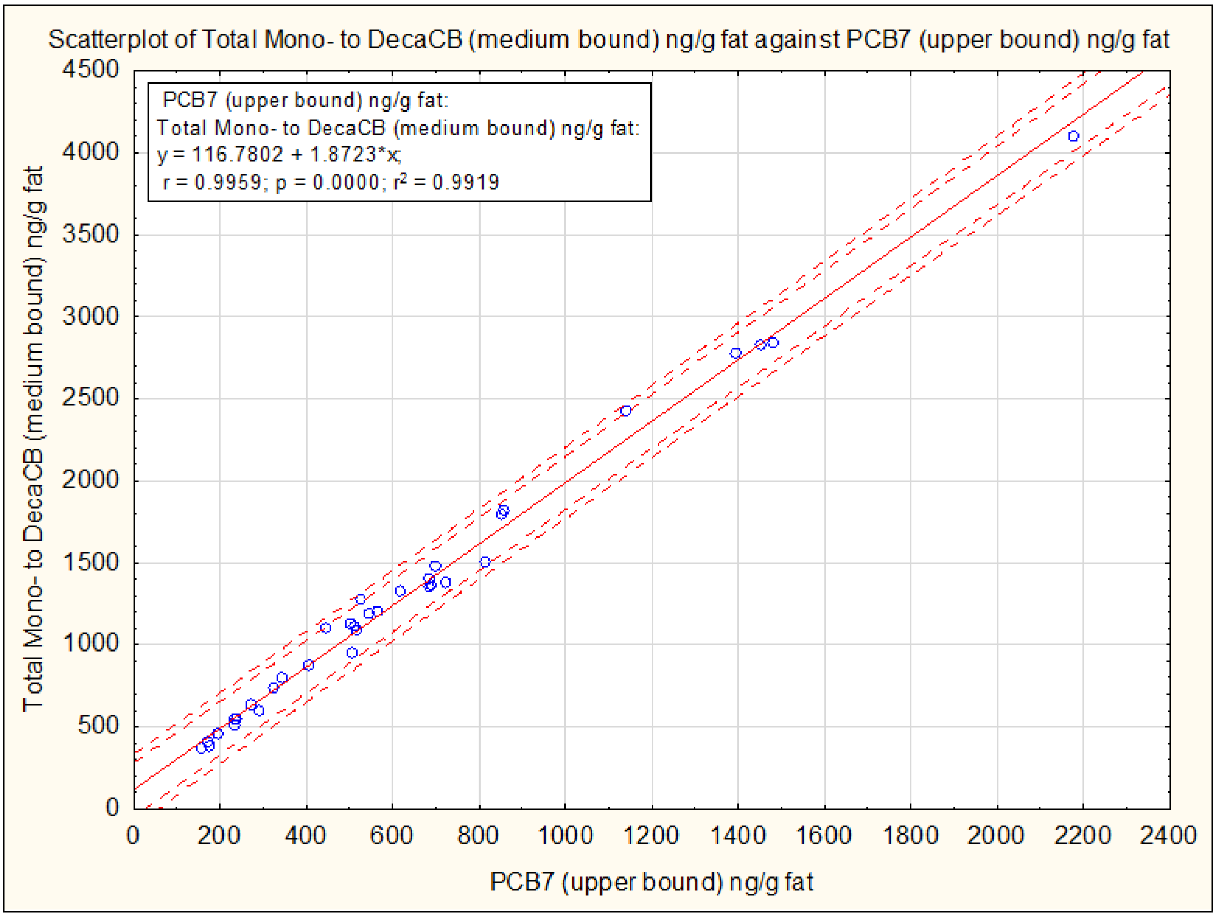

Figure 4: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

3.2.3 Fat weigh – LB total PCB

The linear regression of PCB_7 and total PCB with prediction intervals using all data points is shown in figure 5. In figure 6 the sample outside the 99% prediction interval (JNR 2015-985), previously identified as an outlier based on $\%PCB_7$, was removed before the linear regression analysis was repeated.

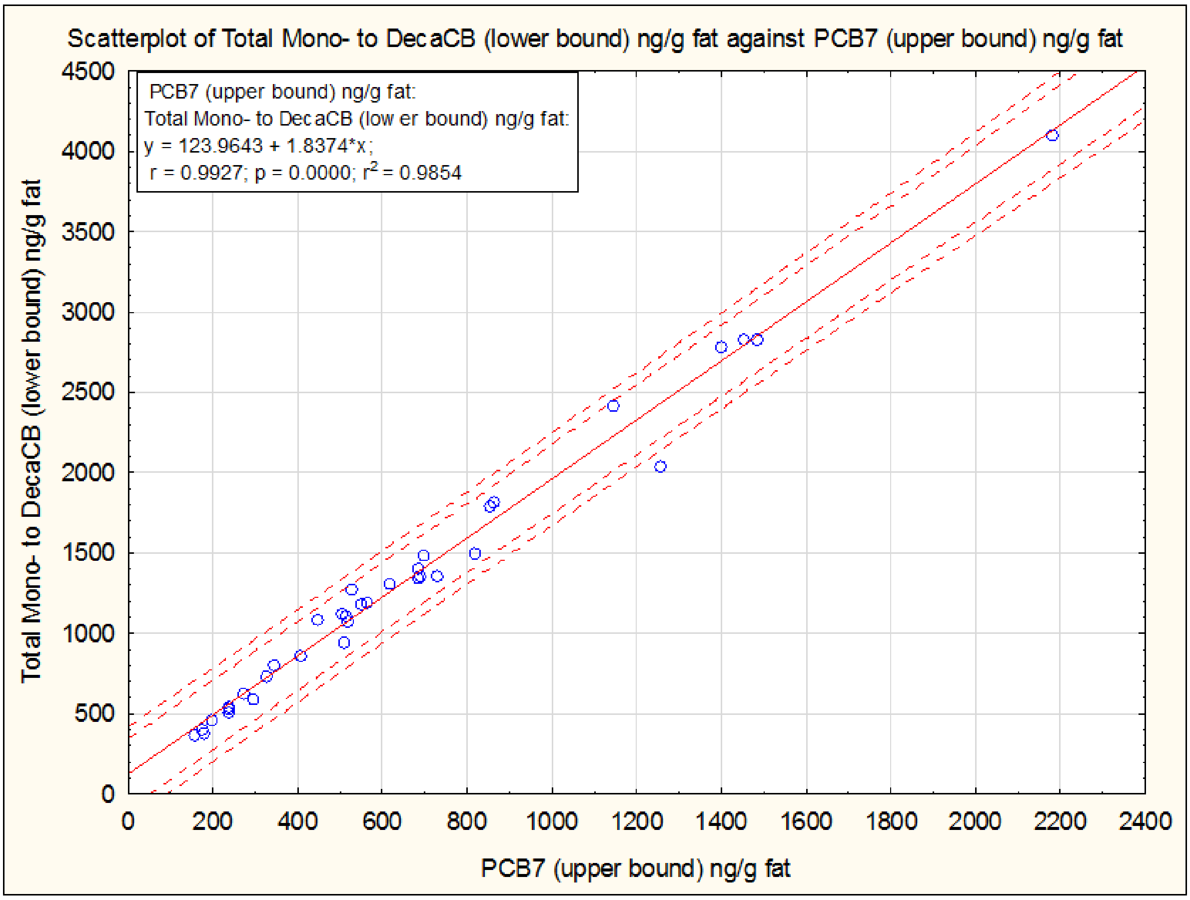

Figure 5: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

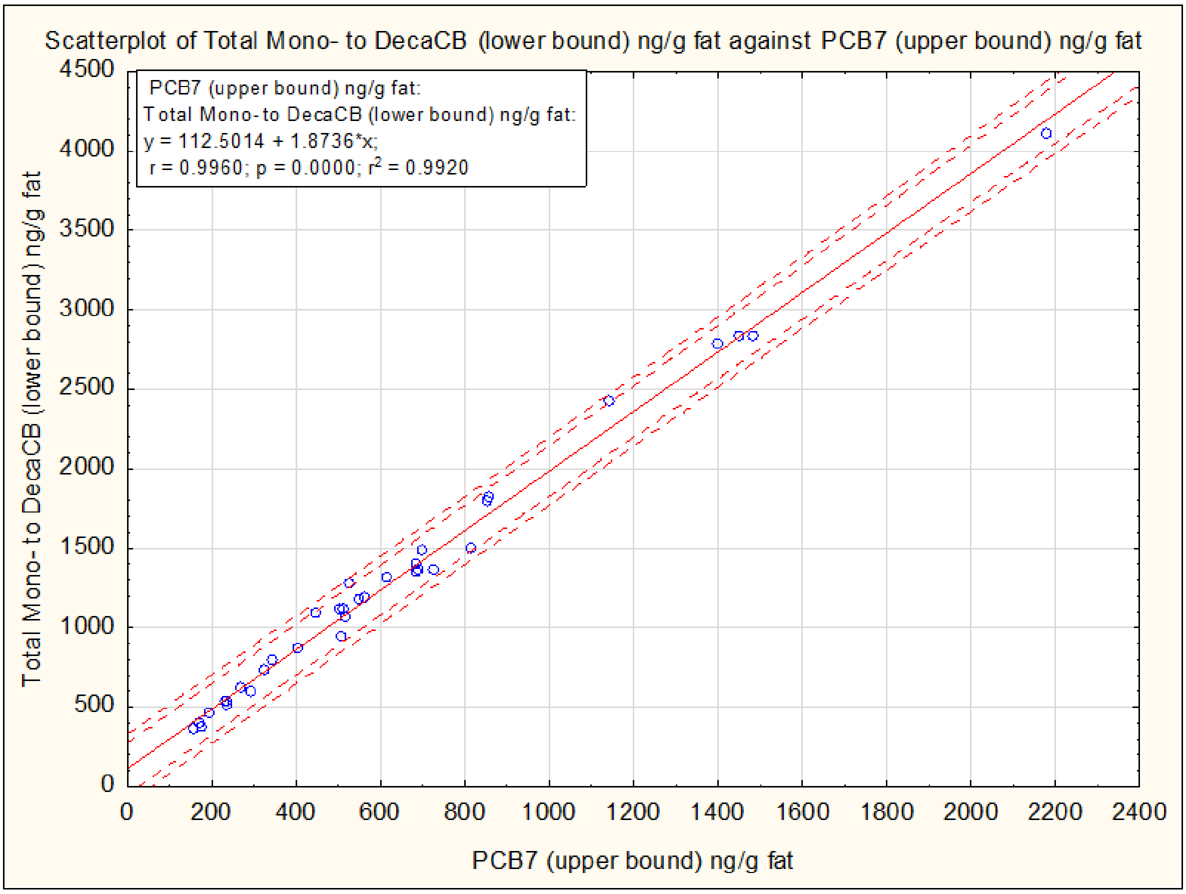

Figure 6: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

3.2.4 Wet weight – UB total PCB

The linear regression of PCB_7 and total PCB (UB) with prediction intervals is shown in figure 7 and figure 8. The data point outside the 99% prediction interval in figure 7 is sample 2015-985, previously identified as an outlier based on %PCB_7. An overview of the regression equations are shown in table 5. Predicted proxy PCB_7 maximum limits for Japanese total PCB maximum limit is shown in table 6 and figure 9. Predicted conversion factor from PCB_7 to total PCB is shown in table 7 and figure 10. Predicted proxy PCB_7 limits ranges from 0.30 to 0.22 if the outlier is included and from 0.28 to 0.23 if the outlier is excluded. Predicted PCB_7 factors ranges from 1.6 to 2.3 if the outlier is included and from 1.8 to 2.2 if the outlier is excluded.

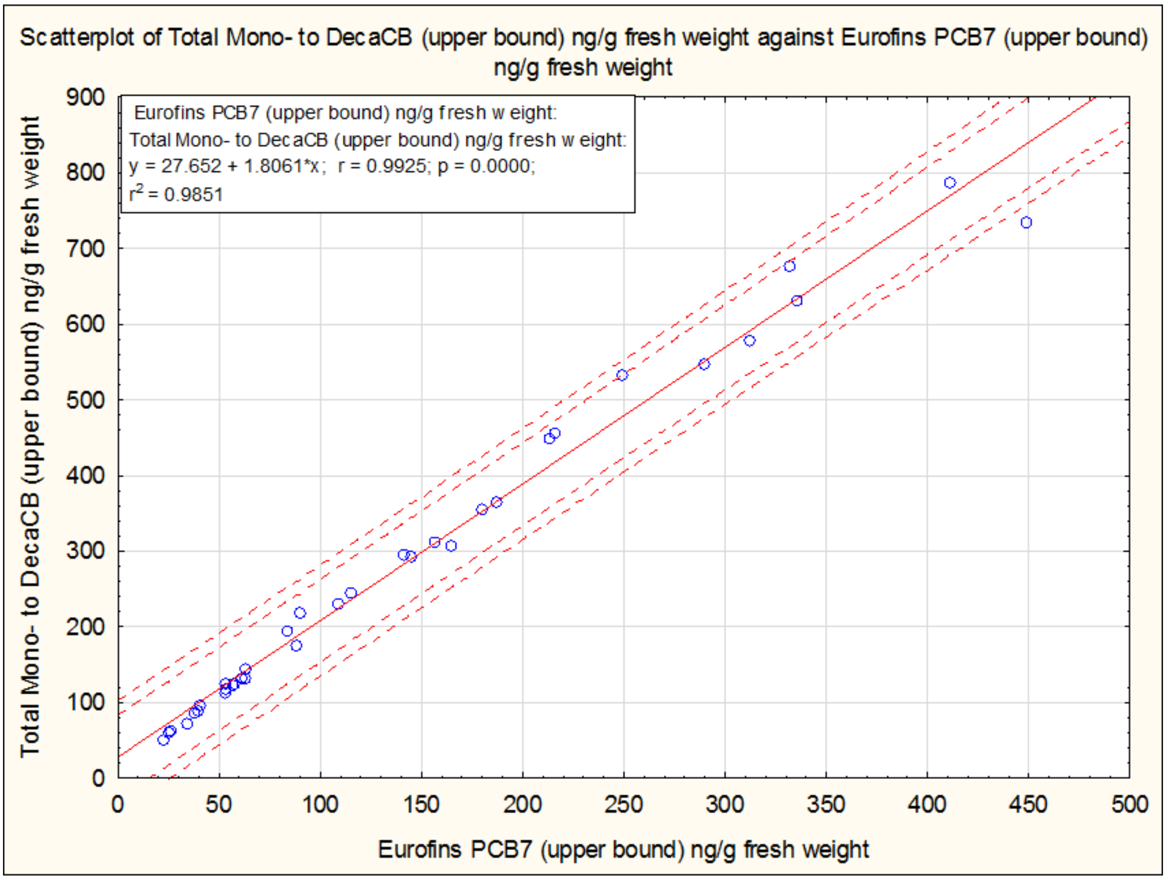

Figure 7: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

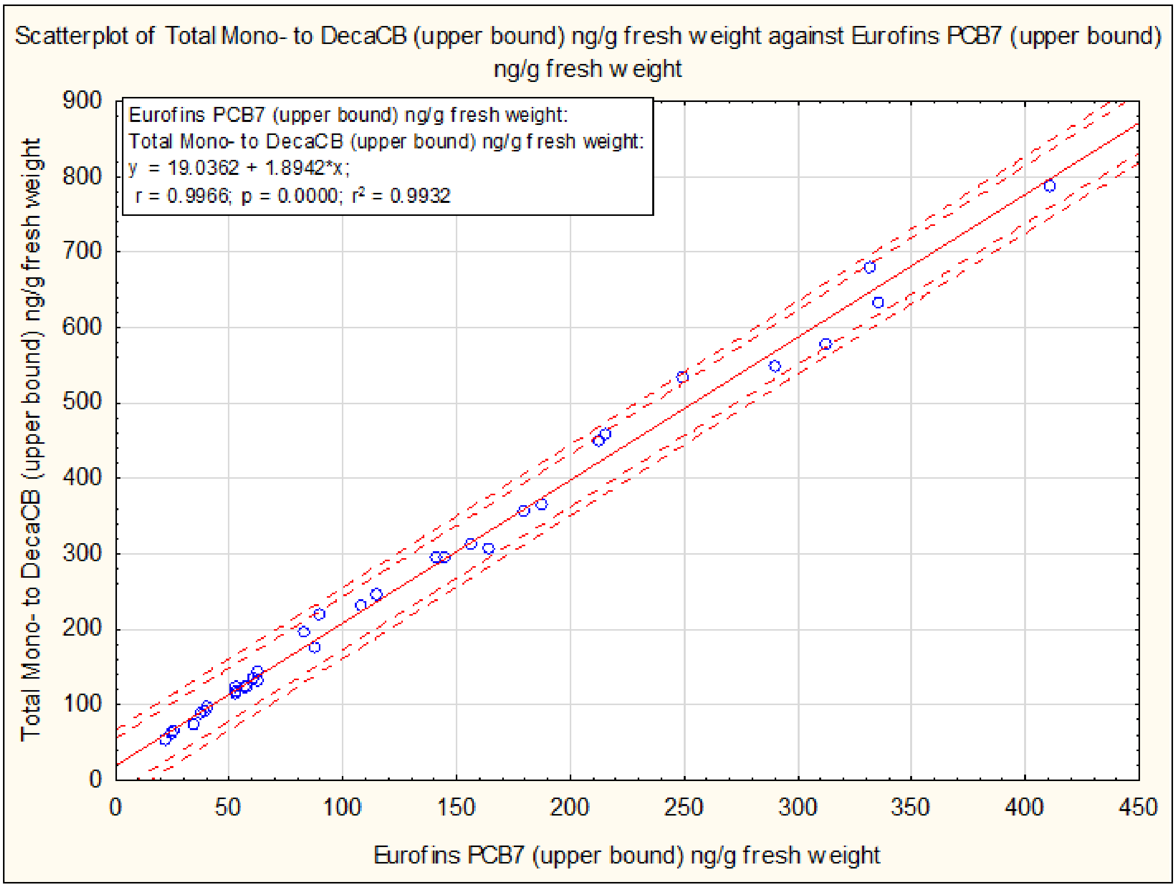

Figure 8: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

Table 5: Overview of regression equations for linear regression and prediction intervals

	UB ww	UB ww minus outlier
at -99% prediction interval	y = 1.797x - 45.419	y = 1.888x - 27.573
at -95% prediction interval	y = 1.7994x - 26.738	y = 1.8896x - 15.658
from linear regression	y = 1.8061x + 27.652	y = 1.8942x + 19.036
at +95% prediction interval	y = 1.8129x + 82.043	y = 1.8989x + 53.73
at +99% prediction interval	y = 1.8152x + 100.72	y = 1.9005x + 65.646

Table 6: Predicted proxy PCB_7 limit at Japanese limit for total PCB (0.5 mg/kg)

	UB ww	UB ww minus outlier
at -99% prediction interval	0.30	0.28
at -95% prediction interval	0.29	0.27
from linear regression	0.26	0.25
at +95% prediction interval	0.23	0.24
at +99% prediction interval	0.22	0.23

Table 7: Predicted PCB_7 factor for conversion into total PCB concentration

	UB ww	UB ww minus outlier
at -99% prediction interval	1.6	1.8
at -95% prediction interval	1.7	1.8
from linear regression	1.9	2.0
at +95% prediction interval	2.2	2.1
at +99% prediction interval	2.3	2.2

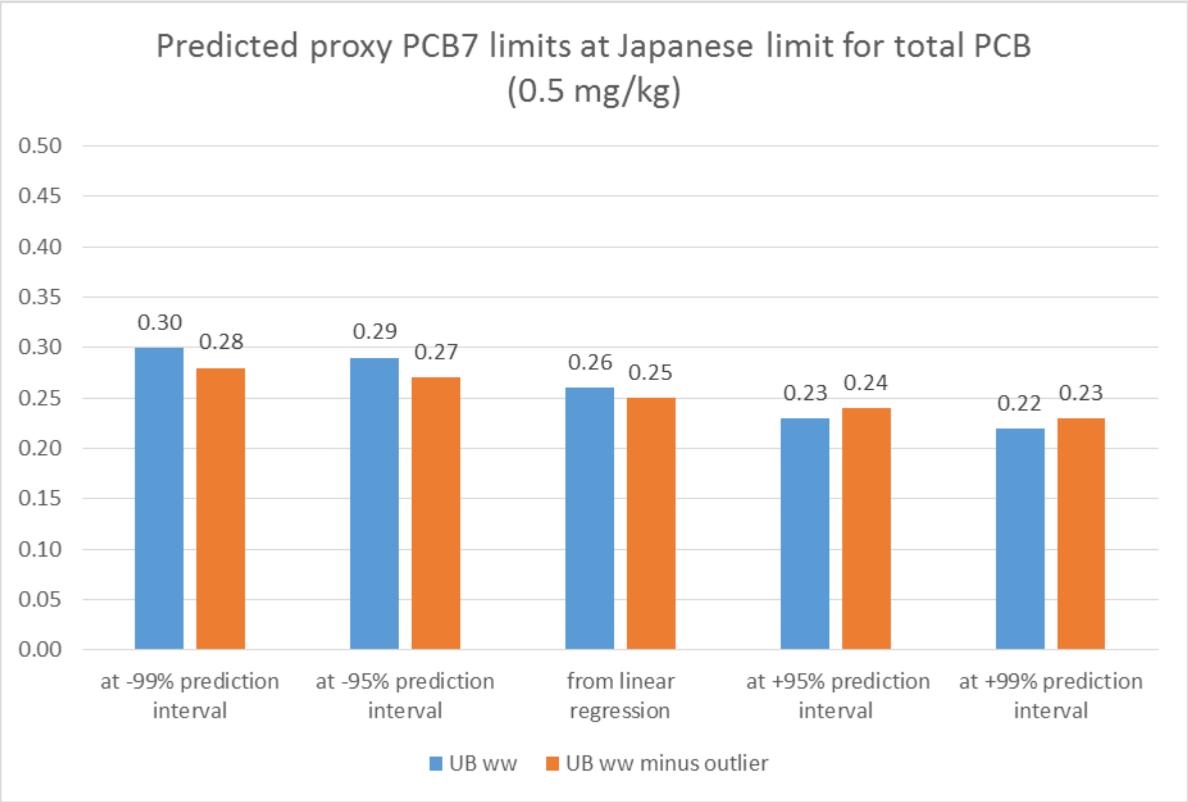

Figure 9: Predicted proxy PCB_7 limits at Japanese limit for total PCB

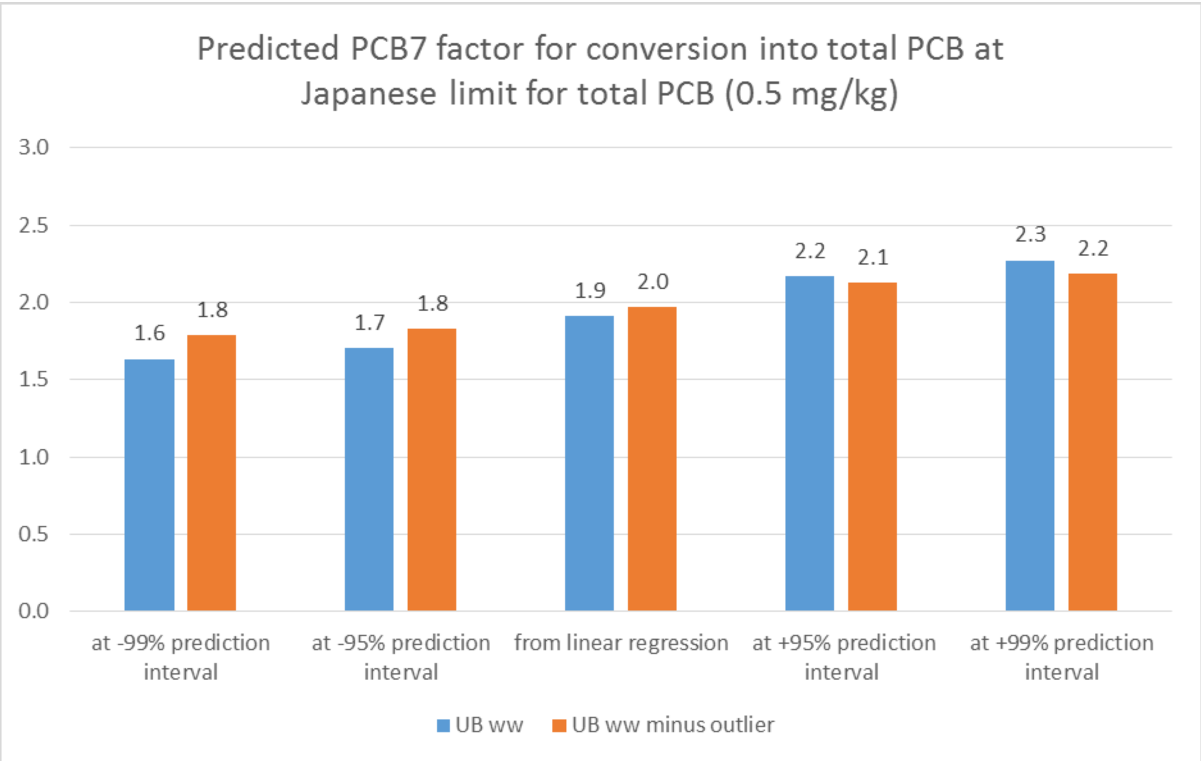

Figure 10: Predicted proxy PCB_7 factor for conversion into total PCB at Japanese limit for total PCB

3.2.5 Wet weight – MB total PCB

The linear regression of PCB_7 and total PCB (UB) with prediction intervals is shown in figure 11 and figure 12. The data point outside the 99% prediction interval in figure 11 is sample 2015-985, previously identified as an outlier based on $\%PCB_7$. An overview of the regression equations are shown in table 8. Predicted proxy PCB_7 maximum limits for Japanese total PCB maximum limit is shown in table 9 and figure 13. Predicted conversion factor from PCB_7 to total PCB is shown in table 10 and figure 14. Predicted proxy PCB_7 limits ranges from 0.30 to 0.22 if the outlier is included and from 0.29 to 0.21 if the outlier is excluded. Predicted PCB_7 factors ranges from 1.6 to 2.3 if the outlier is included and from 1.7 to 2.3 if the outlier is excluded.

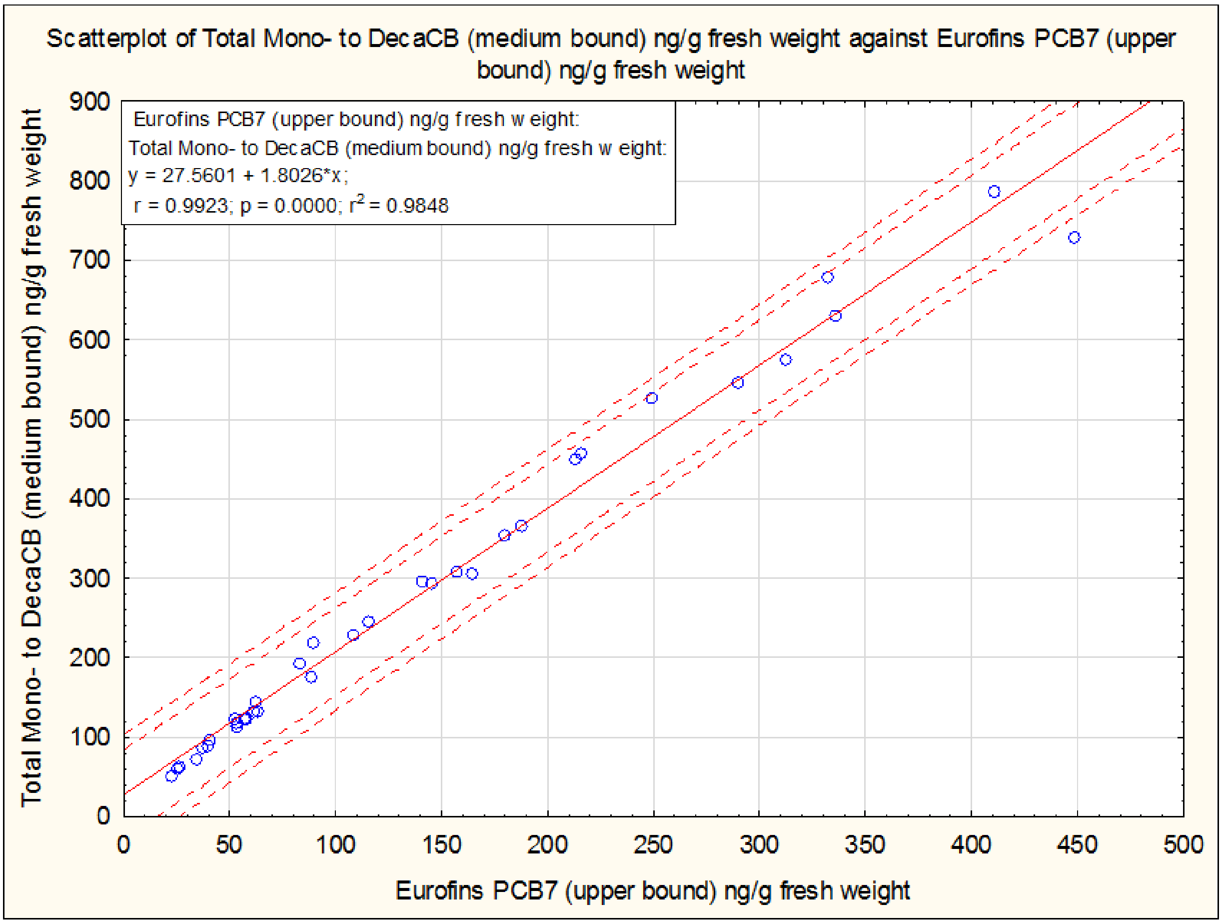

Figure 11: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

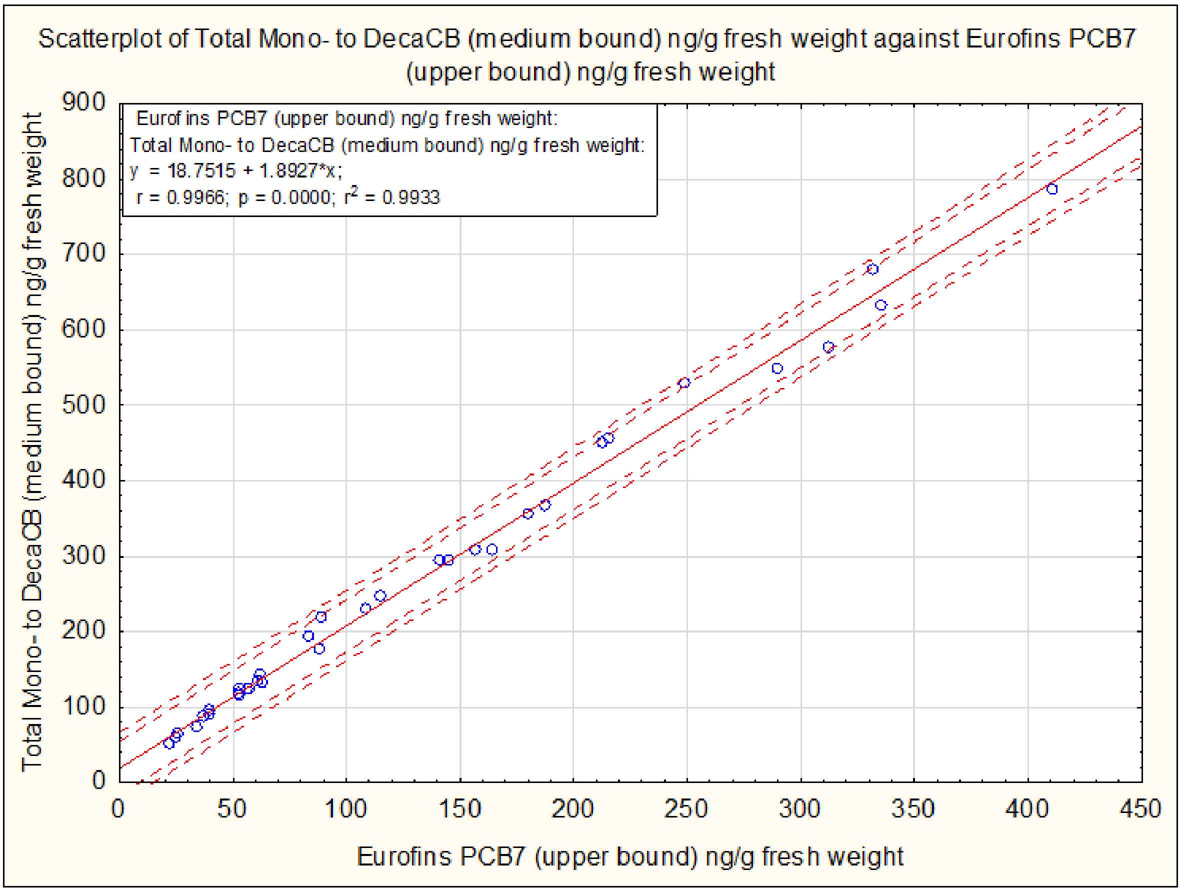

Figure 12: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

Table 8: Overview of regression equations for linear regression and prediction intervals

	MB ww	MB ww minus outlier
at -99% prediction interval	y = 1.7935x - 45.511	y = 1.8861x - 54.572
at -95% prediction interval	y = 1.7958x - 26.83	y = 1.8878x - 35.827
from linear regression	y = 1.8026x + 27.56	y = 1.8927x + 18.751
at +95% prediction interval	y = 1.8094x + 81.951	y = 1.8975x + 73.33
at +99% prediction interval	y = 1.8117x + 100.63	y = 1.8992x + 92.075

Table 9: Predicted proxy PCB_7 limit at Japanese limit for total PCB (0.5 mg/kg)

	MB ww	MB ww minus outlier
at -99% prediction interval	0.30	0.29
at -95% prediction interval	0.29	0.28
from linear regression	0.26	0.25
at +95% prediction interval	0.23	0.22
at +99% prediction interval	0.22	0.21

Table 10: Predicted PCB_7 factor for conversion into total PCB concentration

	MB ww	MB ww minus outlier
at -99% prediction interval	1.6	1.7
at -95% prediction interval	1.7	1.8
from linear regression	1.9	2.0
at +95% prediction interval	2.2	2.2
at +99% prediction interval	2.3	2.3

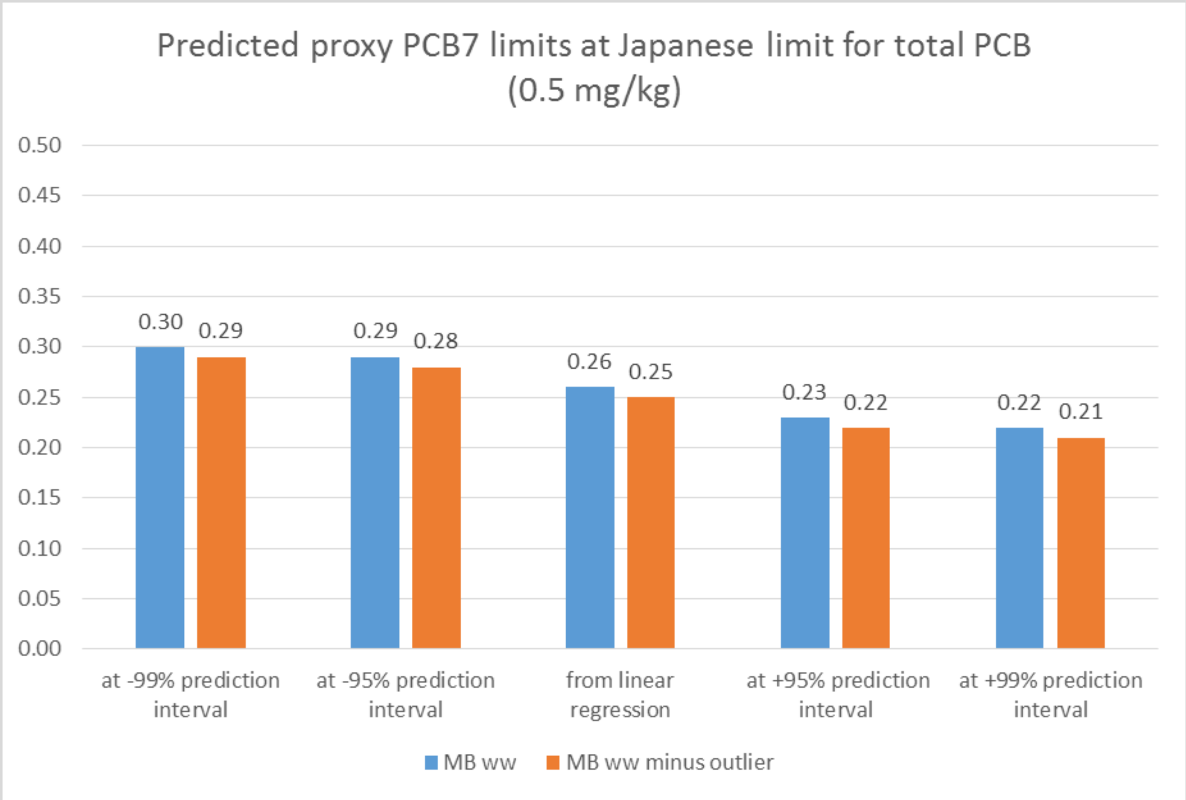

Figure 13: Predicted proxy PCB_7 limits at Japanese limit for total PCB

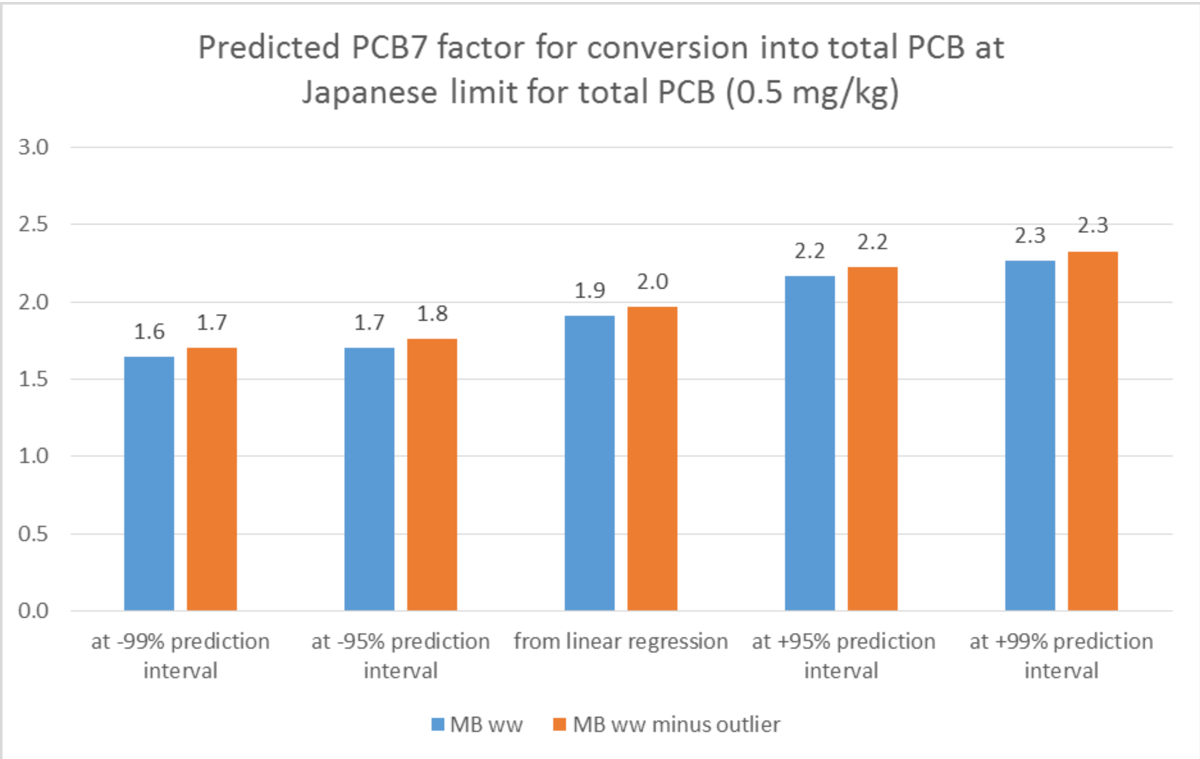

Figure 14: Predicted proxy PCB_7 factor for conversion into total PCB at Japanese limit for total PCB

3.2.6 Wet weight – LB total PCB

The linear regression of PCB_7 and total PCB (UB) with prediction intervals is shown in figure 15 and figure 16. The data point outside the 99% prediction interval in figure 15 is sample 2015-985, previously identified as an outlier based on %PCB_7. An overview of the regression equations are shown in table 11. Predicted proxy PCB_7 maximum limits for Japanese total PCB maximum limit is shown in table 12 and figure 17. Predicted conversion factor from PCB_7 to total PCB is shown in table 13 and figure 18. Predicted proxy PCB_7 limits ranges from 0.30 to 0.22 if the outlier is included and from 0.29 to 0.22 if the outlier is excluded. Predicted PCB_7 factors ranges from 1.6 to 2.3 if the outlier is included and from 1.7 to 2.3 if the outlier is excluded.

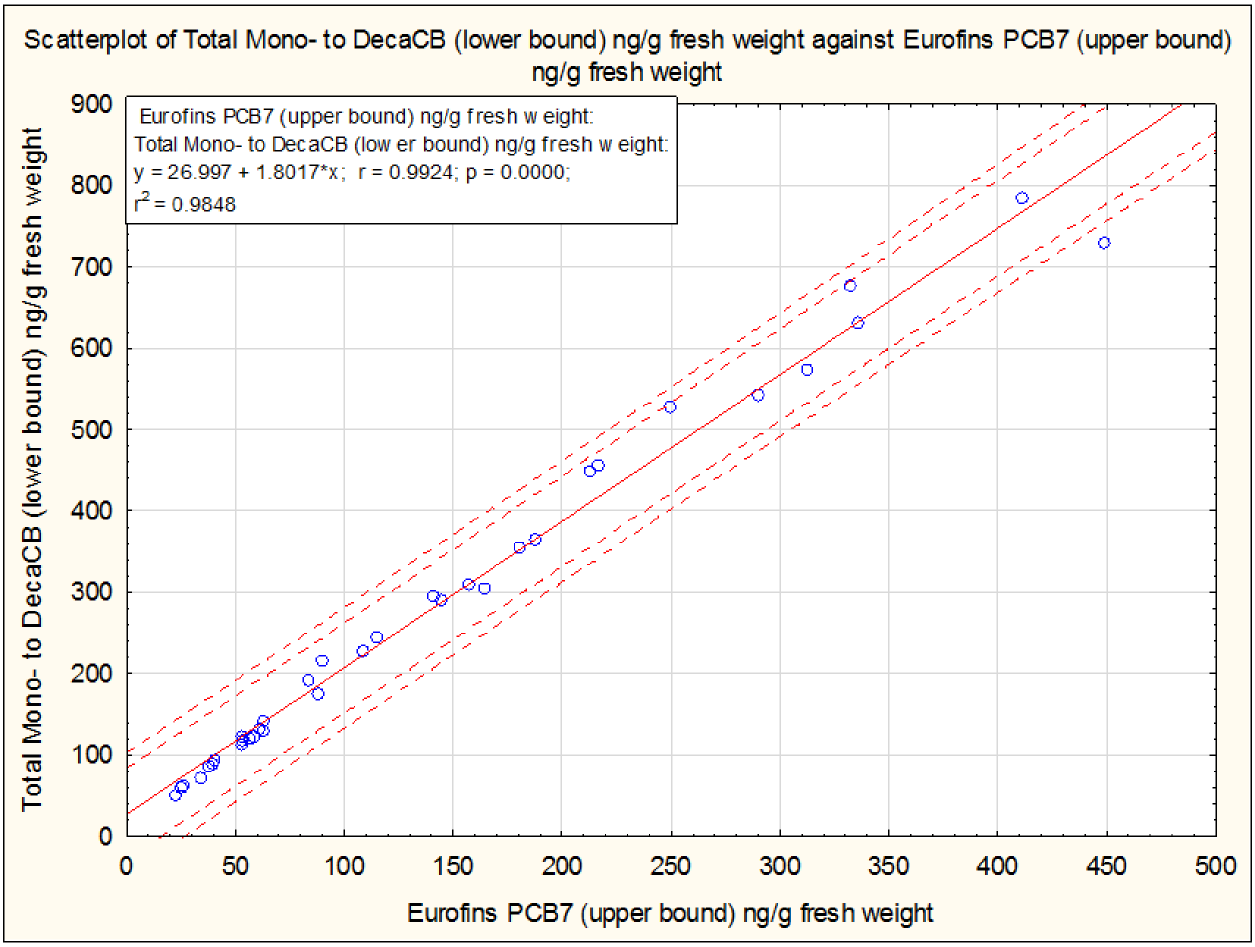

Figure 15: Linear regression analysis with 95% and 99% prediction intervals shown as dotted lines

Figure 16: Linear regression analysis after outlier removal of sample 2015-985, with 95% and 99% prediction intervals shown as dotted lines

Table 11: Overview of regression equations for linear regression and prediction intervals

	LB ww	LB ww minus outlier
at -99% prediction interval	y = 1.7926x - 46.074	y = 1.8844x - 55.055
at -95% prediction interval	y = 1.7949x - 27.393	y = 1.8861x - 36.31
from linear regression	y = 1.8017x + 26.997	y = 1.8909x + 18.269
at +95% prediction interval	y = 1.8085x + 81.388	y = 1.8958x + 72.847
at +99% prediction interval	y = 1.8108x + 100.07	y = 1.8974x + 91.592

Table 12: Predicted proxy PCB_7 limit at Japanese limit for total PCB (0.5 mg/kg)

	LB ww	LB ww minus outlier
at -99% prediction interval	0.30	0.29
at -95% prediction interval	0.29	0.28
from linear regression	0.26	0.25
at +95% prediction interval	0.23	0.23
at +99% prediction interval	0.22	0.22

Table 13: Predicted PCB_7 factor for conversion into total PCB concentration

	LB ww	LB ww minus outlier
at -99% prediction interval	1.6	1.7
at -95% prediction interval	1.7	1.8
from linear regression	1.9	2.0
at +95% prediction interval	2.2	2.2
at +99% prediction interval	2.3	2.3

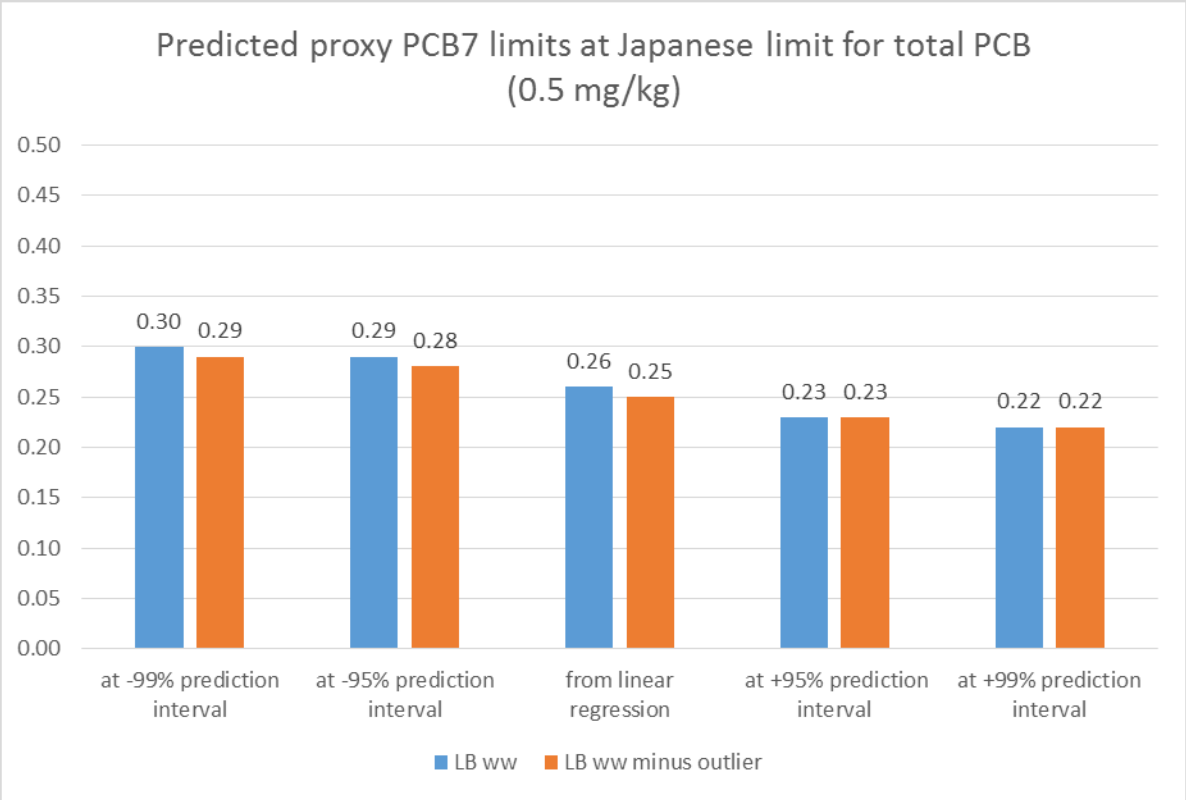

Figure 17: Predicted proxy PCB$_7$ limits at Japanese limit for total PCB

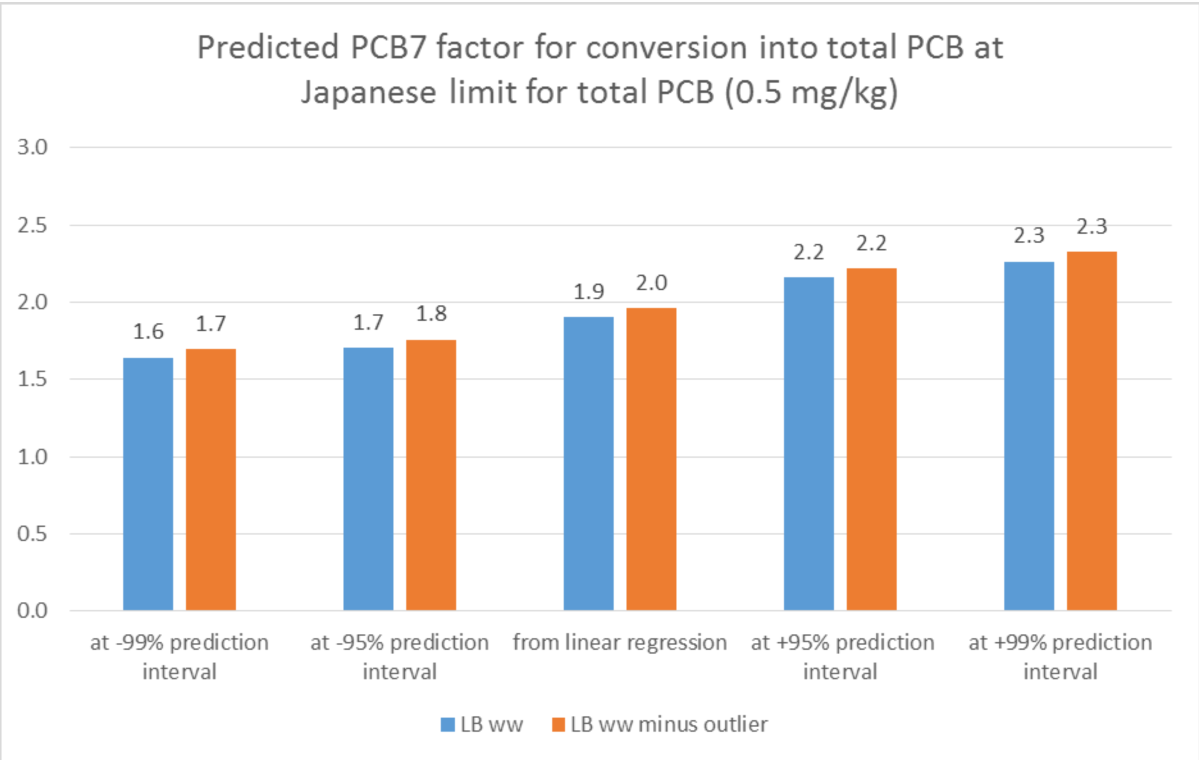

Figure 18: Predicted proxy PCB$_7$ factor for conversion into total PCB at Japanese limit for total PCB

3.3 Collaborative examination of PCB analyses among Norway (NIFES), Iceland (Innovation Center Iceland) and Eurofins Europe

The results from the interlaboratory comparison between Eurofins, Iceland and NIFES is shown in table 14.

Table 14: Results from the interlaboratory comparison

ID	JNR	Eurofins PCB_7 (UB), ng/g ww	Iceland PCB_7, ng/g ww	NIFES PCB_7 (UB), ng/g ww
14	2015-914/2	412	461	490
15	2015-917/2	372	376	460
17	2015-921/2	108	108	100
18	2015-922/2	276	262	260
19	2015-924/2	64	69	68
20	2015-926/2	414	387	360
21	2015-929/2	104	93	90
22	2015-930/2	79	77	73
23	2015-931/2	170	205	180
24	2015-933/2	91	89	83
25	2015-935/2	338	363	300
26	2015-937/2	240	244	240
27	2015-938/2	118	142	140
28	2015-940/2	454	568	470
29	2015-941/2	77	100	97
30	2015-943/2	390	453	420
31	2015-945/2	96	127	120
33	2015-946/2	141	185	180
34	2015-947/2	303	354	320
35	2015-948/2	75	75	86

The results were evaluated statistically using ISO 13528, which describes criteria that should be applied to data obtained in a proficiency test by interlaboratory comparison to see if the results give rise to action or warning signals. The results from the proficiency testing is summarized in Table 15. It is important to be aware that even in a well-run laboratory, with experienced staff, anomalous results may sometimes be obtained. According to ISO 13528 results that give rise to z-score above 3.0 or below 3.0 shall be considered to give an "action signal" and z-score above 2.0 or below 2.0 shall be considered to give "warning signal". A single "action signal", or "warning signals" in two successive rounds, shall be taken as evidence that an anomaly has occurred that requires investigation.

Results from the proficiency testing gave rise to z-scores in the range from -1.0 to + 1.2, demonstrating good agreement between the laboratories. As the histogram of the z-scores in figure 19 shows, there is good agreement on the consensus values among the laboratories with the majority of z-scores centered around 0.

Table 15: Results from the proficiency testing

JNR	Assigned value, ng/g ww	Standard deviation, ng/g ww	Eurofins result, z-score	Iceland result, z-score	NIFES result, z-score
2015-914/2	454	82	-0.52	0.09	0.44
2015-917/2	378	70	-0.08	-0.02	1.18
2015-921/2	108	24	0.01	0.02	-0.31
2015-922/2	266	52	0.20	-0.08	-0.12
2015-924/2	67	16	-0.23	0.12	0.03
2015-926/2	387	71	0.38	0.00	-0.38
2015-929/2	94	21	0.46	-0.05	-0.18
2015-930/2	76	18	0.16	0.02	-0.18
2015-931/2	184	38	-0.35	0.56	-0.10
2015-933/2	88	20	0.16	0.05	-0.25
2015-935/2	334	63	0.06	0.47	-0.53
2015-937/2	240	48	0.00	0.07	-0.01
2015-938/2	139	30	-0.70	0.08	0.02
2015-940/2	477	85	-0.27	1.08	-0.08
2015-941/2	96	22	-0.84	0.21	0.06
2015-943/2	421	77	-0.40	0.42	-0.01
2015-945/2	117	26	-0.83	0.36	0.11
2015-946/2	178	37	-1.00	0.17	0.05
2015-947/2	326	62	-0.37	0.47	-0.09
2015-948/2	75	18	-0.02	-0.01	0.59

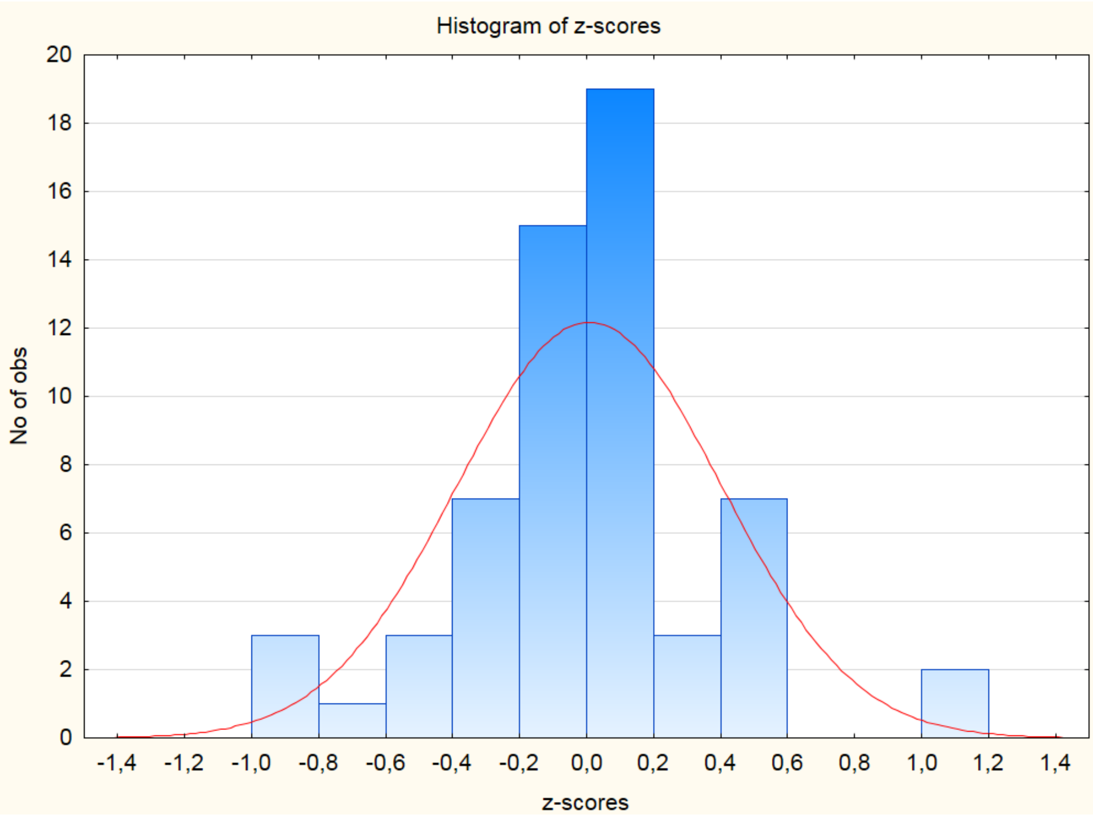

Figure 19: Distribution of z-scores from the proficiency test

3.3.1 Comparison of PCB_7 results from before and after homogenization

A summary of the new results for PCB_7 obtained after homogenization and the previously reported results using non-homogenized samples are shown in table 16.

Table 16: Results from new analyses of homogenized samples and old analyses of non-homogenized samples

ID	JNR	New Eurofins PCB_7 (UB), ng/g ww	New Iceland PCB_7, ng/g ww	New NIFES PCB_7 (UB), ng/g ww	Old NIFES PCB_7 (UB), ng/g ww	Old Eurofins PCB_7 (UB), ng/g ww
14	2015-914/2	412	461	490	360	188
15	2015-917/2	372	376	460	59	-
17	2015-921/2	108	108	100	96	53
18	2015-922/2	276	262	260	120	157
19	2015-924/2	64	69	68	31	-
20	2015-926/2	414	387	360	270	-
21	2015-929/2	104	93	90	120	40
22	2015-930/2	79	77	73	63	26
23	2015-931/2	170	205	180	150	61
24	2015-933/2	91	89	83	38	37
25	2015-935/2	338	363	300	300	180
26	2015-937/2	240	244	240	180	115
27	2015-938/2	118	142	140	56	63
28	2015-940/2	454	568	470	410	411
29	2015-941/2	77	100	97	75	63

ID	JNR	New Eurofins PCB_7 (UB), ng/g ww	New Iceland PCB_7, ng/g ww	New NIFES PCB_7 (UB), ng/g ww	Old NIFES PCB_7 (UB), ng/g ww	Old Eurofins PCB_7 (UB), ng/g ww
30	2015-943/2	390	453	420	220	332
31	2015-945/2	96	127	120	78	90
33	2015-946/2	141	185	180	130	145
34	2015-947/2	303	354	320	250	290
35	2015-948/2	75	75	86	66	83

The combined old and new results for PCB_7 were evaluated statistically using ISO 13528 as in the previous section. The results are summarized in table 17. As the histogram in figure 20 shows, results obtained for non-homogenized samples are more spread out and z-scores are typically more negative than for homogenized samples. The overall distribution of z-scores is clearly not unimodal any more, but appears to be bi or even tri modal.

Table 17:Results from statistical evaluation of old non-homogenized and new homogenized PCB_7 results

JNR	Assigned value, ng/g ww	Standard deviation, ng/g ww	New Eurofins z-score	New Iceland z-score	New NIFES z-score	Old NIFES z-score	Old Eurofins z-score
2015-914/2	404	74	0.10	0.77	1.16	-0.59	-2.92
2015-917/2	371	69	0.01	0.07	1.29	-4.53	-5.38
2015-921/2	99	22	0.39	0.40	0.05	-0.13	-2.06
2015-922/2	249	49	0.55	0.26	0.22	-2.63	-1.88
2015-924/2	65	16	-0.10	0.26	0.17	-2.18	-4.15
2015-926/2	369	69	0.67	0.27	-0.13	-1.44	-5.38
2015-929/2	94	22	0.44	-0.07	-0.20	1.19	-2.54
2015-930/2	70	17	0.52	0.37	0.16	-0.44	-2.65
2015-931/2	166	35	0.13	1.12	0.40	-0.46	-3.01
2015-933/2	78	18	0.71	0.59	0.26	-2.19	-2.23
2015-935/2	303	58	0.59	1.03	-0.06	-0.06	-2.12
2015-937/2	237	47	0.07	0.14	0.06	-1.22	-2.59
2015-938/2	106	24	0.50	1.48	1.41	-2.11	-1.81
2015-940/2	459	83	-0.06	1.33	0.14	-0.59	-0.58
2015-941/2	82	19	-0.26	0.92	0.76	-0.39	-1.03
2015-943/2	371	69	0.28	1.19	0.71	-2.19	-0.57
2015-945/2	102	23	-0.27	1.07	0.78	-1.04	-0.54
2015-946/2	154	33	-1.55	-0.23	-0.37	-0.75	-0.29
2015-947/2	304	58	-0.83	0.06	-0.53	-0.92	-0.23
2015-948/2	77	18	-0.86	-0.85	-0.26	-0.62	0.35

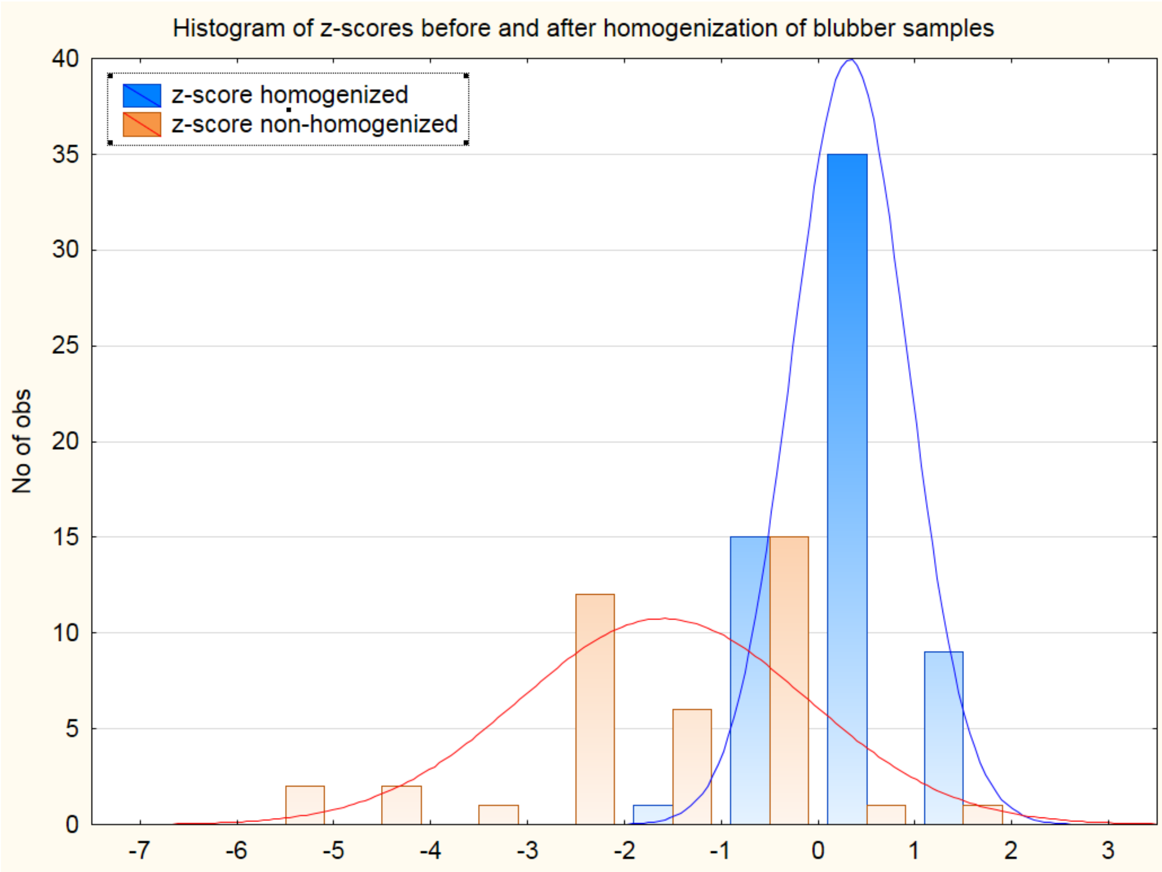

Figure 20: Comparison of z-scores obtained before and after homogenization of whale blubber

4. Conclusion

A summary of the results for predicted proxy limits for PCB_7 and predicted PCB_7 factors for conversion into total PCB is given in table 18 and table 19, respectively. The results for predicted proxy PCB_7 limits ranged from 0.30 to 0.21 mg/kg in the 99% prediction interval, and the results from predicted PCB_7 factors ranged from 1.6 to 2.3 in the 99% prediction interval. The results show that only small changes are seen whether the suspected outliers are included or not. The main differences are seen when the prediction interval (95% or 99%) is added or subtracted. It should be pointed out that the prediction interval could have been narrower if more samples than 35 had been analyzed and included in the data evaluation.

These results show that it is possible to use PCB_7 as a proxy for total PCB. This could be done either by:

1) applying a proxy PCB_7 maximum limit corresponding to the Japanese maximum limit for total PCB, or

2) applying a PCB_7 conversion factor for calculation into total PCB concentration followed by comparison with Japanese maximum limit.

3) using the appropriate equation from the linear regression for conversion of PCB_7 concentrations into total PCB concentrations.

All these approaches results in equal results when evaluating the compliance of samples within the uncertainty expected due to rounding to two significant figures.

Table 18: Predicted proxy PCB_7 limits at Japanese limit for total PCB (0.5 mg/kg)

Analysis	N	at -99% prediction interval	at -95% prediction interval	from linear regression	at +95% prediction interval	at +99% prediction interval
UB ww	35	0.30	0.29	0.26	0.23	0.22
UB ww minus outlier	34	0.28	0.27	0.25	0.24	0.23
MB ww	35	0.30	0.29	0.26	0.23	0.22
MB ww minus outlier	34	0.29	0.28	0.25	0.22	0.21
LB ww	35	0.30	0.29	0.26	0.23	0.22
LB ww minus outlier	34	0.29	0.28	0.25	0.23	0.22
Min	34	0.28	0.27	0.25	0.22	0.21
Max	35	0.30	0.29	0.26	0.24	0.23

Table 19: Predicted PCB_7 factor for conversion into total PCB concentration

Analysis	N	at -99% prediction interval	at -95% prediction interval	from linear regression	at +95% prediction interval	at +99% prediction interval
UB ww	35	1.6	1.7	1.9	2.2	2.3
UB ww minus outlier	34	1.8	1.8	2.0	2.1	2.2

Analysis	N	at -99% prediction interval	at -95% prediction interval	from linear regression	at +95% prediction interval	at +99% prediction interval
MB ww	35	1.6	1.7	1.9	2.2	2.3
MB ww minus outlier	34	1.7	1.8	2.0	2.2	2.3
LB ww	35	1.6	1.7	1.9	2.2	2.3
LB ww minus outlier	34	1.7	1.8	2.0	2.2	2.3
Min	34	1.6	1.7	1.9	2.1	2.2
Max	35	1.8	1.8	2.0	2.2	2.3

Results from the interlaboratory comparison between Eurofins, Iceland and NIFES demonstrated good agreement between the laboratories with z-scores in the range -1.0 to + 1.2. By combining and comparing the new PCB_7 results from homogenized samples with the old PCB_7 data of non-homogenized samples it is clear that sample homogeneity is an important factor when determining the PCB concentration in whale blubber.

5. References

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