Results of Proficiency Test
PCBs in Mineral Oil
November 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. M. Meijer
Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink
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CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 4

2.6 ANALYZES 5

3 RESULTS 5

3.1 STATISTICS 5

3.2 GRAPHICS 6

3.3 Z-SCORES 6

4 EVALUATION 7

4.1 EVALUATION PER TEST 7

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 9

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2020 WITH PREVIOUS PTs 10

Appendices:

1. Data, statistical and graphic results 11
2. z-scores individual PCBs and Aroclors 19
3. Number of participants per country 21
4. Abbreviations and literature 22

1 INTRODUCTION

Since 2001 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for PCBs in Mineral Oil every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis on PCBs in Mineral Oil.
In this interlaboratory study 49 laboratories in 23 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the PCBs in Mineral Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of mineral oil positive on PCB in an 8mL vial labelled #20228. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of approximately 1 liter of mineral oil positive on PCB was obtained from a third-party laboratory. After homogenization 78 amber glass vials of 8 mL were filled and labelled #20228.
The homogeneity of the subsamples was checked by determination of Total Organic Chlorides content in accordance with UOP779 on eight stratified randomly selected subsamples.

	Total Organic Chlorides as Cl in mg/kg
sample #20228-1	25
sample #20228-2	23
sample #20228-3	24
sample #20228-4	24
sample #20228-5	25
sample #20228-6	23
sample #20228-7	23
sample #20228-8	24

Table 1: homogeneity test results of subsamples of #20228

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Total Organic Chlorides as Cl in mg/kg
r (observed)	2
reference method	Horwitz
0.3 * R (reference method)	2

Table 2: evaluation of the repeatability of subsamples #20228

The calculated repeatability was in agreement with 0.3 times the reference reproducibility. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #20228 was sent on October 28, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the oil packed in amber glass vials was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on sample #20228: Total Organohalogenic Compounds (TOX) as Cl and Poly Chlorinated Biphenyls (via seven individual PCBs, via the determination of the total PCB content and/or via Aroclor standards).
It was requested to determine all four Aroclor components and not just the main Aroclor component.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the participants were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs’ or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.
The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test no major problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and four participants did not report any test results. Not all participants were able to report all tests requested.
In total 45 laboratories reported 251 numerical test results. Observed were 9 outlying test results, which is 3.6%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

In the iis PT reports test methods are referred to with a number (e.g. D4059) and an added designation for the year that the method was adopted or revised (e.g. D4059:00).
If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D4059:00(2018)). In the results tables of appendix 1 only the method number and year of adoption or revision (e.g. D4059:00) are used.

For the statistical evaluation of the individual PCBs the test method EN12766-1:00 was used, this test method is equal to IP462-1:01. In the test methods IEC61619:99 and DIN51527:93 only the reproducibilities of the total PCB content are mentioned, while in EN12766-1:00 / IP462-1:01 the reproducibilities for individual congeners are mentioned.

Sample #20228

TOX as Cl: Only two test results were reported. Therefore, no z-scores were calculated.

Individual PCBs: The determination of the individual PCBs may be problematic. In total six statistical outliers were observed over seven congeners and one other test result was excluded.
The calculated reproducibilities of congeners No. 52, 101 and 138 after rejection of the suspect data are not in agreement with requirements of EN12766-1:00 / IP462-1:01.
The calculated reproducibilities of congeners No. 118, 153 and 180 after rejection of the statistical outliers are in agreement with requirements of EN12766-1:00 / IP462-1:01.
For PCB 28 the reported test results were close to the detection limit, therefore no z-scores were calculated.

Individual Aroclors: The determination of the individual Aroclors was problematic. No statistical outliers were observed. The calculated reproducibilities of the Aroclors 1254 and 1260 are not in agreement with the requirements of ASTM D4059:00(2018). For Aroclors 1242 and 1248 the reported test results were close to the detection limit, therefore no z-scores were calculated.

Total PCB, 5 times the sum of 6 PCB congeners: This determination and/or calculation was not problematic. One statistical outlier was observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN12766-2 test method B:2001.
Three laboratories reported 5 times the sum of 7 congeners. Therefore, these test results were excluded from statistical evaluation. PCB118 should not be used in the calculation.

Total PCB, sum of all PCB congeners: This determination and/or calculation was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN61619:99 and EN12766-2 test method A:2001 as this test method is identical to EN61619:99.

Total PCB, sum of all Aroclors: This determination and/or calculation was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4059:00(2018).

Summary: All participants agreed that sample #20228 was positive on PCBs. From the data on total organic halogenic components (TOX) an average concentration of 32.4 mg/kg was calculated. From this concentration, a total content of 56.1 mg PCB/kg was estimated using an average Cl content of 57.7%, assuming the presence of 38.2% Aroclor 1254 (54% Cl) and 61.8% Aroclor 1260 (60% Cl). All values for total PCB are given in the next table.

	total PCB content in mg/kg
estimated by TOX as Cl	57.0
5 times the sum of 6 congeners	46.7
sum of all Congeners	29.7
sum of all Aroclors	28.9

Table 3: comparison of estimations of total PCB content in sample #20228

The total PCB content determined by EN12766-2, method A or IEC61619:99 is in good agreement with the total PCB content as determined by the Aroclor method. The other two estimates, from TOX and from 5 x 6 congeners, are both higher.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu EN or ASTM test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX as Cl	mg/kg	2	32.4	n.e.	n.e.
PCB no. 28	mg/kg	22	<0.3	n.e.	n.e.
PCB no. 52	mg/kg	22	0.52	0.36	0.24
PCB no. 101	mg/kg	23	1.46	0.87	0.71
PCB no. 118	mg/kg	17	0.90	0.30	0.43
PCB no. 138	mg/kg	23	2.60	1.42	1.28
PCB no. 153	mg/kg	23	2.70	1.17	1.33
PCB no. 180	mg/kg	23	1.98	0.75	0.97
Aroclor 1242	mg/kg	8	<2	n.e.	n.e.
Aroclor 1248	mg/kg	3	<1	n.e.	n.e.
Aroclor 1254	mg/kg	14	11.17	11.31	8.18
Aroclor 1260	mg/kg	15	18.05	15.88	11.73
Total PCB, 5 x sum 6 congeners	mg/kg	17	46.06	14.92	20.63
Total PCB, sum of all congeners	mg/kg	20	29.67	13.10	9.42
Total PCB, sum of Aroclors	mg/kg	12	28.85	18.98	16.68

Table 4: reproducibilities of tests on sample #20228

Without further statistical calculations it can be concluded that for most tests there is not a good compliance of the group of participating laboratories with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2020 PROFICIENCY TEST WITH PREVIOUS PTS

	November 2020	November 2019	November 2018	November 2017	November 2016
Number of reporting laboratories	45	45	45	50	45
Number of test results	251	277	247	275	221
Number of statistical outliers	9	14	13	16	12
Percentage of statistical outliers	3.6%	5.1%	5.3%	5.8%	5.4%

Table 5: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the reference test methods. The conclusions are given in the following table.

	November 2020	November 2019	November 2018	November 2017	November 2016
TOX as Cl	n.e.	n.e.	--*	+/-*	--*
PCB individual	-	+/-	-	-	-
Aroclor individual	-	-	--	--	--
Total PCB, 5 x the sum of 6 cong	+	+	+/-	+/-	+/-
Total PCB, sum of all congeners	-	+	-	-	-
Total PCB, sum of Aroclors	-	-	-	+/-	-

Table 6: comparison determinations against the reference test methods
*) based on three or four test results

The following performance categories were used:

++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e. : not evaluated

APPENDIX 1

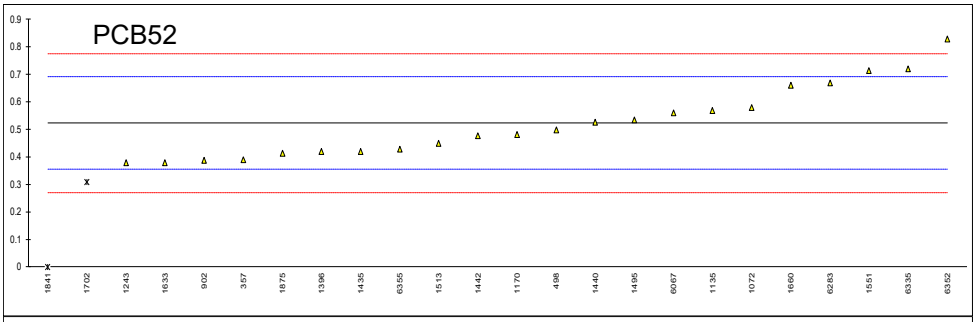
Determination of Total Organohalogenic Compounds (TOX) as Cl on sample #20228; results in mg/kg

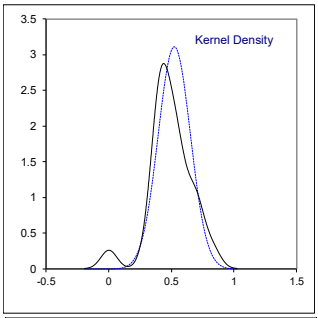
lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072		-----		-----	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495	EN14077	27		-----	
1505		-----		-----	
1513		-----		-----	
1551		-----		-----	
1602		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1743		-----		-----	
1765		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885		-----		-----	
1888		-----		-----	
1965		-----		-----	
6067		-----		-----	
6278		-----		-----	
6283		-----		-----	
6334		-----		-----	
6335		-----		-----	
6352		-----		-----	
6355		37.7		-----	
	n	2			
	mean (n)	32.4			

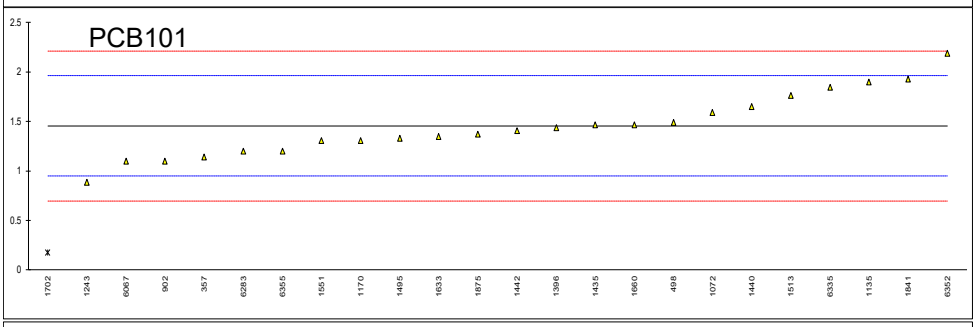
Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #20228; results in mg/kg

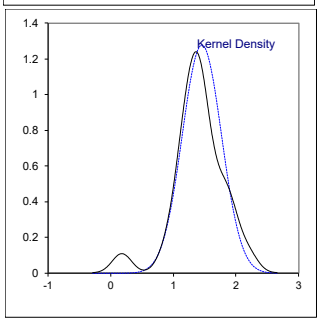
lab	method	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341		-----	-----	-----	-----	-----	-----	-----
343		-----	-----	-----	-----	-----	-----	-----
357	EN12766-1	<0.05	0.39	1.14	0.84	2.38	2.70	2.04
398		-----	-----	-----	-----	-----	-----	-----
498	EN12766-1	<0,3	0.498	1.494	0.888	2.658	2.571	1.805
511		-----	-----	-----	-----	-----	-----	-----
614		-----	-----	-----	-----	-----	-----	-----
902		-----	0.388	1.103	0.934	2.64	2.621	2.051
912		-----	-----	-----	-----	-----	-----	-----
1059		-----	-----	-----	-----	-----	-----	-----
1072	EN12766-1	0.0624	0.5792	1.5936	0.8665	2.8509	3.3604	1.9730
1126		-----	-----	-----	-----	-----	-----	-----
1135	IEC61619	0	0.57 C	1.9	1.0	3.8	2.86 C	2.51 C
1170		0.023	0.482	1.311	0.851	2.428	2.470	1.911
1243	EN12766-1	0.08	0.38	0.89	-----	1.25	1.59	1.32
1304		-----	-----	-----	-----	-----	-----	-----
1306		-----	-----	-----	-----	-----	-----	-----
1352		-----	-----	-----	-----	-----	-----	-----
1367		-----	-----	-----	-----	-----	-----	-----
1374		-----	-----	-----	-----	-----	-----	-----
1396	IP462-1	0.055	0.42	1.44	-----	1.984	2.817	2.312
1435	EN12766-1	0.00	0.42	1.47	0.85	2.79	2.79	1.93
1440	IEC61619	0.0786	0.5264	1.6495	1.0726	2.7457	3.0356	2.1797
1442	EN12766-1	0.018	0.477	1.408	1.015	2.611	3.001	2.024
1458		-----	-----	-----	-----	-----	-----	-----
1495	EN12766-1	0	0.536	1.333	-----	2.803	2.586	2.121
1505		-----	-----	-----	-----	-----	-----	-----
1513	IEC61619	<0,2	0.449	1.765	0.876	2.622	2.390	2.086
1551	EN12766-1	0.0415	0.7144	1.3059	-----	2.0184	2.8030	1.5055
1602		-----	-----	-----	-----	-----	-----	-----
1633		<0.10	0.38	1.35	0.80	2.11	2.23	1.69
1660	IEC61619	0	0.66	1.47	0.74	3.12	2.93	2.00
1702	IEC61619	0.30	0.31 ex	0.18 R(5)	0.21 G(1)	0.28 R(1)	0.44 R(1)	0.19 R(1)
1743		-----	-----	-----	-----	-----	-----	-----
1765		-----	-----	-----	-----	-----	-----	-----
1801		-----	-----	-----	-----	-----	-----	-----
1816		-----	-----	-----	-----	-----	-----	-----
1841	IEC61619	0.00	0.00 R(5)	1.93	1.09	3.41	1.98	2.40
1875	In house	0.044	0.414	1.374	-----	2.232	2.833	1.941
1885		-----	-----	-----	-----	-----	-----	-----
1888		-----	-----	-----	-----	-----	-----	-----
1965		-----	-----	-----	-----	-----	-----	-----
6067		0 C	0.560417	1.10227	0.84957	2.67315	2.51169	1.82215
6278		-----	-----	-----	-----	-----	-----	-----
6283		0.00	0.67 C	1.20	1.01 C	2.50	2.90	1.80
6334		-----	-----	-----	-----	-----	-----	-----
6335	EN12766-1	<0.2	0.720	1.845	-----	2.825	2.850	1.955
6352	DIN51527Mod.	0 C	0.83	2.19	0.94	2.81	3.58	2.23
6355	EN12766-1	0	0.429	1.204	0.736	2.544	2.577	1.890
	normality	OK	OK	OK	OK	not OK	suspect	suspect
	n	22	22	23	17	23	23	23
	outliers	n.a.	1 +1ex	1	1	1	1	1
	mean (n)	<0.3	0.5224	1.4551	0.9035	2.6002	2.6951	1.9781
	st.dev. (n)	n.e.	0.12830	0.31216	0.10582	0.50635	0.41621	0.26648
	R(calc.)	n.e.	0.3592	0.8740	0.2963	1.4178	1.1654	0.7461
	st.dev.(EN12766-1:00)	n.e.	0.08579	0.25298	0.15409	0.45823	0.47523	0.34672
	R(EN12766-1:00)	n.e.	0.2402	0.7083	0.4314	1.2831	1.3307	0.9708

Lab 1135 first reported 0.9 PCB52, 4.1 PCB153, 3.8 PCB180
Lab 1702 test result PCB52 excluded due to statistical outliers in related parameters
Lab 6067 first reported 0.22455 PCB28
Lab 6283 first reported 0.3 PCB52, 1.6 PCB118
Lab 6352 first reported 0.32 PCB28


PCB52


Kernel Density


PCB101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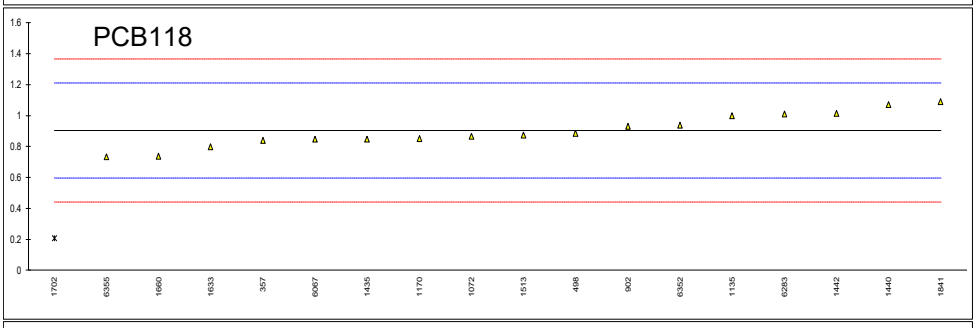
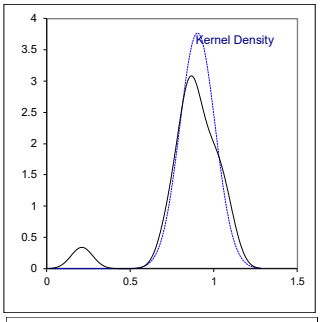
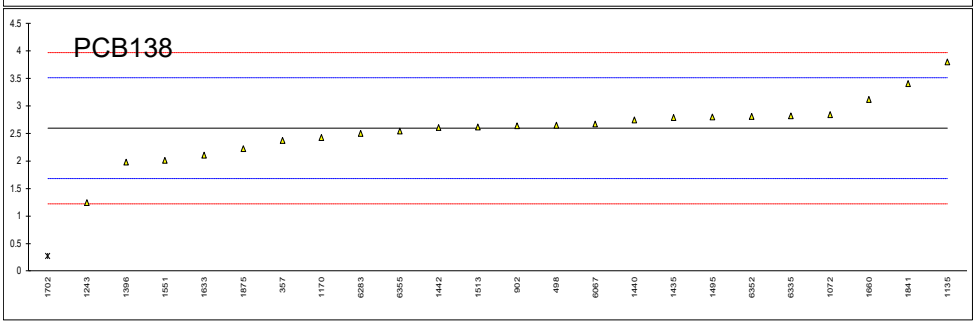
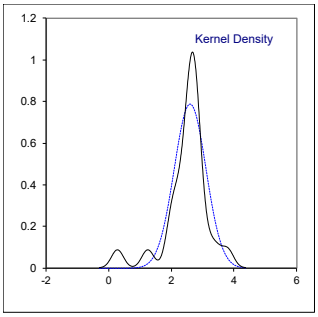
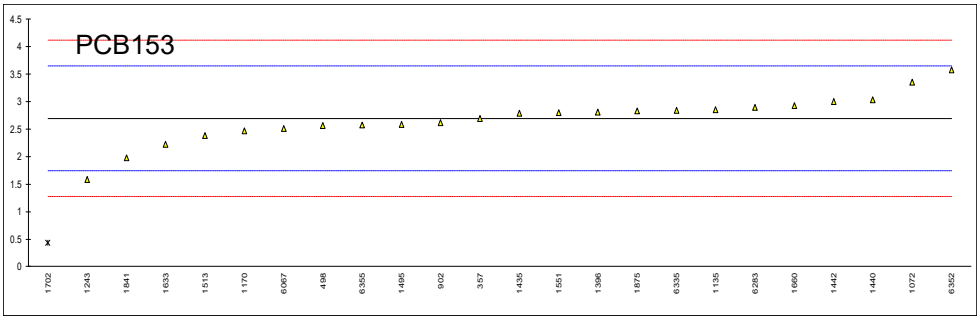
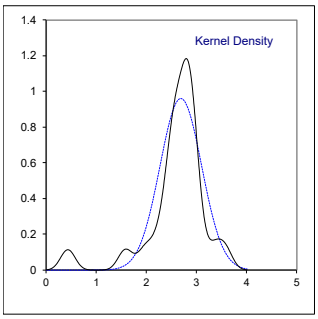

PCB118

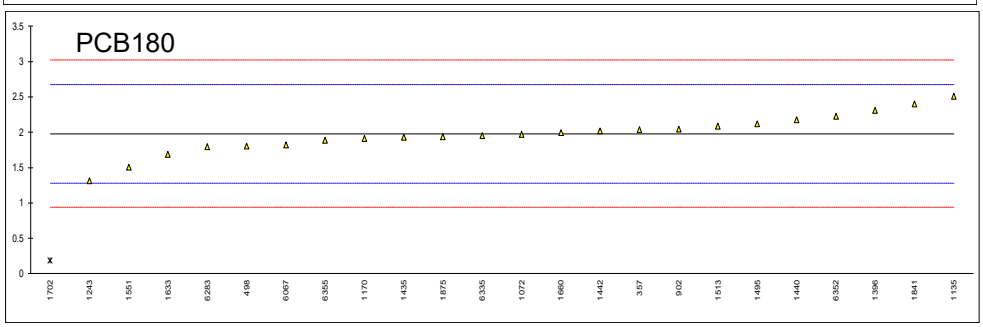

Kernel Density

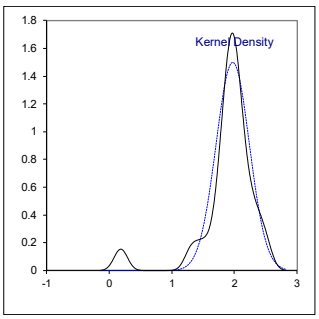

PCB138


Kernel Density


PCB153

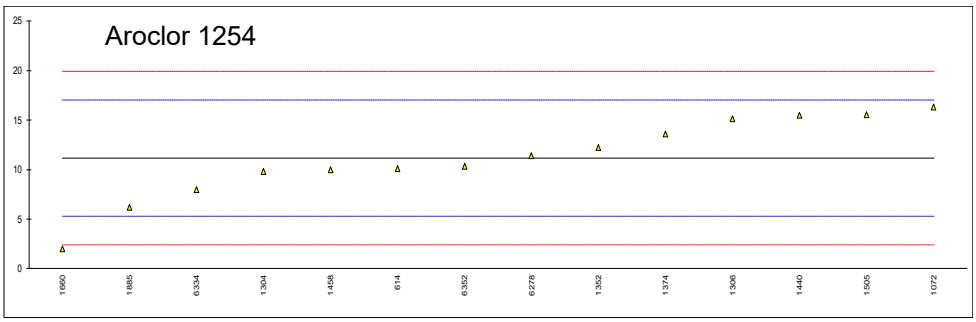

Kernel Density

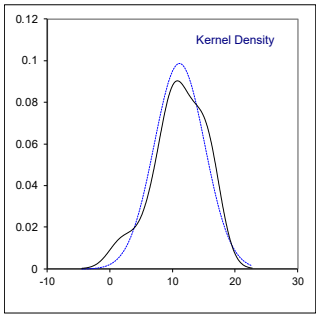

PCB180

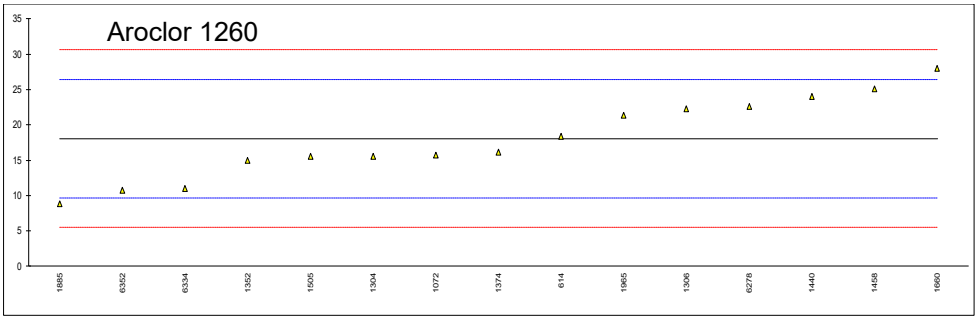

Kernel Density

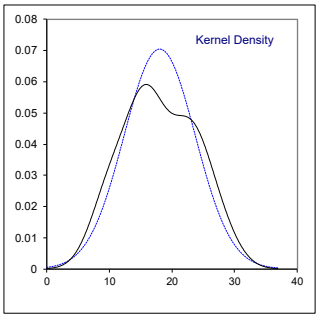
Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #20228; results in mg/kg

lab	method	Aroclor 1242		Aroclor 1248		Aroclor 1254	Aroclor 1260
341		-----		-----		-----	-----
343		-----		-----		-----	-----
357		-----		-----		-----	-----
398		-----		-----		-----	-----
498		-----		-----		-----	-----
511		-----		-----		-----	-----
614	D4059	<2		-----		10.1	18.4
902		-----		-----		-----	-----
912		-----		-----		-----	-----
1059		-----		-----		-----	-----
1072	D4059	0.698		-----		16.348	15.792
1126		-----		-----		-----	-----
1135		-----		-----		-----	-----
1170		-----		-----		-----	-----
1243		-----		-----		-----	-----
1304	In house	-----		-----		9.811	15.600
1306	In house	<1		-----		15.16	22.33
1352	In house	Not detected		-----		12.245	15.039
1367		-----		-----		-----	-----
1374		-----		-----		13.60	16.17
1396		-----		-----		-----	-----
1435		-----		-----		-----	-----
1440	In house	2		-----		15.5	24
1442		-----		-----		-----	-----
1458	D4059	<2		-----		10.0	25.1
1495		-----		-----		-----	-----
1505	D4059	21.47	f+?	-----		15.60	15.59
1513		-----		-----		-----	-----
1551		-----		-----		-----	-----
1602		-----		-----		-----	-----
1633		-----		-----		-----	-----
1660		-----		-----		2	28
1702		-----		-----		-----	-----
1743		-----		-----		-----	-----
1765		-----		-----		-----	-----
1801		-----		-----		-----	-----
1816		-----		-----		-----	-----
1841		-----		-----		-----	-----
1875		-----		-----		-----	-----
1885	EPAA6013	0		0		6.2	8.9
1888		-----		-----		-----	-----
1965	D6160	<0.1		<0.1		<0.1	21.40
6067		-----		-----		-----	-----
6278	EPA8082	0		0		11.4	22.6
6283		-----		-----		-----	-----
6334	IEC61619	<1		-----		8	11
6335		-----		-----		-----	-----
6352	DIN51527Mod.	2.09		3.58	f+?	10.35	10.81
6355		-----		-----		-----	-----
	normality	unknown		n.a.		OK	OK
	n	8		3		14	15
	outliers	n.a.		n.a.		0	0
	mean (n)	<2		<1		11.1653	18.0487
	st.dev. (n)	n.e.		n.e.		4.03914	5.66990
	R(calc.)	n.e.		n.e.		11.3096	15.8757
	st.dev.(D4059:00 (silicone))	n.e.		n.e.		2.92314	4.19065
	R(D4059:00 (silicone))	n.e.		n.e.		8.1848	11.7338


Aroclor 1254
25
20
15
10
5
0
1660
1885
6334
1304
1458
614
6352
6278
1352
1374
1306
1440
1505
1072


Kernel Density
0.12
0.1
0.08
0.06
0.04
0.02
0
-10
0
10
20
30

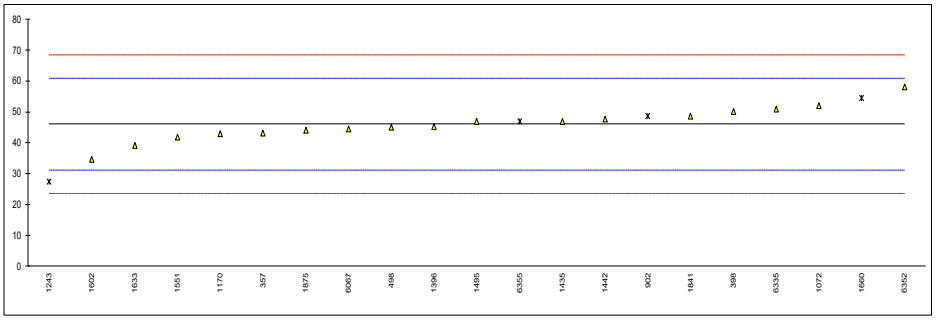

Aroclor 1260
35
30
25
20
15
10
5
0
1885
6352
6334
1352
1505
1304
1072
1374
614
1965
1306
6278
1440
1458
1660

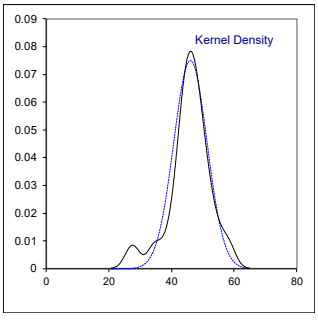

Kernel Density
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
10
20
30
40

Determination of Total PCB, 5 times the sum of 6 congeners on sample #20228; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	43.25		-0.38	
398	EN12766-2-B	50.2		0.56	
498	EN12766-2-B	45.100		-0.13	
511		-----		-----	
614		-----		-----	
902	EN12766-2-B	48.65	ex	0.35	test result excluded, reported 5 times sum of 7 congeners
912		-----		-----	
1059		-----		-----	
1072	EN12766-2-B	52.0975		0.82	
1126		-----		-----	
1135		-----		-----	
1170	EN12766-2-B	43.095		-0.40	
1243	EN12766-2-B	27.55	R(0.05)	-2.51	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	45.3213	E	-0.10	iis calculated 45.1400
1435	EN12766-2-B	46.95		0.12	
1440		-----		-----	
1442	EN12766-2-B	47.697		0.22	
1458		-----		-----	
1495	EN12766-2-B	46.9		0.11	
1505		-----		-----	
1513		-----		-----	
1551	EN12766-2-B	41.9435		-0.56	
1602	EN12766-2-B	34.63		-1.55	
1633		39.29	E	-0.92	iis calculated 38.80
1660	EN12766-2-B	54.6	ex	1.16	test result excluded, reported 5 times sum of 7 congeners
1702		-----		-----	
1743		-----		-----	
1765		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	48.65		0.35	
1875	EN12766-2-B	44.2		-0.25	
1885		-----		-----	
1888		-----		-----	
1965		-----		-----	
6067	IEC61619	44.5	E	-0.21	iis calculated 43.3
6278		-----		-----	
6283		-----		-----	
6334		-----		-----	
6335	EN12766-2-B	51.037		0.68	
6352	DIN51527Mod.	58.2	C	1.65	first reported 11.96
6355	EN12766-2-B	46.901	ex	0.11	test result excluded, reported 5 times sum of 7 congeners

normality	suspect
n	17
outliers	1 +3ex
mean (n)	46.0624
st.dev. (n)	5.32739
R(calc.)	14.9167
st.dev.(EN12766-2B:01)	7.36662
R(EN12766-2B:01)	20.6265

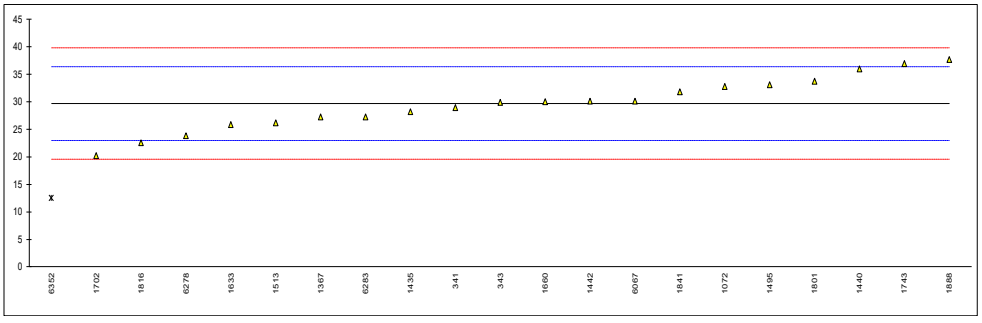


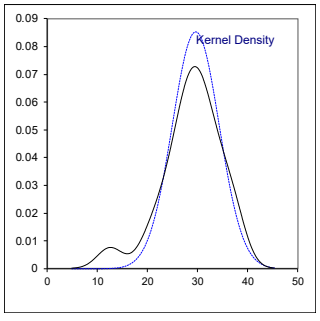


Determination of Total PCB, sum of all congeners on sample #20228; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		29		-0.20	
343	EN61619	30		0.10	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072	EN61619	32.8385		0.94	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367	EN61619	27.25		-0.72	
1374		-----		-----	
1396		-----		-----	
1435	IEC61619	28.29		-0.41	
1440	EN61619	36		1.88	
1442	IEC61619	30.206		0.16	
1458		-----		-----	
1495	EN12766-2A	33.2		1.05	
1505		-----		-----	
1513	IEC61619	26.2		-1.03	
1551		-----		-----	
1602		-----		-----	
1633		25.89		-1.12	
1660	IEC61619	30.02		0.10	
1702	IEC61619	20.21		-2.81	
1743	IEC61619	37		2.18	
1765		-----		-----	
1801	EN61619	33.758		1.21	
1816	EN61619	22.6		-2.10	
1841	IEC61619	31.89		0.66	
1875		-----		-----	
1885		-----		-----	
1888	EN61619	37.7	C	2.39	first reported 46.8
1965		-----		-----	
6067	IEC61619	30.21		0.16	
6278	EPA8082	23.9		-1.72	
6283	IEC61619	27.3		-0.71	
6334		-----		-----	
6335		-----		-----	
6352	DIN51527Mod.	12.58	C,R(0.05)	-5.08	first reported 12.9
6355		-----		-----	

normality	OK
n	20
outliers	1
mean (n)	29.6731
st.dev. (n)	4.68005
R(calc.)	13.1041
st.dev.(EN61619:99)	3.36367
R(EN61619:99)	9.4183

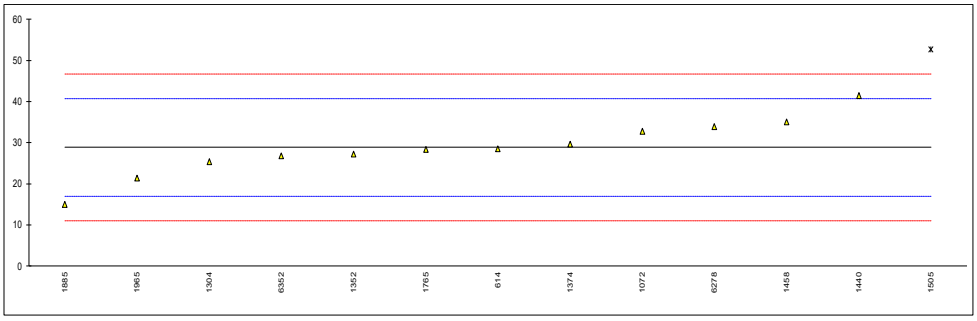


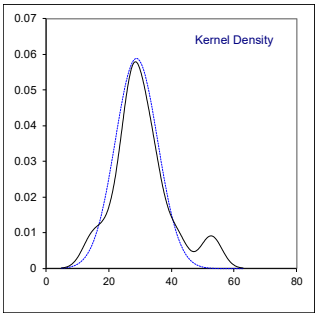


Determination of Total PCB, sum of all Aroclors on sample #20228; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614	D4059	28.5		-0.06	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072	D4059	32.838		0.67	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304	In house	25.41		-0.58	
1306		-----		-----	
1352	In house	27.284		-0.26	
1367		-----		-----	
1374	D4059	29.77		0.15	
1396		-----		-----	
1435		-----		-----	
1440	In house	41.5		2.12	
1442		-----		-----	
1458	D4059	35.1		1.05	
1495		-----		-----	
1505	D4059	52.66	D(0.05)	4.00	
1513		-----		-----	
1551		-----		-----	
1602		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1743		-----		-----	
1765	EN61619	28.469		-0.06	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885	EPA6013	15.1		-2.31	
1888		-----		-----	
1965	D6160	21.40		-1.25	also reported a test result 21.20 acc. to D4059
6067		-----		-----	
6278	EPA8082	34		0.86	
6283		-----		-----	
6334		-----		-----	
6335		-----		-----	
6352	DIN51527Mod.	26.83		-0.34	
6355		-----		-----	

normality	OK
n	12
outliers	1
mean (n)	28.8501
st.dev. (n)	6.77936
R(calc.)	18.9822
st.dev.(D4059:00 (silicone))	5.95740
R(D4059:00 (silicone))	16.6807





APPENDIX 2

z-scores of PCB 28, 52, 101, 118, 138, 153 and 180 determination on sample #20228;

lab	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341	-----	-----	-----	-----	-----	-----	-----
343	-----	-----	-----	-----	-----	-----	-----
357	-----	-1.54	-1.25	-0.41	-0.48	0.01	0.18
398	-----	-----	-----	-----	-----	-----	-----
498	-----	-0.28	0.15	-0.10	0.13	-0.26	-0.50
511	-----	-----	-----	-----	-----	-----	-----
614	-----	-----	-----	-----	-----	-----	-----
902	-----	-1.57	-1.39	0.20	0.09	-0.16	0.21
912	-----	-----	-----	-----	-----	-----	-----
1059	-----	-----	-----	-----	-----	-----	-----
1072	-----	0.66	0.55	-0.24	0.55	1.40	-0.01
1126	-----	-----	-----	-----	-----	-----	-----
1135	-----	0.55	1.76	0.63	2.62	0.35	1.53
1170	-----	-0.47	-0.57	-0.34	-0.38	-0.47	-0.19
1243	-----	-1.66	-2.23	-----	-2.95	-2.33	-1.90
1304	-----	-----	-----	-----	-----	-----	-----
1306	-----	-----	-----	-----	-----	-----	-----
1352	-----	-----	-----	-----	-----	-----	-----
1367	-----	-----	-----	-----	-----	-----	-----
1374	-----	-----	-----	-----	-----	-----	-----
1396	-----	-1.19	-0.06	-----	-1.34	0.26	0.96
1435	-----	-1.19	0.06	-0.35	0.41	0.20	-0.14
1440	-----	0.05	0.77	1.10	0.32	0.72	0.58
1442	-----	-0.53	-0.19	0.72	0.02	0.64	0.13
1458	-----	-----	-----	-----	-----	-----	-----
1495	-----	0.16	-0.48	-----	0.44	-0.23	0.41
1505	-----	-----	-----	-----	-----	-----	-----
1513	-----	-0.86	1.22	-0.18	0.05	-0.64	0.31
1551	-----	2.24	-0.59	-----	-1.27	0.23	-1.36
1602	-----	-----	-----	-----	-----	-----	-----
1633	-----	-1.66	-0.42	-0.67	-1.07	-0.98	-0.83
1660	-----	1.60	0.06	-1.06	1.13	0.49	0.06
1702	-----	-2.48	-5.04	-4.50	-5.06	-4.75	-5.16
1743	-----	-----	-----	-----	-----	-----	-----
1765	-----	-----	-----	-----	-----	-----	-----
1801	-----	-----	-----	-----	-----	-----	-----
1816	-----	-----	-----	-----	-----	-----	-----
1841	-----	-6.09	1.88	1.21	1.77	-1.50	1.22
1875	-----	-1.26	-0.32	-----	-0.80	0.29	-0.11
1885	-----	-----	-----	-----	-----	-----	-----
1888	-----	-----	-----	-----	-----	-----	-----
1965	-----	-----	-----	-----	-----	-----	-----
6067	-----	0.44	-1.39	-0.35	0.16	-0.39	-0.45
6278	-----	-----	-----	-----	-----	-----	-----
6283	-----	1.72	-1.01	0.69	-0.22	0.43	-0.51
6334	-----	-----	-----	-----	-----	-----	-----
6335	-----	2.30	1.54	-----	0.49	0.33	-0.07
6352	-----	3.59	2.90	0.24	0.46	1.86	0.73
6355	-----	-1.09	-0.99	-1.09	-0.12	-0.25	-0.25

z-scores of Aroclor 1242, 1248, 1254 and 1260 determination on sample #20228;

lab	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341	-----	-----	-----	-----
343	-----	-----	-----	-----
357	-----	-----	-----	-----
398	-----	-----	-----	-----
498	-----	-----	-----	-----
511	-----	-----	-----	-----
614	-----	-----	-0.36	0.08
902	-----	-----	-----	-----
912	-----	-----	-----	-----
1059	-----	-----	-----	-----
1072	-----	-----	1.77	-0.54
1126	-----	-----	-----	-----
1135	-----	-----	-----	-----
1170	-----	-----	-----	-----
1243	-----	-----	-----	-----
1304	-----	-----	-0.46	-0.58
1306	-----	-----	1.37	1.02
1352	-----	-----	0.37	-0.72
1367	-----	-----	-----	-----
1374	-----	-----	0.83	-0.45
1396	-----	-----	-----	-----
1435	-----	-----	-----	-----
1440	-----	-----	1.48	1.42
1442	-----	-----	-----	-----
1458	-----	-----	-0.40	1.68
1495	-----	-----	-----	-----
1505	-----	-----	1.52	-0.59
1513	-----	-----	-----	-----
1551	-----	-----	-----	-----
1602	-----	-----	-----	-----
1633	-----	-----	-----	-----
1660	-----	-----	-3.14	2.37
1702	-----	-----	-----	-----
1743	-----	-----	-----	-----
1765	-----	-----	-----	-----
1801	-----	-----	-----	-----
1816	-----	-----	-----	-----
1841	-----	-----	-----	-----
1875	-----	-----	-----	-----
1885	-----	-----	-1.70	-2.18
1888	-----	-----	-----	-----
1965	-----	-----	<-3.79	0.80
6067	-----	-----	-----	-----
6278	-----	-----	0.08	1.09
6283	-----	-----	-----	-----
6334	-----	-----	-1.08	-1.68
6335	-----	-----	-----	-----
6352	-----	-----	-0.28	-1.73
6355	-----	-----	-----	-----

Lab 1965 possibly a false negative test result?

APPENDIX 3

Number of participating laboratories per country

7 labs in	AUSTRALIA
2 labs in	BELGIUM
1 lab in	FINLAND
3 labs in	FRANCE
4 labs in	GERMANY
2 labs in	GREECE
1 lab in	INDIA
3 labs in	ITALY
1 lab in	MALAYSIA
1 lab in	MONTENEGRO
1 lab in	MOROCCO
1 lab in	NETHERLANDS
2 labs in	NORWAY
1 lab in	PERU
1 lab in	PHILIPPINES
1 lab in	POLAND
2 labs in	PORTUGAL
1 lab in	QATAR
1 lab in	SLOVENIA
1 lab in	SOUTH AFRICA
7 labs in	SPAIN
1 lab in	TURKEY
4 labs in	UNITED KINGDOM

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01) / G(1)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01) / R(1)	= outlier in Rosner's outlier test
R(0.05) / R(5)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

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