Results of Proficiency Test
PCB in Mineral Oil
November 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink

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1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for PCB in (mineral) oil every year. During the annual proficiency testing program 2019/2020, it was decided to continue the round robin for the analysis of PCB in (mineral) oil.
In this interlaboratory study, 51 laboratories in 26 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 proficiency test on PCB in (mineral) oil 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. Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one 8mL vial with mineral oil contaminated with PCB, labelled #19243. 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

In this proficiency test the necessary bulk material for the preparation of the subsamples was a mineral oil positive on PCB donated by a third party laboratory. After ultrasonic homogenization 70 amber glass vials of 8mL were filled and labelled #19243.
The homogeneity of the subsamples #19243 was checked by determination of Total Organic Chloride content in accordance with UOP779 on eight stratified randomly selected samples.

	Total Organic Chloride as Cl in mg/kg
sample #19243-1	20.1
sample #19243-2	20.0
sample #19243-3	19.9
sample #19243-4	20.2
sample #19243-5	20.0
sample #19243-6	20.2
sample #19243-7	20.0
sample #19243-8	20.0

Table 1: homogeneity test results of subsamples of #19243

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

	Total Organic Chloride as Cl in mg/kg
r (observed)	0.3
reference test method	UOP779:08
0.3 x R (ref. test method)	1.0

Table 2: evaluation of the repeatability of subsamples of #19243

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

To each of the participating laboratories one amber glass vial of 8mL, labelled #19243, was sent on October 23, 2019. 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 ANALYSES

The participants were requested to determine on sample #19243: 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 more, 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 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 individual laboratories 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 reanalysis). Additional or corrected test results are used for data analysis and 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’ and/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.

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. The Kernel Density Graph 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 and EN 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 problems were encountered with the dispatch of the sample. Six participants did not report any test results. All other participants, except one, reported on time. Not all participants were able to report test results for all tests. In total 45 laboratories reported 277 numerical test results. Observed were 14 outlying test results, which is 5.1% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution. These are referred to as “OK”.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods that are reported by the laboratories are taken into account for explaining the observed differences when possible and applicable. These test methods are also mentioned in the tables in appendix 1 together with the original data. 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 all congeners are mentioned.

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

Individual PCBs: The determination of the individual PCB was problematic. In total eleven statistical outliers were observed over seven congeners and two other test results were excluded because out of six related test results four test results were statistical outliers.
The calculated reproducibilities of congeners No. 28, 101, and 118 after rejection of the suspect data are not in agreement with requirements of EN12766-1:00 / IP462-1:01.
The calculated reproducibility of congeners No. 52 , 138, 153 and 180 after rejection of the statistical outliers is in full agreement with requirements of EN12766-1:00 / IP462-1:01.

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

Total PCB: **Total PCB, “5 times the sum of 6 PCB congeners”**
This determination and/or calculation of total PCB content was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN12766-2 test method B:2001.

Total PCB, “sum of all PCB congeners”
This determination and/or calculation of total PCB content was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is 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 of total PCB content was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D4059:00(2018).

Summary: All participants agreed that sample #19243 was positive on PCBs. From the data on total organic halogenic components (TOX) an average concentration of 12.9 mg/kg was calculated. From this concentration, a total content of 23.3 mg PCB/kg was estimated using an average Cl content of 55.3%, assuming the presence of 14.8% Aroclor 1242 (42% Cl), 34.3% Aroclor 1254 (54% Cl) and 50.9% 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	23.3
5 times the sum of 6 congeners	28.7
sum of all congeners	22.1
using Aroclor method	17.3

Table 3: comparison of estimations of total PCB content in sample #19243.

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

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 result, the calculated reproducibility (2.8*standard deviation) and the target reproducibility derived from literature 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	3	12.9	n.a.	n.a.
PCB no. 28	mg/kg	23	0.21	0.22	0.08
PCB no. 52	mg/kg	23	0.67	0.34	0.31
PCB no. 101	mg/kg	25	1.21	0.91	0.58
PCB no. 118	mg/kg	17	0.53	0.28	0.24
PCB no. 138	mg/kg	25	1.41	0.69	0.69
PCB no. 153	mg/kg	23	1.54	0.56	0.75
PCB no. 180	mg/kg	25	1.03	0.43	0.50
Aroclor 1242	mg/kg	13	2.74	3.51	2.86
Aroclor 1248	mg/kg	2	<2	n.a.	n.a.
Aroclor 1254	mg/kg	14	6.34	8.48	5.35
Aroclor 1260	mg/kg	15	9.41	10.69	7.21
Total PCB, 5 x sum 6 congeners	mg/kg	19	28.70	10.65	12.69
Total PCB, sum of all congeners	mg/kg	20	22.08	6.75	7.52
Total PCB, sum of Aroclors	mg/kg	14	17.25	15.71	11.34

Table 4: reproducibilities of tests on sample #19243

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

4.3 COMPARISON OF THE NOVEMBER 2019 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2019	November 2018	November 2017	November 2016	November 2015
Number of reporting laboratories	45	45	50	45	43
Number of test results reported	277	247	275	221	219
Number of statistical outliers	14	13	16	12	5
Percentage outliers	5.1%	5.3%	5.8%	5.4%	2.3%

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 respective reference test methods. The conclusions are given the following table.

	November 2019	November 2018	November 2017	November 2016	November 2015
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 of observed precisions against requirements of the reference test methods
*) based on three or four test results

To indicate the performance of the determinations against the requirements of the respective reference test methods the following performance categories were used in the table above

++: 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

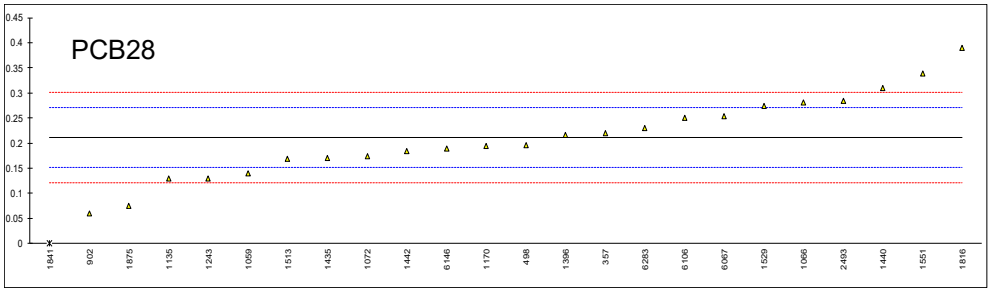
Determination of Total Organohalogenic Compounds (TOX) on sample #19243; results in mg/kg.

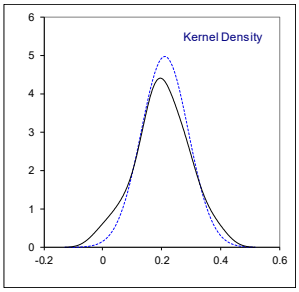
lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
974		-----		-----	
1059		-----		-----	
1066	UOP779	9.7		-----	
1072		-----		-----	
1126		-----		-----	
1135	EN14077	16		-----	
1170		-----		-----	
1243		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495	EN14077	13.02		-----	
1505		-----		-----	
1513		-----		-----	
1529		-----		-----	
1551		-----		-----	
1660		-----		-----	
1702		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885		-----		-----	
1888		-----		-----	
2493		-----		-----	
6067		-----		-----	
6106		-----		-----	
6141		-----		-----	
6146		-----		-----	
6165		-----		-----	
6254		-----		-----	
6278		-----		-----	
6283		-----		-----	
	n	3			
	mean (n)	12.9			

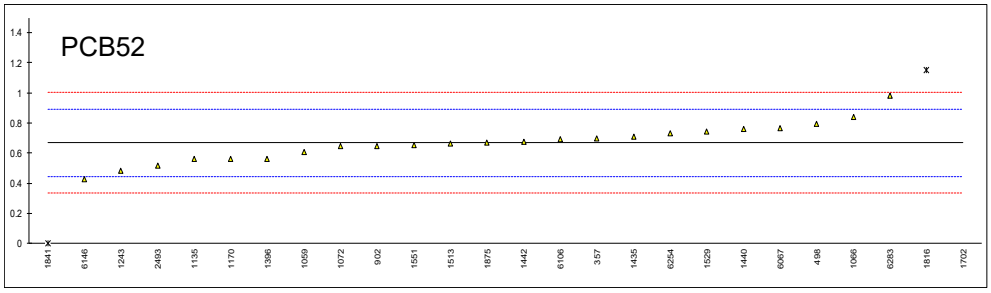
Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #19243; results in mg/kg.

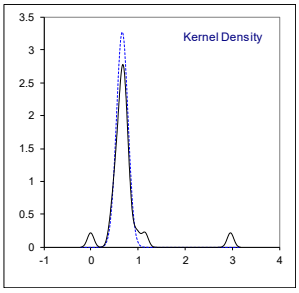
lab	Method	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341		-----	-----	-----	-----	-----	-----	-----
343		-----	-----	-----	-----	-----	-----	-----
357	EN12766-1	0.22	0.70	0.86	0.47	1.14	1.41	0.94
398		-----	-----	-----	-----	-----	-----	-----
498	EN12766-1	0.195	0.793	1.430	0.635	1.683	1.720	1.035
511		-----	-----	-----	-----	-----	-----	-----
614		-----	-----	-----	-----	-----	-----	-----
902	EN12766-1	0.060	0.6450	0.8913	0.8839 DG(5)	1.3104	1.5435	0.896 C
912		-----	-----	-----	-----	-----	-----	-----
974		-----	-----	-----	-----	-----	-----	-----
1059	EN12766-1	0.14	0.61	1.08	0.47 C	1.40	1.38	0.95
1066	EN12766-1	0.28	0.84	1.28	0.60	1.74	1.84	1.18
1072	EN12766-1	0.1742	0.6449	1.6526	0.5669	1.3549	1.4914	1.0116
1126		-----	-----	-----	-----	-----	-----	-----
1135	EN12766-1	0.13	0.56	0.90	0.38	1.10	1.24	0.95
1170	EN12766-1	0.194	0.562	0.981	0.426	1.0756	1.287	0.858
1243		0.13	0.48	0.84	-----	1.56	1.53	1.11
1303		-----	-----	-----	-----	-----	-----	-----
1304		-----	-----	-----	-----	-----	-----	-----
1306		-----	-----	-----	-----	-----	-----	-----
1352		-----	-----	-----	-----	-----	-----	-----
1367		-----	-----	-----	-----	-----	-----	-----
1374		-----	-----	-----	-----	-----	-----	-----
1396	IP462-1	0.2156	0.5643	1.0521	-----	1.3262	1.3010	0.9382
1435	EN12766-1	0.17	0.71	1.12	0.48	1.36	1.56	0.97
1440	IEC61619	0.3098	0.7628	1.5815	0.6356	1.6425	1.8150	1.2003
1442	EN12766-1	0.184	0.678	1.064	0.566	1.221	1.288	0.925
1458		-----	-----	-----	-----	-----	-----	-----
1495		-----	-----	-----	-----	-----	-----	-----
1505		-----	-----	-----	-----	-----	-----	-----
1513	IEC61619	0.169	0.663	1.553	0.539	1.420	1.494	1.095
1529	EN12766-1+EN61619	0.274	0.743	1.127	0.584	1.186	1.605	1.050
1551		0.339	0.652	1.027	-----	1.222	1.734	1.085
1660		-----	-----	-----	-----	-----	-----	-----
1702	IEC61619	ND	2.96 R(1)	0.4 ex	0.94 ex	4.08 R(1)	5.72 R(1)	3.53 R(1)
1743		-----	-----	-----	-----	-----	-----	-----
1801		-----	-----	-----	-----	-----	-----	-----
1816	IEC61619	0.39	1.15	1.75	<1	1.88	2.36 R(5)	1.39
1841	IEC61619	0.00 R(5)	0.00 R(5)	1.46	0.64	1.80	1.32	1.37
1875	EN12766-1	0.0758	0.6710 R(1)	1.1750	-----	1.5600	1.7075	1.0780
1885		-----	-----	-----	-----	-----	-----	-----
1888		-----	-----	-----	-----	-----	-----	-----
2493	EN12766-1	0.285	0.516	0.796	0.454	1.312	0.786 R(5)	0.742
6067	IEC61619	0.2531	0.7639 C	1.2360	0.8639 DG(5)	1.7382	1.7397	1.0808
6106	EN12766-1	0.250	0.694	0.983	0.437	1.33	1.57	1.03
6141		-----	-----	-----	-----	-----	-----	-----
6146	EN12766-1	0.1883	0.4277	0.8044	0.3652	0.9913	1.2687	0.7635
6165		-----	-----	-----	-----	-----	-----	-----
6254		-----	0.73	1.96	-----	1.24	1.81	1.02
6278		-----	-----	-----	-----	-----	-----	-----
6283	IEC61619	0.23	0.98	1.57	0.69	1.63	1.79	1.11
	normality	OK	OK	OK	OK	OK	OK	OK
	n	23	23	25	17	25	23	25
	outliers	1	3	0 (+1ex)	2 (+1ex)	1	3	1
	mean (n)	0.2112	0.6692	1.2070	0.5258	1.4089	1.5411	1.0311
	st.dev. (n)	0.08033	0.12216	0.32668	0.09834	0.24635	0.20093	0.15383
	R(calc.)	0.2249	0.3420	0.9147	0.2754	0.6898	0.5626	0.4307
	st.dev.(EN12766-1:00)	0.02999	0.11209	0.20849	0.08639	0.24469	0.26838	0.17697
	R(EN12766-1:00)	0.0840	0.3138	0.5838	0.2419	0.6851	0.7515	0.4955

Lab 902 first reported for PCB180: 0.4702, Lab 1059 first reported for PCB118: 1.59, Lab 6067 first reported for PCB52: 1.0981






PCB52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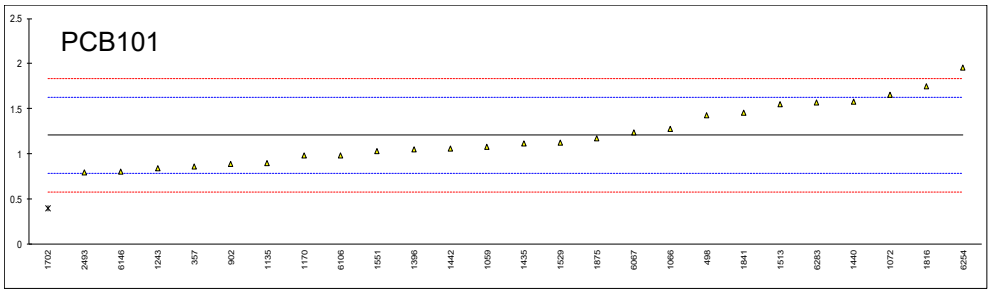
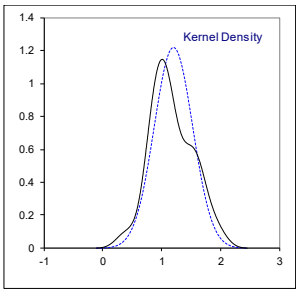
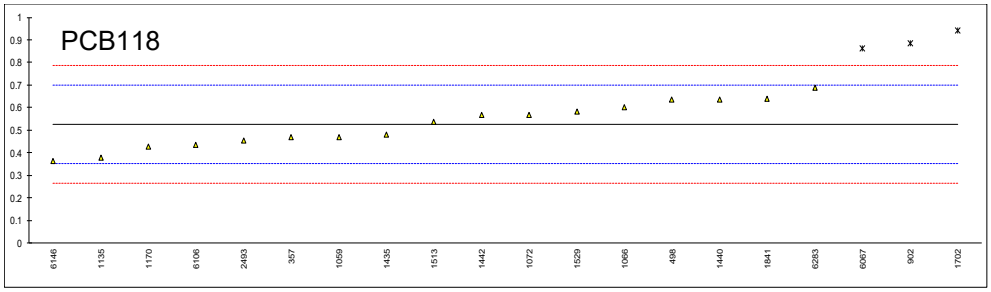
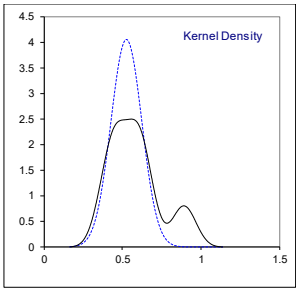
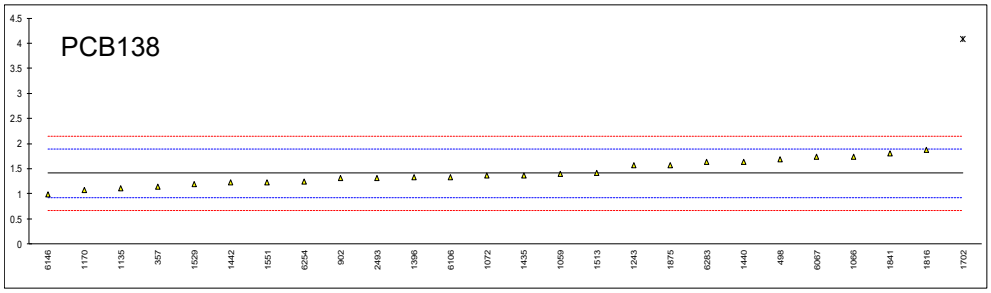
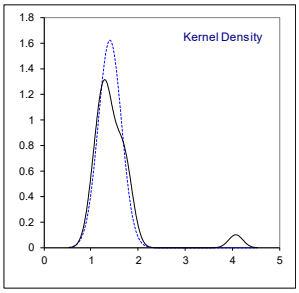

PCB101

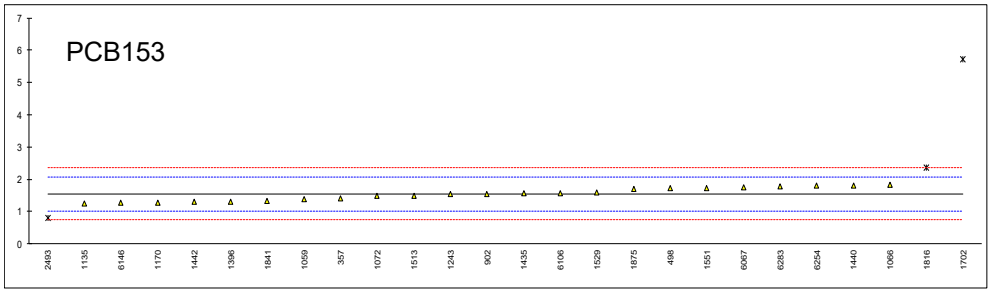

Kernel Density

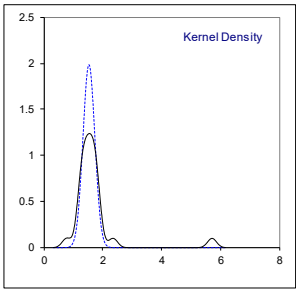

PCB118

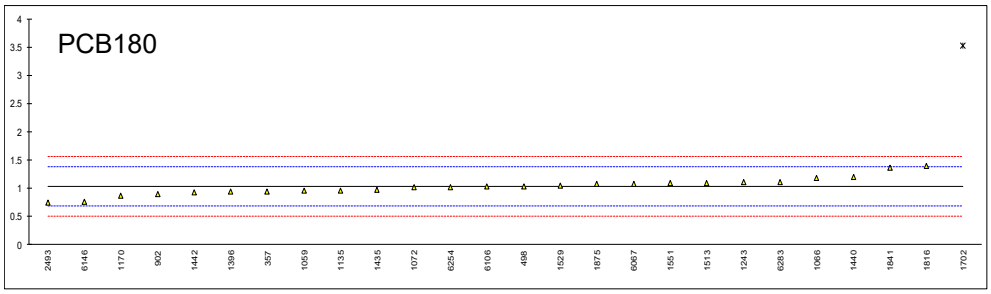

Kernel Density

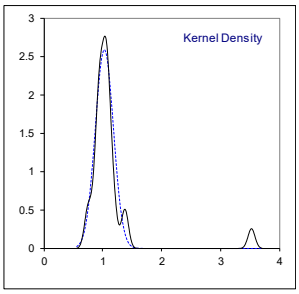

PCB138


Kernel Density


PCB153


Kernel Density

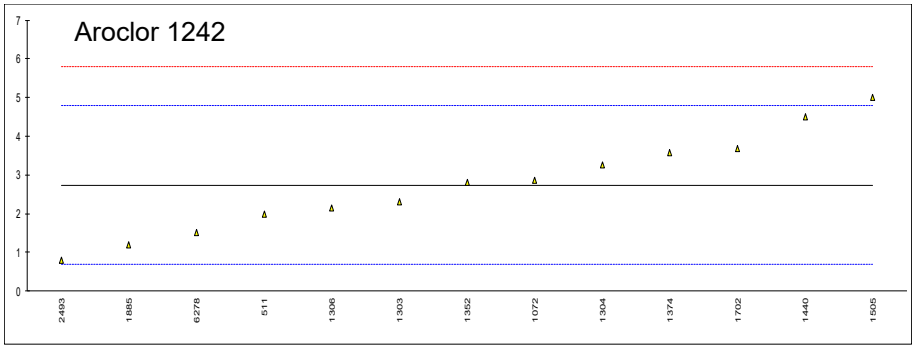

PCB180

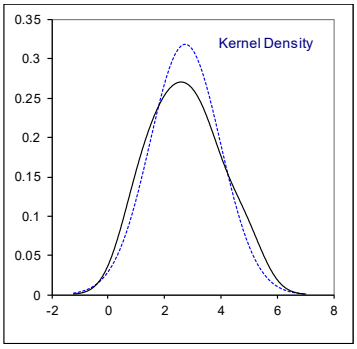

Kernel Density

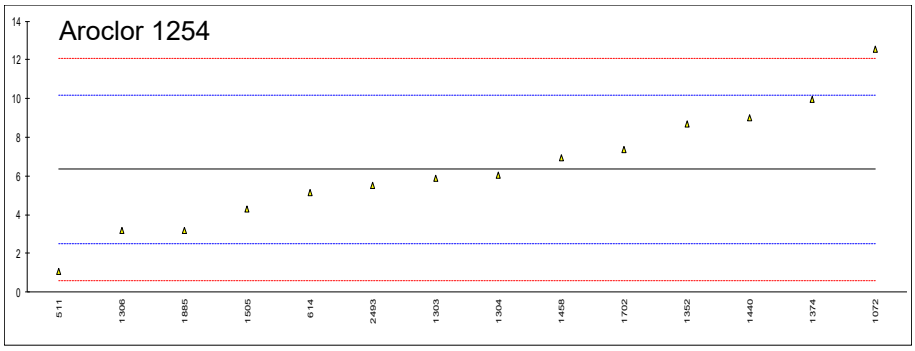
Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #19243; results in mg/kg.

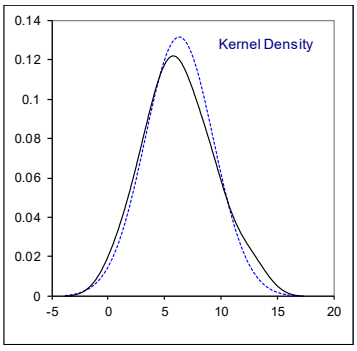
lab	method	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341		-----	-----	-----	-----
343		-----	-----	-----	-----
357		-----	-----	-----	-----
398		-----	-----	-----	-----
498		-----	-----	-----	-----
511	D4059	1.99	-----	1.06	10.98
614	D4059	<2	-----	5.14	4.25
902		-----	-----	-----	-----
912		-----	-----	-----	-----
974		-----	-----	-----	-----
1059		-----	-----	-----	-----
1066		-----	-----	-----	-----
1072	D4059	2.858	-----	12.529	5.883
1126		-----	-----	-----	-----
1135		-----	-----	-----	-----
1170	D4059	-----	-----	-----	-----
1243		-----	-----	-----	-----
1303	D4059	2.31	-----	5.90	6.29
1304	INH-127	3.2635	-----	6.0382	9.4204
1306	EPA600	2.15	-----	3.20	6.46
1352	in house	2.81	-----	8.66	8.25
1367		-----	-----	-----	-----
1374	D4059	3.57	-----	9.93	8.23
1396		-----	-----	-----	-----
1435		-----	-----	-----	-----
1440	in house	4.5	-----	9	12.5
1442		-----	-----	-----	-----
1458	D4059	<3	-----	6.93	10.19
1495		-----	-----	-----	-----
1505	D4059	5.0	-----	4.3	13.8
1513		-----	-----	-----	-----
1529		-----	-----	-----	-----
1551		-----	-----	-----	-----
1660		-----	-----	-----	-----
1702	IEC61619	3.69	-----	7.38	14.78
1743		-----	-----	-----	-----
1801		-----	-----	-----	-----
1816		-----	-----	-----	-----
1841		-----	-----	-----	-----
1875		-----	-----	-----	-----
1885		1.2	0	3.2	2.8
1888		-----	-----	-----	-----
2493		0.8	1.8	5.5	14.88
6067		-----	-----	-----	-----
6106		-----	-----	-----	-----
6141		-----	-----	-----	-----
6146		-----	-----	-----	-----
6165		-----	-----	-----	-----
6254		-----	-----	-----	-----
6278	D4059	1.5 C	-----	-----	12.5
6283		-----	-----	-----	-----
	normality	OK	n.a.	OK	OK
	n	13	2	14	15
	outliers	0	n.a.	0	0
	mean (n)	2.7417	<2	6.3405	9.4142
	st.dev. (n)	1.25359	n.a.	3.02895	3.81848
	R(calc.)	3.5100	n.a.	8.48106	10.6918
	st.dev.(D4059:00 (silicone))	1.01966	n.a.	1.912230	2.57208
	R(D4059:00 (silicone))	2.8551	n.a.	5.35424	7.2018

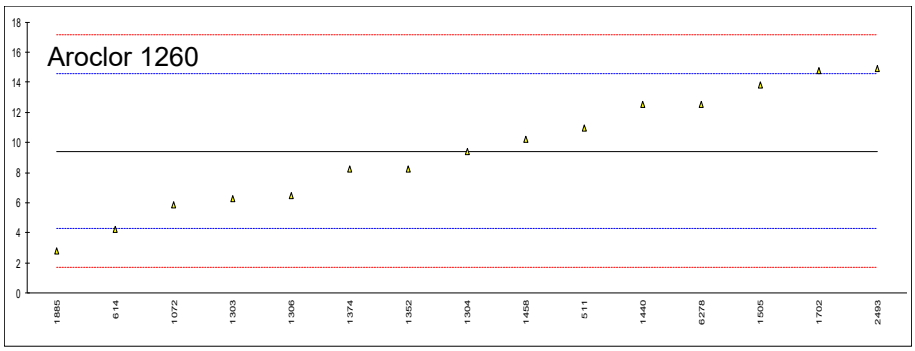
Lab 6278 first reported: 10.8

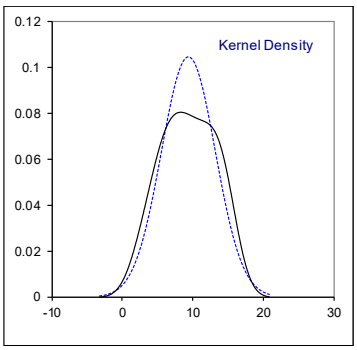

Aroclor 1242
2493
1885
6278
511
1306
1303
1352
1072
1304
1374
1702
1440
1505


Kernel Density


Aroclor 1254
511
1306
1885
1505
614
2493
1303
1304
1458
1702
1352
1440
1374
1072


Kernel Density

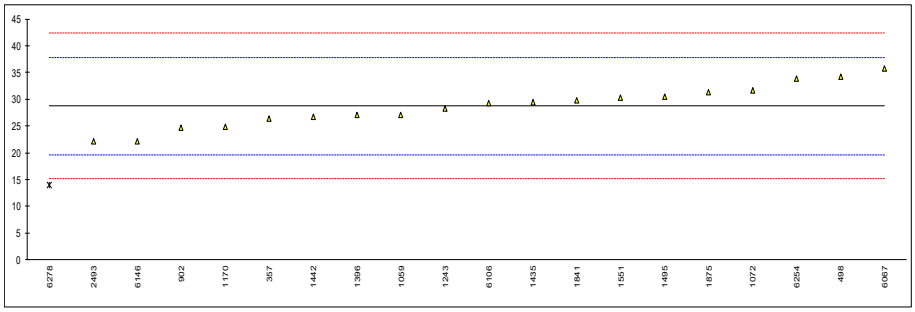

Aroclor 1260
1885
614
1072
1303
1306
1374
1352
1304
1458
511
1440
6278
1505
1702
2493

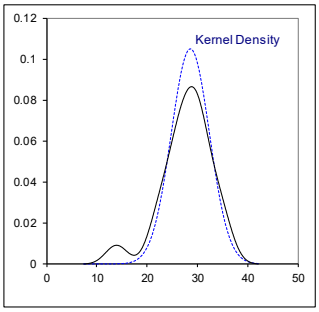

Kernel Density

Determination of Total PCB, 5 times the sum of 6 congeners on sample #19243; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	26.38		-0.51	
398		-----		-----	
498	EN12766-2-B	34.275		1.23	
511		-----		-----	
614		-----		-----	
902	EN12766-2-B	24.602		-0.90	
912		-----		-----	
974		-----		-----	
1059	EN12766-2-B	27.1		-0.35	
1066		-----		-----	
1072	EN12766-2-B	31.648		0.65	
1126		-----		-----	
1135		-----		-----	
1170	EN12766-2-B	24.791		-0.86	
1243	EN12766-2-B	28.25		-0.10	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	26.9878		-0.38	
1435	EN12766-2-B	29.45		0.17	
1440		-----		-----	
1442	EN12766-2-B	26.798		-0.42	
1458		-----		-----	
1495	EN12766-2-B	30.4		0.38	
1505		-----		-----	
1513		-----		-----	
1529		-----		-----	
1551	IP462-2	30.306		0.35	
1660		-----		-----	
1702		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	29.80		0.24	
1875	EN12766-2-B	31.3		0.57	
1885		-----		-----	
1888		-----		-----	
2493	EN12766-2-B	22.185		-1.44	
6067	IEC61619	35.73		1.55	
6106	EN12766-2-B	29.3		0.13	
6141		-----		-----	
6146	EN12766-2-B	22.219		-1.43	
6165		-----		-----	
6254	EN12766-2-B	33.78		1.12	
6278		14	R(0.05)	-3.24	
6283		-----		-----	

normality	OK
n	19
outliers	1
mean (n)	28.7001
st.dev. (n)	3.80480
R(calc.)	10.6534
st.dev.(EN12766-2B:01)	4.53222
R(EN12766-2B:01)	12.6902

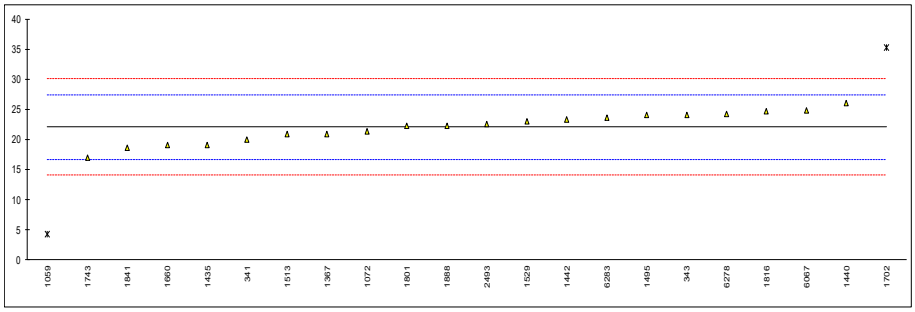


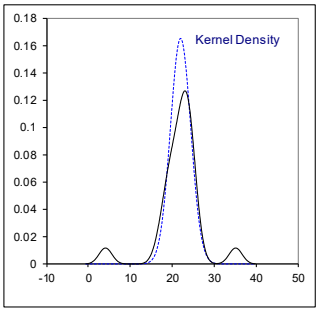


Determination of Total PCB, sum of all congeners on sample #19243; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	20		-0.77	
343	EN61619	24		0.72	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
974		-----		-----	
1059	EN12766-2A	4.19	R(0.01)	-6.66	
1066		-----		-----	
1072	EN61619	21.2709		-0.30	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1367	EN61619	20.95		-0.42	
1374		-----		-----	
1396		-----		-----	
1435	IEC61619	19.04		-1.13	
1440	IEC61619	26		1.46	
1442	IEC61619	23.288		0.45	
1458		-----		-----	
1495	EN12766-2A	24.0		0.72	
1505		-----		-----	
1513	IEC61619	20.896		-0.44	
1529	EN12766-1+EN61619	23		0.34	
1551		-----		-----	
1660	IEC61619	19		-1.15	
1702	IEC61619	35.19	R(0.01)	4.88	
1743	IEC61619	17		-1.89	
1801	EN61619	22.18		0.04	
1816	IEC61619	24.7		0.98	
1841	EN61619	18.68		-1.26	
1875		-----		-----	
1885		-----		-----	
1888	EN61619	22.3	C	0.08	first reported: 11.8
2493	EN61619	22.582	C	0.19	first reported: 4.891
6067	IEC61619	24.83		1.03	
6106		-----		-----	
6141		-----		-----	
6146		-----		-----	
6165		-----		-----	
6254		-----		-----	
6278	EPA8082A	24.2		0.79	
6283	IEC61619	23.61		0.57	

normality	OK
n	20
outliers	2
mean (n)	22.0763
st.dev. (n)	2.41019
R(calc.)	6.7485
st.dev.(EN61619:99)	2.68539
R(EN61619:99)	7.5191

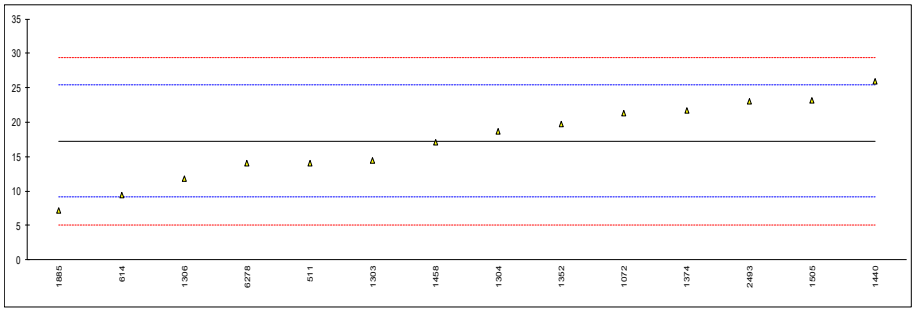


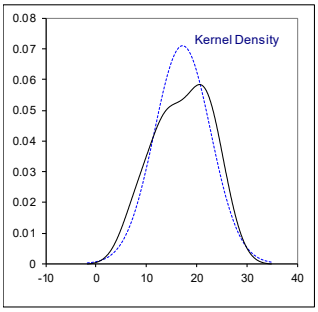


Determination of Total PCB, sum of all Aroclors on sample #19243; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511	D4059	14.02		-0.80	
614	D4059	9.39		-1.94	
902		-----		-----	
912		-----		-----	
974		-----		-----	
1059		-----		-----	
1066		-----		-----	
1072	D4059	21.271		0.99	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1303	D4059	14.50		-0.68	
1304	INH-127	18.7219		0.36	
1306	EPA600	11.80		-1.35	
1352	in house	19.71		0.61	
1367		-----		-----	
1374	D4059	21.73		1.11	
1396		-----		-----	
1435		-----		-----	
1440	in house	26		2.16	
1442		-----		-----	
1458	D4059	17.12		-0.03	
1495		-----		-----	
1505	D4059	23.1		1.44	
1513		-----		-----	
1529		-----		-----	
1551		-----		-----	
1660		-----		-----	
1702		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885	EPA6013	7.2		-2.48	
1888		-----		-----	
2493	D4059	22.98		1.41	
6067		-----		-----	
6106		-----		-----	
6141		-----		-----	
6146		-----		-----	
6165		-----		-----	
6254		-----		-----	
6278	D4059	14	C	-0.80	first reported: 23.3
6283		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	17.2530
st.dev. (n)	5.60700
R(calc.)	15.7079
st.dev.(D4059:00 (silicone))	4.05132
R(D4059:00 (silicone))	11.3437





APPENDIX 2

z-scores of PCB 28, 52, 101, 118, 138, 153 and 180 determination on sample #19243;

lab	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341	-----	-----	-----	-----	-----	-----	-----
343	-----	-----	-----	-----	-----	-----	-----
357	0.29	0.28	-1.66	-0.65	-1.10	-0.49	-0.51
398	-----	-----	-----	-----	-----	-----	-----
498	-0.54	1.10	1.07	1.26	1.12	0.67	0.02
511	-----	-----	-----	-----	-----	-----	-----
\614	-----	-----	-----	-----	-----	-----	-----
902	-5.04	-0.22	-1.51	4.14	-0.40	0.01	-0.76
912	-----	-----	-----	-----	-----	-----	-----
974	-----	-----	-----	-----	-----	-----	-----
1059	-2.37	-0.53	-0.61	-0.65	-0.04	-0.60	-0.46
1066	2.29	1.52	0.35	0.86	1.35	1.11	0.84
1072	-1.23	-0.22	2.14	0.48	-0.22	-0.19	-0.11
1126	-----	-----	-----	-----	-----	-----	-----
1135	-2.71	-0.97	-1.47	-1.69	-1.26	-1.12	-0.46
1170	-0.57	-0.96	-1.08	-1.16	-1.36	-0.95	-0.98
1243	-2.71	-1.69	-1.76	-----	0.62	-0.04	0.45
1303	-----	-----	-----	-----	-----	-----	-----
1304	-----	-----	-----	-----	-----	-----	-----
1306	-----	-----	-----	-----	-----	-----	-----
1352	-----	-----	-----	-----	-----	-----	-----
1367	-----	-----	-----	-----	-----	-----	-----
1374	-----	-----	-----	-----	-----	-----	-----
1396	0.15	-0.94	-0.74	-----	-0.34	-0.89	-0.53
1435	-1.37	0.36	-0.42	-0.53	-0.20	0.07	-0.35
1440	3.29	0.84	1.80	1.27	0.95	1.02	0.96
1442	-0.91	0.08	-0.69	0.47	-0.77	-0.94	-0.60
1458	-----	-----	-----	-----	-----	-----	-----
1495	-----	-----	-----	-----	-----	-----	-----
1505	-----	-----	-----	-----	-----	-----	-----
1513	-1.41	-0.05	1.66	0.15	0.05	-0.18	0.36
1529	2.09	0.66	-0.38	0.67	-0.91	0.24	0.11
1551	4.26	-0.15	-0.86	-----	-0.76	0.72	0.30
1660	-----	-----	-----	-----	-----	-----	-----
1702	-----	20.44	-3.87	4.79	10.92	15.57	14.12
1743	-----	-----	-----	-----	-----	-----	-----
1801	-----	-----	-----	-----	-----	-----	-----
1816	5.96	4.29	2.60	-----	1.93	3.05	2.03
1841	-7.04	-5.97	1.21	1.32	1.60	-0.82	1.91
1875	-4.51	0.02	-0.15	-----	0.62	0.62	0.26
1885	-----	-----	-----	-----	-----	-----	-----
1888	-----	-----	-----	-----	-----	-----	-----
2493	2.46	-1.37	-1.97	-0.83	-0.40	-2.81	-1.63
6067	1.40	0.85	0.14	3.91	1.35	0.74	0.28
6106	1.29	0.22	-1.07	-1.03	-0.32	0.11	-0.01
6141	-----	-----	-----	-----	-----	-----	-----
6146	-0.76	-2.15	-1.93	-1.86	-1.71	-1.01	-1.51
6165	-----	-----	-----	-----	-----	-----	-----
6254	-----	0.54	3.61	-----	-0.69	1.00	-0.06
6278	-----	-----	-----	-----	-----	-----	-----
6283	0.63	2.77	1.74	1.90	0.90	0.93	0.45

z-scores of Aroclor 1242, 1248, 1254 and 1260 determination on sample #19243;

lab	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341	-----	-----	-----	-----
343	-----	-----	-----	-----
357	-----	-----	-----	-----
398	-----	-----	-----	-----
498	-----	-----	-----	-----
511	-0.74	-----	-2.76	0.61
614	-----	-----	-0.63	-2.01
902	-----	-----	-----	-----
912	-----	-----	-----	-----
974	-----	-----	-----	-----
1059	-----	-----	-----	-----
1066	-----	-----	-----	-----
1072	0.11	-----	3.24	-1.37
1126	-----	-----	-----	-----
1135	-----	-----	-----	-----
1170	-----	-----	-----	-----
1243	-----	-----	-----	-----
1303	-0.42	-----	-0.23	-1.21
1304	0.51	-----	-0.16	0.00
1306	-0.58	-----	-1.64	-1.15
1352	0.07	-----	1.21	-0.45
1367	-----	-----	-----	-----
1374	0.81	-----	1.88	-0.46
1396	-----	-----	-----	-----
1435	-----	-----	-----	-----
1440	1.72	-----	1.39	1.20
1442	-----	-----	-----	-----
1458	-----	-----	0.31	0.30
1495	-----	-----	-----	-----
1505	2.21	-----	-1.07	1.71
1513	-----	-----	-----	-----
1529	-----	-----	-----	-----
1551	-----	-----	-----	-----
1660	-----	-----	-----	-----
1702	0.93	-----	0.54	2.09
1743	-----	-----	-----	-----
1801	-----	-----	-----	-----
1816	-----	-----	-----	-----
1841	-----	-----	-----	-----
1875	-----	-----	-----	-----
1885	-1.51	-----	-1.64	-2.57
1888	-----	-----	-----	-----
2493	-1.90	-----	-0.44	2.13
6067	-----	-----	-----	-----
6106	-----	-----	-----	-----
6141	-----	-----	-----	-----
6146	-----	-----	-----	-----
6165	-----	-----	-----	-----
6254	-----	-----	-----	-----
6278	-1.22	-----	-----	1.20
6283	-----	-----	-----	-----

APPENDIX 3

Number of participating laboratories per country

7 labs in	AUSTRALIA
1 lab in	AUSTRIA
2 labs in	BELGIUM
1 lab in	ESTONIA
2 labs in	FINLAND
3 labs in	FRANCE
4 labs in	GERMANY
1 lab in	GREECE
1 lab in	HUNGARY
1 lab in	INDIA
3 labs in	ITALY
1 lab in	MALAYSIA
1 lab in	MOROCCO
3 labs in	NETHERLANDS
1 lab in	NORWAY
1 lab in	PERU
1 lab in	PHILIPPINES
1 lab in	PORTUGAL
1 lab in	QATAR
1 lab in	SAUDI ARABIA
1 lab in	SLOVENIA
1 lab in	SOUTH AFRICA
6 labs in	SPAIN
1 lab in	TURKEY
1 lab in	UNITED ARAB EMIRATES
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)	= 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	= possibly an error in calculations
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

Literature:

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