Results of Proficiency Test
PCBs in Mineral Oil
November 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. E.R. Montenij-Bos
Correctors: ing. R.J. Starink & ing. A.S. Noordman-de Neef
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1 INTRODUCTION

Since 2001 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Poly Chlorinated Biphenyls (PCBs) in Mineral Oil every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of PCBs in Mineral Oil.

In this interlaboratory study 55 laboratories in 25 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 #21243.
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 PCBs was obtained from a third-party laboratory. After homogenization 71 amber glass vials of 8 mL were filled and labelled #21243.
The homogeneity of the subsamples was checked by determination of Total Organic Chlorides content in accordance with UOP779 on 8 stratified randomly selected subsamples.

	Total Organic Chlorides as Cl in mg/kg
sample #21243-1	35.3
sample #21243-2	35.3
sample #21243-3	35.2
sample #21243-4	35.1
sample #21243-5	35.2
sample #21243-6	35.4
sample #21243-7	35.3
sample #21243-8	35.0

Table 1: homogeneity test results of subsamples of #21243

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

	Total Organic Chlorides as Cl in mg/kg
r (observed)	0.36
reference test method	UOP779:08
0.3 x R (reference test method)	1.79

Table 2: evaluation of the repeatability of subsamples #21243

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

To each of the participating laboratories one sample PCB in Mineral Oil labelled #21243 was sent on October 27, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the PCB in Mineral Oil 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 #21243: 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 only 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 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in 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. Three 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 51 laboratories reported 288 numerical test results. Observed were 11 outlying test results, which is 3.8%. 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 ASTM 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) will be 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 reproducibility of the total PCB content is mentioned while in EN12766-1:00 / IP462-1:01 the reproducibilities for individual congeners are mentioned.

sample #21243

Total Organohalogenic Compounds TOX as Cl: This determination may be problematic. Only four test results were reported. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of UOP779:08.

Poly Chlorinated Biphenyls as PCB: This determination was problematic. In total eight statistical outliers were observed over seven congeners (no. 28, 52, 101,118, 138,153,180). The calculated reproducibilities after rejection of the statistical outliers are not in agreement with the requirements of EN12766-1:00 / IP462-1:01.

Poly Chlorinated Biphenyls as Aroclor: This determination was problematic. No statistical outliers were observed. 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 no test results were reported.

Total PCB, 5 times the sum of 6 PCB congeners: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12766-2 test method B:01.

Total PCB, sum of all congeners: Some participants reported the sum of the seven congeners which are asked in the PT while the total sum PCB of **all** congeners present in the PT sample is requested for this parameter. Therefore, the test results based on the sum of the reported congeners are excluded from the statistical analysis.
This determination was problematic. Three statistical outliers were observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with

the requirements of EN61619:99 and EN12766-2 test method A:01 as this test method is identical to EN61619:99.

Total PCB, sum of all Aroclors: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D4059:00(2018).

All participants agree that sample #21243 is positive on PCBs. From the data on total organic halogenic components (TOX) an average concentration of 37.5 mg/kg was found in this PT. From this concentration, a total content of 75.7 mg PCB/kg is estimated using an average Cl content of 49.6%, assuming the presence of 45.8% Aroclor 1242 (42% Cl), 36.2% Aroclor 1254 (54% Cl) and 18.0% 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	75.7
5 times the sum of 6 congeners	50.4
sum of all congeners	57.3
sum of all Aroclors	46.3

Table 3: comparison of estimations of total PCB content in sample #21243

The total PCB content is in agreement with the calculation of 5 times the sum of 6 congeners, sum of all congeners and sum of all Aroclor. The estimate from TOX as Cl is much higher.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the 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 ASTM or EN test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX as Cl	mg/kg	4	37.5	14.6	6.4
PCB no. 28	mg/kg	26	1.61	1.42	0.79
PCB no. 52	mg/kg	28	1.61	1.75	0.79
PCB no. 101	mg/kg	28	2.27	2.15	1.12
PCB no. 118	mg/kg	17	1.51	1.33	0.74
PCB no. 138	mg/kg	25	2.18	1.73	1.07
PCB no. 153	mg/kg	28	1.86	1.61	0.91
PCB no. 180	mg/kg	26	0.95	0.71	0.45
Aroclor 1242	mg/kg	12	22.3	25.4	13.8
Aroclor 1248	mg/kg	1	<1	n.e.	n.e.
Aroclor 1254	mg/kg	12	17.6	18.1	11.5
Aroclor 1260	mg/kg	12	8.80	8.50	6.85

Parameter	unit	n	average	2.8 * sd	R(lit)
Total PCB, 5 x sum 6 congeners	mg/kg	25	50.4	37.6	22.6
Total PCB, sum of all congeners	mg/kg	17	57.3	24.2	16.3
Total PCB, sum of Aroclors	mg/kg	13	46.3	45.4	23.8

Table 4: reproducibilities of tests on sample #21243

Without further statistical calculations it can be concluded that 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 2021 PROFICIENCY TEST WITH PREVIOUS PTS

	November 2021	November 2020	November 2019	November 2018	November 2017
Number of reporting laboratories	51	45	45	45	50
Number of test results	288	251	277	247	275
Number of statistical outliers	11	9	14	13	16
Percentage of statistical outliers	3.8%	3.6%	5.1%	5.3%	5.8%

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 2021	November 2020	November 2019	November 2018	November 2017
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:

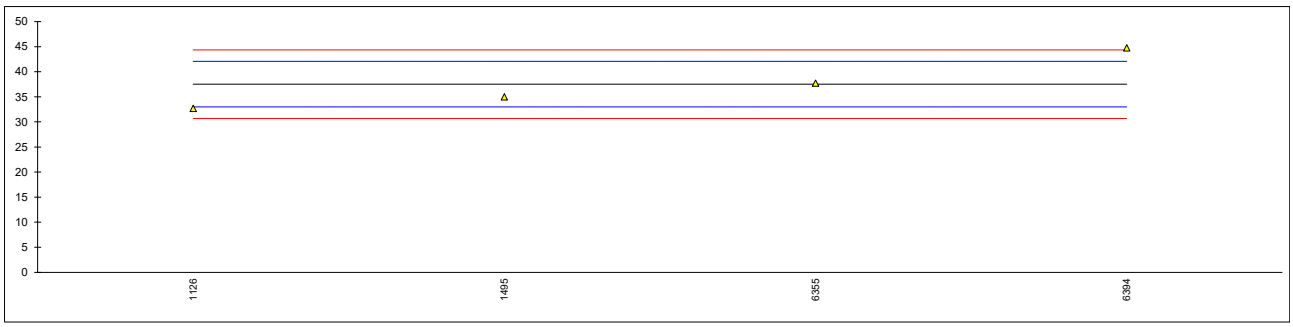
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

APPENDIX 1

Determination of Total Organohalogenic Compounds (TOX) as Cl on sample #21243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
392		-----		-----	
398		-----		-----	
455		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072		-----		-----	
1126	EN14077	32.69		-2.13	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1374		-----		-----	
1396		-----		-----	
1397		-----		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495	EN14077	35		-1.11	
1505		-----		-----	
1513		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885		-----		-----	
1888		-----		-----	
1912		-----		-----	
6067		-----		-----	
6275		-----		-----	
6278		-----		-----	
6283		-----		-----	
6334		-----		-----	
6335		-----		-----	
6352		-----		-----	
6355	In house	37.7		0.07	
6382		-----		-----	
6394		44.75		3.17	
6414		-----		-----	
6417		-----		-----	
6427		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	37.535
st.dev. (n)	5.2276
R(calc.)	14.637
st.dev.(UOP779:08)	2.2741
R(UOP779:08)	6.368

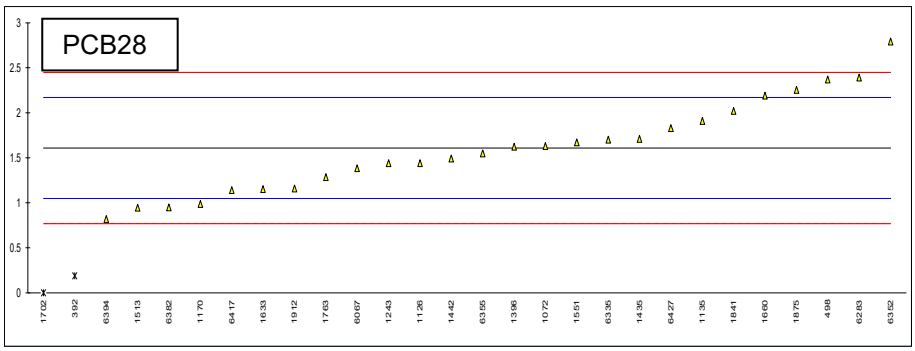

50
45
40
35
30
25
20
15
10
5
0
1126
1495
6355
6394

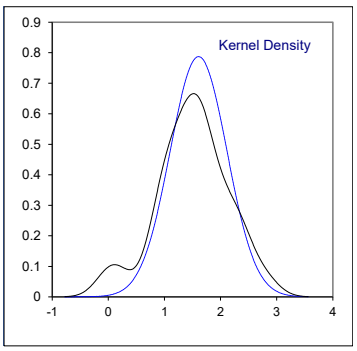
Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #21243; results in mg/kg

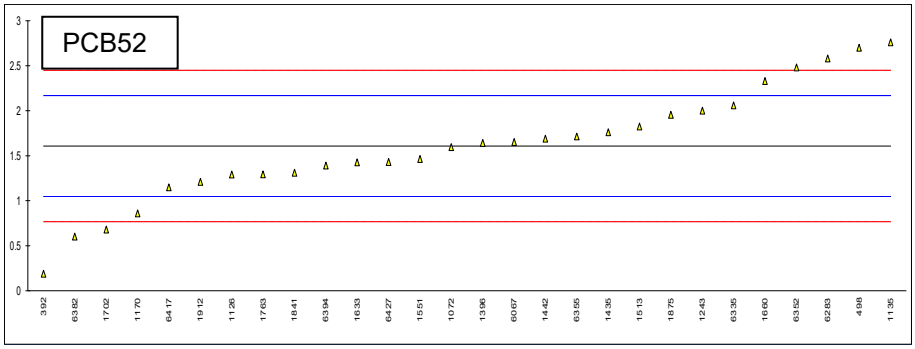
lab	method	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341		-----	-----	-----	-----	-----	-----	-----
343		-----	-----	-----	-----	-----	-----	-----
357		-----	-----	-----	-----	-----	-----	-----
392	EN12766-1	***0.18842***	0.18842	1.88419	-----	***4.73559***	1.54503	***2.22334***
398		-----	-----	-----	-----	-----	-----	-----
455		-----	-----	-----	-----	-----	-----	-----
498	EN12766-1	2.37	2.70	3.15	2.09	3.21	2.48	1.30
511		-----	-----	-----	-----	-----	-----	-----
614		-----	-----	-----	-----	-----	-----	-----
902		-----	-----	-----	-----	-----	-----	-----
912		-----	-----	-----	-----	-----	-----	-----
1059		-----	-----	-----	-----	-----	-----	-----
1072	EN12766-1	1.6305	1.5957	2.5588	1.3465	2.1414	2.0255	0.8927
1126	EN12766-1	1.44	1.29	2.12	1.35	2.01	1.54	0.84
1135	EN12766-1	1.91	2.76	3.10	***3.29***	1.61	1.70	0.84
1170	EN12766-1	0.987	0.860	1.175	0.812	1.156	1.031	0.593
1243	EN12766-1	1.44	2.00	1.96	-----	1.57	1.88	1.12
1304		-----	-----	-----	-----	-----	-----	-----
1306		-----	-----	-----	-----	-----	-----	-----
1352		-----	-----	-----	-----	-----	-----	-----
1374		-----	-----	-----	-----	-----	-----	-----
1396	IP462-1	1.622	1.640	1.986	-----	2.270	2.582	1.0419
1397		-----	-----	-----	-----	-----	-----	-----
1435	EN12766-1	1.71	1.76	2.13	1.38	1.97	1.78	0.91
1440		-----	-----	-----	-----	-----	-----	-----
1442	EN12766-1	1.49	1.69	2.10	1.91	2.10	2.04	1.02
1458		-----	-----	-----	-----	-----	-----	-----
1495		-----	-----	-----	-----	-----	-----	-----
1505		-----	-----	-----	-----	-----	-----	-----
1513	IEC61619	0.944 C	1.824	2.533	1.268	2.443	1.766	1.103
1551	IP462-1	1.6701	1.4625	0.9641	-----	2.4655	2.9532	0.9950
1633	IEC61619	1.151	1.427	2.652	1.770	2.468	2.113	1.025
1660	IEC61619	2.19	2.33	2.92	1.35	2.75	2.24	1.25
1702	IEC61619	***0***	0.68	1.03	0.91	***0***	1.41	***0***
1763	EN12766-1	1.286	1.292	3.267	-----	1.746	1.775	0.703
1801		-----	-----	-----	-----	-----	-----	-----
1816		-----	-----	-----	-----	-----	-----	-----
1841	IEC61619	2.02	1.31	2.60	2.13	2.91	1.50	1.30
1875	EN12766-1	2.254	1.955	2.403	-----	2.602	2.023	0.869
1885		-----	-----	-----	-----	-----	-----	-----
1888		-----	-----	-----	-----	-----	-----	-----
1912	EN12766-1	1.15905	1.20841	1.85682	1.15989	1.99353	1.44234	0.84887
6067	EN12766-1	1.384	1.652	1.227	1.186	2.025	1.417	0.808
6275		-----	-----	-----	-----	-----	-----	-----
6278		-----	-----	-----	-----	-----	-----	-----
6283		2.39	2.58	3.09	2.17	2.61	2.17	1.09
6334		-----	-----	-----	-----	-----	-----	-----
6335	EN12766-1	1.70	2.06	3.24	-----	2.14	1.16	0.90
6352	DIN51527Mod.	2.79	2.48	3.33	2.40	3.69	3.04	1.62
6355	EN12766-1	1.549	1.714	2.661	1.460	2.491	2.267	1.019
6382	EN12766-1	0.948	0.602	1.036	-----	0.982	0.822	0.472
6394		0.82 C	1.39 C	1.77 C	-----	1.5 C	1.256 C	0.866 C
6414		-----	-----	-----	-----	-----	-----	-----
6417	EN12766-1	1.14	1.15	1.49	1.05	1.69	1.32	0.75
6427	EN12766-1	1.83	1.43	3.38	-----	***0.26***	2.88	0.53
	normality	OK	OK	OK	OK	OK	OK	OK
	n	26	28	28	17	25	28	26
	outliers	2	0	0	1	3	0	2
	mean (n)	1.6086	1.6083	2.2719	1.5143	2.1817	1.8628	0.9502
	st.dev. (n)	0.50672	0.62526	0.76751	0.47553	0.61621	0.57587	0.25223
	R(calc.)	1.4188	1.7507	2.1490	1.3315	1.7254	1.6124	0.7062
	st.dev.(EN12766-1:00)	0.28049	0.28042	0.39939	0.26357	0.38322	0.32605	0.16247
	R(EN12766-1:00)	0.7854	0.7852	1.1183	0.7380	1.0730	0.9129	0.4549

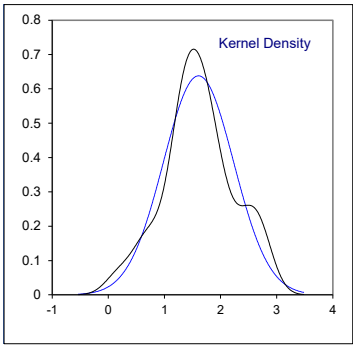
Underlined, Italic and bold test results are statistical outliers according Grubbs outlier test.

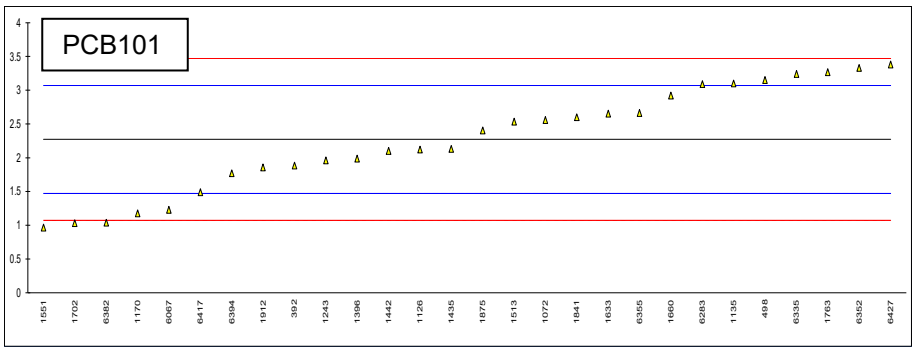
Lab 1513 first reported <0.2 PCB28
Lab 6394 first reported 4.10 PCB28, 6.95 PCB52, 8.87 PCB101, 7.51 PCB138, 6.28 PCB153, 4.33 PCB180

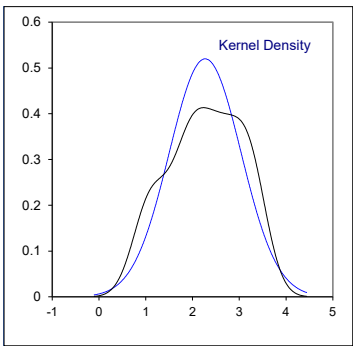

PCB28

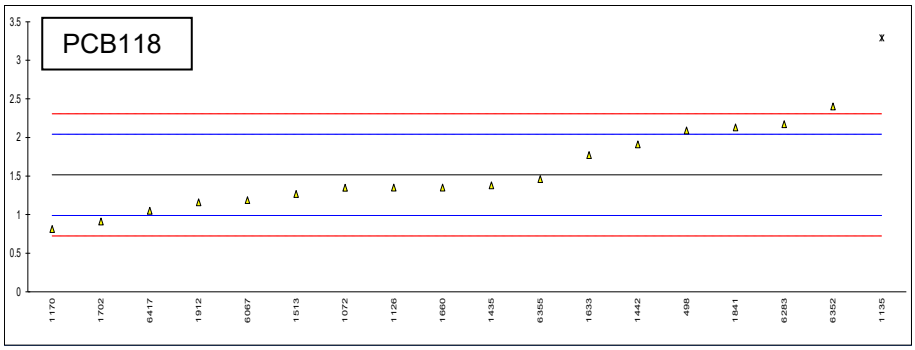

Kernel Density

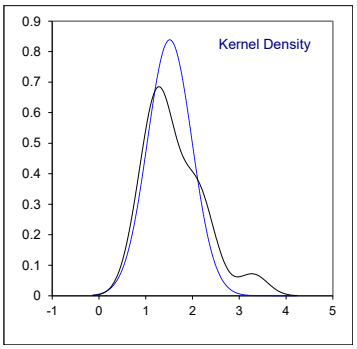

PCB52

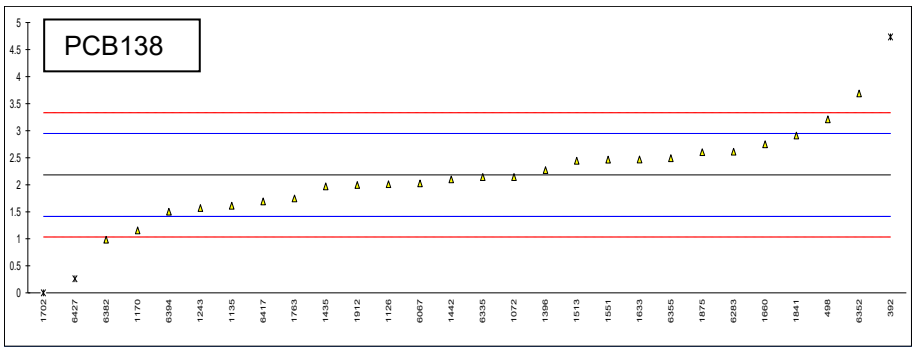

Kernel Density

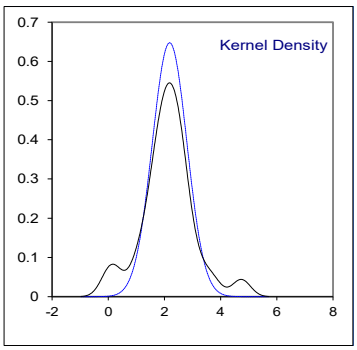

PCB101

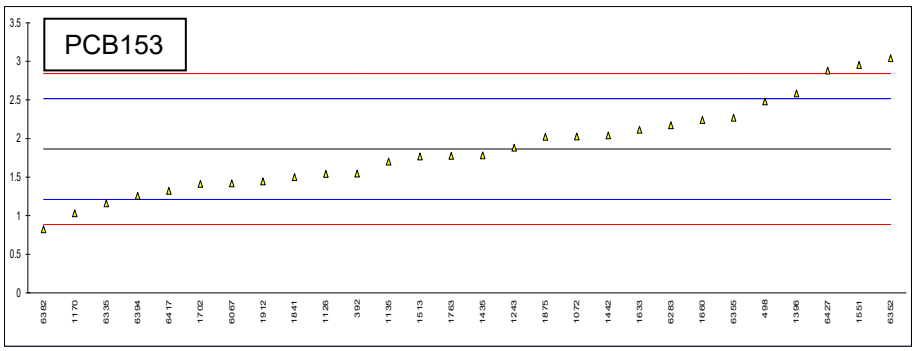

Kernel Density

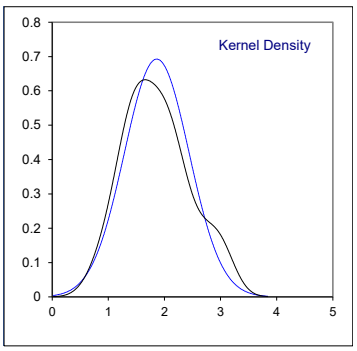

PCB118

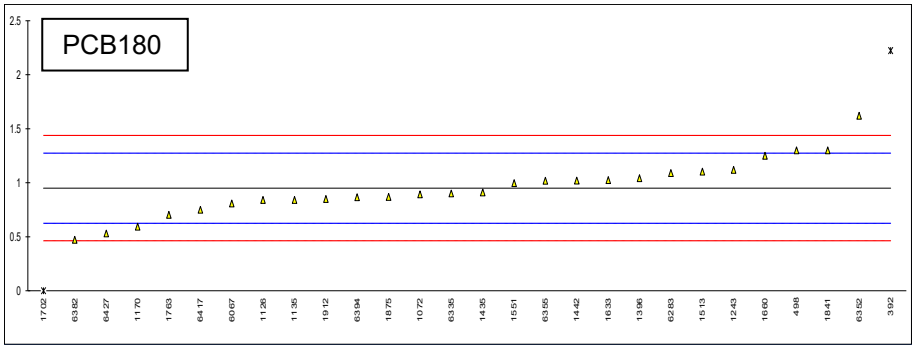

Kernel Density

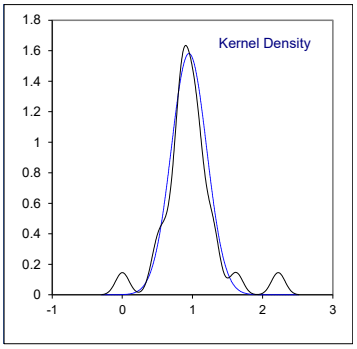

PCB138


Kernel Density


PCB153
3.5
3
2.5
2
1.5
1
0.5
0
63.82
11.70
63.35
63.94
64.17
17.02
60.67
19.12
18.41
11.26
3.92
11.35
15.13
17.63
14.35
12.43
18.75
10.72
14.42
16.33
62.83
16.60
63.55
4.98
13.96
64.27
15.51
63.52


Kernel Density
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
1
2
3
4
5

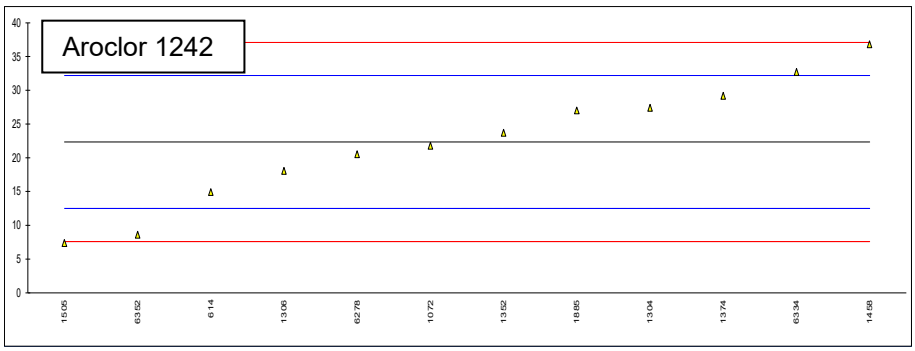

PCB180
2.5
2
1.5
1
0.5
0
17.02
63.82
64.27
11.70
17.63
64.17
60.67
11.26
11.35
19.12
63.94
18.75
10.72
63.35
14.35
15.51
63.55
14.42
16.33
13.96
62.83
15.13
12.43
16.60
4.98
18.41
63.52
3.92

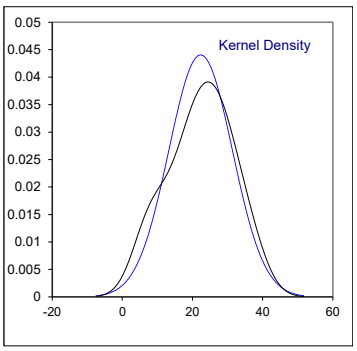

Kernel Density
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
-1
0
1
2
3

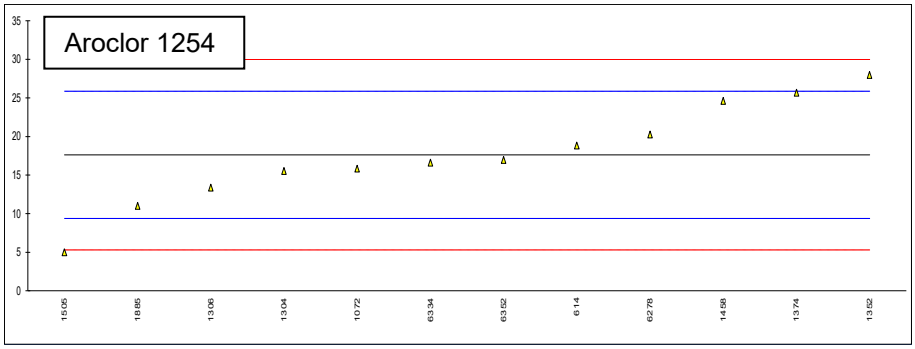
Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #21243; results in mg/kg

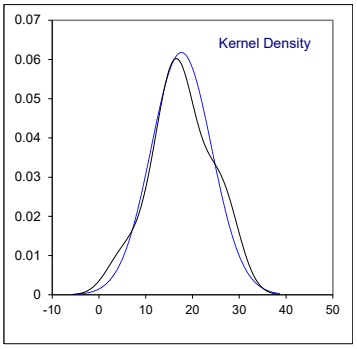
lab	method	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341		-----	-----	-----	-----
343		-----	-----	-----	-----
357		-----	-----	-----	-----
392		-----	-----	-----	-----
398		-----	-----	-----	-----
455		-----	-----	-----	-----
498		-----	-----	-----	-----
511		-----	-----	-----	-----
614	D4059	14.92	-----	18.82	9.33
902		-----	-----	-----	-----
912		-----	-----	-----	-----
1059		-----	-----	-----	-----
1072	D4059	21.792	-----	15.812	10.506
1126		-----	-----	-----	-----
1135		-----	-----	-----	-----
1170		-----	-----	-----	-----
1243		-----	-----	-----	-----
1304	In house	27.38	-----	15.53	9.38
1306	EPA600	18.0614	-----	13.3659	11.2791
1352	In house	23.699	-----	27.972	12.440
1374	D4059	29.17	-----	25.66	7.93
1396		-----	-----	-----	-----
1397		-----	-----	-----	-----
1435		-----	-----	-----	-----
1440		-----	-----	-----	-----
1442		-----	-----	-----	-----
1458	D4059	36.8	-----	24.6 C	11.8
1495		-----	-----	-----	-----
1505	D4059	7.37	-----	5.00	2.67
1513		-----	-----	-----	-----
1551		-----	-----	-----	-----
1633		-----	-----	-----	-----
1660		-----	-----	-----	-----
1702		-----	-----	-----	-----
1763		-----	-----	-----	-----
1801		-----	-----	-----	-----
1816		-----	-----	-----	-----
1841		-----	-----	-----	-----
1875		-----	-----	-----	-----
1885	EPA6013	27	0	11	5.0
1888		-----	-----	-----	-----
1912		-----	-----	-----	-----
6067		-----	-----	-----	-----
6275		-----	-----	-----	-----
6278	EPA8082A	20.533	-----	20.241	6.464
6283		-----	-----	-----	-----
6334	IEC61619	32.7	-----	16.6	7.1
6335		-----	-----	-----	-----
6352	DIN51527Mod.	8.60	-----	16.95	11.68
6355		-----	-----	-----	-----
6382		-----	-----	-----	-----
6394		-----	-----	-----	-----
6414		-----	-----	-----	-----
6417		-----	-----	-----	-----
6427		-----	-----	-----	-----
	normality	OK		OK	OK
	n	12		12	12
	outliers	0		0	0
	mean (n)	22.3354		17.6292	8.7983
	st.dev. (n)	9.05464		6.46070	3.03522
	R(calc.)	25.3530		18.0900	8.4986
	st.dev.(D4059:00 (silicone))	4.91692		4.11739	2.44481
	R(D4059:00 (silicone))	13.7674		11.5287	6.8455

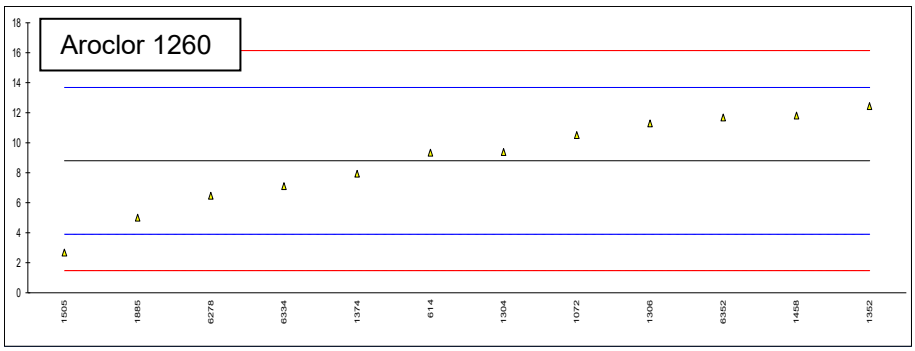
Lab 1458 first reported 44.6

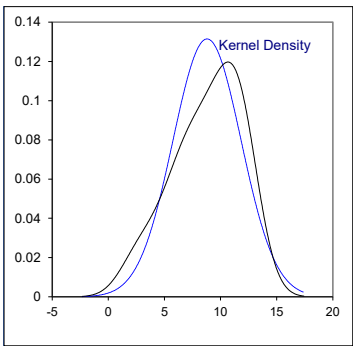

Aroclor 1242
40
35
30
25
20
15
10
5
0
1505
6352
614
1306
6278
1072
1352
1885
1304
1374
6334
1458


Kernel Density
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-20
0
20
40
60


Aroclor 1254
35
30
25
20
15
10
5
0
1505
1885
1306
1304
1072
6334
6352
614
6278
1458
1374
1352


Kernel Density
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-10
0
10
20
30
40
50

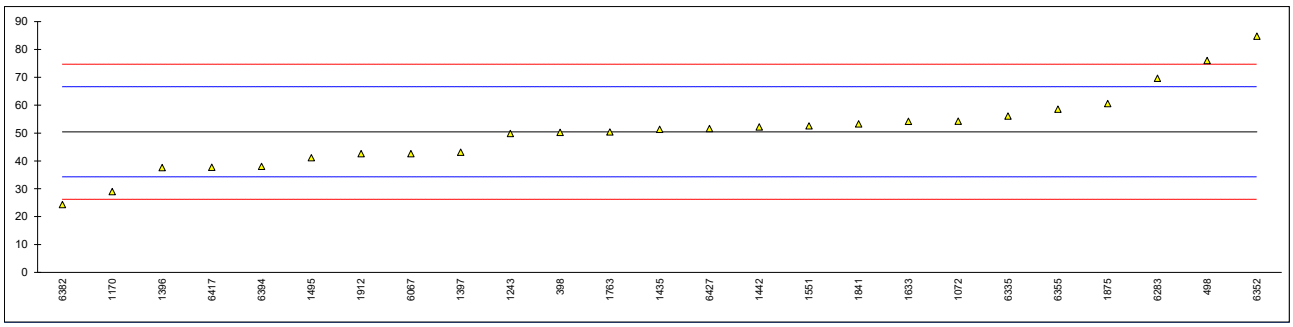

Aroclor 1260
18
16
14
12
10
8
6
4
2
0
1505
1885
6278
6334
1374
614
1304
1072
1306
6352
1458
1352

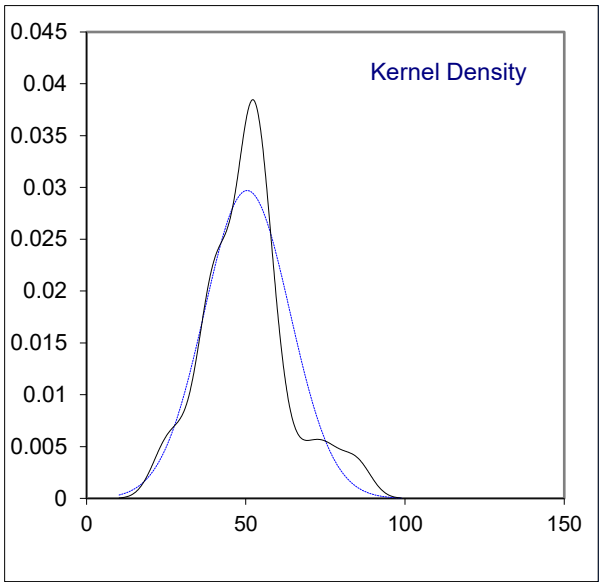

Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
-5
0
5
10
15
20

Determination of Total PCB, 5 times the sum of 6 congeners on sample #21243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
392		-----		-----	
398	EN12766-2-B	50.2		-0.03	
455		-----		-----	
498	EN12766-2-B	76.01		3.16	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072	EN12766-2-B	54.2228		0.47	
1126		-----		-----	
1135		-----		-----	
1170	EN12766-2-B	29.01		-2.65	
1243	EN12766-2-B	49.85		-0.07	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1374		-----		-----	
1396	IP462-2	37.560	E	-1.59	calculation difference, iis calculated 55.71
1397	EN12766-2-B	43.1		-0.91	
1435	EN12766-2-B	51.3		0.11	
1440		-----		-----	
1442	EN12766-2-B	52.18		0.21	
1458		-----		-----	
1495	EN12766-2-B	41.1		-1.16	
1505		-----		-----	
1513		-----		-----	
1551	IP462-2	52.5522		0.26	
1633	EN12766-2-B	54.170		0.46	
1660		-----		-----	
1702		-----		-----	
1763	EN12766-2-B	50.346		-0.01	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	53.23	E	0.34	calculated difference, iis calculated 58.2
1875	EN12766-2-B	60.53		1.25	
1885		-----		-----	
1888		-----		-----	
1912	EN12766-2-B	42.6		-0.97	
6067	EN12766-2-B	42.6		-0.97	
6275		-----		-----	
6278		-----		-----	
6283	EN12766-2-B	69.65		2.38	
6334		-----		-----	
6335	EN12766-2-B	56.054		0.69	
6352	DIN51527Mod.	84.75		4.24	
6355	EN12766-2-B	58.511		1.00	
6382	EN12766-2-B	24.31		-3.23	
6394	EN12766-2-B	38.04		-1.54	
6414		-----		-----	
6417	EN12766-2-B	37.70		-1.58	
6427	EN12766-2-B	51.6		0.14	

normality	suspect
n	25
outliers	0
mean (n)	50.4470
st.dev. (n)	13.43582
R(calc.)	37.6203
st.dev.(EN12766-2B:01)	8.08240
R(EN12766-2B:01)	22.6307

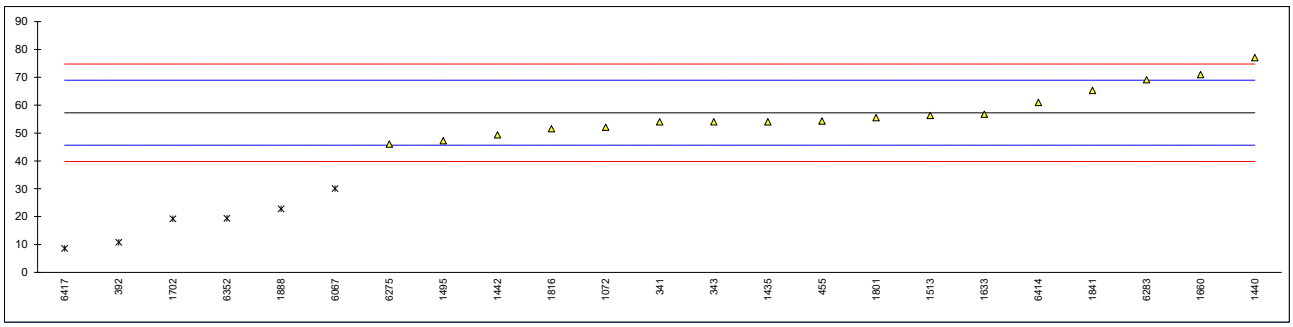

90
80
70
60
50
40
30
20
10
0
6382
1170
1396
6417
6394
1495
1912
6067
1397
1243
398
1763
1435
6427
1442
1551
1841
1633
1072
6335
6355
1875
6283
498
6352

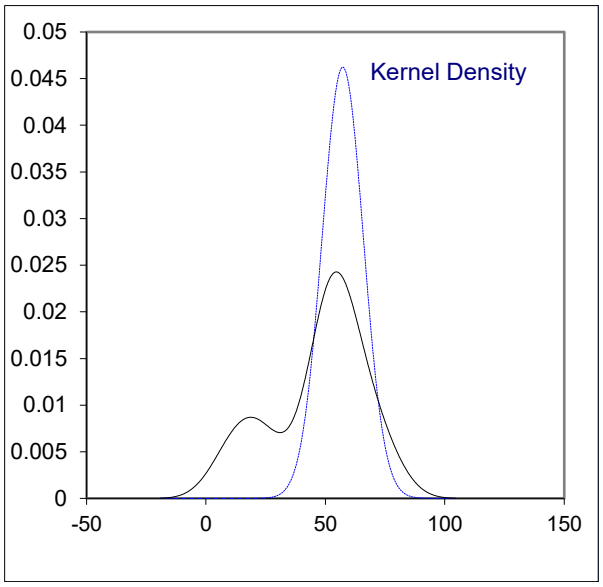

Kernel Density
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150

Determination of Total PCB, sum of all congeners on sample #21243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341	EN61619	54		-0.57	
343	EN61619	54		-0.57	
357		-----		-----	
392	EN12766-2A	10.76499	ex	-7.98	test result excluded, see § 4.1
398		-----		-----	
455	EN61619	54.3		-0.52	
498		-----		-----	
511		-----		-----	
614		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072	EN61619	52.0276		-0.90	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1374		-----		-----	
1396		-----		-----	
1397		-----		-----	
1435	EN61619	54.02		-0.56	
1440	EN61619	77	C	3.38	first reported 81
1442	EN61619	49.31		-1.37	
1458		-----		-----	
1495	EN12766-2A	47.2		-1.73	
1505		-----		-----	
1513	IEC61619	56.3	C	-0.17	first reported 52.4
1551		-----	W	-----	test result withdrawn, reported 10.5104
1633	IEC61619	56.740		-0.10	
1660	EN61619	70.92		2.34	
1702	IEC61619	19.20	G(0.05)	-6.54	
1763		-----		-----	
1801	EN61619	55.49		-0.31	
1816	EN61619	51.5		-1.00	
1841	IEC61619	65.26		1.36	
1875		-----		-----	
1885		-----		-----	
1888	IEC61619	22.8	C, DG(0.05)	-5.92	first reported 28.02
1912		-----		-----	
6067	EN61619	30.06	DG(0.05)	-4.67	
6275	IEC61619	46		-1.94	
6278		-----		-----	
6283	IEC61619	69.1		2.02	
6334		-----		-----	
6335		-----		-----	
6352	DIN51527Mod.	19.35	ex	-6.51	test result excluded, see § 4.1
6355		-----		-----	
6382		-----		-----	
6394		-----	W	-----	test result withdrawn, reported 24.03
6414	IEC61619	61		0.63	
6417	EN12766-2A	8.59	ex	-8.35	test result excluded, see § 4.1
6427		-----		-----	

normality	OK
n	17
outliers	3 + 3ex
mean (n)	57.3040
st.dev. (n)	8.62769
R(calc.)	24.1575
st.dev.(EN61619:99)	5.83071
R(EN61619:99)	16.3260

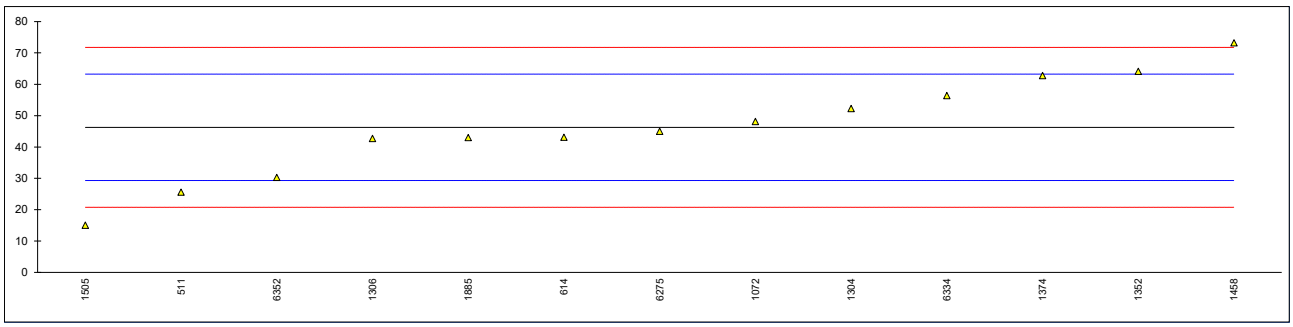

90
80
70
60
50
40
30
20
10
0
6417
392
1702
6352
1888
6067
6275
1495
1442
1816
1072
341
343
1435
455
1801
1513
1633
6414
1841
6283
1660
1440

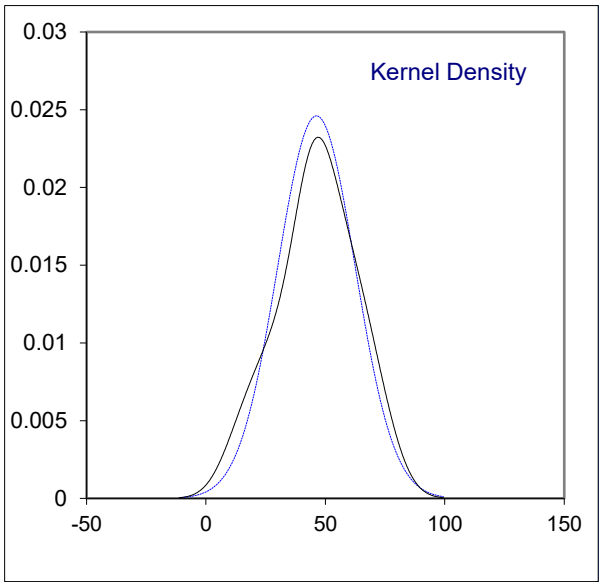

Kernel Density
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100
150

Determination of Total PCB, sum of all Aroclors on sample #21243; results in mg/kg

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
392		-----		-----	
398		-----		-----	
455		-----		-----	
498		-----		-----	
511	D4059	25.56		-2.44	
614	D4059	43.07		-0.38	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072	D4059	48.1107		0.22	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1243		-----		-----	
1304	In house	52.29		0.71	
1306	EPA600	42.7064		-0.42	
1352	In house	64.111		2.10	
1374	D4059	62.76		1.94	
1396		-----		-----	
1397		-----		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458	D4059	73.2	C	3.17	first reported 93.2
1495		-----		-----	
1505	D4059	15.04		-3.68	
1513		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885	EPA6013	43		-0.39	
1888		-----		-----	
1912		-----		-----	
6067		-----		-----	
6275	D4059	45		-0.15	
6278		-----		-----	
6283		-----		-----	
6334	IEC61619	56.4		1.19	
6335		-----		-----	
6352	DIN51527Mod.	30.27		-1.88	
6355		-----		-----	
6382		-----		-----	
6394		-----		-----	
6414		-----		-----	
6417		-----		-----	
6427		-----		-----	

normality	OK
n	13
outliers	0
mean (n)	46.2706
st.dev. (n)	16.20996
R(calc.)	45.3879
st.dev.(D4059:00 (silicone))	8.49035
R(D4059:00 (silicone))	23.7730


80
70
60
50
40
30
20
10
0
1505
511
6352
1306
1885
614
6275
1072
1304
6334
1374
1352
1458


Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100
150

APPENDIX 2

z-scores of PCB 28, 52, 101, 118, 138, 153 and 180 determination on sample #21243;

lab	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341	-----	-----	-----	-----	-----	-----	-----
343	-----	-----	-----	-----	-----	-----	-----
357	-----	-----	-----	-----	-----	-----	-----
392	-5.06	-5.06	-0.97	-----	6.66	-0.97	7.84
398	-----	-----	-----	-----	-----	-----	-----
455	-----	-----	-----	-----	-----	-----	-----
498	2.71	3.89	2.20	2.18	2.68	1.89	2.15
511	-----	-----	-----	-----	-----	-----	-----
614	-----	-----	-----	-----	-----	-----	-----
902	-----	-----	-----	-----	-----	-----	-----
912	-----	-----	-----	-----	-----	-----	-----
1059	-----	-----	-----	-----	-----	-----	-----
1072	0.08	-0.04	0.72	-0.64	-0.11	0.50	-0.35
1126	-0.60	-1.13	-0.38	-0.62	-0.45	-0.99	-0.68
1135	1.07	4.11	2.07	6.74	-1.49	-0.50	-0.68
1170	-2.22	-2.67	-2.75	-2.66	-2.68	-2.55	-2.20
1243	-0.60	1.40	-0.78	-----	-1.60	0.05	1.04
1304	-----	-----	-----	-----	-----	-----	-----
1306	-----	-----	-----	-----	-----	-----	-----
1352	-----	-----	-----	-----	-----	-----	-----
1374	-----	-----	-----	-----	-----	-----	-----
1396	0.05	0.11	-0.72	-----	0.23	2.21	0.56
1397	-----	-----	-----	-----	-----	-----	-----
1435	0.36	0.54	-0.36	-0.51	-0.55	-0.25	-0.25
1440	-----	-----	-----	-----	-----	-----	-----
1442	-0.42	0.29	-0.43	1.50	-0.21	0.54	0.43
1458	-----	-----	-----	-----	-----	-----	-----
1495	-----	-----	-----	-----	-----	-----	-----
1505	-----	-----	-----	-----	-----	-----	-----
1513	-2.37	0.77	0.65	-0.93	0.68	-0.30	0.94
1551	0.22	-0.52	-3.27	-----	0.74	3.34	0.28
1633	-1.63	-0.65	0.95	0.97	0.75	0.77	0.46
1660	2.07	2.57	1.62	-0.62	1.48	1.16	1.84
1702	-5.74	-3.31	-3.11	-2.29	-5.69	-1.39	-5.85
1763	-1.15	-1.13	2.49	-----	-1.14	-0.27	-1.52
1801	-----	-----	-----	-----	-----	-----	-----
1816	-----	-----	-----	-----	-----	-----	-----
1841	1.47	-1.06	0.82	2.34	1.90	-1.11	2.15
1875	2.30	1.24	0.33	-----	1.10	0.49	-0.50
1885	-----	-----	-----	-----	-----	-----	-----
1888	-----	-----	-----	-----	-----	-----	-----
1912	-1.60	-1.43	-1.04	-1.34	-0.49	-1.29	-0.62
6067	-0.80	0.16	-2.62	-1.25	-0.41	-1.37	-0.88
6275	-----	-----	-----	-----	-----	-----	-----
6278	-----	-----	-----	-----	-----	-----	-----
6283	2.79	3.47	2.05	2.49	1.12	0.94	0.86
6334	-----	-----	-----	-----	-----	-----	-----
6335	0.33	1.61	2.42	-----	-0.11	-2.16	-0.31
6352	4.21	3.11	2.65	3.36	3.94	3.61	4.12
6355	-0.21	0.38	0.97	-0.21	0.81	1.24	0.42
6382	-2.36	-3.59	-3.09	-----	-3.13	-3.19	-2.94
6394	-2.81	-0.78	-1.26	-----	-1.78	-1.86	-0.52
6414	-----	-----	-----	-----	-----	-----	-----
6417	-1.67	-1.63	-1.96	-1.76	-1.28	-1.66	-1.23
6427	0.79	-0.64	2.77	-----	-5.01	3.12	-2.59

z-scores of Aroclor 1242, 1248, 1254 and 1260 determination on sample #21243;

lab	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341	-----	-----	-----	-----
343	-----	-----	-----	-----
357	-----	-----	-----	-----
392	-----	-----	-----	-----
398	-----	-----	-----	-----
455	-----	-----	-----	-----
498	-----	-----	-----	-----
511	-----	-----	-----	-----
614	-1.51	-----	0.29	0.22
902	-----	-----	-----	-----
912	-----	-----	-----	-----
1059	-----	-----	-----	-----
1072	-0.11	-----	-0.44	0.70
1126	-----	-----	-----	-----
1135	-----	-----	-----	-----
1170	-----	-----	-----	-----
1243	-----	-----	-----	-----
1304	1.03	-----	-0.51	0.24
1306	-0.87	-----	-1.04	1.01
1352	0.28	-----	2.51	1.49
1374	1.39	-----	1.95	-0.36
1396	-----	-----	-----	-----
1397	-----	-----	-----	-----
1435	-----	-----	-----	-----
1440	-----	-----	-----	-----
1442	-----	-----	-----	-----
1458	2.94	-----	1.69	1.23
1495	-----	-----	-----	-----
1505	-3.04	-----	-3.07	-2.51
1513	-----	-----	-----	-----
1551	-----	-----	-----	-----
1633	-----	-----	-----	-----
1660	-----	-----	-----	-----
1702	-----	-----	-----	-----
1763	-----	-----	-----	-----
1801	-----	-----	-----	-----
1816	-----	-----	-----	-----
1841	-----	-----	-----	-----
1875	-----	-----	-----	-----
1885	0.95	-----	-1.61	-1.55
1888	-----	-----	-----	-----
1912	-----	-----	-----	-----
6067	-----	-----	-----	-----
6275	-----	-----	-----	-----
6278	-0.37	-----	0.63	-0.95
6283	-----	-----	-----	-----
6334	2.11	-----	-0.25	-0.69
6335	-----	-----	-----	-----
6352	-2.79	-----	-0.16	1.18
6355	-----	-----	-----	-----
6382	-----	-----	-----	-----
6394	-----	-----	-----	-----
6414	-----	-----	-----	-----
6417	-----	-----	-----	-----
6427	-----	-----	-----	-----

APPENDIX 3

Number of participants per country

6 labs in	AUSTRALIA
2 labs in	BELGIUM
2 labs in	CROATIA
1 lab in	CZECH REPUBLIC
1 lab in	FINLAND
2 labs in	FRANCE
7 labs in	GERMANY
2 labs in	GREECE
1 lab in	INDIA
1 lab in	INDONESIA
1 lab in	IRELAND
4 labs in	ITALY
1 lab in	MALAYSIA
1 lab in	MOROCCO
1 lab in	NETHERLANDS
2 labs in	NORWAY
1 lab in	PERU
1 lab in	PHILIPPINES
2 labs in	PORTUGAL
1 lab in	QATAR
1 lab in	SLOVENIA
1 lab in	SOUTH AFRICA
7 labs in	SPAIN
2 labs 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)	= 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)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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