Results of Proficiency Test
PCB in (Mineral) Oil
November 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2018/2019, it was decided to continue the round robin for the analysis PCB on (mineral) oil.
In this interlaboratory study, 48 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 2018 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one 8 ml vial with mineral oil contaminated with PCB, labelled #18233. 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/IEC 17043:2010 (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 60 amber glass vials of 8 mL were filled and labelled #18233.
The homogeneity of the subsamples #18233 was checked by determination of Total Organic Chloride content in accordance with UOP779 on seven stratified randomly selected samples.

	Total Organic Chloride as Cl in mg/kg
sample #18233-1	56.9
sample #18233-2	57.9
sample #18233-3	57.5
sample #18233-4	58.1
sample #18233-5	57.0
sample #18233-6	59.1
sample #18233-7	59.6

Table 1: homogeneity test results of subsamples of #18233

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 ISO 13528, Annex B2 in the next table.

	Total Organic Chloride as Cl in mg/kg
r (observed)	2.9
reference test method	UOP779:08
0.3 x R (ref.test method)	3.0

Table 2: evaluation of the repeatability of subsamples of #18233

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18233 was assumed.

To each of the participating laboratories one amber glass vial of 8 ml, labelled #18233, was sent on October 31, 2018. 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 #18233: Total Organic Halogenic 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 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 calculations.

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 ISO 5725 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. Three participants did not report any test results. All other participants reported on time. Not all participants were able to report test results for all tests. In total 45 laboratories reported 247 numerical test results. Observed were 13 outlying test results, which is 5.3% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original 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 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 listed 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 Based on only four numerical test results the determination may be problematic. The calculated reproducibility is not in agreement with the requirements of UOP779:08.

Individual PCBs: The determination of the individual PCB was problematic. In total eight statistical outliers were observed over seven congeners. The calculated reproducibilities of congeners No. 28, 52, 101, 118, 138 and 153 after rejection of the statistical outliers are not in agreement with requirements of EN12766-1:00 / IP462-1:01.
The calculated reproducibility of congener No. 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. In total one statistical outlier was observed and one other test result was excluded. The calculated reproducibilities of the Aroclors 1254 and 1260 after rejection of the suspect data are not in agreement with the requirements of ASTM D4059:00(2018). For Aroclor 1242 and 1248 regretfully only a few test results were reported, therefore no significant conclusions were drawn.

Total PCB:

Total PCB, “5 times the sum of 6 PCB congeners”
This determination and/or calculation of total PCB content was not problematic. Two statistical outliers were observed and two other test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in full 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 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 of total PCB content 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 #18233 was positive on PCBs. From the data on total organic halogenic components (TOX) an average concentration of 56.0 mg/kg was calculated. From this concentration, a total content of 99.6 mg PCB/kg was estimated using an average Cl content of 56.2%, assuming the presence of 63.7% Aroclor 1254 (54%Cl) and 36.3% 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	99.6
5 times the sum of 6 congeners	65.4
sum of all congeners	54.1
using Aroclor method	51.1

Table 3: comparison of estimations of total PCB content in sample #18233.

The total PCB content as 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 significantly 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 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	4	56.0	17.2	9.5
PCB no. 28	mg/kg	19	0.06	0.16	n.a.
PCB no. 52	mg/kg	25	1.67	1.38	0.82
PCB no. 101	mg/kg	22	3.45	2.10	1.71
PCB no. 118	mg/kg	16	2.19	1.51	1.08
PCB no. 138	mg/kg	24	3.72	4.00	1.84
PCB no. 153	mg/kg	25	3.34	2.96	1.66
PCB no. 180	mg/kg	23	1.77	0.91	0.87
Aroclor 1254	mg/kg	12	32.7	31.1	18.3
Aroclor 1260	mg/kg	13	18.7	28.5	12.0
Total PCB, 5 times the sum of 6 congeners	mg/kg	17	65.4	29.3	29.5
Total PCB, sum of all congeners	mg/kg	19	54.1	23.1	15.5
Total PCB, sum of Aroclors	mg/kg	12	51.1	28.4	25.6

Table 4: reproducibilities of tests on sample #18233

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 2018 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2018	*November 2017*	*November 2016*	*November 2015*	*November 2014*
Number of reporting labs	45	50	45	43	48
Number of test results reported	247	275	221	219	239
Statistical outliers	13	16	12	5	5
Percentage outliers	5.3%	5.8%	5.4%	2.3%	2.1%

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 2018	*November 2017*	*November 2016*	*November 2015*	*November 2014*
TOX as Cl	--*	+/-*	--*	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

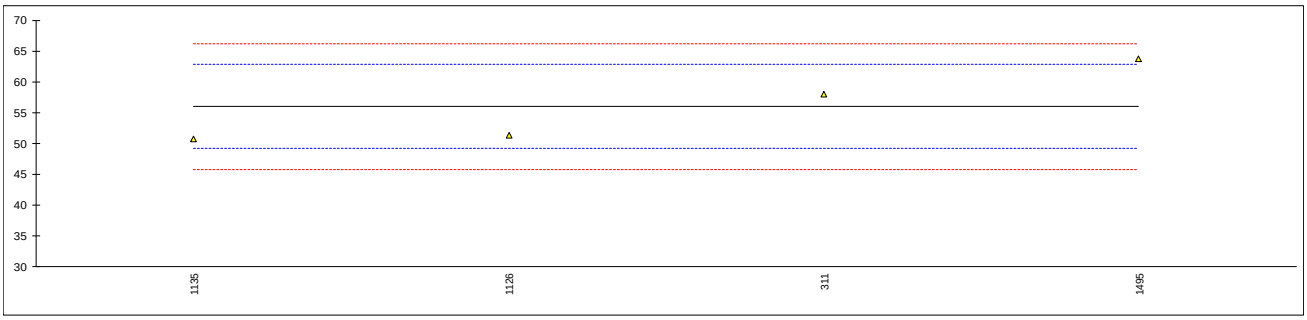
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

APPENDIX 1

Determination of Total Organohalogenic Compounds (TOX) on sample #18233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
311	UOP779	58.0		0.59	
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840		-----		-----	
902		-----		-----	
912		-----		-----	
1059		-----		-----	
1072		-----		-----	
1126	EN14077	51.4		-1.35	
1135	EN14077	50.8		-1.53	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271		-----		-----	
1303		-----		-----	
1304		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1435		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495	EN14077	63.77		2.29	
1513		-----		-----	
1516		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885		-----		-----	
1888		-----		-----	
1899		-----		-----	
2622		-----		-----	
3195		-----		-----	
6067		-----		-----	
6089		-----		-----	
6141		-----		-----	

	normality	unknown
	n	4
	outliers	0
	mean (n)	55.99
	st.dev. (n)	6.126
	R(calc.)	17.15
	st.dev.(UOP779:08)	3.392
	R(UOP779:08)	9.50
Compare		
	R(EN14077:03)	4.21

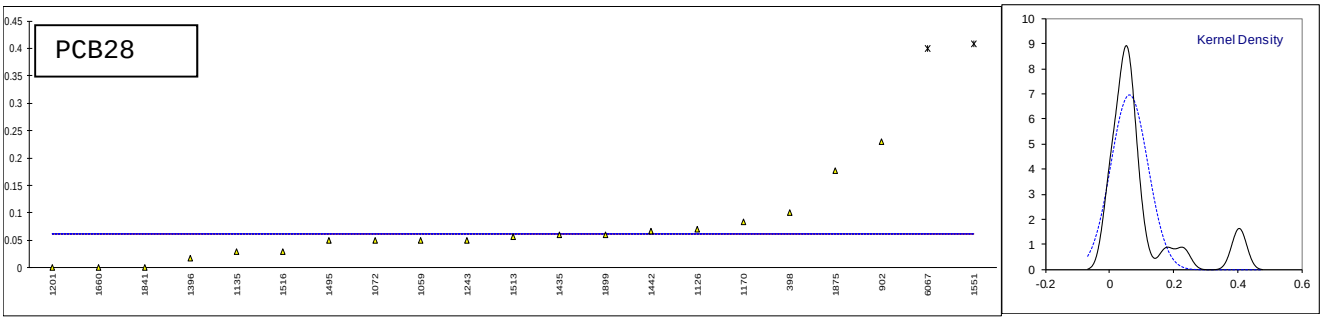


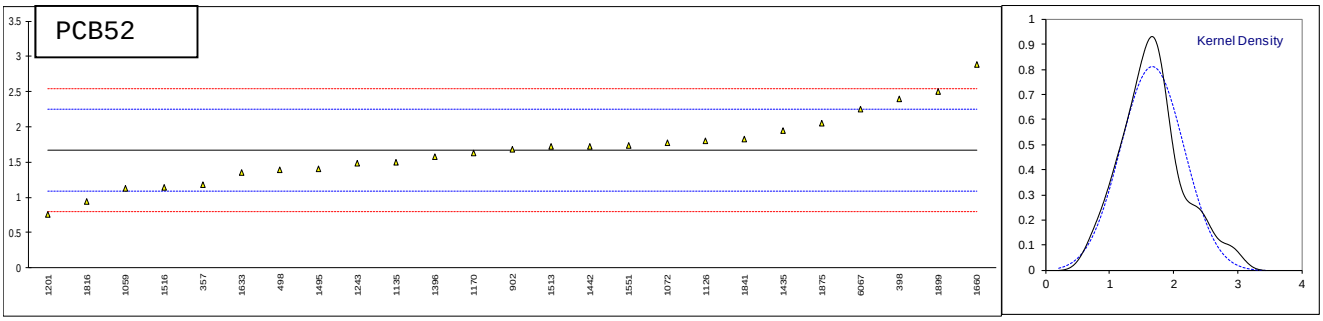
Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #18233; results in mg/kg.

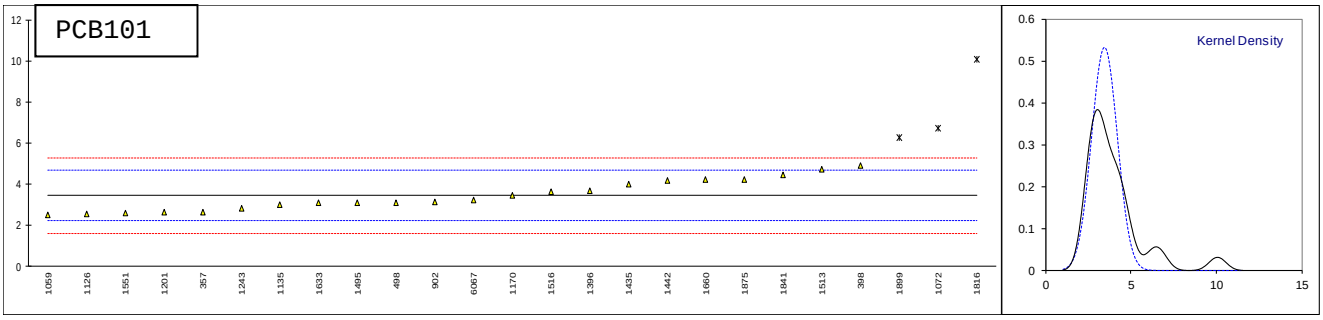
lab	Method	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341		-----	-----	-----	-----	-----	-----	-----
343		-----	-----	-----	-----	-----	-----	-----
357	EN12766-1	<0.05	1.18	2.64	1.94	3.24	2.73	1.62
398	EN12766-1	0.1	2.4	4.9	-----	6.5	4.8	2.1
498		<0,3	1.39	3.11	1.98	3.09	2.36	1.24
511		-----	-----	-----	-----	-----	-----	-----
614		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
902	EN12766-1	0.23	1.68	3.14	2.57	2.76	2.28	1.56
912		-----	-----	-----	-----	-----	-----	-----
1059	EN12766-1	0.05	1.13	2.52	1.56	3.05	2.23	1.36
1072	EN12766-1	0.0499	1.7713	**6.7355**	3.0944	5.6794	3.5599	1.8457
1126	EN12766-1	0.07	1.80	2.55	1.63	2.89	2.13	1.32
1135	EN12766-1	0.03	1.5	3.0	-----	2.4	4.7	2.3 C
1170	EN12766-1	0.084	1.625	3.434	2.359	3.20	2.528	1.340
1201	EN12766-1	0	0.76	2.62	1.80	0.49	5.93	1.73
1243		0.05	1.48	2.82	-----	2.8	3.01	1.85
1271		-----	-----	-----	-----	-----	-----	-----
1303		-----	-----	-----	-----	-----	-----	-----
1304		-----	-----	-----	-----	-----	-----	-----
1352		-----	-----	-----	-----	-----	-----	-----
1358		-----	-----	-----	-----	-----	-----	-----
1367		-----	-----	-----	-----	-----	-----	-----
1374		-----	-----	-----	-----	-----	-----	-----
1396		0.0181	1.574	3.665	-----	3.096	3.148	1.910
1435	EN12766-1	0.06	1.94	4.00	2.54	4.90	3.88	2.17
1442	EN12766-1	0.067	1.727	4.175	2.702	4.337	4.506	2.424
1458		-----	-----	-----	-----	-----	-----	-----
1495	EN12766-1	0.049	1.408	3.088	-----	3.879	3.028	2.023
1513	IEC61619	0.056	1.723	4.744	2.422	4.255	3.094	1.953
1516	IEC61619	0.03	1.14	3.65	2.49	3.07	2.52	1.67
1551		**0.40865** f+?	1.73565	2.5733	-----	2.80015	3.31345	1.37755
1633	IEC61619	<0.10	1.35	3.08	2.13	3.38	2.92	1.60
1660		0	2.89	4.24	0.88	5.36	4.24	1.86
1743		-----	-----	-----	-----	-----	-----	-----
1801		-----	-----	-----	-----	-----	-----	-----
1816	IEC61619	<1	0.94	**10.09**	<1 f-?	2.65	1.93	**3.11**
1841	IEC61619	0.00	1.83	4.43	2.35	4.10	2.13	1.91
1875		0.1765	2.0475	4.2410	-----	4.2500	3.5940	2.0033
1885		-----	-----	-----	-----	-----	-----	-----
1888		-----	-----	-----	-----	-----	-----	-----
1899	EN12766-1	0.06	2.5	**6.25**	**4.66**	7.07	4.92	**2.94**
2622		-----	-----	-----	-----	-----	-----	-----
3195		-----	-----	-----	-----	-----	-----	-----
6067	IEC61619	**0.40** C,f+?	2.25 C	3.24 C	2.53 C	-----	4.09 C	1.56 C
6089		-----	-----	-----	-----	-----	-----	-----
6141		-----	-----	-----	-----	-----	-----	-----
	normality	not OK	OK	OK	suspect	suspect	OK	OK
	n	19	25	22	16	24	25	23
	outliers	2	0	3	1	0	0	2
	mean (n)	0.0621	1.6709	3.4482	2.1861	3.7186	3.3429	1.7707
	st.dev. (n)	0.05750	0.49263	0.75003	0.53847	1.42778	1.05552	0.32547
	R(calc.)	0.1610	1.3794	2.1001	1.5077	3.9978	2.9555	0.9113
	st.dev.(EN12766-1:00)	n.a.	0.29164	0.61023	0.38400	0.60873	0.65870	0.30954
	R(EN12766-1:00)	n.a.	0.8166	1.7086	1.0752	1.8444	1.6558	0.8667

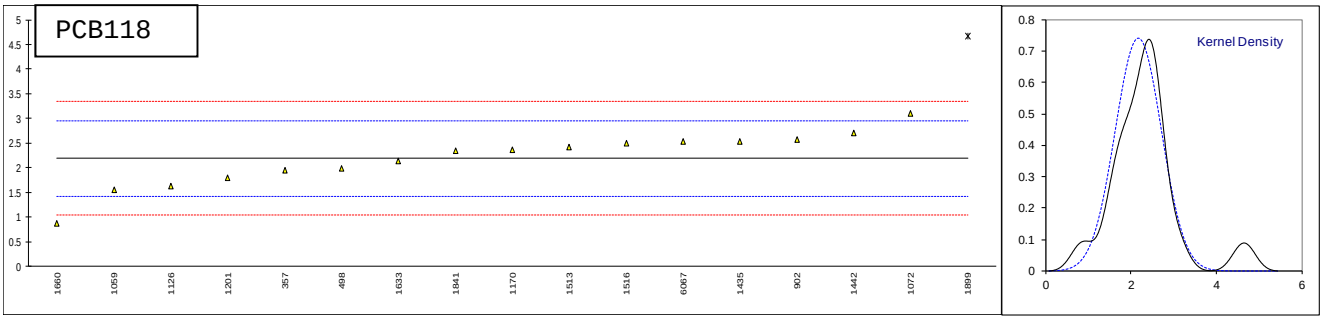
Lab 1135: first reported 3.3
Lab 6067: first reported respectively 0.7724, 3.8184, 67240, 5.1789, 8.6244, 1.4410

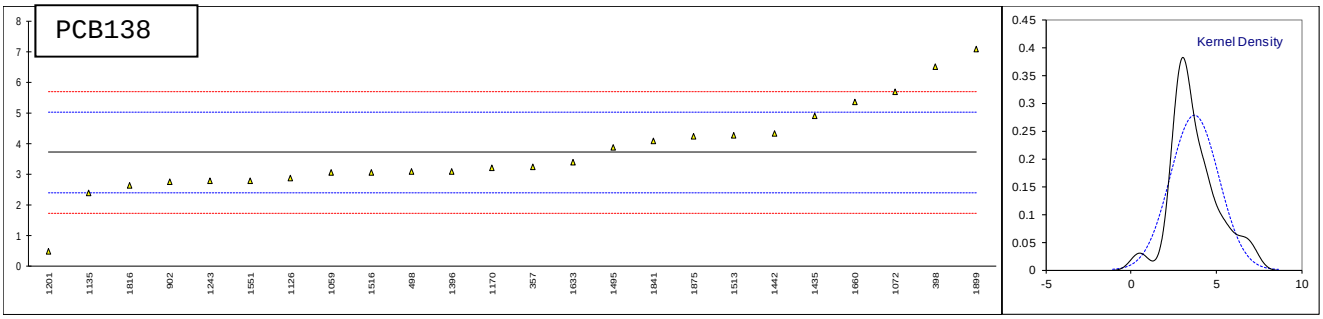
Bold and underlined test results are marked as statistical outliers

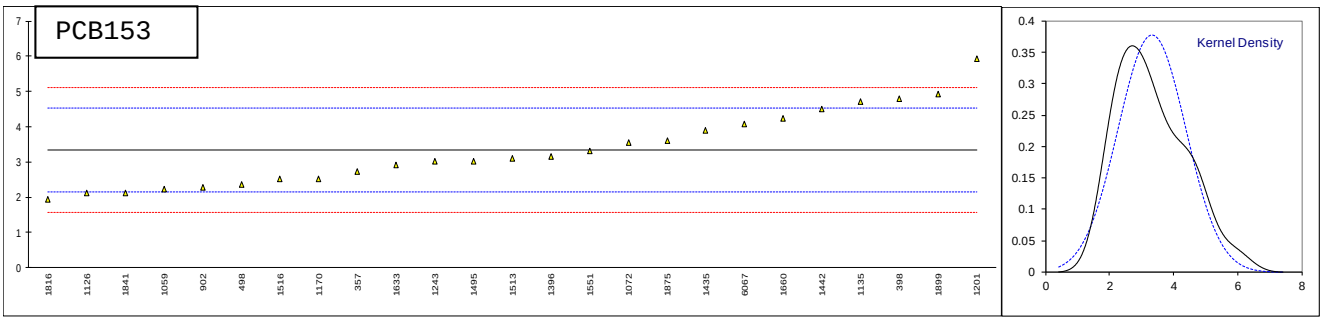


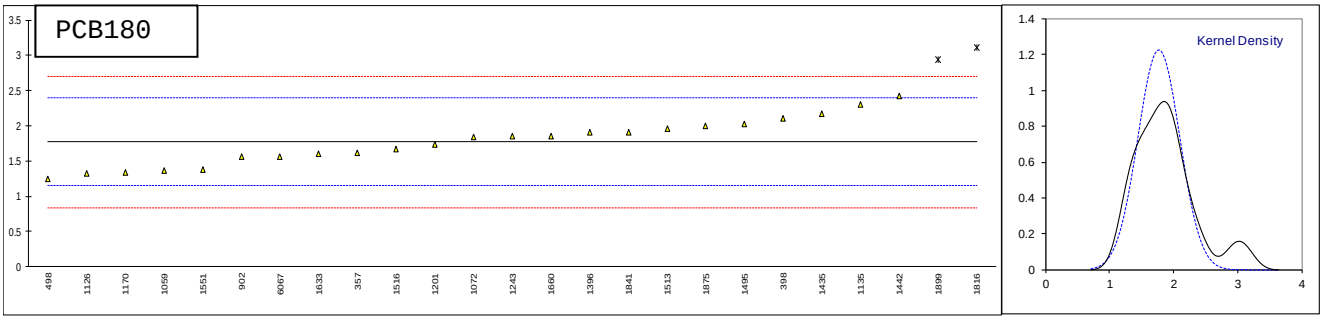

PCB52
Kernel Density


PCB101
Kernel Density


PCB118
Kernel Density


PCB138
Kernel Density

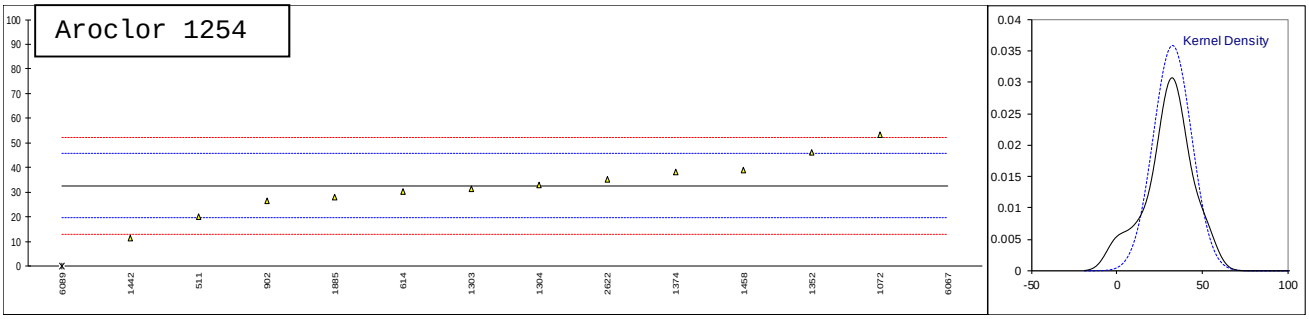

PCB153
Kernel Density

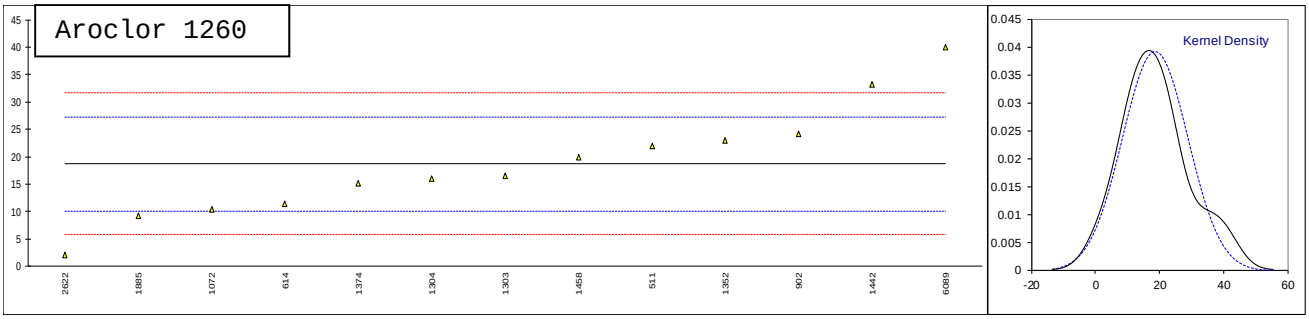

PCB180
Kernel Density

Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #18233; results in mg/kg.

lab	method	Aroclor 1242		Aroclor 1248	Aroclor 1254		Aroclor 1260
341		-----		-----	-----		-----
343		-----		-----	-----		-----
357		-----		-----	-----		-----
398		-----		-----	-----		-----
498		-----		-----	-----		-----
511	D4059	0		-----	20		22
614	D4059	<2		-----	30.2		11.4
840		-----		-----	-----		-----
902	D4059	4.2	f+?	-----	26.57		24.20
912		-----		-----	-----		-----
1059		-----		-----	-----		-----
1072	D4059	0.824		-----	53.189		10.432
1126		-----		-----	-----		-----
1135		-----		-----	-----		-----
1170		-----		-----	-----		-----
1201		-----		-----	-----		-----
1243		-----		-----	-----		-----
1271		-----		-----	-----		-----
1303	D4059	<2		-----	31.581		16.530
1304	INH-127	<0.5		-----	33.12		15.97
1352	D4059	1.340		-----	46.345		23.022
1358		-----		-----	-----		-----
1367		-----		-----	-----		-----
1374		-----		-----	38.05		15.08
1396		-----		-----	-----		-----
1435		-----		-----	-----		-----
1442	D4059	6.724	f+?	-----	11.522		33.218
1458	D4059	<2		-----	38.85		19.89
1495		-----		-----	-----		-----
1513		-----		-----	-----		-----
1516		-----		-----	-----		-----
1551		-----		-----	-----		-----
1633		-----		-----	-----		-----
1660		-----		-----	-----		-----
1743		-----		-----	-----		-----
1801		-----		-----	-----		-----
1816		-----		-----	-----		-----
1841		-----		-----	-----		-----
1875		-----		-----	-----		-----
1885	EPA6013Mod.	<1		<1	28		9.2
1888		-----		-----	-----		-----
1899		-----		-----	-----		-----
2622	EN61619	2		2	35.3		2
3195		-----		-----	-----		-----
6067		-----		-----	225	C,G(0.05)	-----
6089	In house	0		0	0	ex	40
6141		-----		-----	-----		-----
	normality	n.a.		n.a.	OK		OK
	n	10		3	12		13
	outliers	n.a.		n.a.	1 (+1excl)		0
	mean (n)	<2		<2	32.7272		18.6878
	st.dev. (n)	n.a.		n.a.	11.10415		10.17248
	R(calc.)	n.a.		n.a.	31.0916		28.4829
	st.dev.(D4059:00 (silicone))	n.a.		n.a.	6.54830		4.30146
	R(D4059:00 (silicone))	n.a.		n.a.	18.3353		12.0441

Lab 6067: first reported 264.027
Lab 6089: result excluded as zero is not a real test result

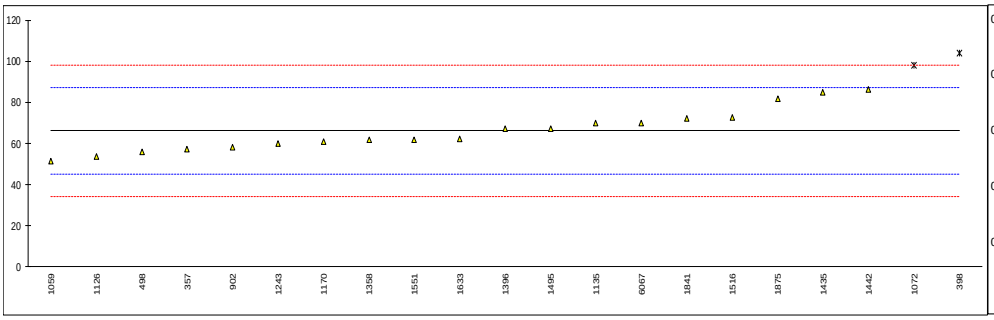

Aroclor 1254
6089
1442
511
902
1885
614
1303
1304
2622
1374
1458
1352
1072
6067
Kernel Density

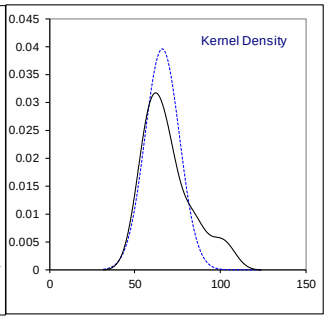

Aroclor 1260
2622
1885
1072
614
1374
1304
1303
1458
511
1352
902
1442
6089
Kernel Density

Determination of Total PCB, 5 times the sum of 6 congeners on sample #18233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	57.29		-0.77	
398	EN12766-2-B	104	DG(0.05)	3.66	
498	EN12766-2-B	55.96		-0.90	
511		-----		-----	
614		-----		-----	
840		-----		-----	
902	EN12766-2-B	58.25		-0.68	
912		-----		-----	
1059	EN12766-2-B	51.5		-1.32	
1072	EN12766-2-B	98.209	DG(0.05)	3.11	
1126	EN12766-2-B	53.8		-1.10	
1135	EN12766-2-B	69.7	C	0.41	First reported 74.7
1170	EN12766-2-B	61.05		-0.42	
1201		-----		-----	
1243	EN12766-2-B	60.05		-0.51	
1271		-----		-----	
1303		-----		-----	
1304		-----		-----	
1352		-----		-----	
1358	IP462-2	61.66331324		-0.36	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	67.1437		0.16	
1435	EN12766-2-B	84.70		1.83	
1442	EN12766-2-B	86.18		1.97	
1458		-----		-----	
1495	EN12766-2-B	67.4		0.19	
1513		-----		-----	
1516	IEC61619	72.85	ex	0.71	Result excluded, reported 5 times sum 7 congeners
1551	IP462-2	61.760		-0.35	
1633	IEC61619	62.15		-0.31	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	72.00		0.62	
1875	EN12766-2-B	81.5617		1.53	
1885		-----		-----	
1888		-----		-----	
1899		-----		-----	
2622		-----		-----	
3195		-----		-----	
6067	IEC61619	70	ex	0.43	Result excluded, reported 5 times sum 7 congeners
6089		-----		-----	
6141		-----		-----	

normality	OK
n	17
outliers	2 (+2excl)
mean (n)	65.4211
st.dev. (n)	10.46924
R(calc.)	29.3139
st.dev.(EN12766-2B:01)	10.52692
R(EN12766-2B:01)	29.4754

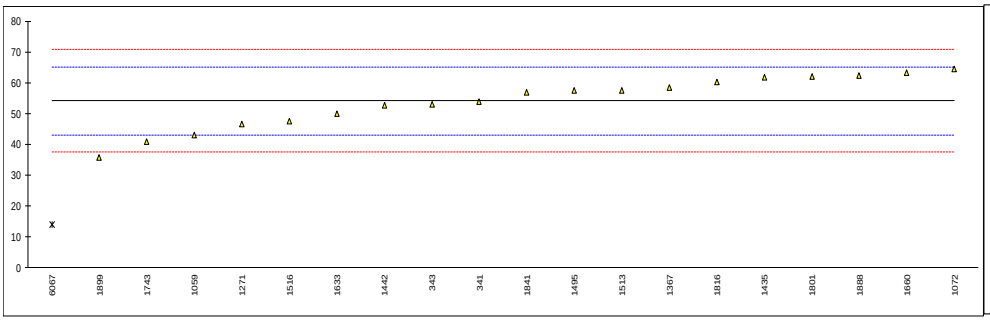


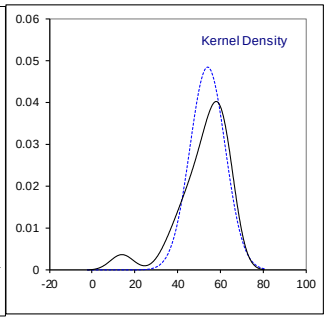


Determination of Total PCB, sum of all congeners on sample #18233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	54		-0.02	
343	EN61619	53		-0.20	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840		-----		-----	
902		-----		-----	
912		-----		-----	
1059	EN12766-2A	42.9		-2.02	
1072	EN61619	64.445		1.87	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271	EN61619	46.5800	C	-1.35	First reported 1.6775
1303		-----		-----	
1304		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367	EN61619	58.397		0.78	
1374		-----		-----	
1396		-----		-----	
1435	IEC61619	61.83		1.40	
1442	IEC61619	52.79		-0.23	
1458		-----		-----	
1495	EN12766-2A	57.4		0.60	
1513	IEC61619	57.525		0.62	
1516	IEC61619	47.46		-1.20	
1551		-----		-----	
1633	IEC61619	50.00		-0.74	
1660	IEC61619	63.3		1.66	
1743	IEC61619	41		-2.36	
1801	EN61619	61.92		1.41	
1816	EN61619	60.2		1.10	
1841	IEC61619	56.81		0.49	
1875		-----		-----	
1885		-----		-----	
1888	EN61619	62.4		1.50	
1899	EN12766-2A	35.69		-3.32	
2622		-----		-----	
3195		-----		-----	
6067	IEC61619	14	C,R(0.01)	-7.23	First reported 26.5591
6089		-----		-----	
6141		-----		-----	

normality	OK
n	19
outliers	1
mean (n)	54.0867
st.dev. (n)	8.244372
R(calc.)	23.0842
st.dev.(EN61619:99)	5.543454
R(EN61619:99)	15.5217

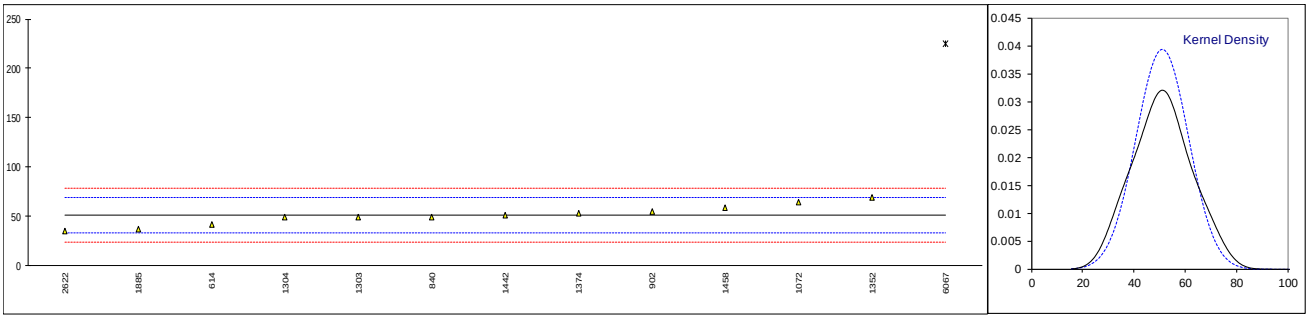




Determination of Total PCB, sum of all Aroclors on sample #18233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614	D4059	41.6		-1.04	
840	D4059	49.3		-0.20	
902	D4059	55		0.42	
912		-----		-----	
1059		-----		-----	
1072	D4059	64.445		1.45	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271		-----		-----	
1303	D4059	49.156		-0.22	
1304	INH-127	49.09		-0.22	
1352	D4059	69.367		1.99	
1358		-----		-----	
1367		-----		-----	
1374	D4059	53.13		0.22	
1396		-----		-----	
1435		-----		-----	
1442	D4059	51.464		0.03	
1458	D4059	58.74		0.83	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885	EPA6013Mod.	37.2		-1.52	
1888		-----		-----	
1899		-----		-----	
2622	EN61619	35.3		-1.73	
3195		-----		-----	
6067		225	C,G(0.01)	18.99	First reported 264.0265
6089		-----		-----	
6141		-----		-----	

normality	OK
n	12
outliers	1
mean (n)	51.1493
st.dev. (n)	10.15218
R(calc.)	28.4261
st.dev.(D4059:00 (silicone))	9.15328
R(D4059:00 (silicone))	25.6292



APPENDIX 2

z-scores of PCB 28, 52, 101, 118, 138, 153 and 180 determination on sample #18233;

lab	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341	-----	-----	-----	-----	-----	-----	-----
343	-----	-----	-----	-----	-----	-----	-----
357	-----	-1.68	-1.32	-0.64	-0.73	-1.04	-0.49
398	-----	2.50	2.38	-----	4.22	2.46	1.06
498	-----	-0.96	-0.55	-0.54	-0.95	-1.66	-1.71
511	-----	-----	-----	-----	-----	-----	-----
614	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
902	-----	0.03	-0.51	1.00	-1.46	-1.80	-0.68
912	-----	-----	-----	-----	-----	-----	-----
1059	-----	-1.85	-1.52	-1.63	-1.02	-1.88	-1.33
1072	-----	0.34	5.39	2.37	2.98	0.37	0.24
1126	-----	0.44	-1.47	-1.45	-1.26	-2.05	-1.46
1135	-----	-0.59	-0.73	-----	-2.00	2.29	1.71
1170	-----	-0.16	-0.02	0.45	-0.79	-1.38	-1.39
1201	-----	-3.12	-1.36	-1.01	-4.90	4.37	-0.13
1243	-----	-0.65	-1.03	-----	-1.39	-0.56	0.26
1271	-----	-----	-----	-----	-----	-----	-----
1303	-----	-----	-----	-----	-----	-----	-----
1304	-----	-----	-----	-----	-----	-----	-----
1352	-----	-----	-----	-----	-----	-----	-----
1358	-----	-----	-----	-----	-----	-----	-----
1367	-----	-----	-----	-----	-----	-----	-----
1374	-----	-----	-----	-----	-----	-----	-----
1396	-----	-0.33	0.36	-----	-0.95	-0.33	0.45
1435	-----	0.92	0.90	0.92	1.79	0.91	1.29
1442	-----	0.19	1.19	1.34	0.94	1.97	2.11
1458	-----	-----	-----	-----	-----	-----	-----
1495	-----	-0.90	-0.59	-----	0.24	-0.53	0.82
1513	-----	0.18	2.12	0.61	0.81	-0.42	0.59
1516	-----	-1.82	0.33	0.79	-0.98	-1.39	-0.33
1551	-----	0.22	-1.43	-----	-1.39	-0.05	-1.27
1633	-----	-1.10	-0.60	-0.15	-0.51	-0.72	-0.55
1660	-----	4.18	1.30	-3.40	2.49	1.52	0.29
1743	-----	-----	-----	-----	-----	-----	-----
1801	-----	-----	-----	-----	-----	-----	-----
1816	-----	-2.51	10.88	<-3.09	-1.62	-2.39	4.33
1841	-----	0.55	1.61	0.43	0.58	-2.05	0.45
1875	-----	1.29	1.30	-----	0.81	0.42	0.75
1885	-----	-----	-----	-----	-----	-----	-----
1888	-----	-----	-----	-----	-----	-----	-----
1899	-----	2.84	4.59	6.44	5.09	2.67	3.78
2622	-----	-----	-----	-----	-----	-----	-----
3195	-----	-----	-----	-----	-----	-----	-----
6067	-----	1.99	-0.34	0.90	-----	1.26	-0.68
6089	-----	-----	-----	-----	-----	-----	-----
6141	-----	-----	-----	-----	-----	-----	-----

z-scores of Aroclor 1242, 1248, 1254 and 1260 determination on sample #18233;

lab	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341	-----	-----	-----	-----
343	-----	-----	-----	-----
357	-----	-----	-----	-----
398	-----	-----	-----	-----
498	-----	-----	-----	-----
511	-----	-----	-1.94	0.77
614	-----	-----	-0.39	-1.69
840	-----	-----	-----	-----
902	-----	-----	-0.94	1.28
912	-----	-----	-----	-----
1059	-----	-----	-----	-----
1072	-----	-----	3.12	-1.92
1126	-----	-----	-----	-----
1135	-----	-----	-----	-----
1170	-----	-----	-----	-----
1201	-----	-----	-----	-----
1243	-----	-----	-----	-----
1271	-----	-----	-----	-----
1303	-----	-----	-0.18	-0.50
1304	-----	-----	0.06	-0.63
1352	-----	-----	2.08	1.01
1358	-----	-----	-----	-----
1367	-----	-----	-----	-----
1374	-----	-----	0.81	-0.84
1396	-----	-----	-----	-----
1435	-----	-----	-----	-----
1442	-----	-----	-3.24	3.38
1458	-----	-----	0.94	0.28
1495	-----	-----	-----	-----
1513	-----	-----	-----	-----
1516	-----	-----	-----	-----
1551	-----	-----	-----	-----
1633	-----	-----	-----	-----
1660	-----	-----	-----	-----
1743	-----	-----	-----	-----
1801	-----	-----	-----	-----
1816	-----	-----	-----	-----
1841	-----	-----	-----	-----
1875	-----	-----	-----	-----
1885	-----	-----	-0.72	-2.21
1888	-----	-----	-----	-----
1899	-----	-----	-----	-----
2622	-----	-----	0.39	-3.88
3195	-----	-----	-----	-----
6067	-----	-----	29.36	-----
6089	-----	-----	-5.00	4.95
6141	-----	-----	-----	-----

APPENDIX 3

Number of participating laboratories per country

7 labs in	AUSTRALIA
2 labs in	BELGIUM
1 lab in	BOSNIA and HERZEGOVINA
1 lab in	CROATIA
1 lab in	FINLAND
2 labs in	FRANCE
4 labs in	GERMANY
1 lab in	GREECE
1 lab in	INDIA
3 labs in	ITALY
1 lab in	MOROCCO
3 labs in	NETHERLANDS
1 lab in	NORWAY
1 lab in	PERU
2 labs in	PORTUGAL
1 lab in	QATAR
1 lab in	SAUDI ARABIA
2 labs in	SLOVENIA
1 lab in	SOUTH AFRICA
6 labs in	SPAIN
1 lab in	TURKEY
4 labs in	UNITED KINGDOM
1 lab in	VIETNAM

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	= possibly an error in calculations
U	= test result probably reported in a different unit
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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, June 2018
2 NEN 12766-2:04
3 ASTM E178:02
4 ASTM E1301:95(2003)
5 ISO 5725:86
6 ISO 5725, parts 1-6, 1994
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4, January 2001
14 P.J. Lowthian and M. Thompson, the Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)