Results of Proficiency Test
PCB in (Mineral) Oil
November 2017

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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Report: iis17L11

February 2018

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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 2017/2018, it was decided to continue the round robin for the analysis PCB on (mineral) oil.
In this interlaboratory study, 51 laboratories in 22 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 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 organiser 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 #17233). 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 March 2017 (iis-protocol, version 3.4). 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 sub samples was a mineral oil positive on PCB donated by a third party laboratory. After ultrasonic homogenisation, 78 glass vials of 8 mL were filled, capped and labelled #17233.
The homogeneity of the sub samples #17233 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 #17233-1	41.63
sample #17233-2	42.12
sample #17233-3	41.81
sample #17233-4	41.82
sample #17233-5	42.08
sample #17233-6	41.86
sample #17233-7	40.99
sample #17233-8	41.63

Table 1: homogeneity test results of sub samples of #17233

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)	0.99
reference test method	UOP779:08
0.3 x R (ref.test method)	2.12

Table 2: evaluation of the repeatability of sub samples of #17233

The calculated repeatability was in agreement with 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the sub samples #17233 was assumed.

To each of the participating laboratories, one glass vial of 8 ml, labelled #17233, was sent on November 1, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the oil packed in brown 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 #17233: 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 samples as if they were routine samples 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 March 2017 (iis-protocol, version 3.4).
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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, EN or ISO 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:

	$\lvert z \rvert < 1$	good
1 <	$\lvert z \rvert < 2$	satisfactory
2 <	$\lvert z \rvert < 3$	questionable
3 <	$\lvert z \rvert$	unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the sample. Five participants reported test results after the final reporting date and one participant did not report any test result at all. Not all participants were able to report test results for all tests. In total 50 laboratories reported 275 numerical test results. Observed were 16 outlying test results, which is 5.8% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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 3.

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.

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. D4007:100(2010)). 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 Based on only three numerical test results the determination was not problematic. The calculated reproducibility is in full agreement with the requirements of UOP779:08.

Individual PCBs: The determination of the individual PCB was problematic. In total seven statistical outliers were observed over seven congeners. The calculated reproducibilities of congeners No. 28, 52, 101, 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. 118 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. In total five statistical outliers were observed and two test results were excluded from the statistical calculations as the other reported tests results for Aroclors by the same laboratories were marked as statistical outliers. The calculated reproducibilities of the Aroclors 1242 and 1254 after rejection of the suspect data are not in agreement with the requirements of ASTM D4059:00(2010). However, the calculated reproducibility of Aroclor 1260 after rejection of the suspect data is in full agreement with the requirements of ASTM D4059:00(2010). For Aroclor 1248 regretfully only one test result was 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. One statistical outlier was observed and one test result was excluded as this test result contained a calculation error. 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 as 5 times the sum of 6 congeners was also calculated by iis from all reported individual congener test results. The consensus value of the reported sum results is in agreement with the consensus value (78.37 vs 80.98 mg/kg) calculated by iis.

Total PCB, "sum of all PCB congeners"

This determination and/or calculation of total PCB content was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D4059:00(2010).

Summary:

All participants agreed that sample #17233 was positive on PCBs. From the data on total organic halogenic components (TOX) an average concentration of 39.3 mg/kg was calculated. From this concentration, a total content of 71.2 mg PCB/kg was estimated using an average Cl content of 55.2%, assuming the presence of 14.4% Aroclor 1242 (41%Cl), 36.9% Aroclor 1254 (54%Cl) and 48.7% Aroclor 1260 (60%Cl).

All estimates for total PCB are given in the next table:

	#17233
total PCB content, estimated by TOX data, in mg/kg	71.2
total PCB content, 5 times the sum of 6 congeners, in mg/kg	78.4
total PCB content, sum of all congeners, in mg/kg	54.8
total PCB content, using Aroclor method, in mg/kg	46.6

Table 3: comparison of estimations of total PCB content in sample #17233.

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 average results, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu EN or ASTM test methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX as Cl	mg/kg	3	39.3	6.9	6.7
PCB no. 28	mg/kg	24	0.47	0.47	0.21
PCB no. 52	mg/kg	25	1.66	1.01	0.81
PCB no. 101	mg/kg	26	3.36	1.88	1.66
PCB no. 118	mg/kg	19	1.39	0.71	0.68
PCB no. 138	mg/kg	26	3.75	2.39	1.86
PCB no. 153	mg/kg	26	4.15	2.63	2.06
PCB no. 180	mg/kg	26	2.60	1.34	1.28
Aroclor 1242	mg/kg	9	6.68	9.39	5.57
Aroclor 1254	mg/kg	11	17.2	32.6	11.3
Aroclor 1260	mg/kg	9	21.7	13.4	13.5
Total PCB, 5 times the sum of 6 congeners	mg/kg	20	78.4	35.3	35.4
Total PCB, sum of all congeners	mg/kg	22	54.8	26.4	15.7
Total PCB, sum of Aroclors	mg/kg	9	46.6	23.5	23.9

Table 4: reproducibilities of tests on sample #17233

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

4.3 COMPARISON OF THE NOVEMBER 2017 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2017	*November 2016*	*November 2015*	*November 2014*	*November 2013*
Number of reporting labs	50	45	43	48	44
Number of test results reported	275	221	219	239	254
Statistical outliers	16	12	5	5	6
Percentage outliers	5.8%	5.4%	2.3%	2.1%	2.4%

Table 5: comparison with previous proficiency tests

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

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

Determination	*November 2017*	*November 2016*	*November 2015*	*November 2014*	*November 2013*
TOX	+/-*	--*	n.e.	++*	--
PCB (individual)	-	-	+/-	-	--
Aroclor (individual)	--	--	+/-	+	--
Total PCB, 5 x sum of 6 congeners	+/-	+/-	-	-	+/-
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 test results

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. 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) on sample #17233; results in mg/kg.

lab	method	value	Mark	z(targ)	remarks
311	UOP779	41.7		1.02	
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840		-----		-----	
912		-----		-----	
1059		-----		-----	
1072		-----		-----	
1126	EN14077	36.8		-1.04	
1135	UOP779	39.3		0.01	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1442		-----		-----	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1704		-----		-----	
1743		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885		-----		-----	
1888		-----		-----	
2320		-----		-----	
3150		-----		-----	
3195		-----		-----	
6086		-----		-----	
6106		-----		-----	
6133		-----		-----	
6157		-----		-----	

normality	n.a.
n	3
outliers	0
mean (n)	39.27
st.dev. (n)	2.450
R(calc.)	6.86
st.dev.(UOP779:08)	2.379
R(UOP779:08)	6.66

Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #17233; results in mg/kg.

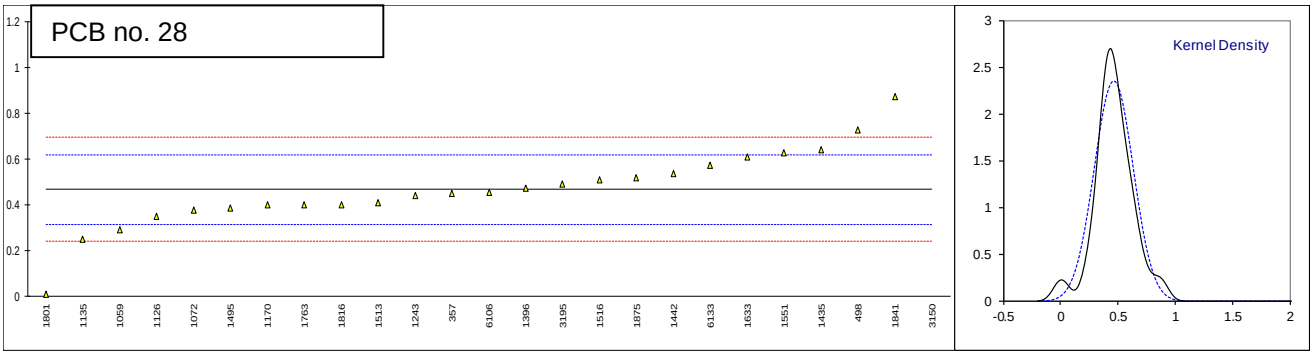
lab	Method	PCB28		PCB52		PCB101		PCB118		PCB138		PCB153		PCB180	
341		-----		-----		-----		-----		-----		-----		-----	
343		-----		-----		-----		-----		-----		-----		-----	
357	EN12766-1	0.45		1.62		2.81		1.27		3.75		4.31		2.87	
398		-----		-----		-----		-----		-----		-----		-----	
498	EN12766-1	0.726		2.525		3.788		1.777		4.601		4.779		2.869	
511		-----		-----		-----		-----		-----		-----		-----	
614		-----		-----		-----		-----		-----		-----		-----	
840		-----		-----		-----		-----		-----		-----		-----	
912		-----		-----		-----		-----		-----		-----		-----	
1059	EN12766-1	0.29		1.31		3.48		1.01		3.74		3.59		2.22	
1072	EN12766-1	0.3791		1.9353		5.2150		1.4059		4.8973		5.1649		3.1086	
1126		0.35		0.89		3.21		1.08		4.56		4.77		2.72	
1135	EN12766-1Mod.	0.25	C	1.72		3.55		1.30		5.16		5.05		2.96	
1170	EN12766-1	0.398		1.268		2.50		0.905		2.981		3.311		1.932	
1201	EN12766-1	<0.5		<0.5	f-?	2.512		1.570		2.427		6.275		2.313	
1243		0.44		1.72		2.66		-----		2.82		3.99		2.49	
1271	IEC61619	-----	W	-----	W	-----	W	-----	W	-----	W	-----	W	-----	W
1303		-----		-----		-----		-----		-----		-----		-----	
1304		-----		-----		-----		-----		-----		-----		-----	
1306		-----		-----		-----		-----		-----		-----		-----	
1352		-----		-----		-----		-----		-----		-----		-----	
1358		-----		-----		-----		-----		-----		-----		-----	
1367		-----		-----		-----		-----		-----		-----		-----	
1374		-----		-----		-----		-----		-----		-----		-----	
1396		0.472		1.675		3.250		n/a		3.07		3.934		2.5438	
1429		-----		-----		-----		-----		-----		-----		-----	
1435	IEC61619	0.64		2.26		3.15		1.32		3.46		4.16		2.37	
1442	EN12766-1	0.538		2.108		3.237		1.865		3.396		3.917		2.274	
1495		0.386		1.622		2.865		-----		3.678		4.132		2.588	
1513	IEC61619	0.40751		1.63341		3.81747		1.24059		3.58312		3.68341		2.60322	
1516	IEC61619	0.51		1.55		4.01		1.50		3.31		3.51		2.50	
1551		0.6264		1.2293		1.7310		-----		2.1521		2.9880		1.4529	
1633	IEC61619	0.61		1.60		3.34		1.49		4.11		1.53		2.65	
1660		-----		-----		-----		-----		-----		-----		-----	
1702	EN12766-1	ND		1.2		3.1		NB		4.7		5.1		***4.4***	
1704		-----		-----		-----		-----		-----		-----		-----	
1743		-----		-----		-----		-----		-----		-----		-----	
1763		0.40		1.55		3.29		-----		2.84		3.16		1.96	
1801		0.00937		1.69049		4.0329		1.27033		***6.17009***		***7.57903***		3.64358	
1816		0.40		1.48		***7.89***		<1		3.88		3.92		3.69	
1841	IEC61619	0.87		1.32		4.40		1.75		5.22		3.58		***4.13***	
1875	DIN61619	0.5198		1.7380		3.6137		-----		2.7865		4.9813		2.8077	
1885		-----		-----		-----		-----		-----		-----		-----	
1888		-----		-----		-----		-----		-----		-----		-----	
2320		-----		-----		-----		-----		-----		-----		-----	
3150	EN15803	***5.61***		***51.8***		3.49		1.48		4.50		5.31		2.53	
3195	EN12766-1	0.49		1.71		3.55		1.26		4.37		4.83		2.69	
6086		-----		-----		-----		-----		-----		-----		2.9	
6106	EN12766-1	0.454		2.00		3.42		1.59		4.40		4.25		2.66	
6133		0.57		2.05		3.3		1.33		3.05		3.8		2.25	
6157		-----		-----		-----		-----		-----		-----		-----	
	Normality	not OK		OK		not OK		OK		OK		suspect		suspect	
	n	24		25		26		19		26		26		26	
	Outliers	1		1		1		0		1		1		2	
	mean (n)	0.4661		1.6562		3.3585		1.3902		3.7478		4.1548		2.5998	
	st.dev. (n)	0.16945		0.36025		0.67177		0.25475		0.85250		0.93892		0.47677	
	R(calc.)	0.4745		1.0087		1.8810		0.7133		2.3870		2.6290		1.3350	
	st.dev.(EN12766-1:00)	0.07569		0.28901		0.59416		0.24134		0.66393		0.73690		0.45816	
	R(EN12766-1:00)	0.2119		0.8092		1.6637		0.6757		1.8590		2.0633		1.2829	

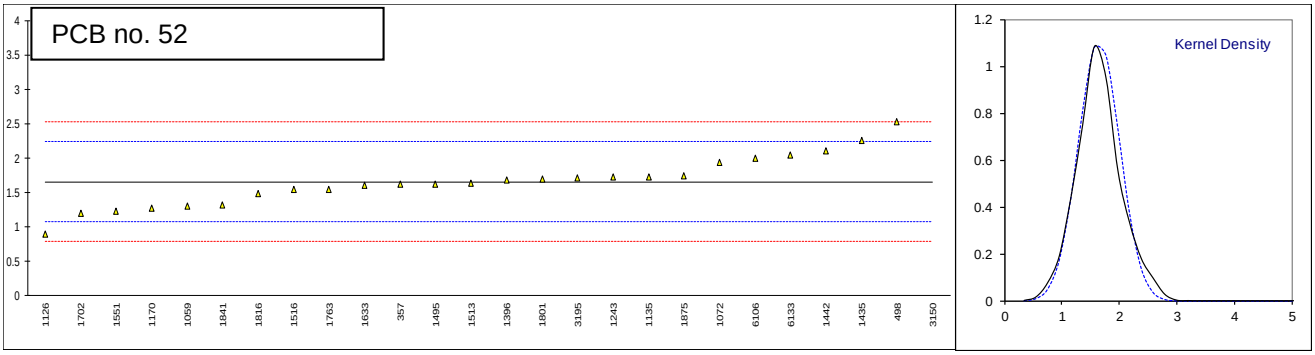
Test results which are marked “Bold, underlined and Italic” are statistical outliers

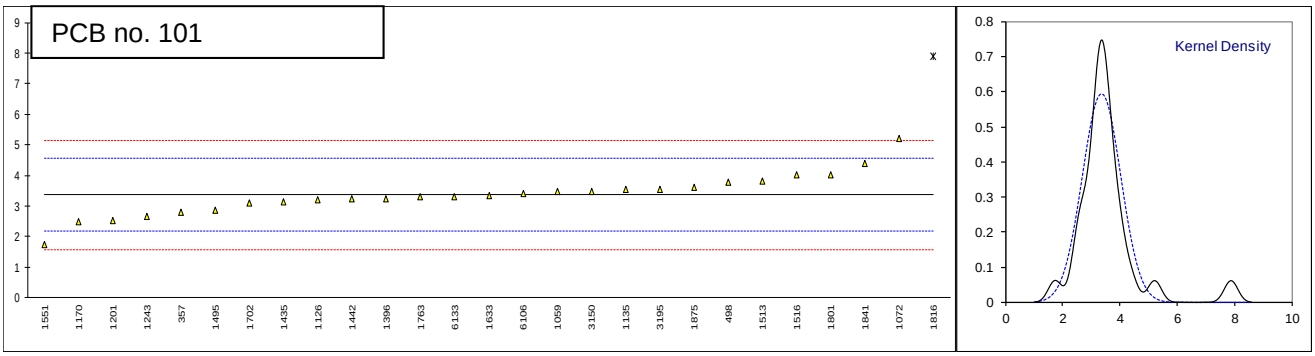
Lab 1135: first reported 0.25
Lab 1271: test results withdrawn, first reported 0.016, 0.013, 0.085, 0.026, 0, 0.082, 0.029

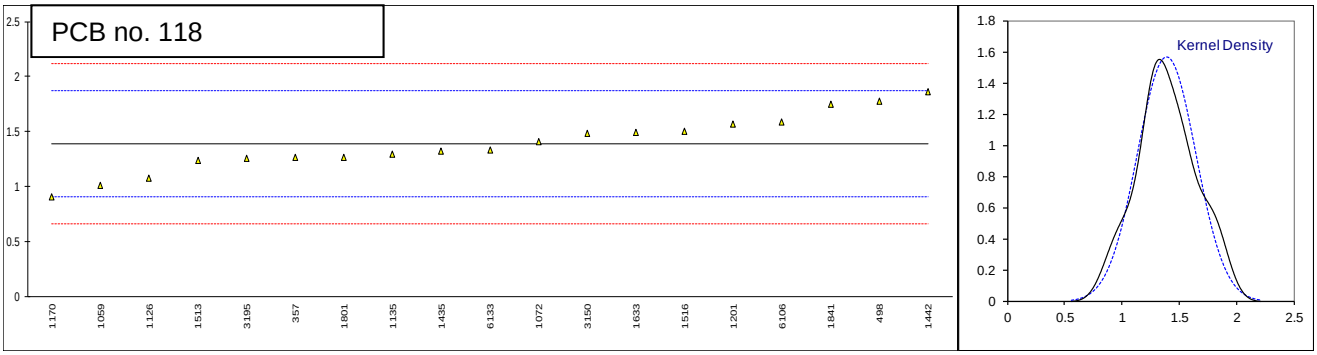
Determination of PCB 28, 52, 101, 118, 138, 153 and 180 on sample #17233; z-scores.

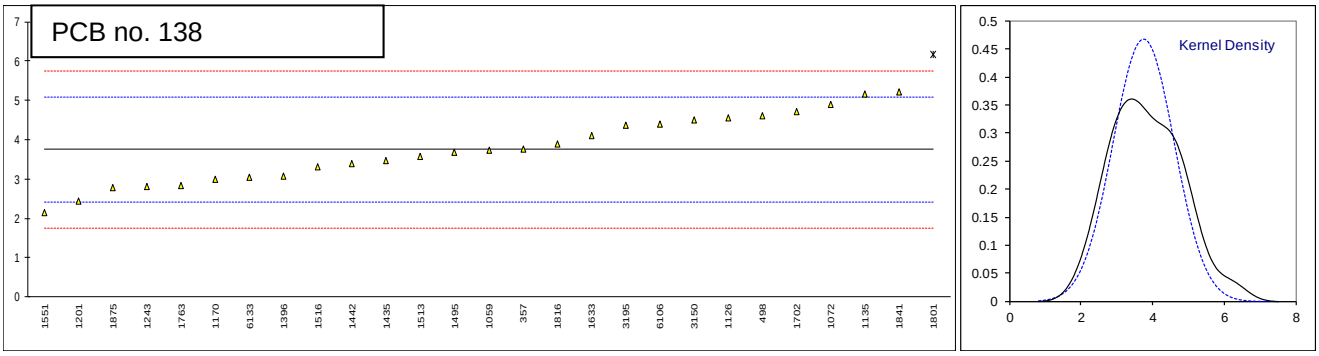
lab	PCB28	PCB52	PCB101	PCB118	PCB138	PCB153	PCB180
341	-----	-----	-----	-----	-----	-----	-----
343	-----	-----	-----	-----	-----	-----	-----
357	-0.21	-0.13	-0.92	-0.50	0.00	0.21	0.59
398	-----	-----	-----	-----	-----	-----	-----
498	3.43	3.01	0.72	1.60	1.29	0.85	0.59
511	-----	-----	-----	-----	-----	-----	-----
614	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
912	-----	-----	-----	-----	-----	-----	-----
1059	-2.33	-1.20	0.20	-1.58	-0.01	-0.77	-0.83
1072	-1.15	0.97	3.12	0.07	1.73	1.37	1.11
1126	-1.53	-2.65	-0.25	-1.29	1.22	0.83	0.26
1135	-2.85	0.22	0.32	-0.37	2.13	1.21	0.79
1170	-0.90	-1.34	-1.44	-2.01	-1.15	-1.15	-1.46
1201	-----	<-4.00	-1.42	0.75	-1.99	2.88	-0.63
1243	-0.34	0.22	-1.18	-----	-1.40	-0.22	-0.24
1271	-----	-----	-----	-----	-----	-----	-----
1303	-----	-----	-----	-----	-----	-----	-----
1304	-----	-----	-----	-----	-----	-----	-----
1306	-----	-----	-----	-----	-----	-----	-----
1352	-----	-----	-----	-----	-----	-----	-----
1358	-----	-----	-----	-----	-----	-----	-----
1367	-----	-----	-----	-----	-----	-----	-----
1374	-----	-----	-----	-----	-----	-----	-----
1396	0.08	0.07	-0.18	-----	-1.02	-0.30	-0.12
1429	-----	-----	-----	-----	-----	-----	-----
1435	2.30	2.09	-0.35	-0.29	-0.43	0.01	-0.50
1442	0.95	1.56	-0.20	1.97	-0.53	-0.32	-0.71
1495	-1.06	-0.12	-0.83	-----	-0.11	-0.03	-0.03
1513	-0.77	-0.08	0.77	-0.62	-0.25	-0.64	0.01
1516	0.58	-0.37	1.10	0.45	-0.66	-0.88	-0.22
1551	2.12	-1.48	-2.74	-----	-2.40	-1.58	-2.50
1633	1.90	-0.19	-0.03	0.41	0.55	-3.56	0.11
1660	-----	-----	-----	-----	-----	-----	-----
1702	-----	-1.58	-0.44	-----	1.43	1.28	3.93
1704	-----	-----	-----	-----	-----	-----	-----
1743	-----	-----	-----	-----	-----	-----	-----
1763	-0.87	-0.37	-0.12	-----	-1.37	-1.35	-1.40
1801	-6.03	0.12	1.13	-0.50	3.65	4.65	2.28
1816	-0.87	-0.61	7.63	-----	0.20	-0.32	2.38
1841	5.34	-1.16	1.75	1.49	2.22	-0.78	3.34
1875	0.71	0.28	0.43	-----	-1.45	1.12	0.45
1885	-----	-----	-----	-----	-----	-----	-----
1888	-----	-----	-----	-----	-----	-----	-----
2320	-----	-----	-----	-----	-----	-----	-----
3150	67.96	173.50	0.22	0.37	1.13	1.57	-0.15
3195	0.32	0.19	0.32	-0.54	0.94	0.92	0.20
6086	-----	-----	-----	-----	-----	-----	0.66
6106	-0.16	1.19	0.10	0.83	0.98	0.13	0.13
6133	1.37	1.36	-0.10	-0.25	-1.05	-0.48	-0.76
6157	-----	-----	-----	-----	-----	-----	-----

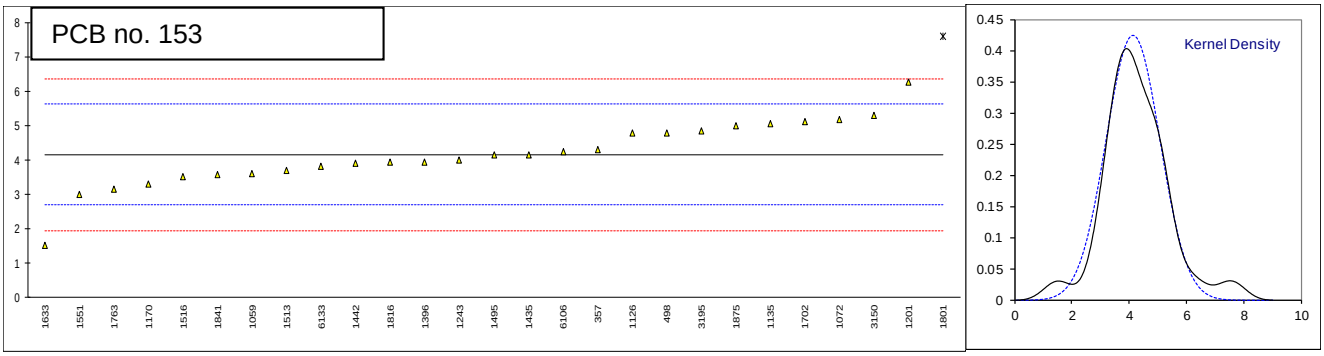

PCB no. 28
Kernel Density

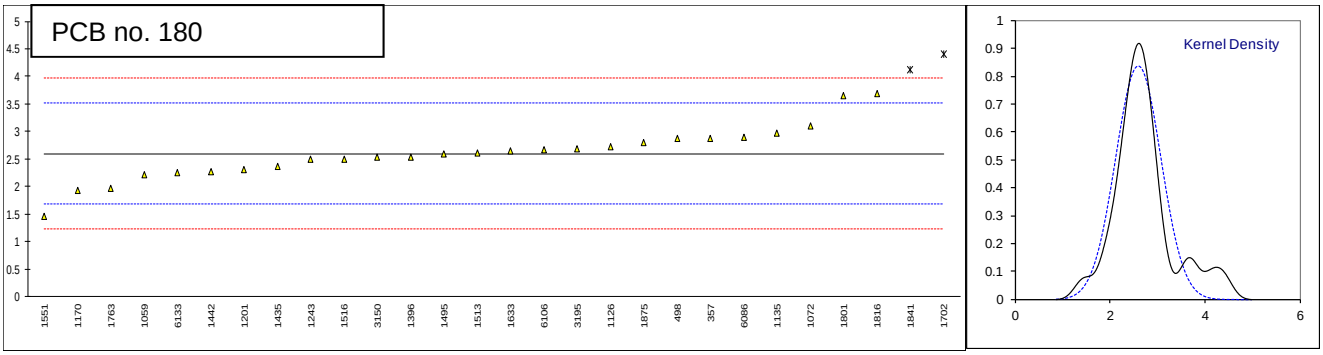

PCB no. 52
Kernel Density


PCB no. 101
Kernel Density


PCB no. 118
Kernel Density


PCB no. 138
Kernel Density


PCB no. 153
x
0
1
2
3
4
5
6
7
8
1633
1551
1763
1170
1516
1841
1059
1513
6133
1442
1816
1396
1243
1495
1435
6106
357
1126
498
3195
1875
1135
1702
1072
3150
1201
1801
Kernel Density
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
0
2
4
6
8
10


PCB no. 180
x
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
1551
1170
1763
1059
6133
1442
1201
1435
1243
1516
3150
1396
1495
1513
1633
6106
3195
1126
1875
498
357
6086
1135
1072
1801
1816
1841
1702
Kernel Density
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
2
4
6

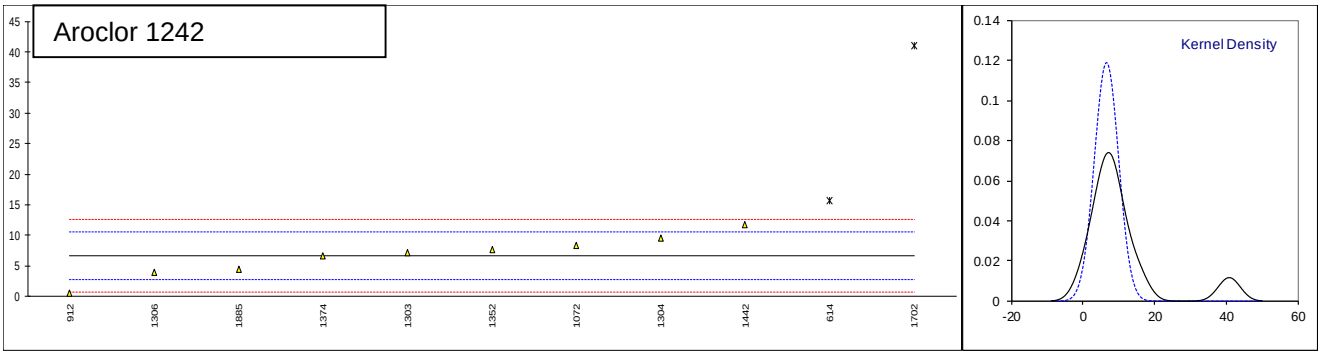
Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #17233; results in mg/kg.

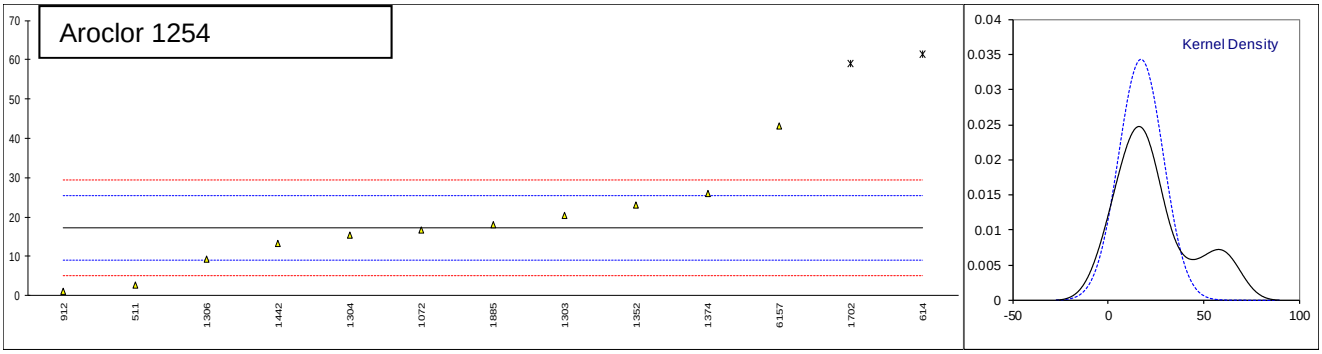
lab	method	Aroclor 1242		Aroclor 1248	Aroclor 1254		Aroclor 1260	
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357		-----		-----	-----		-----	
398		-----		-----	-----		-----	
498		-----		-----	-----		-----	
511	D4059	<1.0	f-?	-----	2.73		-----	
614	D4059	15.6	ex	-----	61.4	G(0.05)	76.6	DG(0.05)
840		-----		-----	-----		-----	
912	D4059	0.501		-----	1.058		-----	
1059		-----		-----	-----		-----	
1072	D4059	8.3		12.9	16.8		19.1	
1126		-----		-----	-----		-----	
1135		-----		-----	-----		-----	
1170		-----		-----	-----		-----	
1201		-----		-----	-----		-----	
1243		-----		-----	-----		-----	
1271		-----		-----	-----		-----	
1303		7.239		-----	20.528		23.692	
1304	INH-127	9.603		-----	15.407		15.858	
1306		3.89		-----	9.38		26.72	
1352	In house	7.640		-----	23.097		19.370	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1374		6.69		-----	26.00		21.00	
1396		-----		-----	-----		-----	
1429		-----		-----	-----		-----	
1435		-----		-----	-----		-----	
1442	D4059	11.823		-----	13.285		28.249	
1495		-----		-----	-----		-----	
1513		-----		-----	-----		-----	
1516		-----		-----	-----		-----	
1551		-----		-----	-----		-----	
1633		-----		-----	-----		-----	
1660		-----		-----	-----		-----	
1702	D4059	41	G(0.01)	-----	59	G(0.05)	41	ex
1704		-----		-----	-----		-----	
1743		-----		-----	-----		-----	
1763		-----		-----	-----		-----	
1801		-----		-----	-----		-----	
1816		-----		-----	-----		-----	
1841		-----		-----	-----		-----	
1875		-----		-----	-----		-----	
1885	INH-6013	4.4		-----	18		15	
1888		-----		-----	-----		-----	
2320		-----		-----	-----		77.0877	C,DG(0.05)
3150		-----		-----	-----		-----	
3195		-----		-----	-----		-----	
6086		-----		-----	-----		-----	
6106		-----		-----	-----		-----	
6133		-----		-----	-----		-----	
6157		-----		-----	43.2566		26	
	normality	OK		unknown	suspect		OK	
	n	9		1	11		9	
	outliers	1 (+1 ex)		n.a.	2		2 (+1 ex)	
	mean (n)	6.6762		n.a.	17.2311		21.6654	
	st.dev. (n)	3.35439		n.a.	11.63041		4.77277	
	R(calc.)	9.3923		n.a.	32.5652		13.3638	
	st.dev.(D4059:00 (silicone)	1.98767		n.a.	4.04744		4.80587	
	R(D4059:00 (silicone))	5.5655		n.a.	11.3328		13.4565	

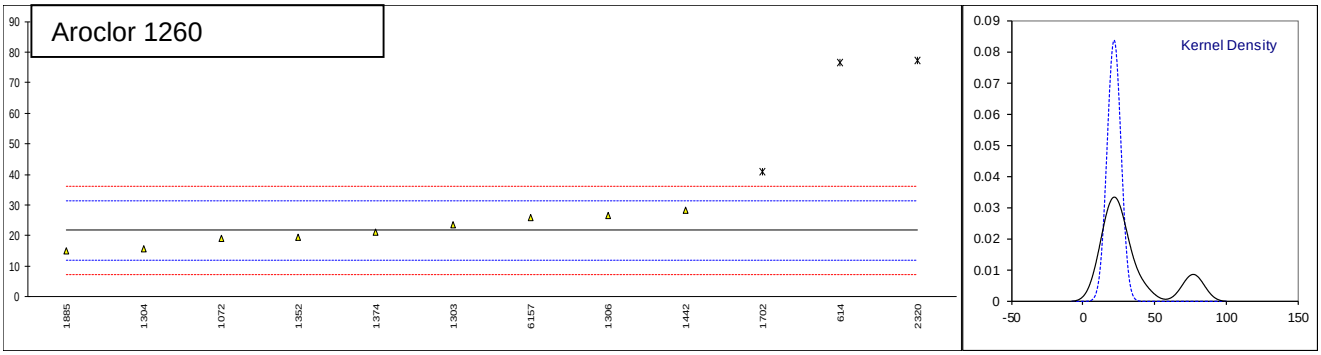
Lab511: false negative test result?
Lab 614: see §4.1
Lab1702: see §4.1
Lab 2320: first reported 391.58

Determination of Aroclor 1242, 1248, 1254 and 1260 on sample #17233; z-scores

lab	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260
341	-----	-----	-----	-----
343	-----	-----	-----	-----
357	-----	-----	-----	-----
398	-----	-----	-----	-----
498	-----	-----	-----	-----
511	<-2.85	-----	-3.58	-----
614	4.49	-----	10.91	11.43
840	-----	-----	-----	-----
912	-3.11	-----	-4.00	-----
1059	-----	-----	-----	-----
1072	0.82	-----	-0.11	-0.53
1126	-----	-----	-----	-----
1135	-----	-----	-----	-----
1170	-----	-----	-----	-----
1201	-----	-----	-----	-----
1243	-----	-----	-----	-----
1271	-----	-----	-----	-----
1303	0.28	-----	0.81	0.42
1304	1.47	-----	-0.45	-1.21
1306	-1.40	-----	-1.94	1.05
1352	0.48	-----	1.45	-0.48
1358	-----	-----	-----	-----
1367	-----	-----	-----	-----
1374	0.01	-----	2.17	-0.14
1396	-----	-----	-----	-----
1429	-----	-----	-----	-----
1435	-----	-----	-----	-----
1442	2.59	-----	-0.97	1.37
1495	-----	-----	-----	-----
1513	-----	-----	-----	-----
1516	-----	-----	-----	-----
1551	-----	-----	-----	-----
1633	-----	-----	-----	-----
1660	-----	-----	-----	-----
1702	17.27	-----	10.32	4.02
1704	-----	-----	-----	-----
1743	-----	-----	-----	-----
1763	-----	-----	-----	-----
1801	-----	-----	-----	-----
1816	-----	-----	-----	-----
1841	-----	-----	-----	-----
1875	-----	-----	-----	-----
1885	-1.15	-----	0.19	-1.39
1888	-----	-----	-----	-----
2320	-----	-----	-----	11.53
3150	-----	-----	-----	-----
3195	-----	-----	-----	-----
6086	-----	-----	-----	-----
6106	-----	-----	-----	-----
6133	-----	-----	-----	-----
6157	-----	-----	6.43	0.90

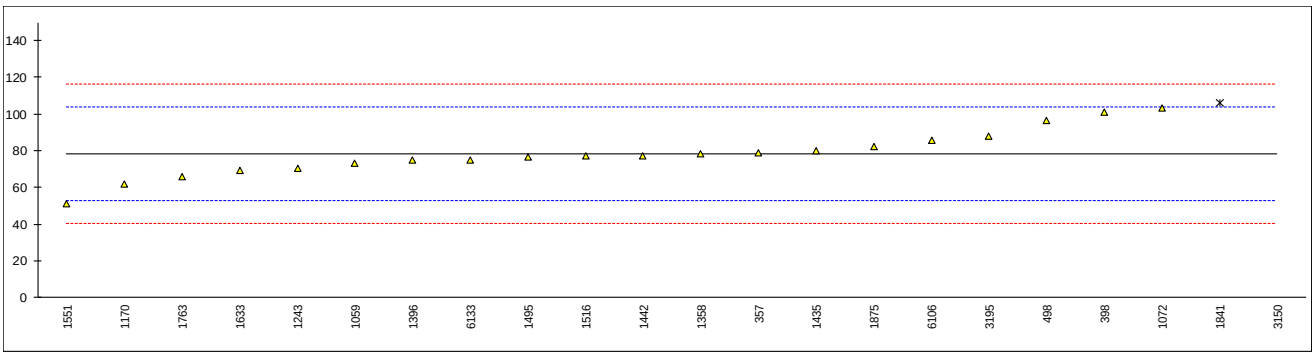

Aroclor 1242
912
1306
1885
1374
1303
1352
1072
1304
1442
614
1702
Kernel Density

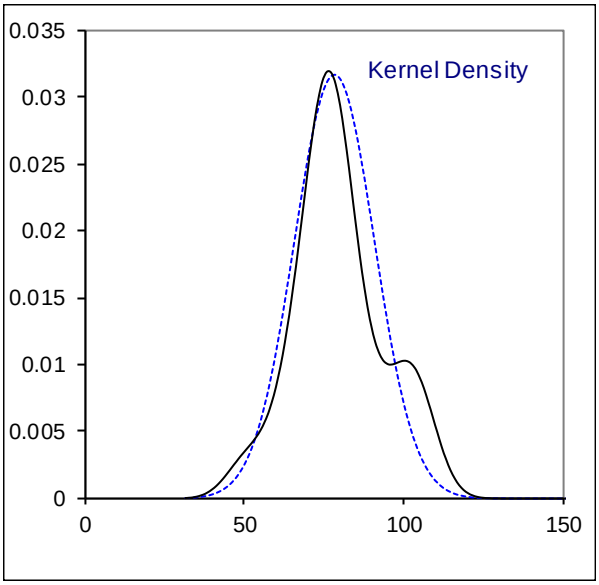

Aroclor 1254
912
511
1306
1442
1304
1072
1885
1303
1352
1374
6157
1702
614
Kernel Density


Aroclor 1260
1885
1304
1072
1352
1374
1303
6157
1306
1442
1702
614
2320
Kernel Density

Determination of Total PCB, 5 times the sum of 6 congeners on sample #17233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	79.05		0.05	
398	EN12766-2-B	101		1.79	
498	EN12766-2-B	96.440		1.43	
511		-----		-----	
614		-----		-----	
840		-----		-----	
912		-----		-----	
1059	EN12766-2-B	73.1		-0.42	
1072	EN12766-2-B	103.501		1.99	
1126		-----		-----	
1135		-----		-----	
1170	EN12766-2-B	61.95		-1.30	
1201		-----		-----	
1243	EN12766-2-B	70.6		-0.61	
1271		-----	W	-----	First reported 1.125
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358	IP462-2	78.13		-0.02	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	75.0025	C	-0.27	First reported 15.0005
1429		-----		-----	
1435	EN12766-2-B	80.1	C	0.14	First reported 16.04
1442	EN12766-2-B	77.35		-0.08	
1495	EN12766-2-B	76.4		-0.16	
1513		-----		-----	
1516	EN12766-2-B	76.95		-0.11	
1551	IP462-2	50.899		-2.17	
1633	EN12766-2-B	69.50		-0.70	
1660		-----		-----	
1702		-----		-----	
1704		-----		-----	
1743		-----		-----	
1763		66.05		-0.97	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	106.35	E,ex	2.21	Excluded: iis calculated 97.6
1875	EN12766-2-B	82.2350		0.31	
1885		-----		-----	
1888		-----		-----	
2320		-----		-----	
3150	EN15803	366	R(0.01)	22.75	
3195	EN12766-2-B	88.22		0.78	
6086		-----		-----	
6106	EN12766-2-B	85.9		0.60	
6133	EN12766-2-B	75.1		-0.26	
6157		-----		-----	
					iis calculated
	normality	OK			OK
	n	20			25
	outliers	1 (+1 ex)			2 (+1 ex)
	mean (n)	78.3739			80.9791
	st.dev. (n)	12.59539			14.53164
	R(calc.)	35.2671			40.6886
	st.dev.(EN12766-2B:01)	12.64146			13.06677
	R(EN12766-2B:01)	35.3961			36.5869

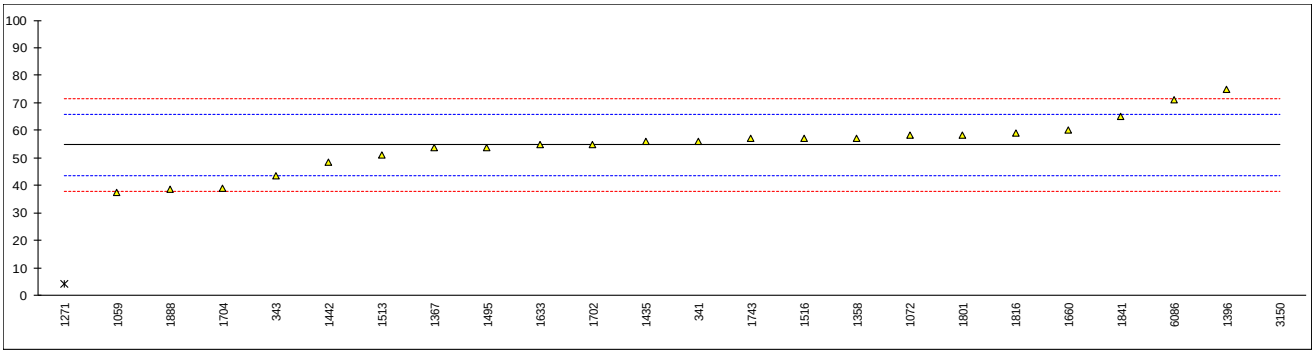

140
120
100
80
60
40
20
0
1551
1170
1763
1633
1243
1059
1396
6133
1495
1516
1442
1358
357
1435
1875
6106
3195
498
398
1072
1841
3150

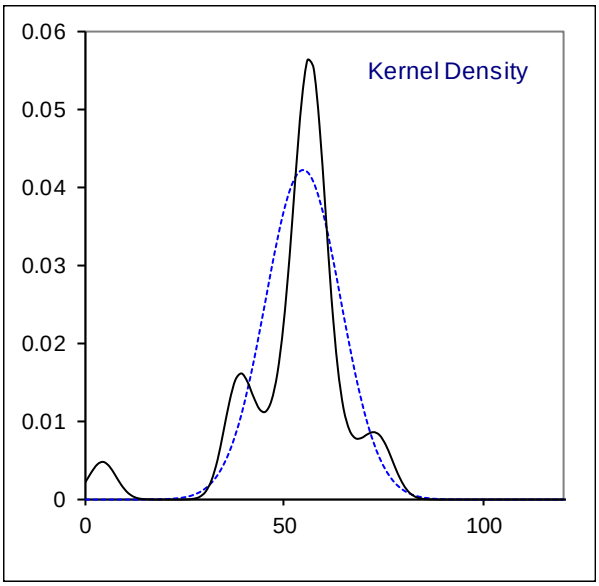

Kernel Density
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150

Determination of Total PCB, sum of all congeners on sample #17233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	56		0.22	
343	EN61619	43.5		-2.01	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840		-----		-----	
912		-----		-----	
1059	EN12766-2-A	37.5	C	-3.08	First reported 31.6
1072	EN61619	58.066		0.59	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271	IEC61619	4.39	C,R(0.01)	-8.99	First reported 0.448
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358	IP462-2	57.21		0.43	
1367	EN61619	53.78		-0.18	
1374		-----		-----	
1396	IP462-2	75.0025		3.61	
1429		-----		-----	
1435	EN12766-2-A	55.93		0.20	
1442	IEC61619	48.575		-1.11	
1495	EN12766-2-A	53.9		-0.16	
1513	IEC61619	50.8890		-0.69	
1516	EN61619	57.04		0.40	
1551		-----		-----	
1633	IEC61619	54.94		0.03	
1660	IEC61619	60		0.93	
1702	EN61619	55		0.04	
1704	EN61619	38.95		-2.82	
1743	IEC61619	57		0.40	
1763		-----		-----	
1801	EN61619	58.25		0.62	
1816	EN61619	59.0		0.75	
1841	IEC61619	65.00		1.82	
1875		-----		-----	
1885		-----		-----	
1888	EN61619	38.5		-2.90	
2320		-----		-----	
3150	EN15803	368	R(0.01)	55.88	
3195		-----		-----	
6086	EN61619	71.16		2.92	
6106		-----		-----	
6133		-----		-----	
6157		-----		-----	

normality	OK
n	22
outliers	2
mean (n)	54.7815
st.dev. (n)	9.42465
R(calc.)	26.3890
st.dev.(EN61619:99)	5.60549
R(EN61619:99)	15.6954

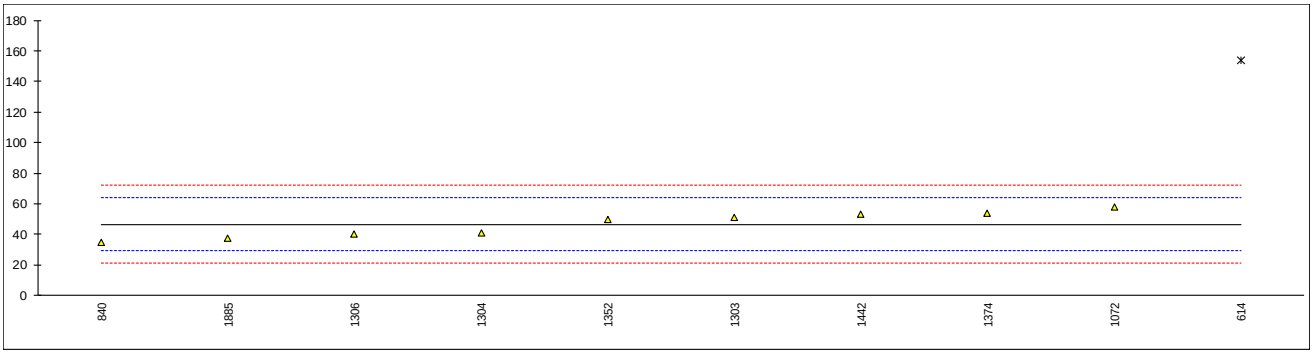

100
90
80
70
60
50
40
30
20
10
0
1271
1059
1888
1704
343
1442
1513
1367
1495
1633
1702
1435
341
1743
1516
1358
1072
1801
1816
1660
1841
6086
1396
3150

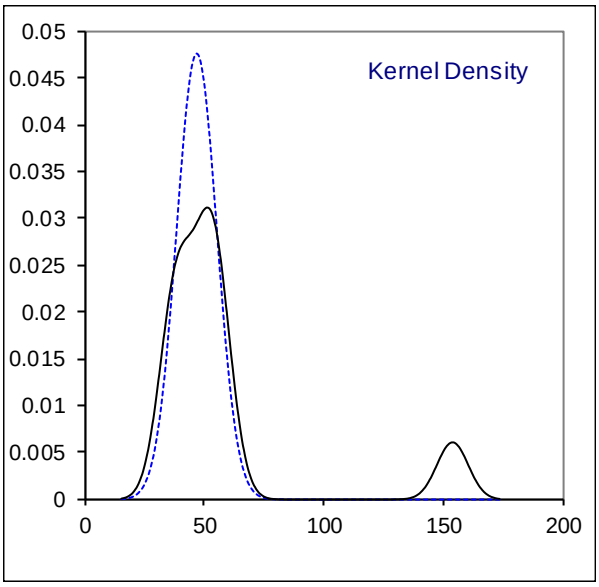

Kernel Density
0.06
0.05
0.04
0.03
0.02
0.01
0
0
50
100

Determination of Total PCB, sum of all Aroclors on sample #17233; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
498		-----		-----	
511		-----		-----	
614	D4059	153.6	G(0.01)	12.52	
840	D4059	34.8		-1.39	
912		-----		-----	
1059		-----		-----	
1072	D4059	58.066		1.34	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1271		-----		-----	
1303	D4059	51.459		0.56	
1304	INH-127	40.868		-0.68	
1306	INH-122	39.99		-0.78	
1352	In house	50.107		0.41	
1358		-----		-----	
1367		-----		-----	
1374	D4059	53.69		0.83	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1442	D4059	53.357		0.79	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1551		-----		-----	
1633		-----		-----	
1660		-----		-----	
1702		-----		-----	
1704		-----		-----	
1743		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1885	INH-6013	37.4		-1.08	
1888		-----		-----	
2320		-----		-----	
3150		-----		-----	
3195		-----		-----	
6086		-----		-----	
6106		-----		-----	
6133		-----		-----	
6157		-----		-----	

normality	OK
n	9
outliers	1
mean (n)	46.6374
st.dev. (n)	8.39433
R(calc.)	23.5041
st.dev.(D4059:00 (silicone))	8.54079
R(D4059:00 (silicone))	23.9142


180
160
140
120
100
80
60
40
20
0
840
1885
1306
1304
1352
1303
1442
1374
1072
614


Kernel Density
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150
200

APPENDIX 2

Number of participating laboratories per country

6 labs in	AUSTRALIA
2 labs in	BELGIUM
1 lab in	BOSNIA and HERZEGOVINA
1 lab in	CROATIA
2 lab in	FINLAND
2 labs in	FRANCE
6 labs in	GERMANY
1 lab in	GREECE
2 labs in	INDIA
3 labs in	ITALY
1 lab in	MALAYSIA
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NORWAY
1 lab in	PERU
2 labs in	PORTUGAL
2 labs in	SLOVENIA
1 lab in	SOUTH AFRICA
6 labs in	SPAIN
1 lab in	SRI LANKA
6 labs in	UNITED KINGDOM
1 lab in	VIETNAM

APPENDIX 3

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	= probably 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
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

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