Results of Proficiency Test
PCB in (Mineral) Oil
November 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Appendices:

1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies organizes a proficiency test for PCB in (mineral) oil every year. During the annual proficiency testing program 2014/2015, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.
In this interlaboratory study, 50 laboratories from 20 different countries have participated. See appendix 3 for the number of participants per country. In this report the results of the 2014 proficiency test on PCB are presented and discussed. This report is also electronically available through the iis internet site http://www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. It was decided to send one 8 ml vial with mineral oil contaminated with PCB (labelled #14225). Sample analyses for fit-for-use and homogeneity testing were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded 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, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, R007). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 organisation 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 April 2014 (iis-protocol, version 3.3). The protocol can be downloaded from iis website http://www.iisnl.com.

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 only one sample was used. The necessary bulk material for the sample, was a mineral oil which was spiked with 40 mg/kg PCBs. This was done using a PCB contaminated oil (positive on PCBs). This contaminated oil was donated by a third party laboratory. After ultrasonic homogenisation, 112 subsamples were transferred to 8 mL amber glass vials, all labelled #14225.
The homogeneity of the subsamples #14225 was checked by determination of the organic chloride content in accordance with UOP779-08 on eight stratified randomly selected samples:

	Organic chloride in mg/kg
sample #14225-1	46
sample #14225-2	46
sample #14225-3	46
sample #14225-4	46
sample #14225-5	45
sample #14225-6	46
sample #14225-7	46
sample #14225-8	46

Table 1: homogeneity test results of subsample #14225

From the above test results, the repeatability was calculated and compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Organic chloride in mg/kg
r (samples #14225)	1.0
reference method	UOP779-08
0.3 x $R_{(reference\ method)}$	3.2

Table 2: evaluation of the observed repeatability

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

To each of the participating laboratories, one vial of 8 mL (labelled #14225) was sent on November 5, 2014.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine Total Organo Halogenic Compounds (TOX) and Poly Chlorinated Biphenyls (via seven individual PCBs, via the determination of the total PCB content and/or via Aroclors standards) on the sample.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1. Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation 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 April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or 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 (ref. 15). 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 these 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

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.

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 some no problems were encountered during execution. In total three participants reported results after the final reporting date and two participants did not report any results at all. Not all participants were able to report results for all tests. In total 48 participating laboratories reported 239 numerical results. Observed were 5 outlying results, which is 2.1% of the numerical results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

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

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. For the statistical evaluation of the individual PCBs the method EN12766-1:00 was used, this method is equal to IP462-1:01.

In the iis PT reports, ASTM 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. D2086:00(2010)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2086:00 will be used.

For the results on sample #14225, the following was concluded:

TOX — This determination may not be problematic. No statistical outliers were observed, however only three test results were reported. The calculated reproducibility is in agreement with the estimated reproducibility calculated using UOP779:08.

Individual PCBs: This determination was problematic for all seven congeners. For the evaluation of the individual congeners method EN12766-1:00 / IP462-1:01 was used. In the methods IEC61619:98 and DIN51527:93 only the reproducibilities of the total PCB content are mentioned, while in EN12766-1:00 / IP462-1:01 the reproducibilities for each individual congener are mentioned. In total only one statistical outlier was observed and nine test results were excluded. The test results of labcode 1201 were excluded for reporting low values for all congeners. Labcode 1841 reported zero as a test result, which statistically is not considered a value and was therefore excluded. The calculated reproducibilities of all seven congeners after rejection of the suspect data are not in agreement with requirements of EN12766-1:00 / IP462-1:01.

Individual Aroclors: The determination of the individual Aroclors may be very problematic depending on the method used. In total two statistical outliers were observed and two results were excluded for reporting a zero value.
In total 18 participants reported the presence of Aroclor 1260 between 29 and 46 mg/kg. Of these 18 participants five participants reported test results between 4.3 and 24.5 for Aroclor 1254, while ten other participants reported Aroclor 1254 being absent (values < 2 mg/kg).
The reported test results for Aroclor 1242 are all low (<4.1 mg/kg) and the spread is large (values between 1.7 and 4.1). Therefore it was concluded that for both components inconsistent test results were reported and no z-scores should be calculated for Aroclor 1242 and Aroclor 1254. The calculated reproducibility of Aroclor 1260 after rejection of the outlying results is in agreement with all three requirements of ASTM D4059:00(2010).

The majority of the laboratories identified Aroclor 1260 as the main compound in the sample. ASTM D4059 describes two ways of determining the Aroclor compounds. The first determines all three compounds by using three specifically defined windows within the chromatogram. The second checks whether the chromatogram is similar to the chromatogram of one of the three compounds and then only determines this ‘main’ compound. Since Aroclor 1260 was found to be the main compound, some laboratories only reported the content for this Aroclor, while others reported the content of all three. This may account for the inconsistency in values found for the test results of Aroclor 1242 and Aroclor 1254.

Some participants used EN61619 or IEC61619 to determine the content of Aroclor in the sample. However in this method, Aroclor is only used for qualitative analysis, not for quantitative analysis. So this test is in principle not applicable to determine Aroclor compounds. Some of the inconsistency found for the test results for Aroclor 1242 and Aroclor 1254 may be due to using this qualitative method as a quantative method.

Total PCB:

Total PCB, "5 times of sum 6 PCB congeners"
This determination and/or calculation of total PCB content was problematic. No statistical outliers were observed. However, the calculated reproducibility is not agreement with the requirements of EN12766-2 method B:2001.
iis also calculated the total PCB as 5 times the sum of 6 congeners. An error in calculation was found for the test results of four laboratories. The test results of labcode 1899 were excluded for using all seven congeners in the calculation. The consensus value of the group was in agreement with the consensus value found with the individual summation of the six congeners calculated by iis (58.23 vs 60.12 mg/kg).

Total PCB, "sum of all PCB congeners"
This determination and/or calculation of total PCB content was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12766-2 method A:2001 and EN61619:98.

Total PCB, "sum of all Aroclors"
This determination and/or calculation of total PCB content was not problematic. Two statistical outliers were observed. However, the calculated reproducibility is in full agreement with the requirements of ASTM D4059:00(2010). iis also calculated the total PCB as the sum of all Aroclors. An error in calculation was found for the test results of two laboratories. The consensus value of the group was in close range with the consensus value found with the individual summation of the Aroclors calculated by iis (37.422 vs 42.73 mg/kg).

Summary:

All participants agreed that sample #14225 was positive on PCBs.
From the data on total organic chloride (TOX) an average concentration of 36.8 mg/kg was calculated. From this concentration a total content of 61.3 mg PCB/kg was estimated using an average Cl content of 60%, assuming the presence of only Aroclor 1260. This content is somewhat higher than the estimated total PCB content using the other methods.

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

	#14225
total PCB content, estimated by TOX data, in mg/kg	61.3
total PCB content, 5 times the sum of 6 congeners, in mg/kg	58.2 – 59.7
total PCB content, sum of all congeners, in mg/kg	40.4
total PCB content, using Aroclor method, in mg/kg	37.4 -37.8

Table 3: Comparison of estimations of total PCB content in sample #14225.

The total PCB content as determined by EN12766-2, method A (or IEC61619:98) is in good agreement with the total PCB content as determined by the Aroclor method. The range of all four above estimates for total PCB content is quite acceptable in view of the required precision.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu IEC, EN, or ASTM standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX	mg/kg	3	36.8	1.4	6.3
PCB no. 28	mg/kg	17	0.18	0.21	0.07
PCB no. 52	mg/kg	15	0.11	0.13	0.03
PCB no. 101	mg/kg	23	1.12	0.88	0.54
PCB no. 118	mg/kg	14	0.21	0.17	0.08
PCB no. 138	mg/kg	23	3.18	2.16	1.58
PCB no. 153	mg/kg	23	3.59	2.33	1.78
PCB no. 180	mg/kg	23	3.85	2.77	1.91
Aroclor 1242	mg/kg	10	2.88	2.12	(2.96)
Aroclor 1254	mg/kg	10	<2	n.a.	n.a.
Aroclor 1260	mg/kg	16	33.04	7.27	18.47
Total PCB 5 times sum of 6 congeners	mg/kg	16	58.23	36.25	26.19
Total PCB sum of all congeners	mg/kg	18	40.40	10.36	12.10
Total PCB sum of Aroclors	mg/kg	11	37.42	10.75	20.27

table 4: Performance of the group of participating laboratories on sample #14225

() Values between brackets should be used with due care, see also paragraph 4.1

Without further statistical calculations it can be concluded that for many components there is not a good compliance of the group of participating laboratories with the relevant standards, except for Aroclor 1242, Aroclor 1260, Total PCB sum of all congeners and sum of Aroclors. The problematic components have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2014 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2014	*November 2013*	*October 2012*	*November 2011*
Number of reporting labs	48	44	41	38
Number of results reported	239	254	204	195
Statistical outliers	5	6	10	4
Percentage outliers	2.1%	2.4%	4.9%	2.0%

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 standards. The conclusions are given the following table:

Determination	*November 2014*	*November 2013*	*October 2012*	*November 2011*
TOX	++*	--	n.e.	n.e
PCB (individual)	-	--	--	+/-
Aroclor (individual)	+	--	-	+/-
Total PCB 5 * sum of 6 congeners	-	+/-	-	-
Total PCB sum of all congeners	+	-	--	--
Total PCB sum of Aroclors	++	+/-	+	+

Table 6: comparison of observed precisions against standard requirements
*) based on three results

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Total Organohalogenic Compounds (TOX) on sample #14225; results in mg/kg.

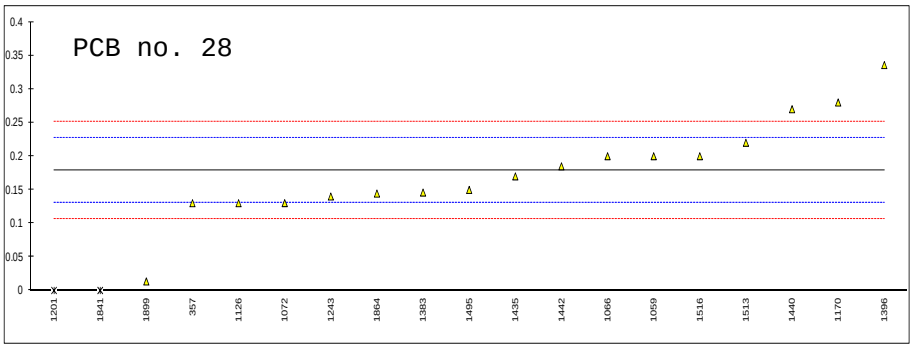
lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
1059		-----		-----	
1066	UOP779	37		0.10	
1072		-----		-----	
1126	EN14077	36.2		-0.25	
1170		-----		-----	
1201	UOP779	37.1		0.15	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1383		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1548		-----		-----	
1568		-----		-----	
1660		-----		-----	
1743		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1864		-----		-----	
1875		-----		-----	
1888		-----		-----	
1899		-----		-----	
1956		-----		-----	
1965		-----		-----	
2122		-----		-----	
3195		-----		-----	

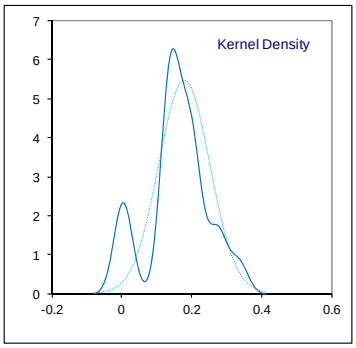
normality	unknown
n	3
outliers	0
mean (n)	36.8
st.dev. (n)	0.49
R(calc.)	1.4
R(Horwitz)	6.3

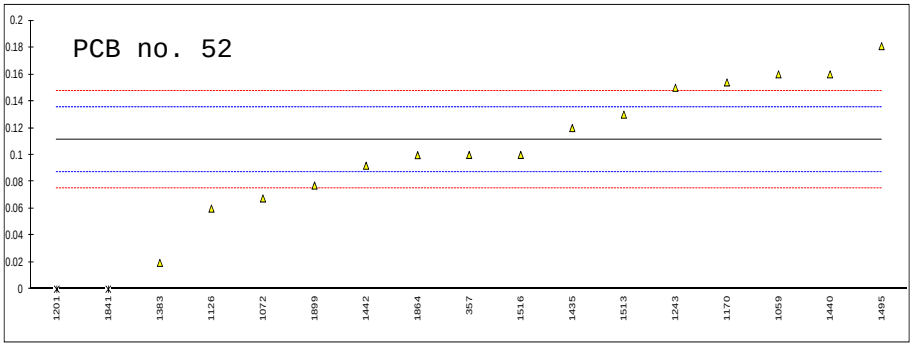
Determination of PCB 28, 52 and 101 on sample #14225; results in mg/kg.

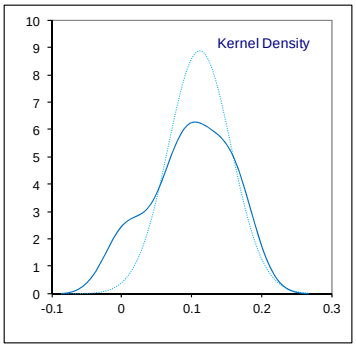
lab	Method	No.28	mark	z(targ)	No.52	mark	z(targ)	No.101	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357	EN12766-1	0.13		-2.02	0.10		-0.94	0.86		-1.35
398		-----		-----	-----		-----	-----		-----
445		-----		-----	-----		-----	-----		-----
498	EN12766	<0.3		-----	<0.3		-----	1.27		0.78
511		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
1059	EN12766-1	0.20		0.86	0.16		4.01	1.21		0.47
1066	EN12766-1	0.2		0.86	<0.2		-----	1.2		0.42
1072	IEC61619	0.1302		-2.02	0.0676		-3.62	1.8934		4.01
1126	EN12766	0.13		-2.02	0.06		-4.24	0.85		-1.40
1170	EN12766-1	0.28	C	4.16	0.154		3.52	1.113		-0.03
1201	EN12766-1	0	ex	-7.39	0	ex	-9.20	0.3	ex	-4.25
1243	EN12766-2	0.14		-1.61	0.15		3.19	0.9		-1.14
1258		-----		-----	-----		-----	-----		-----
1303		-----		-----	-----		-----	-----		-----
1304		-----		-----	-----		-----	-----		-----
1306		-----		-----	-----		-----	-----		-----
1338		-----		-----	-----		-----	-----		-----
1352		-----		-----	-----		-----	-----		-----
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374		-----		-----	-----		-----	-----		-----
1383	EN12766	0.1459		-1.37	0.0196		-7.58	1.0337		-0.45
1396	IP462	0.335746		6.46	<0.1		-----	1.19023		0.37
1429		-----		-----	-----		-----	-----		-----
1435	EN12766-1	0.17	C	-0.37	0.12	C	0.71	1.14	C	0.11
1440	EN12766	0.27		3.75	0.16		4.01	1.43		1.61
1442	EN12766-1	0.185		0.24	0.092		-1.60	1.11		-0.05
1458		-----		-----	-----		-----	-----		-----
1495	EN12766	0.1498		-1.21	0.1810		5.74	0.7276		-2.03
1513	IEC61619	0.22		1.69	0.13		1.53	1.67		2.85
1516	IEC61619	0.20		0.86	0.10		-0.94	1.38		1.35
1548		-----		-----	-----		-----	-----		-----
1568		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1743		-----		-----	-----		-----	-----		-----
1763	EN12766-1	<0,2		-----	<0,2		-----	0.75		-1.92
1801		-----		-----	-----		-----	-----		-----
1816	IEC61619	<2		-----	<2		-----	1.36		1.25
1841	IEC61619	0.00	ex	-7.39	0.00	ex	-9.20	1.39		1.40
1864	EN12766-1	0.1444		-1.43	0.0998		-0.96	0.9803		-0.72
1875	EN12766	<0.1		-----	<0.1		-----	0.74		-1.97
1888		-----		-----	-----		-----	-----		-----
1899	EN12766	0.0135		-6.83	0.0772		-2.82	0.6440		-2.47
1956		-----		-----	-----		-----	-----		-----
1965		-----		-----	-----		-----	-----		-----
2122		-----		-----	-----		-----	-----		-----
3195	EN12766-1	<0.20		-----	<0.20		-----	0.91		-1.09
	normality	suspect			OK			OK		
	n	17			15			23		
	outliers	0 (+2ex)			0 (+2ex)			0 (+1ex)		
	mean (n)	0.179			0.111			1.120		
	st.dev. (n)	0.0731			0.0449			0.3133		
	R(calc.)	0.205			0.126			0.877		
	R(EN12766-1:00)	0.068			0.034			0.540		

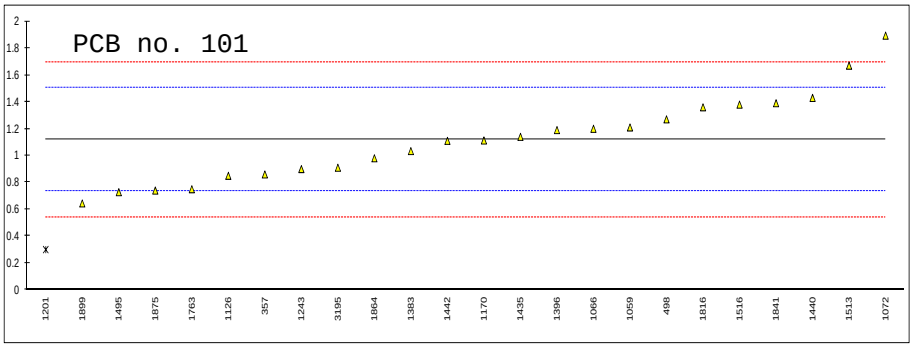
Lab 117 first reported for PCB no. 28: 0.217
Lab 1201 was excluded: see §4.1
Lab 1841 was excluded for PCB no. 28 and PCB no. 52, for zero is not a real value
Lab 1435 first reported for PCB no. 28: 0.13 , for PCB no. 52: 0.10 and for PCB no. 101: 0.86

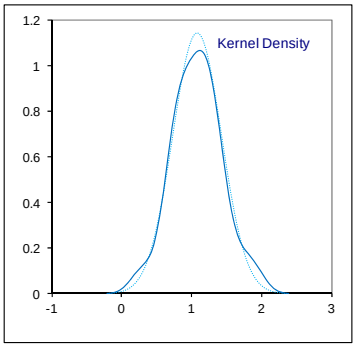

PCB no. 28
1201
1841
1899
357
1126
1072
1243
1864
1383
1495
1435
1442
1066
1059
1516
1513
1440
1170
1396


Kernel Density


PCB no. 52
1201
1841
1383
1126
1072
1899
1442
1864
357
1516
1435
1513
1243
1170
1059
1440
1495

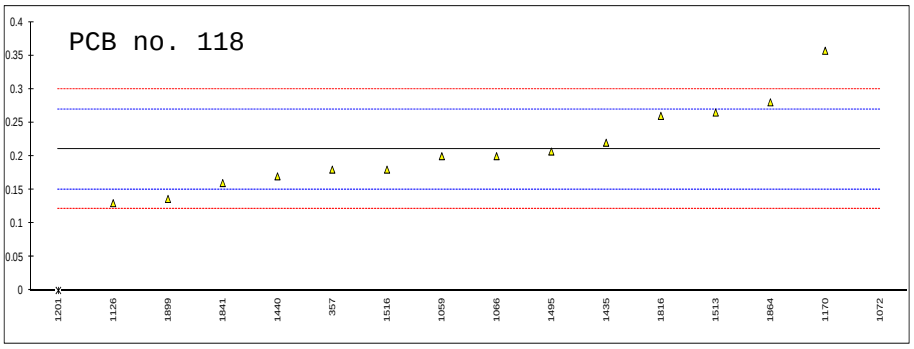

Kernel Density

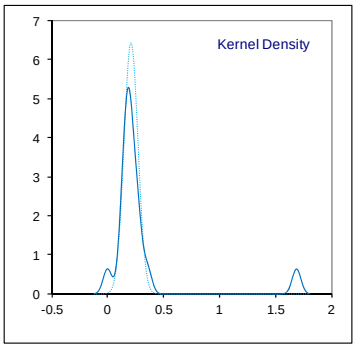

PCB no. 101
1201
1899
1495
1875
1763
1126
357
1243
3195
1864
1383
1442
1170
1435
1396
1066
1059
498
1816
1516
1841
1440
1513
1072

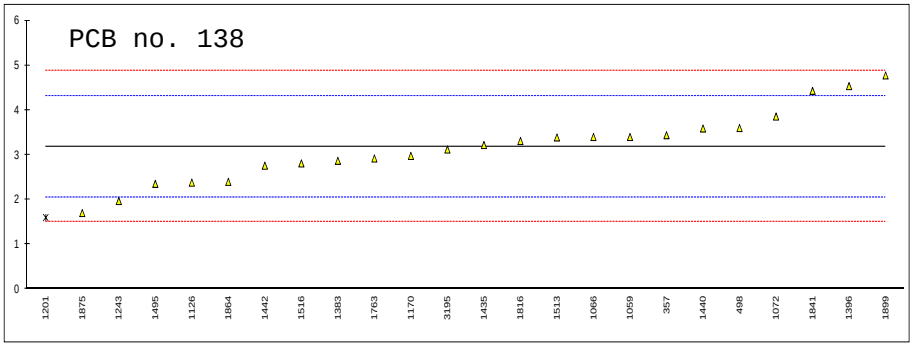

Kernel Density

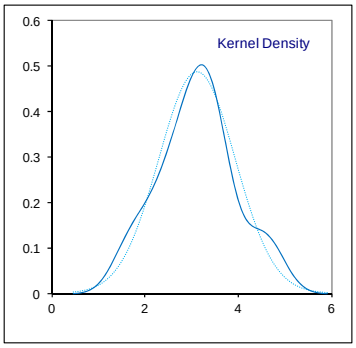
Determination of PCB 118 and 138 on sample #14225; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766-1	0.18		-1.02	3.44		0.46	
398		-----		-----	-----		-----	
445		-----		-----	-----		-----	
498	EN12766	<0.3		-----	3.60		0.74	
511		-----		-----	-----		-----	
614		-----		-----	-----		-----	
1059	EN12766-1	0.20		-0.35	3.40		0.39	
1066	EN12766-1	0.2		-0.35	3.4		0.39	
1072	IEC61619	1.6891	G(0.01)	49.53	3.8571		1.20	
1126	EN12766	0.13		-2.69	2.38		-1.43	
1170	EN12766-1	0.357		4.91	2.978		-0.36	
1201	EN12766-1	0	ex	-7.05	1.6	ex	-2.81	Excluded, see §4.1
1243	EN12766-2	-----		-----	1.97		-2.16	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1374		-----		-----	-----		-----	
1383	EN12766	-----		-----	2.8681		-0.56	
1396	IP462	-----		-----	4.53663		2.41	
1429		-----		-----	-----		-----	
1435	EN12766-1	0.22	C	0.32	3.22	C	0.07	First reported: 0.20 and 2.42
1440	EN12766	0.17		-1.35	3.59		0.72	
1442	EN12766-1	-----		-----	2.76		-0.75	
1458		-----		-----	-----		-----	
1495	EN12766	0.2069		-0.12	2.353		-1.47	
1513	IEC61619	0.265		1.83	3.39		0.37	
1516	IEC61619	0.18		-1.02	2.81		-0.66	
1548		-----		-----	-----		-----	
1568		-----		-----	-----		-----	
1660		-----		-----	-----		-----	
1743		-----		-----	-----		-----	
1763	EN12766-1	-----		-----	2.92		-0.47	
1801		-----		-----	-----		-----	
1816	IEC61619	0.26		1.66	3.31		0.23	
1841	IEC61619	0.16		-1.69	4.43		2.22	
1864	EN12766-1	0.2802	C	2.34	2.3974		-1.40	First reported: 2.6818
1875	EN12766	-----		-----	1.70		-2.64	
1888		-----		-----	-----		-----	
1899	EN12766	0.1363		-2.48	4.7742		2.83	
1956		-----		-----	-----		-----	
1965		-----		-----	-----		-----	
2122		-----		-----	-----		-----	
3195	EN12766-1	<0.20		-----	3.12		-0.11	
	normality	OK			OK			
	n	14			23			
	outliers	1 (+1ex)			0 (+1ex)			
	mean (n)	0.210			3.183			
	st.dev. (n)	0.0621			0.7713			
	R(calc.)	0.174			2.160			
	R(EN12766-1:00)	0.084			1.575			


PCB no. 118
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
1201
1126
1899
1841
1440
357
1516
1059
1066
1495
1435
1816
1513
1864
1170
1072

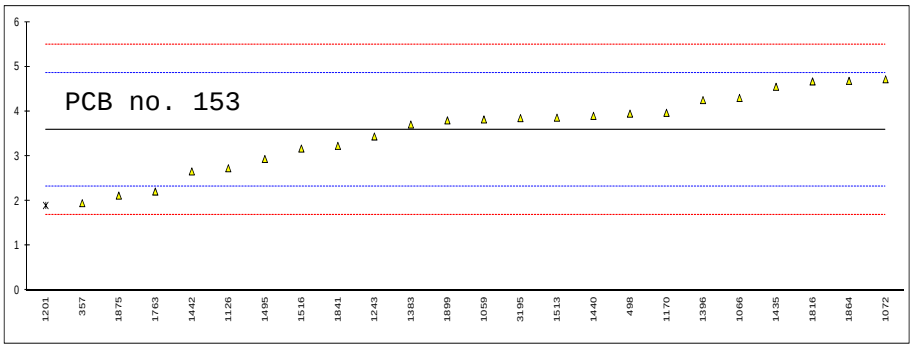

Kernel Density
7
6
5
4
3
2
1
0
-0.5
0
0.5
1
1.5
2

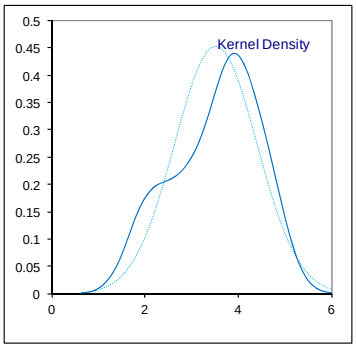

PCB no. 138
6
5
4
3
2
1
0
1201
1875
1243
1495
1126
1864
1442
1516
1383
1763
1170
3195
1435
1816
1513
1066
1059
357
1440
498
1072
1841
1396
1899

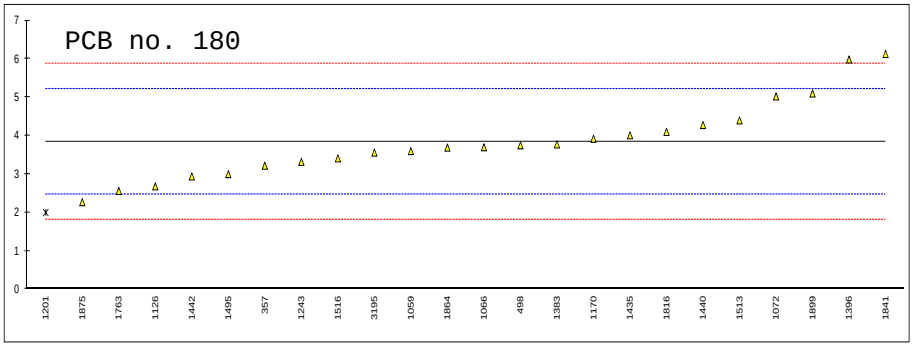

Kernel Density
0.6
0.5
0.4
0.3
0.2
0.1
0
0
2
4
6

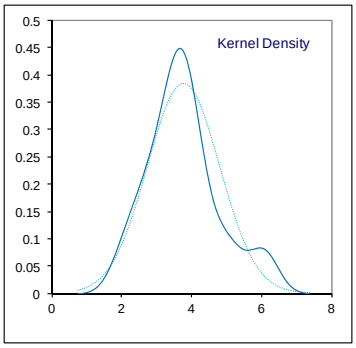
Determination of PCB 153 and 180 on sample #14225; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766-1	1.95		-2.58	3.22		-0.92	
398		-----		-----	-----		-----	
445		-----		-----	-----		-----	
498	EN12766	3.95		0.57	3.75		-0.14	
511		-----		-----	-----		-----	
614		-----		-----	-----		-----	
1059	EN12766-1	3.82	C	0.37	3.60	C	-0.36	First reported: 1.00 and 0.19
1066	EN12766-1	4.3		1.13	3.7		-0.21	
1072	IEC61619	4.7170		1.78	5.0239		1.73	
1126	EN12766	2.73		-1.35	2.68		-1.71	
1170	EN12766-1	3.968		0.60	3.921		0.11	
1201	EN12766-1	1.9	ex	-2.66	2.0	ex	-2.71	
1243	EN12766-2	3.44		-0.23	3.32		-0.77	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1374		-----		-----	-----		-----	
1383	EN12766	3.7066		0.19	3.7738		-0.10	
1396	IP462	4.25216		1.05	5.98768		3.15	
1429		-----		-----	-----		-----	
1435	EN12766-1	4.55	C	1.52	4.01	C	0.24	First reported: 3.43 and 2.99
1440	EN12766	3.90		0.49	4.28		0.64	
1442	EN12766-1	2.66		-1.46	2.94		-1.33	
1458		-----		-----	-----		-----	
1495	EN12766	2.937		-1.02	3.000		-1.24	
1513	IEC61619	3.86		0.43	4.40		0.82	
1516	IEC61619	3.17		-0.65	3.41		-0.64	
1548		-----		-----	-----		-----	
1568		-----		-----	-----		-----	
1660		-----		-----	-----		-----	
1743		-----		-----	-----		-----	
1763	EN12766-1	2.21		-2.17	2.56		-1.89	
1801		-----		-----	-----		-----	
1816	IEC61619	4.67		1.71	4.10		0.37	
1841	IEC61619	3.23		-0.56	6.13		3.35	
1864	EN12766-1	4.6814		1.73	3.6910		-0.23	
1875	EN12766	2.12		-2.31	2.27		-2.31	
1888		-----		-----	-----		-----	
1899	EN12766	3.7997		0.34	5.1003		1.84	
1956		-----		-----	-----		-----	
1965		-----		-----	-----		-----	
2122		-----		-----	-----		-----	
3195	EN12766-1	3.85		0.42	3.56		-0.42	
	normality	OK			OK			
	n	23			23			
	outliers	0 (+1ex)			0 (+1ex)			
	mean (n)	3.586			3.845			
	st.dev. (n)	0.8319			0.9874			
	R(calc.)	2.329			2.765			
	R(EN12766-1:00)	1.778			1.908			


PCB no. 153
0
1
2
3
4
5
6
1201 357 1875 1763 1442 1126 1495 1516 1841 1243 1383 1899 1059 3195 1513 1440 498 1170 1396 1066 1435 1816 1864 1072


Kernel Density
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
0.5
0
2
4
6

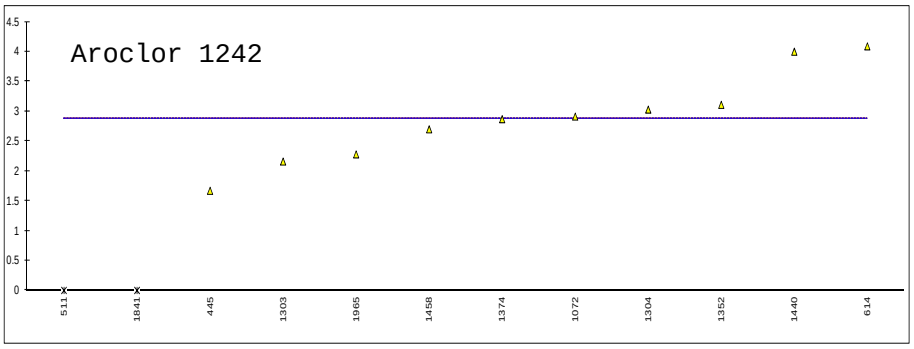

PCB no. 180
0
1
2
3
4
5
6
7
1201 1875 1763 1126 1442 1495 357 1243 1516 3195 1059 1864 1066 498 1383 1170 1435 1816 1440 1513 1072 1899 1396 1841

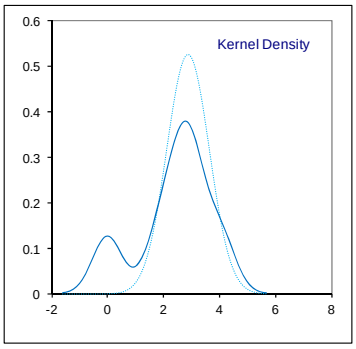

Kernel Density
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
0.5
0
2
4
6
8

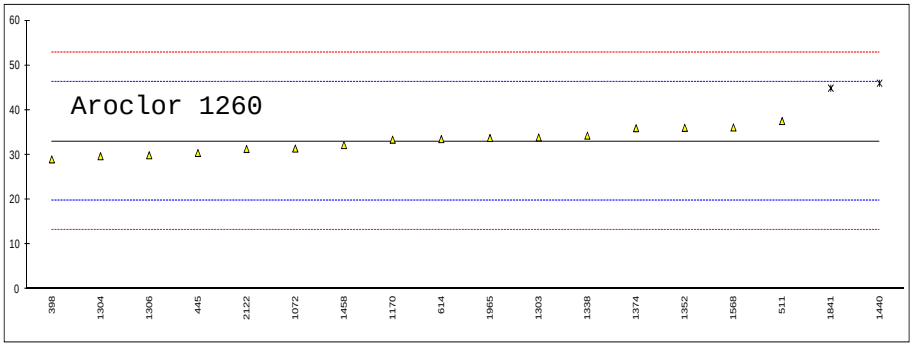
Determination of Aroclor 1242, 1254 and 1260 on sample #14225; results in mg/kg.

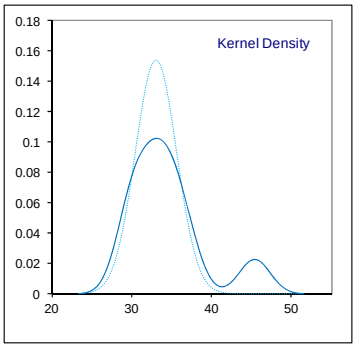
lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357		-----		-----	-----		-----	-----		-----
398	D4059	<1		-----	<1		-----	29		-0.61
445	EN61619	1.67		-----	5.32		-----	30.43		-0.40
498		-----		-----	-----		-----	-----		-----
511	D4059	0.00	ex	-----	0.00		-----	37.56		0.69
614	D4059	4.09		-----	<2	C	-----	33.52		0.07
1059		-----		-----	-----		-----	-----		-----
1066		-----		-----	-----		-----	-----		-----
1072	in house	2.913		-----	7.282		-----	31.418		-0.25
1126		-----		-----	-----		-----	-----		-----
1170	in house	-----		-----	24.5	C	-----	33.4		0.06
1201		-----		-----	-----		-----	-----		-----
1243		-----		-----	-----		-----	-----		-----
1258		-----		-----	-----		-----	-----		-----
1303	in house	2.16		-----	<1		-----	33.9		0.13
1304	in house	3.03		-----	-----		-----	29.70		-0.51
1306	in house	-----		-----	-----		-----	29.917		-0.47
1338	in house	-----		-----	-----		-----	34.3		0.19
1352	IEC61619	3.11		-----	4.34		-----	36.05		0.46
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374	D4059	2.87		-----	n.d.		-----	35.97		0.44
1383		-----		-----	-----		-----	-----		-----
1396		-----		-----	-----		-----	-----		-----
1429		-----		-----	-----		-----	-----		-----
1435		-----		-----	-----		-----	-----		-----
1440	in house	4		-----	n.d.		-----	46	DG(0.01)	1.97
1442		-----		-----	-----		-----	-----		-----
1458	D4059	2.7		-----	<0.4		-----	32.2		-0.13
1495		-----		-----	-----		-----	-----		-----
1513		-----		-----	-----		-----	-----		-----
1516		-----		-----	-----		-----	-----		-----
1548		-----		-----	-----		-----	-----		-----
1568	D4059	n.d.		-----	n.d.		-----	36.1254	C	0.47
1660		-----		-----	-----		-----	-----		-----
1743		-----		-----	-----		-----	-----		-----
1763		-----		-----	-----		-----	-----		-----
1801		-----		-----	-----		-----	-----		-----
1816		-----		-----	-----		-----	-----		-----
1841	EN61619	0.0	ex	-----	0.0		-----	44.9	DG(0.01)	1.80
1864		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
1888		-----		-----	-----		-----	-----		-----
1899		-----		-----	-----		-----	-----		-----
1956		-----		-----	-----		-----	-----		-----
1965	D4059	2.28		-----	<1		-----	33.77		0.11
2122	EN61619	-----		-----	10.31		-----	31.32		-0.26
3195		-----		-----	-----		-----	-----		-----
	normality	OK			unknown			OK		
	n	10			10			16		
	outliers	0 (+2ex)			n.a.			2		
	mean (n)	2.882			<2			33.036		
	st.dev. (n)	0.7579			n.a.			2.5968		
	R(calc.)	2.122			n.a.			7.271		
	R(D4059:00 (silicone))	(2.964)			n.a.			18.465		
	R(D4059: 00 (packed))	(2.278)			n.a.			14.193		
	R(D4059:00 (megabore))	(1.748)			n.a.			10.886		

Lab 511 and 1841 were excluded for PCB no. 28 for zero is not a real value
Lab 614 first reported for Aroclor 1254: 29.12
Lab 1170 first reported for Aroclor 1254: 35.1
Lab 1568 first reported for Aroclor 1260: 48.6660


Aroclor 1242
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
511
1841
445
1303
1965
1458
1374
1072
1304
1352
1440
614


Kernel Density
0.6
0.5
0.4
0.3
0.2
0.1
0
-2
0
2
4
6
8

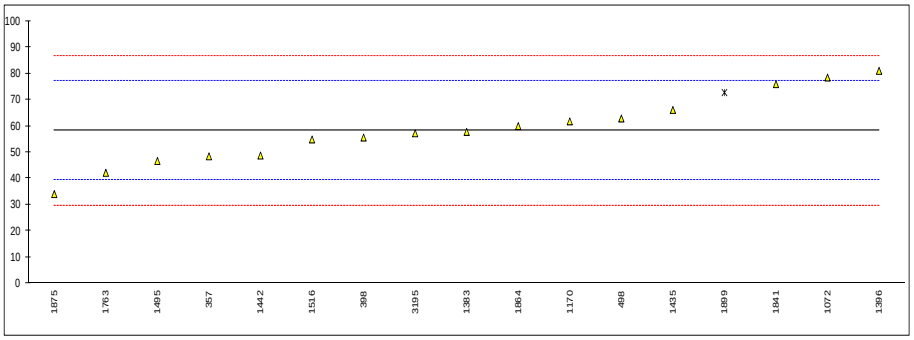

Aroclor 1260
60
50
40
30
20
10
0
398
1304
1306
445
2122
1072
1458
1170
614
1965
1303
1338
1374
1352
1568
511
1841
1440

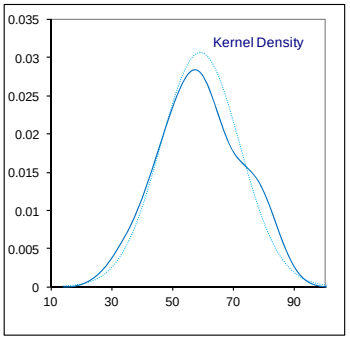

Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
20
30
40
50

Determination of Total PCB, 5 times the sum of 6 congeners on sample #14225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	48.47		-1.03	
398	EN12766-2-B	55.61		-0.28	
445		-----		-----	
498	EN12766	62.85		0.49	
511		-----		-----	
614		-----		-----	
1059		-----		-----	
1066		-----		-----	
1072	EN12766-2-B	78.446		2.13	
1126		-----		-----	
1170	EN12766-2-B	61.76	E	0.37	Error in calculation, iis calc. 62.07
1201		-----		-----	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1383	EN12766-2-B	57.7393		-0.05	
1396	IP462-2	80.9820	E	2.40	Error in calculation, iis calc. 81.5122
1429		-----		-----	
1435	EN12766-2-B	66.1	C	0.83	First reported: 9.94
1440		-----		-----	
1442	EN12766-2-B	48.74		-1.00	
1458		-----		-----	
1495	EN12766-2-B	46.7		-1.21	
1513		-----		-----	
1516	EN12766-2-B	54.87	E	-0.35	Error in calculation, iis calc. 55.35
1548		-----		-----	
1568		-----		-----	
1660		-----		-----	
1743		-----		-----	
1763	EN12766-2-B	42.20		-1.69	
1801		-----		-----	
1816		-----		-----	
1841	IEC61619	75.9		1.86	
1864	EN12766-2-B	59.9715	C	0.18	First reported: 17.4765
1875	EN12766-B	34.1		-2.54	
1888		-----		-----	
1899	EN61619	72.72	C, E, ex	1.53	Error in calculation, iis calc. 72.0445, excluded as 5*sum of 7 cong.
1956		-----		-----	
1965		-----		-----	
2122		-----		-----	
3195	EN12766-2-B	57.21		-0.11	
	normality	OK			
	n	16			
	outliers	0 (+1ex)			
	mean (n)	58.228			
	st.dev. (n)	12.9467			
	R(calc.)	36.251			
	R(EN12766-2B:01)	26.187			

Lab 1899: First reported 13405.17, reported this value as sum of all congeners.

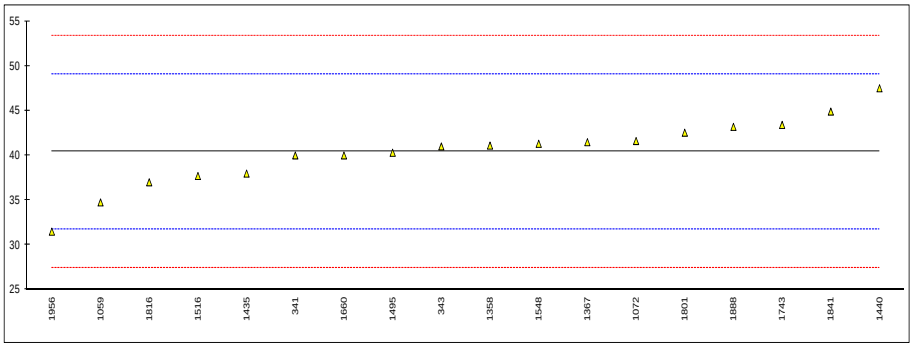


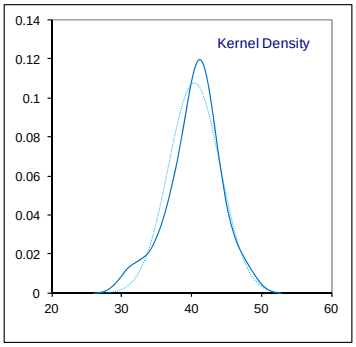


Determination of Total PCB, sum of all congeners on sample #14225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	40		-0.09	
343	EN61619	41	C	0.14	Reported as Total PCB, sum of all Aroclors
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
1059	EN12766-2-A	34.75		-1.31	
1066		-----		-----	
1072	EN61619	41.6130		0.28	
1126		-----		-----	
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358	IP462-2	41.1		0.16	
1367	EN61619	41.48		0.25	
1374		-----		-----	
1383		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435	EN61619	37.97	C	-0.56	First reported: 29.35
1440	EN61619	47.5		1.64	
1442		-----		-----	
1458		-----		-----	
1495	EN12766-2-A	40.3		-0.02	
1513		-----		-----	
1516	IEC61619	37.70		-0.63	
1548	EN61619	41.3		0.21	
1568		-----		-----	
1660	EN61619	40		-0.09	
1743	IEC61619	43.41		0.70	
1763		-----		-----	
1801	EN61619	42.54		0.49	
1816	EN61619	37.0		-0.79	
1841	IEC61619	44.9		1.04	
1864		-----		-----	
1875		-----		-----	
1888	EN61619	43.19		0.65	
1899		-----		-----	
1956	EN61619	31.4886		-2.06	
1965		-----		-----	
2122		-----		-----	
3195		-----		-----	

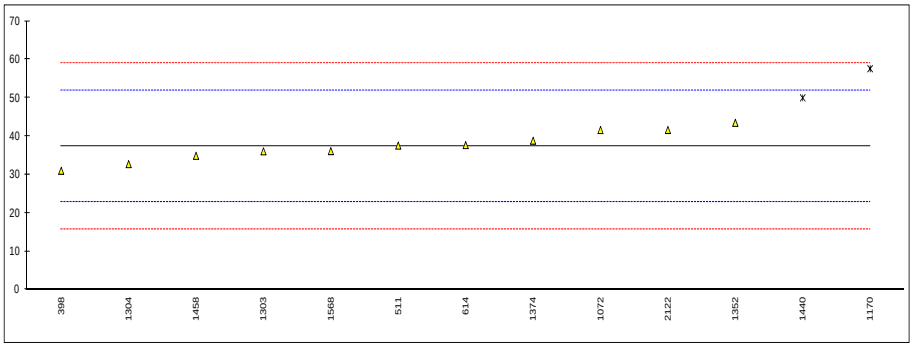
normality	suspect
n	18
outliers	0
mean (n)	40.402
st.dev. (n)	3.7006
R(calc.)	10.362
R(EN61619:98)	12.101

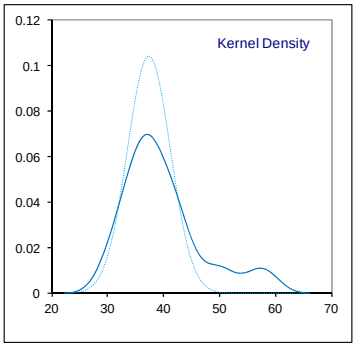




Determination of Total PCB, sum of all Aroclors" on sample #14225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398	D4059	31		-0.89	
445		-----		-----	
498		-----		-----	
511	D4059	37.56		0.02	
614	D4059	37.68	C, E	0.04	First reported: 66.74, Error in calculation, iis calc. 37.61
1059		-----		-----	
1066		-----		-----	
1072	in house	41.6130		0.58	
1126		-----		-----	
1170	in house	57.6	C,DG(0.05),E	2.79	First reported: 68.5, Error in calculation, iis calc. 57.9
1201		-----		-----	
1243		-----		-----	
1258		-----		-----	
1303	in house	36.06		-0.19	
1304	in house	32.73		-0.65	
1306		-----		-----	
1338		-----		-----	
1352	D4059	43.50		0.84	
1358		-----		-----	
1367		-----		-----	
1374	D4059	38.84		0.20	
1383		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1440	in house	50	DG(0.05)	1.74	
1442		-----		-----	
1458	D4059	34.9		-0.35	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1548		-----		-----	
1568	D4059	36.1254	C	-0.18	First reported: 40.6660
1660		-----		-----	
1743		-----		-----	
1763		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1864		-----		-----	
1875		-----		-----	
1888		-----		-----	
1899		-----		-----	
1956		-----		-----	
1965		-----		-----	
2122	INH-61619	41.63		0.58	
3195		-----		-----	
	normality	OK			
	n	11			
	outliers	2			
	mean (n)	37.422			
	st.dev. (n)	3.8401			
	R(calc.)	10.752			Compare R(D4059:10(oil-packed)): 15.584
	R(D4059:10)	20.274	(silicone)		Compare R(D4059:10(oil-megabore)): 11.953





APPENDIX 2

Total PCB by summation of the reported results by iis on sample #14225; results in mg/kg.

lab	method	Sum of 6 congeners	mark	5 x (sum of 6 congeners)	mark	sum of Aroclors	
341		-----		-----		-----	
343		-----		-----		-----	
357	calc by iis	9.7		48.5		-----	
398	calc by iis	-----		-----		29	
445	calc by iis	-----		-----		37.42	
498	calc by iis	12.57		62.85		-----	
511	calc by iis	-----		-----		37.56	
614	calc by iis	-----		-----		37.61	
1059	calc by iis	12.39		61.95		-----	
1066	calc by iis	12.8		64		-----	
1072	calc by iis	15.6892		78.446		41.613	
1126	calc by iis	8.83		44.15		-----	
1170	calc by iis	12.414		62.07		57.9	G(0.05)
1201	calc by iis	5.8	ex	29	ex	-----	
1243	calc by iis	9.92		49.6		-----	
1258		-----		-----		-----	
1303	calc by iis	-----		-----		36.06	
1304	calc by iis	-----		-----		32.73	
1306	calc by iis	-----		-----		29.917	
1338	calc by iis	-----		-----		34.3	
1352	calc by iis	-----		-----		43.5	
1358		-----		-----		-----	
1367		-----		-----		-----	
1374	calc by iis	-----		-----		38.84	
1383	calc by iis	11.5477		57.7385		-----	
1396	calc by iis	16.302446		81.51223		-----	
1429		-----		-----		-----	
1435	calc by iis	13.21		66.05		-----	
1440	calc by iis	13.63		68.15		50	
1442	calc by iis	9.747		48.735		-----	
1458	calc by iis	-----		-----		34.9	
1495	calc by iis	9.3484		46.742		-----	
1513	calc by iis	13.67		68.35		-----	
1516	calc by iis	11.07		55.35		-----	
1548		-----		-----		-----	
1568	calc by iis	-----		-----		36.1254	
1660		-----		-----		-----	
1743		-----		-----		-----	
1763	calc by iis	8.44		42.2		-----	
1801		-----		-----		-----	
1816	calc by iis	13.44		67.2		-----	
1841	calc by iis	15.18		75.9		44.9	
1864	calc by iis	11.9943		59.9715		-----	
1875	calc by iis	6.83		34.15		-----	
1888		-----		-----		-----	
1899	calc by iis	14.4089		72.0445		-----	
1956		-----		-----		-----	
1965	calc by iis	-----		-----		36.05	
2122	calc by iis	-----		-----		41.63	
3195	calc by iis	11.44		57.2		-----	
	normality	OK		OK		OK	
	n	23		23		17	
	outliers	0 (+1ex)		0 (+1ex)		1	
	mean (n)	11.938		59.690		37.774	
	st.dev. (n)	2.4381		12.1906		5.3450	
	R(calc.)	6.827		34.134		14.966	
	R(EN61619:98)	4.984					
	R(EN12766-2-B:01)			26.855			
	R(D4059:10-silicone)					20.417	

APPENDIX 3

Number of participating laboratories per country

1 lab in FINLAND
1 lab in PERU
1 lab in NORWAY
1 lab in ESTONIA
1 lab in SOUTH AFRICA
1 lab in BELGIUM
1 lab in SERBIA
1 lab in MEXICO
1 lab in TURKEY
1 lab in MOROCCO
2 labs in SLOVENIA
2 labs in PORTUGAL
2 labs in CROATIA
3 labs in ITALY
3 labs in NETHERLANDS
4 labs in GERMANY
4 labs in FRANCE
6 labs in SPAIN
7 labs in UNITED KINGDOM

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
fr	= first reported result (only when corrected result was entered)
n.a.	= not applicable
W	= withdrawn on request participant
U	= probably reported in wrong unit
E	= probably error in calculations
SDS	= Material Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, April 2014

2 prNEN 12766-2:2000.

3 ASTM E178-02

4 ASTM E1301-03

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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, First reported Z. Anal. Chem, 331, 513, (1988)

12 J.N. Miller, Analyst, 118, 455, (1993)

13 Analytical Methods Committee Technical Brief, No4 January 2001

14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)

15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)