Results of Proficiency Test
PCB in (Mineral) Oil
November 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2015/2016, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.
In this interlaboratory study, 47 laboratories from 17 different countries have participated. See appendix 3 for the number of participants per country. In this report the results of the 2015 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. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send one 8 ml vial with mineral oil contaminated with PCB (labelled #15225). Participants were requested to report analytical results using the indicated units and 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 (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 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 is electronically available through the iis internet site 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 only one sample was used. The necessary bulk material for the sample, was a mineral oil which was spiked to approximate 30 mg/kg PCB. This was done using a PCB contaminated oil (positive on PCB). This contaminated oil was donated by a third party laboratory. After ultrasonic homogenisation, 80 glass vials of 8 mL were filled, capped and labelled #15225.
The homogeneity of the subsamples #15225 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 #15225-1	27.7
sample #15225-2	27.6
sample #15225-3	27.5
sample #15225-4	27.5
sample #15225-5	27.6
sample #15225-6	27.6
sample #15225-7	27.5
sample #15225-8	27.5

Table 1: homogeneity test results of subsample #15225

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 #15225)	0.2
reference method	UOP779-08
0.3 x $R_{(reference\ method)}$	1.4

Table 2: evaluation of the observed repeatability

The calculated repeatability was less than 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the subsamples #15225 was assumed.

To each of the participating laboratories, one glass vial of 8 mL (labelled #15225) was sent on November 4, 2015.

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/. A SDS and a form to confirm receipt of the samples were added to the sample.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. 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 a reminder was sent to those laboratories that had not reported results at that moment.
Shortly 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 participants that reported 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 problems were encountered during execution. In total eight participants reported results after the final reporting date and four participants did not report any results at all. Not all participants were able to report results for all tests. In total 43 participating laboratories reported 219 numerical results. Observed were 5 outlying results, which is 2.3% 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 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.

Lab 511 and 1841 reported zero as a test result, which statistically is not considered a value and were therefore excluded for statistical evaluation.

TOX Only three test results were reported. Therefore no significant conclusions were drawn.

Individual PCBs: The consensus value for three of the seven congeners (No. 28, 52 and 118) was below the application range of EN12766-1 (0.2 mg/kg, see note1). Therefore no significant conclusions were drawn for the three congeners (No.28, 52, 118).
For the congeners No.101, 138, 153 and 180 in total two statistical outliers were observed. The calculated reproducibilities of congeners No. 101 and 138 are not in agreement with requirements of EN12766-1:00 / IP462-1:01. The calculated reproducibilities of the congeners No. 153 and 180 after rejection of the statistical outliers are in agreement with the 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 test results were excluded (zero values).
In total 14 participants reported the presence of Aroclor 1260 between 15.9 and 36.1 mg/kg. Of these 14 participants five participants reported test results between 2.16 and 11.54 for Aroclor 1254, while eight other participants reported Aroclor 1254 being absent (values < 2 mg/kg).
The reported test results for Aroclor 1242 are all low (<2.9 mg/kg) and the spread is small (values between 0.94 and 2.9). 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 is not in agreement with the three different 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 to determine the Aroclor compounds. The first method determines all three compounds by using three specifically defined windows within the chromatogram. The second method 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.

One participant used 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.

Total PCB: **Total PCB, “5 times the sum of 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.

Total PCB as 5 times the sum of 6 congeners was also calculated by iis (see appendix 2). No errors in calculation were found for the test results. The consensus value of the group was in agreement with the consensus value found with the value calculated by iis (41.76 vs 42.88 mg/kg).

Total PCB, "sum of all PCB congeners"
This determination and/or calculation of total PCB content was problematic. Four laboratories reported the sum of only 7 congeners instead of all congeners, the test result of these four laboratories were excluded for statistical evaluation. One statistical outlier was observed. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN61619:98 and EN12766-2 method A:2001.

Total PCB, "sum of all Aroclors"
This determination and/or calculation of total PCB content was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D4059:00(2010).
The total PCB as the sum of all Aroclors was also calculated by iis (see appendix 2). The consensus value of the group was in agreement with the consensus value found with the value calculated by iis (27.73 vs 28.93 mg/kg).

Summary:

All participants agreed that sample #15225 was positive on PCBs.
From the data on total organic chloride (TOX) an average concentration of 24.4 mg/kg was calculated. From this concentration a total content of 40.7 mg PCB/kg was estimated using an average Cl content of 60%, assuming the presence of only Aroclor 1260. This content is equal to the estimated total PCB content (5 times the sum of 6 congeners).

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

	#15225
total PCB content, estimated by TOX data, in mg/kg	40.7
total PCB content, 5 times the sum of 6 congeners, in mg/kg	41.5 – 42.9
total PCB content, sum of all congeners, in mg/kg	27.8
total PCB content, using Aroclor method, in mg/kg	27.7 – 28.9

Table 3: Comparison of estimations of total PCB content in sample #15225.

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.

Lab 1201 reported as a postive value for congener No 209 (1.1 mg/kg).

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 EN or ASTM standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX	mg/kg	3	24.4	n.e.	n.e.
PCB no. 28	mg/kg	14	0.12	(0.07)	(0.04)
PCB no. 52	mg/kg	13	0.08	(0.09)	(0.02)
PCB no. 101	mg/kg	22	0.82	0.59	0.39
PCB no. 118	mg/kg	11	0.15	(0.11)	(0.05)
PCB no. 138	mg/kg	22	2.16	1.60	1.06
PCB no. 153	mg/kg	22	2.69	0.92	1.33
PCB no. 180	mg/kg	20	2.56	1.01	1.26
Aroclor 1242	mg/kg	11	1.83	1.49	(2.11)
Aroclor 1254	mg/kg	8	<2	n.e.	n.e.
Aroclor 1260	mg/kg	14	24.30	16.20	14.67
Total PCB 5 times the sum of 6 congeners	mg/kg	19	41.76	23.23	18.66
Total PCB sum of all congeners	mg/kg	17	27.79	10.63	8.95
Total PCB sum of Aroclors	mg/kg	8	27.73	17.50	16.19

Table 4: Performance of the group of participating laboratories on sample #15225

() 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. The problematic components have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2015 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2015	*November 2014*	*November 2013*	*October 2012*
Number of reporting labs	43	48	44	41
Number of results reported	219	239	254	204
Statistical outliers	5	5	6	10
Percentage outliers	2.3%	2.1%	2.4%	4.9%

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 2015*	*November 2014*	*November 2013*	*October 2012*
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 #15225; results in mg/kg.

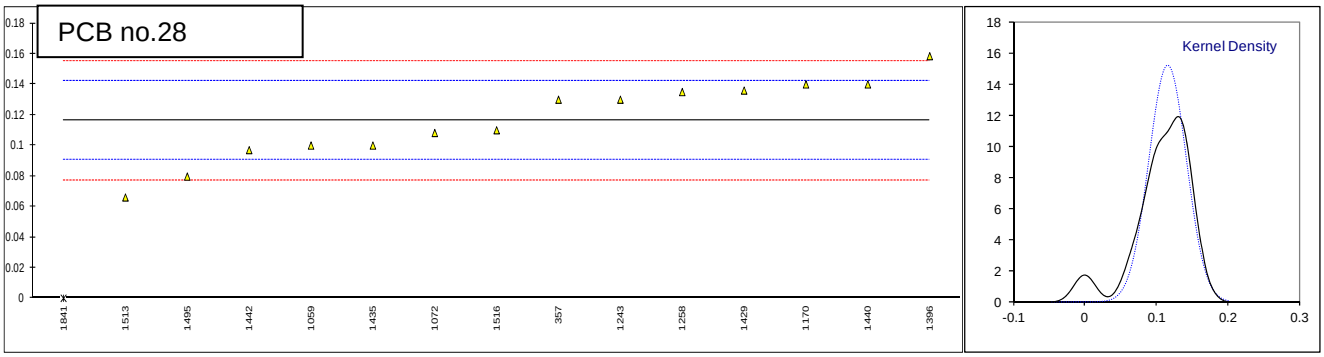
lab	method	value	mark	z(targ)	remarks
311		27.6		-----	
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
1059		-----		-----	
1066		-----		-----	
1072		-----		-----	
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201	UOP779	16.6		-----	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1417		-----		-----	
1429	D7359	29.0		-----	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1452		-----		-----	
1458		-----		-----	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1660		-----		-----	
1704		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1888		-----		-----	
1956		-----		-----	
2122		-----		-----	
3195		-----		-----	
6017		-----		-----	

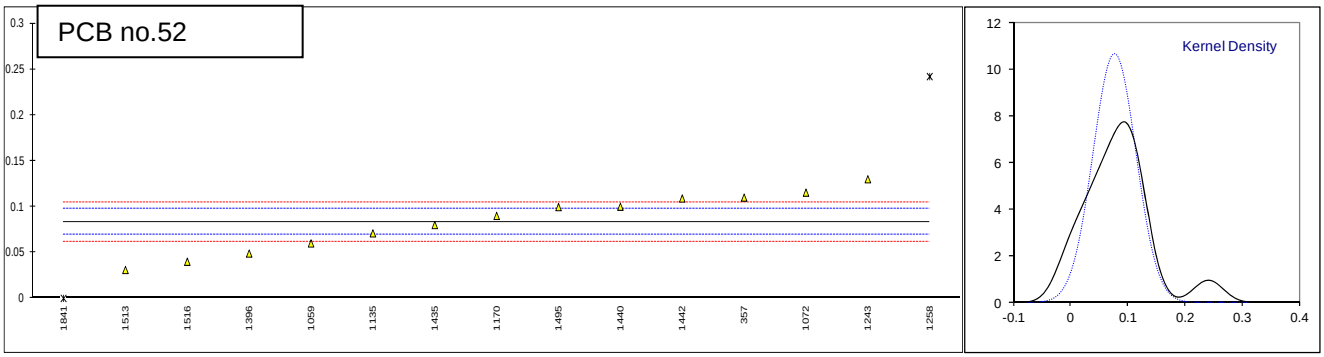
normality	unknown
n	3
outliers	n.a.
mean (n)	24.40
st.dev. (n)	n.a.
R(calc.)	n.a.
R(UOP779:08)	n.a.

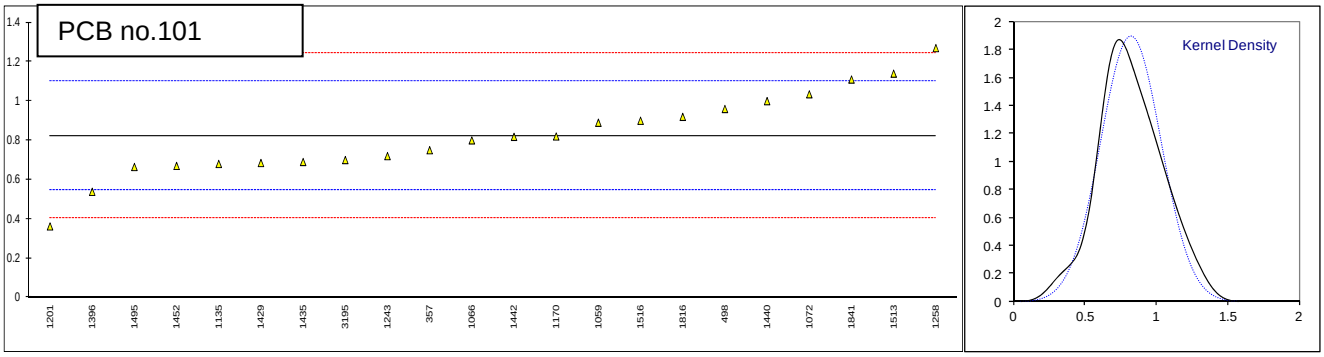
Determination of PCB 28, 52 and 101 on sample #15225; results in mg/kg.

Lab	Method	No.28	mark	z(targ)	No.52	mark	z(targ)	No.101	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357	EN12766-1	0.13		-----	0.11		-----	0.75		-0.53
398		-----		-----	-----		-----	-----		-----
445		-----		-----	-----		-----	-----		-----
498	EN12766-1	<0.3		-----	<0.3		-----	0.96		0.97
511		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
1059	EN12766-1	0.10		-----	0.06		-----	0.89		0.47
1066	EN12766-1	<0.2		-----	<0.2		-----	0.8		-0.17
1072	IEC61619	0.1082		-----	0.1154		-----	1.0348		1.51
1126		-----		-----	-----		-----	-----		-----
1135	in house	<0.1		-----	0.071		-----	0.68		-1.03
1170	EN12766-1	0.14		-----	0.09		-----	0.82		-0.03
1201	EN12766-1	<0.1		-----	<0.1		-----	0.362		-3.30
1243	EN12766-1	0.13		-----	0.13		-----	0.72		-0.74
1258	EN12766-1	0.135		-----	0.242	G(0.05)	-----	1.270		3.19
1303		-----		-----	-----		-----	-----		-----
1304		-----		-----	-----		-----	-----		-----
1306		-----		-----	-----		-----	-----		-----
1338		-----		-----	-----		-----	-----		-----
1352		-----		-----	-----		-----	-----		-----
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374		-----		-----	-----		-----	-----		-----
1396	IP462-1	0.1585		-----	0.04893		-----	0.5383		-2.04
1417		-----		-----	-----		-----	-----		-----
1429	EN12766-1	0.136		-----	<0.2	C	-----	0.685		-0.99
1435	EN12766-1	0.10		-----	0.08		-----	0.69		-0.96
1440	IEC61619	0.14		-----	0.10		-----	1.00		1.26
1442	EN12766-1	0.097		-----	0.109		-----	0.818		-0.04
1452	EN12766-1	<0.13		-----	<0.13		-----	0.67		-1.10
1458		-----		-----	-----		-----	-----		-----
1495	EN12766-1	0.07968		-----	0.09957		-----	0.66464		-1.14
1513	IEC61619	0.066		-----	0.031		-----	1.140		2.26
1516	IEC61619	0.11		-----	0.04		-----	0.90		0.55
1660		-----		-----	-----		-----	-----		-----
1704		-----		-----	-----		-----	-----		-----
1743		-----		-----	-----		-----	-----		-----
1801		-----		-----	-----		-----	-----		-----
1816	IEC61619	<2		-----	<2		-----	0.92		0.69
1841	IEC61619	0.00	ex	-----	0.00	ex	-----	1.11		2.05
1888		-----		-----	-----		-----	-----		-----
1956		-----		-----	-----		-----	-----		-----
2122		-----		-----	-----		-----	-----		-----
3195	EN12766-1	<0.20		-----	<0.20		-----	0.70		-0.89
6017		-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK		
	n	14			13			22		
	outliers	0 (+1 excl)			1 (+1 excl)			0		
	mean (n)	0.1165			0.0835			0.8238		
	st.dev. (n)	(0.02616)			(0.03118)			0.21025		
	R(calc.)	(0.0733)			(0.0873)			0.5887		
	R(EN12766-1:00)	(0.0364)			(0.0199)			0.3914		

Lab 1429: first reported 0.18
Lab 1841: results for PCB no. 28 and 52 were excluded, as zero is not a real result.


PCB no.28
Kernel Density

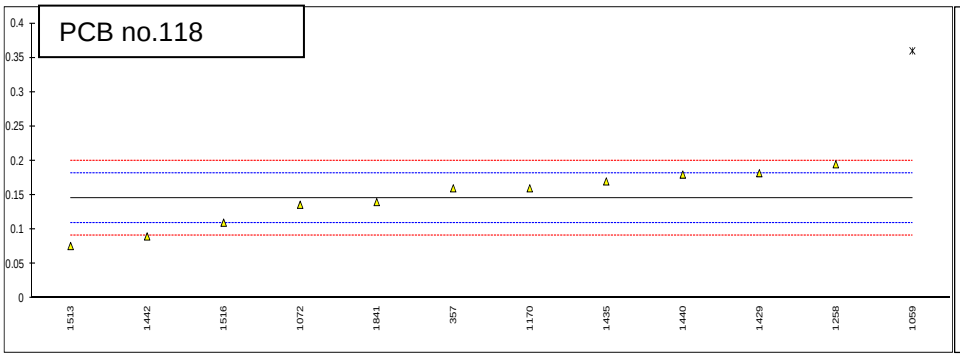

PCB no.52
Kernel Density

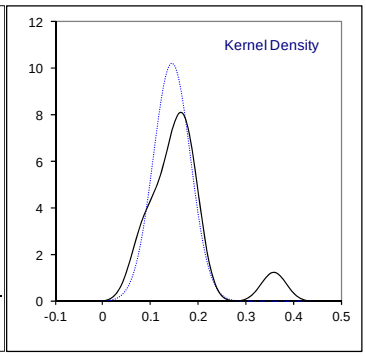

PCB no.101
Kernel Density

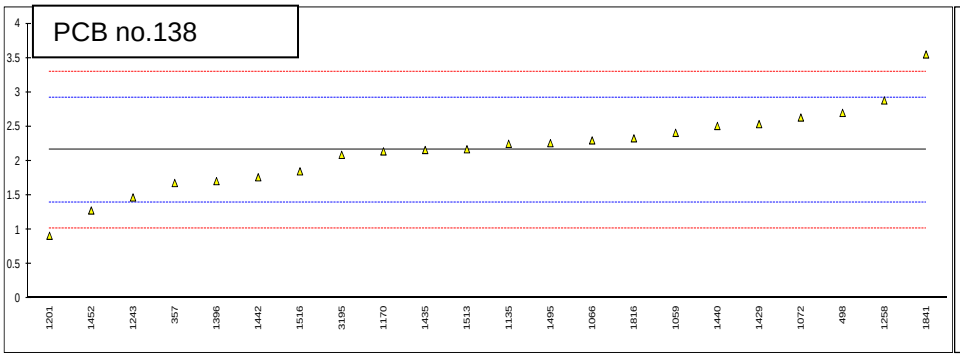
Determination of PCB 118 and 138 on sample #15225; results in mg/kg.

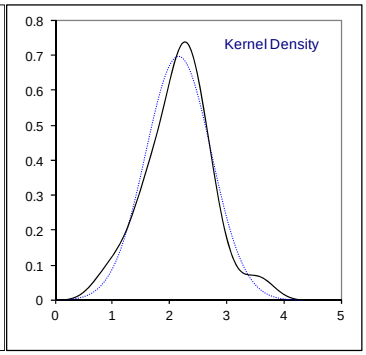
lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766-1	0.16		-----	1.68		-1.27	
398		-----		-----	-----		-----	
445		-----		-----	-----		-----	
498	EN12766-1	<0.3		-----	2.70		1.41	
511		-----		-----	-----		-----	
614		-----		-----	-----		-----	
1059	EN12766-1	0.36	C,G(0.01)	-----	2.41		0.65	
1066	EN12766-1	<0.2		-----	2.3		0.36	
1072	IEC61619	0.1361		-----	2.6331		1.24	
1126		-----		-----	-----		-----	
1135	in house	-----		-----	2.25		0.23	
1170	EN12766-1	0.16		-----	2.14		-0.06	
1201	EN12766-1	<0.1		-----	0.912		-3.29	
1243	EN12766-1	-----		-----	1.47		-1.82	
1258	EN12766-1	0.195		-----	2.881		1.89	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1374		-----		-----	-----		-----	
1396	IP462-1	-----		-----	1.707		-1.20	
1417		-----		-----	-----		-----	
1429	EN12766-1	0.182		-----	2.537		0.98	
1435	EN12766-1	0.17		-----	2.16		-0.01	
1440	IEC61619	0.18		-----	2.51		0.91	
1442	EN12766-1	0.090		-----	1.764		-1.05	
1452	EN12766-1	-----		-----	1.28		-2.32	
1458		-----		-----	-----		-----	
1495	EN12766-1	-----		-----	2.2598		0.25	
1513	IEC61619	0.076		-----	2.171		0.02	
1516	IEC61619	0.11		-----	1.85		-0.82	
1660		-----		-----	-----		-----	
1704		-----		-----	-----		-----	
1743		-----		-----	-----		-----	
1801		-----		-----	-----		-----	
1816	IEC61619	<2		-----	2.33		0.44	
1841	IEC61619	0.14		-----	3.55		3.65	
1888		-----		-----	-----		-----	
1956		-----		-----	-----		-----	
2122		-----		-----	-----		-----	
3195	EN12766-1	<0.20		-----	2.09		-0.19	
6017		-----		-----	-----		-----	
	normality	OK			suspect			
	n	11			22			
	outliers	1			0			
	mean (n)	0.1454			2.1629			
	st.dev. (n)	(0.03909)			0.57166			
	R(calc.)	(0.1095)			1.6007			
	R(EN12766-1:00)	(0.0510)			1.0636			

Lab 1059: first reported 0.28


PCB no.118
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
1513
1442
1516
1072
1841
357
1170
1435
1440
1429
1258
1069

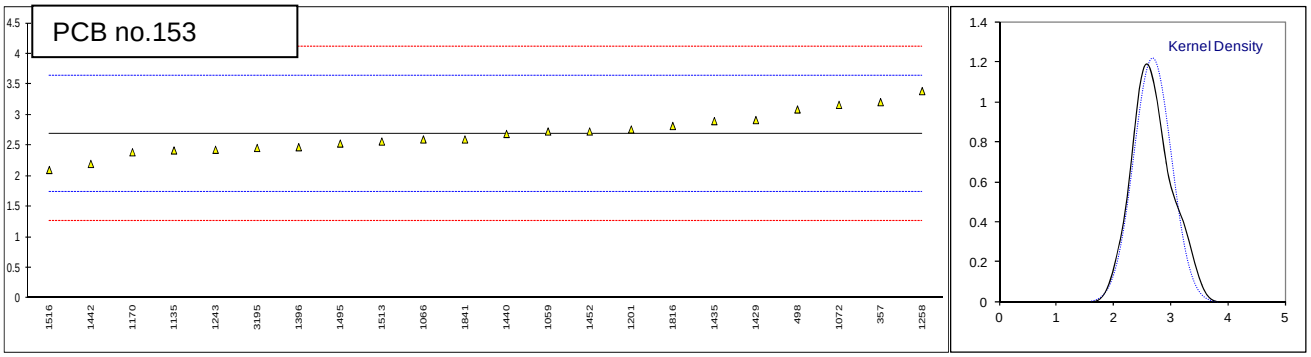

Kernel Density
12
10
8
6
4
2
0
-0.1
0
0.1
0.2
0.3
0.4
0.5

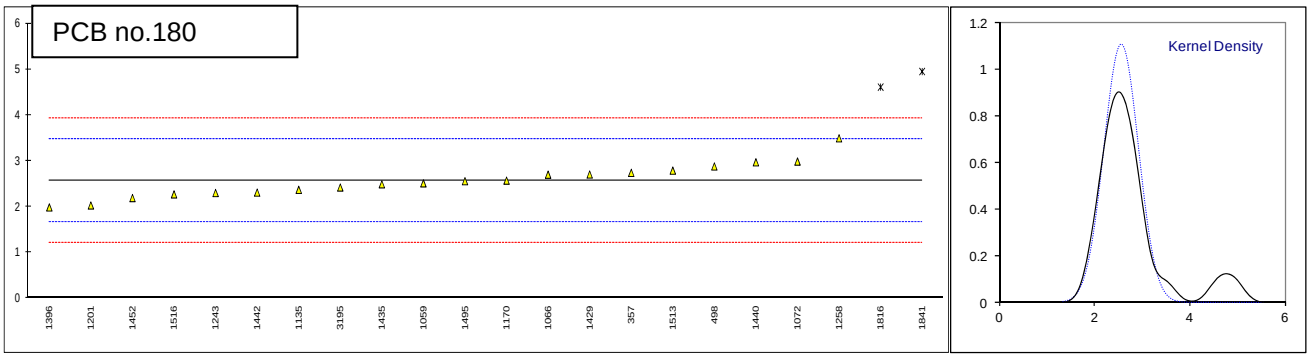

PCB no.138
4
3.5
3
2.5
2
1.5
1
0.5
0
1201
1452
1243
357
1396
1442
1516
3195
1170
1435
1513
1135
1495
1066
1816
1059
1440
1429
1072
498
1258
1841


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

Determination of PCB 153 and 180 on sample #15225; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766-1	3.21		1.10	2.74		0.39	
398		-----		-----	-----		-----	
445		-----		-----	-----		-----	
498	EN12766-1	3.09		0.84	2.88		0.70	
511		-----		-----	-----		-----	
614		-----		-----	-----		-----	
1059	EN12766-1	2.73		0.08	2.51		-0.12	
1066	EN12766-1	2.6		-0.19	2.7		0.30	
1072	IEC61619	3.1658		1.00	2.9864		0.94	
1126		-----		-----	-----		-----	
1135	in house	2.42		-0.57	2.37		-0.43	
1170	EN12766-1	2.39		-0.63	2.57		0.02	
1201	EN12766-1	2.763		0.15	2.028		-1.19	
1243	EN12766-1	2.43		-0.55	2.30		-0.58	
1258	EN12766-1	3.391		1.48	3.494		2.06	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1374		-----		-----	-----		-----	
1396	IP462-1	2.473		-0.46	1.9853		-1.28	
1417		-----		-----	-----		-----	
1429	EN12766-1	2.917		0.48	2.704		0.31	
1435	EN12766-1	2.90		0.44	2.49		-0.16	
1440	IEC61619	2.69		0.00	2.97		0.90	
1442	EN12766-1	2.199		-1.03	2.309		-0.56	
1452	EN12766-1	2.73		0.08	2.19		-0.83	
1458		-----		-----	-----		-----	
1495	EN12766-1	2.5326		-0.33	2.5570		-0.01	
1513	IEC61619	2.566		-0.26	2.789		0.50	
1516	IEC61619	2.10		-1.24	2.27		-0.65	
1660		-----		-----	-----		-----	
1704		-----		-----	-----		-----	
1743		-----		-----	-----		-----	
1801		-----		-----	-----		-----	
1816	IEC61619	2.82		0.27	4.61	R(0.01)	4.53	
1841	IEC61619	2.60		-0.19	4.95	R(0.01)	5.29	
1888		-----		-----	-----		-----	
1956		-----		-----	-----		-----	
2122		-----		-----	-----		-----	
3195	EN12766-1	2.46		-0.48	2.42		-0.32	
6017		-----		-----	-----		-----	
	normality	OK			OK			
	n	22			20			
	outliers	0			2			
	mean (n)	2.6899			2.5631			
	st.dev. (n)	0.32698			0.36112			
	R(calc.)	0.9156			1.0111			
	R(EN12766-1:00)	1.3281			1.2644			

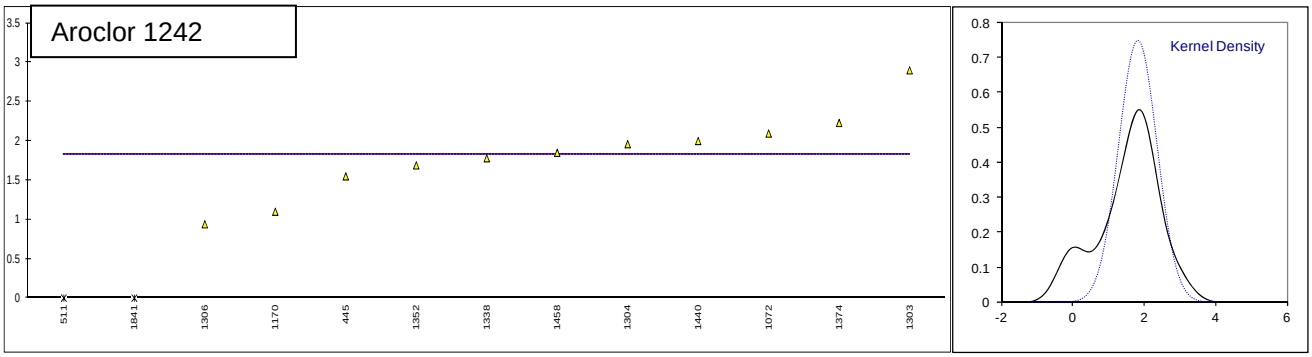

PCB no.153
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
1516 1442 1170 1135 1243 3195 1396 1495 1513 1066 1841 1440 1059 1452 1201 1816 1435 1429 498 1072 357 1258
Kernel Density
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0 1 2 3 4 5

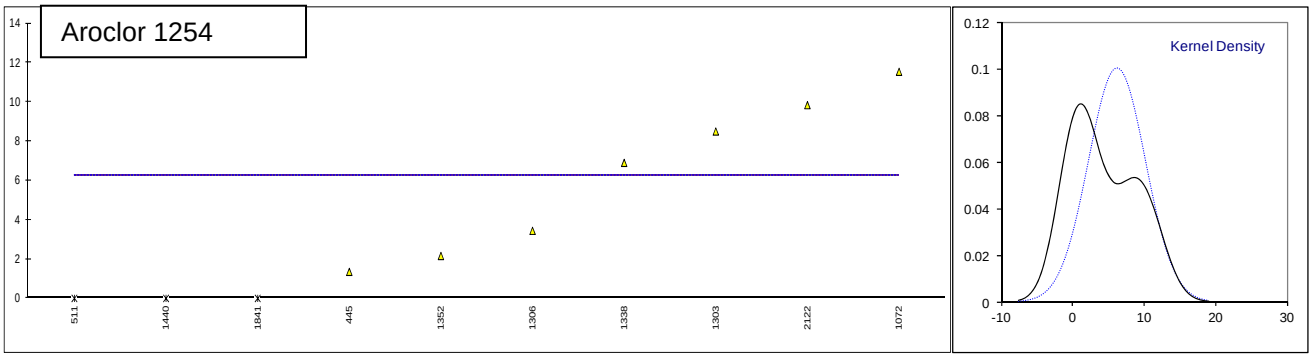

PCB no.180
6
5
4
3
2
1
0
1396 1201 1452 1516 1243 1442 1135 3195 1435 1059 1495 1170 1066 1429 357 1513 498 1440 1072 1258 1816 1841
Kernel Density
1.2
1
0.8
0.6
0.4
0.2
0
0 2 4 6

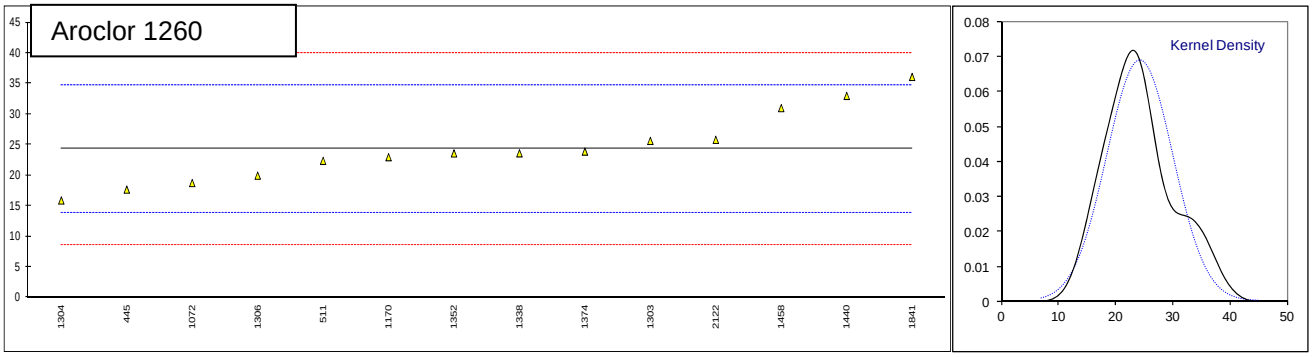
Determination of Aroclor 1242, 1254 and 1260 on sample #15225; results in mg/kg.

lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
445	D4059	1.55		-----	1.35		-----	17.66		-1.27
498		-----		-----	-----		-----	-----		-----
511	D4059	0	ex	-----	0		-----	22.37		-0.37
614		-----		-----	-----		-----	-----		-----
1059		-----		-----	-----		-----	-----		-----
1066		-----		-----	-----		-----	-----		-----
1072	in house	2.095		-----	11.539		-----	18.735		-1.06
1126		-----		-----	-----		-----	-----		-----
1135		-----		-----	-----		-----	-----		-----
1170	D4059	1.1		-----	<1.1		-----	22.97		-0.25
1201		-----		-----	-----		-----	-----		-----
1243		-----		-----	-----		-----	-----		-----
1258		-----		-----	-----		-----	-----		-----
1303	D4059	2.9		-----	8.5		-----	25.63	C	0.25
1304	INH-127	1.96		-----	<0.2		-----	15.88		-1.61
1306		0.94		-----	3.44		-----	19.95		-0.83
1338	INH-4059	1.78		-----	6.9		-----	23.6		-0.13
1352	INH-1767	1.69		-----	2.16		-----	23.59		-0.14
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374	D4059	2.23		-----	n.d.		-----	23.88		-0.08
1396		-----		-----	-----		-----	-----		-----
1417		-----		-----	-----		-----	-----		-----
1429		-----		-----	-----		-----	-----		-----
1435		-----		-----	-----		-----	-----		-----
1440	in house	2.00		-----	0.00		-----	33.0		1.66
1442		-----		-----	-----		-----	-----		-----
1452		-----		-----	-----		-----	-----		-----
1458	D4059	1.85		-----	<0.4		-----	31.0		1.28
1495		-----		-----	-----		-----	-----		-----
1513		-----		-----	-----		-----	-----		-----
1516		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1704		-----		-----	-----		-----	-----		-----
1743		-----		-----	-----		-----	-----		-----
1801		-----		-----	-----		-----	-----		-----
1816		-----		-----	-----		-----	-----		-----
1841	IEC61619	0	ex	-----	0		-----	36.1		2.25
1888		-----		-----	-----		-----	-----		-----
1956		-----		-----	-----		-----	-----		-----
2122		-----		-----	9.84		-----	25.81		0.29
3195		-----		-----	-----		-----	-----		-----
6017		-----		-----	-----		-----	-----		-----
	normality	OK			unknown			OK		
	n	11			8			14		
	outliers	0 (+2 excl)			n.a.			0		
	mean (n)	1.8268			<2			24.2982		
	st.dev. (n)	0.53355			n.a.			5.78733		
	R(calc.)	1.4939			n.a.			16.2045		
	R(D4059:00 (silicone))	(2.1056)			n.a.			14.6651		
	R(D4059:00 (packed))	(1.6185)			n.a.			11.2724		
	R(D4059:00 (megabore))	(1.2414)			n.a.			8.6459		

Lab 1303: first reported 46.5
Lab 511: result reported for Aroclor 1242 was excluded, as zero is not a real result
Lab 1841: result reported for Aroclor 1242 was excluded, as zero is not a real result


Aroclor 1242
Kernel Density

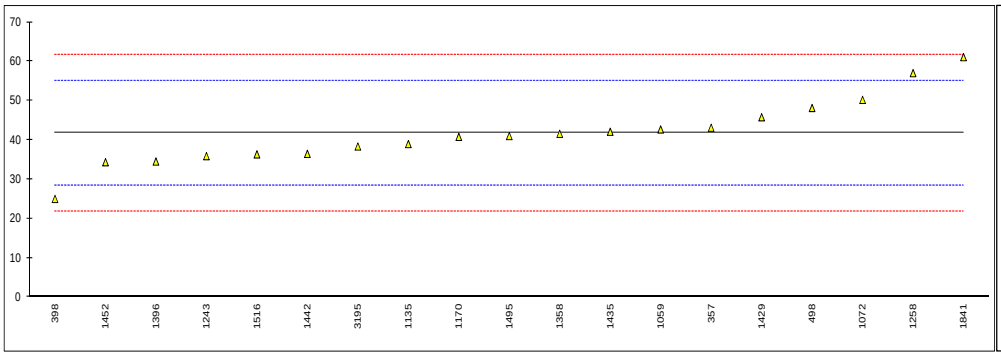

Aroclor 1254
Kernel Density

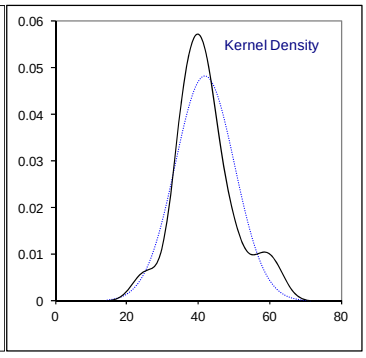

Aroclor 1260
Kernel Density

Determination of Total PCB, 5 times the sum of 6 congeners on sample #15225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	43.12		0.20	
398	EN12766-2-B	25		-2.52	
445		-----		-----	
498	EN12766-2-B	48.16		0.96	
511		-----		-----	
614		-----		-----	
1059	EN12766-2-B	42.7		0.14	
1066		-----		-----	
1072	EN12766-2-B	50.2185		1.27	
1126		-----		-----	
1135	in house	38.96		-0.42	
1170	EN12766-2-B	40.8		-0.14	
1201		-----		-----	
1243	EN12766-2-B	35.9		-0.88	
1258	EN12766-2-B	57.03		2.29	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358	IP462-2	41.580		-0.03	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	34.5248		-1.09	
1417		-----		-----	
1429	EN12766-2-B	45.8		0.61	
1435	EN12766-2-B	42.10		0.05	
1440		-----		-----	
1442	EN12766-2-B	36.48		-0.79	
1452	EN12766-2-B	34.35		-1.11	
1458		-----		-----	
1495	EN12766-2-B	41.0		-0.11	
1513		-----		-----	
1516	EN12766-2-B	36.35		-0.81	
1660		-----		-----	
1704		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841	IEC61619	61.1		2.90	
1888		-----		-----	
1956		-----		-----	
2122		-----		-----	
3195	EN12766-2-B	38.35		-0.51	
6017		-----		-----	

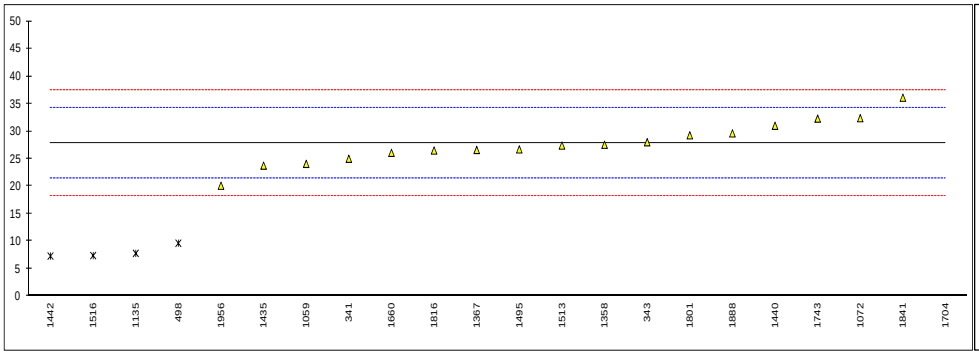
normality	suspect
n	19
outliers	0
mean (n)	41.764
st.dev. (n)	8.2983
R(calc.)	23.235
R(EN12766-2B:01)	18.662

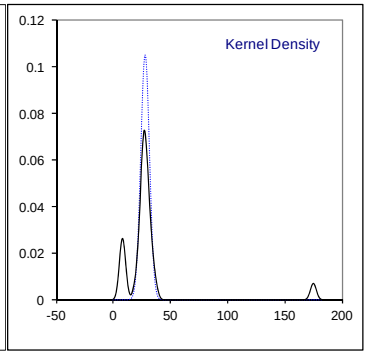




Determination of Total PCB, sum of all congeners on sample #15225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	25		-0.87	
343	EN61619	28		0.07	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498	EN12766-2-A	9.63	ex	-5.68	Reported only the sum of 7 congeners
511		-----		-----	
614		-----		-----	
1059	EN12766-2-A	24.07		-1.16	
1066		-----		-----	
1072	EN61619	32.3685		1.43	
1126		-----		-----	
1135	in house	7.79	ex	-6.26	Reported only the sum of 7 congeners
1170		-----		-----	
1201		-----		-----	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358	IP462-2	27.54		-0.08	
1367	EN61619	26.6		-0.37	
1374		-----		-----	
1396		-----		-----	
1417		-----		-----	
1429		-----		-----	
1435	EN61619	23.75		-1.26	
1440	EN61619	31.0		1.01	
1442	EN12766-2-A	7.296	ex	-6.41	Reported only the sum of 7 congeners
1452		-----		-----	
1458		-----		-----	
1495	EN12766-2-A	26.7		-0.34	
1513	IEC61619	27.4		-0.12	
1516	EN61619	7.38	ex	-6.39	Reported only the sum of 7 congeners
1660	EN61619	26.07		-0.54	
1704	IEC61619	175.105	R(0.01)	46.11	
1743	IEC61619	32.3		1.41	
1801	EN61619	29.26		0.46	
1816	EN61619	26.5		-0.40	
1841	IEC61619	36.1		2.60	
1888	IEC61619	29.61		0.57	
1956	EN61619	20.1		-2.41	
2122		-----		-----	
3195		-----		-----	
6017		-----		-----	
	normality	OK			
	n	17			
	outliers	1 (+4 excl)			
	mean (n)	27.7864			
	st.dev. (n)	3.79722			
	R(calc.)	10.6322			
	R(EN61619:98)	8.9466			EN61619:98 is identical to EN12766-2-A:01

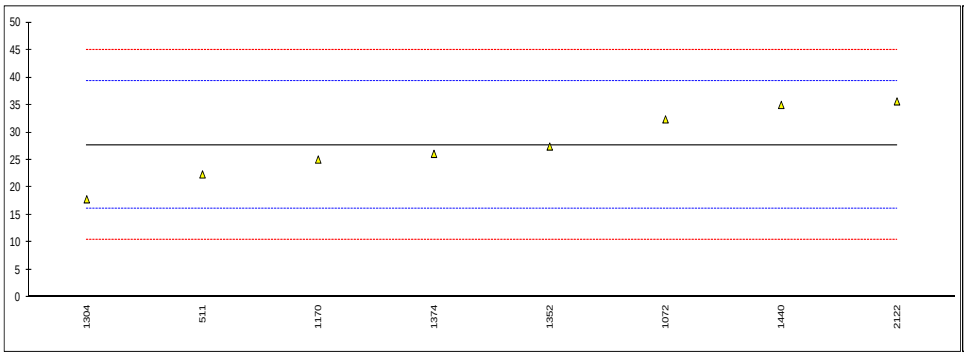


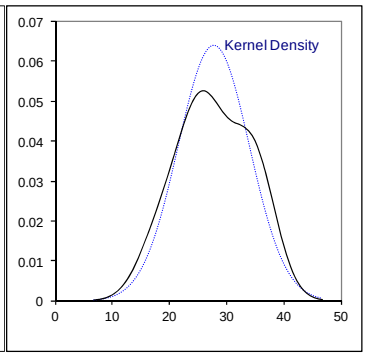


Determination of Total PCB, sum of all Aroclors on sample #15225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511	D4059	22.37		-0.93	
614		-----		-----	
1059		-----		-----	
1066		-----		-----	
1072	in house	32.3685		0.80	
1126		-----		-----	
1135		-----		-----	
1170	D4059	25.07		-0.46	
1201		-----		-----	
1243		-----		-----	
1258		-----		-----	
1303		-----		-----	
1304	INH-127	17.84		-1.71	
1306		-----		-----	
1338		-----		-----	
1352	INH-1767	27.44		-0.05	
1358		-----		-----	
1367		-----		-----	
1374	D4059	26.11		-0.28	
1396		-----		-----	
1417		-----		-----	
1429		-----		-----	
1435		-----		-----	
1440	in house	35.0		1.26	
1442		-----		-----	
1452		-----		-----	
1458		-----		-----	
1495		-----		-----	
1513		-----		-----	
1516		-----		-----	
1660		-----		-----	
1704		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1888		-----		-----	
1956		-----		-----	
2122	D4059	35.65		1.37	
3195		-----		-----	
6017		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	27.7311
st.dev. (n)	6.24981
R(calc.)	17.4995
R(D4059:10)	16.1931





APPENDIX 2

Total PCB by summation of the reported results by iis on sample #15225; results in mg/kg.

lab	method	Sum of 6 congeners	mark	5 x (sum of 6 congeners)	mark	sum of Aroclors
341		-----		-----		-----
343		-----		-----		-----
357	EN12766-2-B	8.62		43.1		-----
398	EN12766-2-B	-----		-----		-----
445	D4059	-----		-----		20.56
498	EN12766-2-B	9.63		48.15		-----
511		-----		-----		22.37
614		-----		-----		-----
1059	EN12766-2-B	8.7		43.5		-----
1066		8.4		42		-----
1072	EN12766-2-B	10.0437		50.2185		32.369
1126		-----		-----		-----
1135	in house	7.791		38.955		-----
1170	EN12766-2-B	8.15		40.75		25.07
1201		6.065		30.325		-----
1243	EN12766-2-B	7.18		35.9		-----
1258	EN12766-2-B	11.413		57.065		-----
1303		-----		-----		37.03
1304		-----		-----		17.84
1306		-----		-----		24.33
1338		-----		-----		32.28
1352		-----		-----		27.44
1358	IP462-2	-----		-----		-----
1367		-----		-----		-----
1374		-----		-----		26.11
1396	IP462-2	6.91103		34.55515		-----
1417		-----		-----		-----
1429	EN12766-2-B	8.979		44.895		-----
1435	EN12766-2-B	8.42		42.1		-----
1440		9.41		47.05		35
1442	EN12766-2-B	7.296		36.48		-----
1452	EN12766-2-B	6.87		34.35		-----
1458		-----		-----		32.85
1495	EN12766-2-B	8.19329		40.96645		-----
1513		8.763		43.815		-----
1516	EN12766-2-B	7.27		36.35		-----
1660		-----		-----		-----
1704		-----		-----		-----
1743		-----		-----		-----
1801		-----		-----		-----
1816		10.68		53.4		-----
1841	IEC61619	12.21		61.05		36.1
1888	IEC61619	-----		-----		-----
1956		-----		-----		-----
2122		-----		-----		35.65
3195	EN12766-2-B	7.67		38.35		-----
6017		-----		-----		-----
	normality	OK		OK		OK
	n	22		22		14
	outliers	0		0		0
	mean (n)	8.5757		42.8784		28.9285
	st.dev. (n)	1.52686		7.63431		6.32303
	R(calc.)	4.2752		21.3761		17.7045
	R(EN61619:98)	4.1439				
	R(EN12766-2-B:01)			19.1711		
	R(D4059:10-silicone)					16.7147

APPENDIX 3

Number of participating laboratories per country

6 labs in AUSTRALIE
2 labs in BELGIUM
1 lab in ESTONIA
1 lab in FINLAND
5 labs in FRANCE
4 labs in GERMANY
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
2 labs in SLOVENIA
1 lab in SOUTH AFRICA
6 labs in SPAIN
7 labs in UNITED KINGDOM

APPENDIX 4

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:

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