Results of Proficiency Test
PCB in (Mineral) Oil
November 2016

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

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CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 4

2.6 ANALYSES 5

3 RESULTS 5

3.1 STATISTICS 5

3.2 GRAPHICS 6

3.3 Z-SCORES 6

4 EVALUATION 7

4.1 EVALUATION PER TEST 7

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 10

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2016 WITH PREVIOUS PTs 11

Appendices:

1. Data and statistical results 12
2. Number of participants per country 28
3. Abbreviations and literature 29

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 2016/2017, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.
In this interlaboratory study, 47 laboratories from 20 different countries did register for participation. See appendix 2 for the number of participants per country.
In this report the results of the 2016 proficiency test on PCB in (mineral) oil are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one 8 ml vial with mineral oil contaminated with PCB (labelled #16253). The participants were requested to report analytical test results using the indicated units and to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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 website www.iisnl.com from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

In this proficiency test only one sample was used. The necessary bulk material for the preparation of the sub samples was a mineral oil which was spiked to approximately 17 mg/kg PCB with 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 #16253.
The homogeneity of the sub samples #16253 was checked by determination of Total Organic Chloride content in accordance with UOP779 on eight stratified randomly selected samples:

	Total Organic Chloride as Cl in mg/kg
sample #16253-1	12.2
sample #16253-2	12.1
sample #16253-3	12.0
sample #16253-4	12.1
sample #16253-5	11.9
sample #16253-6	12.0
sample #16253-7	12.1
sample #16253-8	12.0

Table 1: homogeneity test results of sub samples of #16253

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

	Total Organic Chloride as Cl in mg/kg
r (observed)	0.3
reference test method	UOP779:08
0.3 x R(ref.test method)	0.6

Table 2: evaluation of the repeatability of sub samples of #16253

The calculated repeatability was less than 0.3 times the estimated reproducibility of the reference test method. Therefore, homogeneity of the sub samples #16253 was assumed.

To each of the participating laboratories, one glass vial of 8 mL (labelled #16253) was sent on November 2, 2016.

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 on the sample #16253: Total Organo Halogenic Compounds (TOX) as Cl and Poly Chlorinated Biphenyls (via seven individual PCBs, via the determination of the total PCB content and/or via Aroclor standards).

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results a detailed report form, on which the units were prescribed as well as the required reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The laboratories were also requested to confirm the sample receipt on the same data entry portal. A SDS was added to the sample.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the

visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, EN or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to

recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	| z | < 1	good
1 <	| z | < 2	satisfactory
2 <	| z | < 3	questionable
3 <	| z |	unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the sample. Two participants reported test results after the final reporting date and two participants did not report any test result at all. Not all participants were able to report test results for all tests. In total 45 laboratories reported 221 numerical test results. Observed were 12 outlying test results, which is 5.4% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section the results are discussed per test. The test methods reported by the laboratories are taken into account for explaining the observed differences when possible and applicable. These test methods are also listed in the tables together with the original data (see appendix 1). The abbreviations used in these tables are listed in appendix 3.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care.

For the statistical evaluation of the individual PCBs the test method EN12766-1:00 was used, this test method is equal to IP462-1:01. In the test methods IEC61619:98 and DIN51527:93 only the reproducibilities of the total PCB content are mentioned, while in EN12766-1:00 / IP462-1:01 the reproducibilities for all congeners are mentioned.

TOX Based on only three numerical test results the determination may be problematic. Another test result was excluded as reported value was not in line with the PCB-content (see §2.4). The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of UOP779:08.

Individual PCBs: The determination of the individual PCB was problematic. In total eight statistical outliers were observed over seven congeners. The calculated reproducibilities of congeners No. 101, 138 and 153 after rejection of the statistical outliers are not in agreement with requirements of EN12766-1:00 / IP462-1:01.
The calculated reproducibility of congener No. 180 after rejection of the statistical outliers is in agreement with requirements of EN12766-1:00 / IP462-1:01.
The consensus values for three of the seven congeners (No. 28, 52 and 118) were below the application range of EN12766-1 (0.2 mg/kg). Therefore no significant conclusions were drawn for these three congeners.

Individual Aroclors: The determination of the individual Aroclors was problematic. In total three statistical outliers were observed. The calculated reproducibilities of the three Aroclors after rejection of the statistical outliers are not in agreement with the three different requirements of ASTM D4059:00(2010).

Total PCB: **Total PCB, “5 times the sum of 6 PCB congeners”**
This determination and/or calculation of total PCB content was not problematic. No statistical outliers were observed, but one test result was excluded as this test result contained a calculation error. However, the calculated reproducibility after rejection of the suspect data is agreement with the requirements of EN12766-2 test method B:2001.
Total PCB as 5 times the sum of 6 congeners was also calculated by iis from all reported individual congener test results. The consensus value of the reported sum results is in agreement with the consensus value (27.76 vs 27.74 mg/kg) calculated by iis.

Total PCB, “sum of all PCB congeners”
This determination and/or calculation of total PCB content was problematic. One statistical outlier was observed. Another two laboratories reported the sum of all Aroclors instead of all congeners and another laboratory reported an identical test result as for Aroclor 1260. These three laboratories were excluded for the statistical evaluation. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN61619:97 (and EN12766-2 test method A:2001 as this test method is identical to EN61619:97).

Total PCB, “sum of all Aroclors”
This determination and/or calculation of total PCB content was problematic. No statistical outliers were observed. Three test results were excluded as an outlier was observed in one of the underlying individual Aroclor test results.

The calculated reproducibility after rejection of the suspect data is not agreement with the requirements of ASTM D4059:00(2010).
The total PCB as the sum of all Aroclors was also calculated by iis from all reported individual Aroclor test results. The consensus value of the reported sum results is in agreement with the consensus value calculated by iis (17.17 vs 16.69 mg/kg).

Summary: All participants agreed that sample #16253 was positive on PCBs. From the data on total organic chloride (TOX) an average concentration of 13.4 mg/kg was calculated. From this concentration a total content of 22.3 mg PCB/kg was estimated using an average Cl content of 60%, assuming the presence of only Aroclor 1260.

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

	#16253
total PCB content, estimated by TOX data, in mg/kg	22.3
total PCB content, 5 times the sum of 6 congeners, in mg/kg	27.8
total PCB content, sum of all congeners, in mg/kg	17.0
total PCB content, using Aroclor method, in mg/kg	17.2

Table 3: comparison of estimations of total PCB content in sample #16253.

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 other two estimates (from TOX and from 5 x 6 congeners) are both significantly higher.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The average results, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu EN or ASTM test methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX	mg/kg	3	13.4	4.2	2.3
PCB no. 28	mg/kg	13	(0.08)	(0.08)	(0.02)
PCB no. 52	mg/kg	12	(0.06)	(0.08)	(0.01)
PCB no. 101	mg/kg	20	0.53	0.34	0.24
PCB no. 118	mg/kg	12	(0.11)	(0.15)	(0.03)
PCB no. 138	mg/kg	21	1.53	0.89	0.75
PCB no. 153	mg/kg	22	1.77	1.09	0.87
PCB no. 180	mg/kg	19	1.65	0.52	0.80
Aroclor 1242	mg/kg	11	1.00	2.33	1.34
Aroclor 1254	mg/kg	7	0.53	1.79	0.84
Aroclor 1260	mg/kg	16	15.8	15.0	10.6
Total PCB, 5 times the sum of 6 congeners	mg/kg	17	27.8	11.2	12.3
Total PCB, sum of all congeners	mg/kg	17	17.0	9.3	6.3
Total PCB, sum of Aroclors	mg/kg	12	17.2	16.2	11.3

Table 4: performance of the group of participating laboratories on sample #16253

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 or parameters there is not a good compliance of the group of participating laboratories with the relevant reference test methods. The problematic components have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2016 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2016	*November 2015*	*November 2014*	*November 2013*	*October 2012*
Number of reporting labs	45	43	48	44	41
Number of test results reported	221	219	239	254	204
Statistical outliers	12	5	5	6	10
Percentage outliers	5.4%	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 reference test methods. The conclusions are given the following table:

Determination	*November 2016*	*November 2015*	*November 2014*	*November 2013*	*October 2012*
TOX	--*	n.e.	++*	--	n.e.
PCB (individual)	-	+/-	-	--	--
Aroclor (individual)	--	+/-	+	--	-
Total PCB, 5 x sum of 6 congeners	+/-	-	-	+/-	-
Total PCB, sum of all congeners	-	-	+	-	--
Total PCB, sum of Aroclors	-	+/-	++	+/-	+

Table 6: comparison of observed precisions against requirements of the reference test methods
*) based on three test results

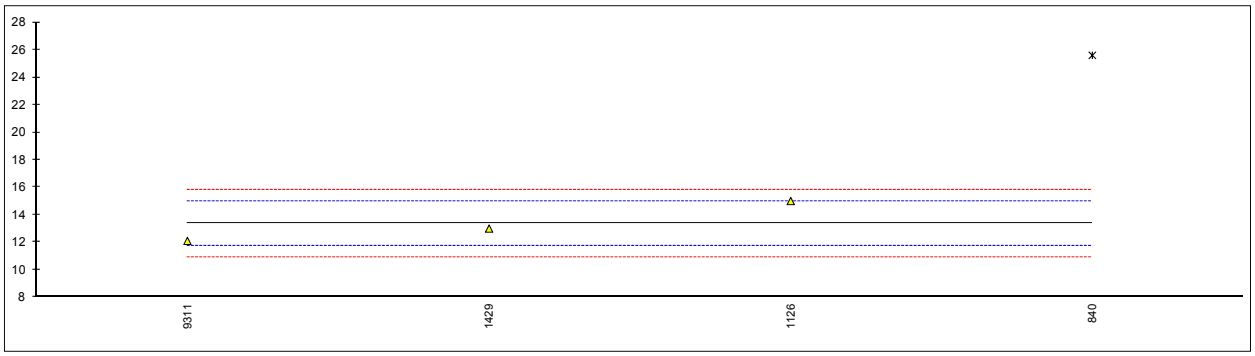
The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Total Organohalogenic Compounds (TOX) on sample #16253; results in mg/kg.

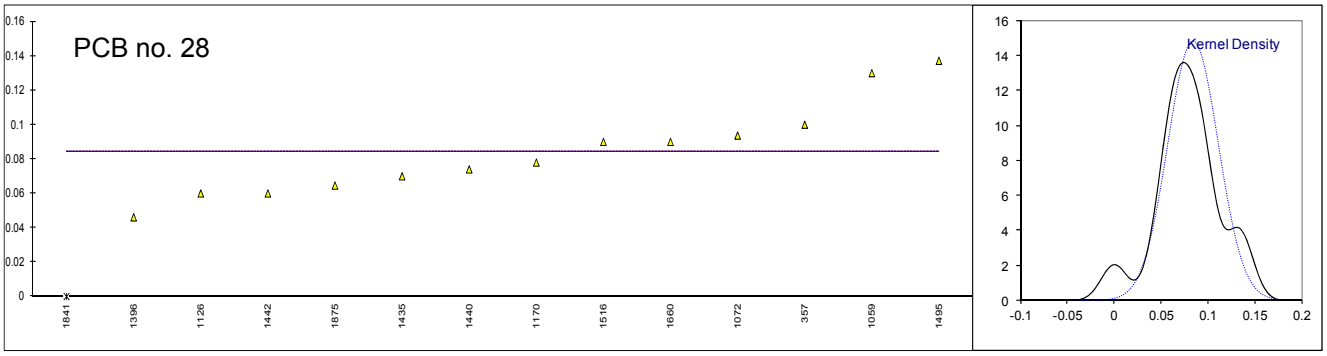
lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840	EN14582	25.6	ex	15.11	Possibly a false positive test result, see §4.1?
912		-----		-----	
1059		-----		-----	
1072		-----		-----	
1126	EN14077	15.0		2.02	
1135	In house	<0.1		-----	Possibly a false negative test result, see §4.1?
1170		-----		-----	
1201		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358		-----		-----	
1367		-----		-----	
1374		-----		-----	
1396		-----		-----	
1429	D7359	13.0		-0.45	
1435		-----		-----	
1440		-----		-----	
1442		-----		-----	
1458		-----		-----	
1495		-----		-----	
1505		-----		-----	
1513		-----		-----	
1516		-----		-----	
1568		-----		-----	
1633		-----		-----	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		-----		-----	
1875		-----		-----	
1888		-----		-----	
2122		-----		-----	
3132		-----		-----	
3195		-----		-----	
4091		-----		-----	
6089		-----		-----	
9311	UOP779	12.1		-1.56	
	normality	unknown			
	n	3			
	outliers	0+1ex			
	mean (n)	13.37			
	st.dev. (n)	1.484			
	R(calc.)	4.16			
	R(UOP779:08)	2.27			

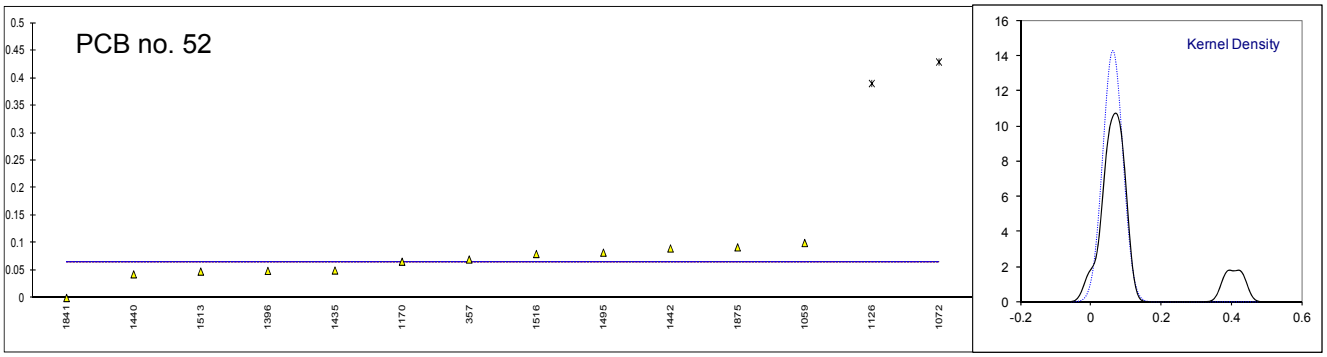

28
26
24
22
20
18
16
14
12
10
8
9311
1429
1126
840

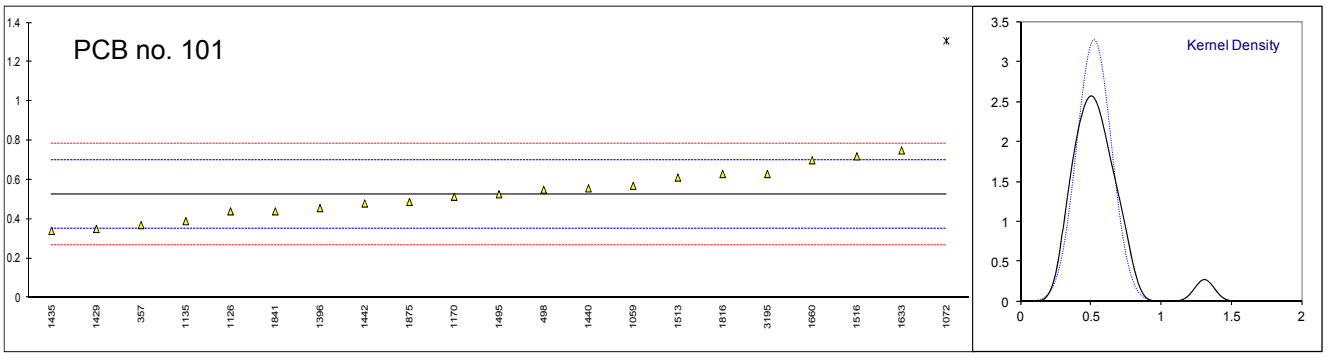
Determination of PCB 28, 52 and 101 on sample #16253; results in mg/kg.

Lab	Method	No.28	mark	z(targ)	No.52	mark	z(targ)	No.101	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357	EN12766-1	0.10		-----	0.07		-----	0.37		-1.80
398		-----		-----	-----		-----	-----		-----
445		-----		-----	-----		-----	-----		-----
498	EN12766-1	<0,3		-----	<0,3		-----	0.55		0.28
511		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
840		-----		-----	-----		-----	-----		-----
912		-----		-----	-----		-----	-----		-----
1059		0.13		-----	0.10		-----	0.57		0.51
1072	IEC61619	0.0937		-----	0.4291	DG(0.01)	-----	1.3081	R(0.01)	9.06
1126	EN12766-1	0.06		-----	0.39	DG(0.01)	-----	0.44		-0.99
1135	In house	<0.1		-----	<0.1		-----	0.39		-1.57
1170	EN12766-1	0.078		-----	0.066		-----	0.514		-0.14
1201	EN12766-1	<0.5		-----	<0.5		-----	<0.5		-----
1303		-----		-----	-----		-----	-----		-----
1304		-----		-----	-----		-----	-----		-----
1306		-----		-----	-----		-----	-----		-----
1352		-----		-----	-----		-----	-----		-----
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374		-----		-----	-----		-----	-----		-----
1396	IP462-1	0.046109		-----	0.0492468		-----	0.456399		-0.80
1429		< 0.2		-----	< 0.2		-----	0.35		-2.03
1435		0.07		-----	0.05		-----	0.34		-2.15
1440	IEC61619	0.074		-----	0.043		-----	0.557		0.36
1442	EN12766-1	0.06		-----	0.09		-----	0.48		-0.53
1458		-----		-----	-----		-----	-----		-----
1495		0.1372		-----	0.0824		-----	0.5267		0.01
1505		-----		-----	-----		-----	-----		-----
1513	IEC61619	<0,2		-----	0.048		-----	0.612		1.00
1516	IEC61619	0.09		-----	0.08		-----	0.72		2.25
1568		-----		-----	-----		-----	-----		-----
1633	EN12766-1	<0.10		-----	<0.10		-----	0.75		2.60
1660	IEC61619	0.09		-----	<0.2		-----	0.7		2.02
1743		-----		-----	-----		-----	-----		-----
1801		-----		-----	-----		-----	-----		-----
1816		<1		-----	<1		-----	0.63		1.21
1841	IEC61619	0.00	D(0.05)	-----	0.00		-----	0.44		-0.99
1875	In house	0.0646		-----	0.0920		-----	0.4875		-0.44
1888		-----		-----	-----		-----	-----		-----
2122		-----		-----	-----		-----	-----		-----
3132		-----		-----	-----		-----	-----		-----
3195		<0.20		-----	<0.20		-----	0.63		1.21
4091		-----		-----	-----		-----	-----		-----
6089		-----		-----	-----		-----	-----		-----
9311		-----		-----	-----		-----	-----		-----
	normality	OK			suspect			OK		
	n	13			12			20		
	outliers	1			2			1		
	mean (n)	(0.0841)			(0.0642)			0.5257		
	st.dev. (n)	(0.02687)			(0.02796)			0.12191		
	R(calc.)	(0.0752)			(0.0783)			0.3413		
	R(EN12766-1:00)	(0.0202)			(0.0102)			0.2418		
	Compare R(Horwitz)	(0.0547)			(0.0435)			0.2594		

Lab 1072 reported possibly a false positive test result for PCB no. 52?
Lab 1126 reported possibly a false positive test result for PCB no. 52?


PCB no. 28
Kernel Density

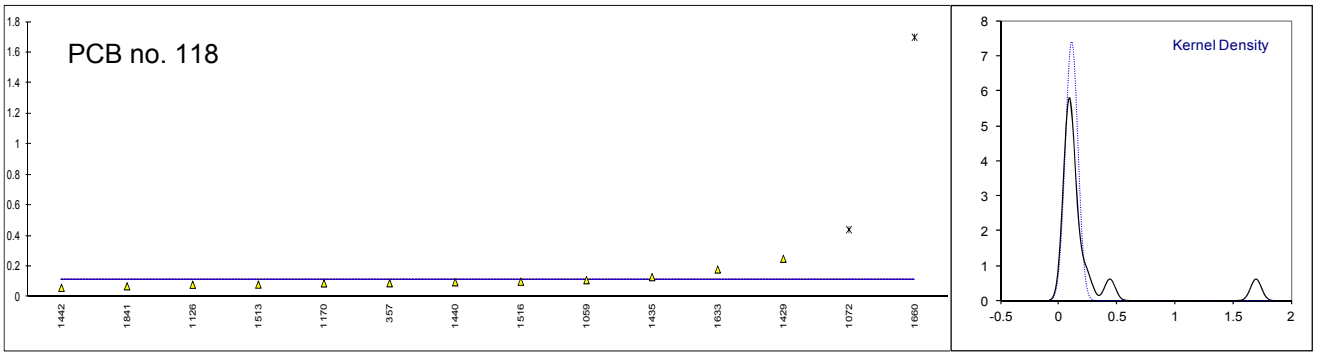

PCB no. 52
Kernel Density

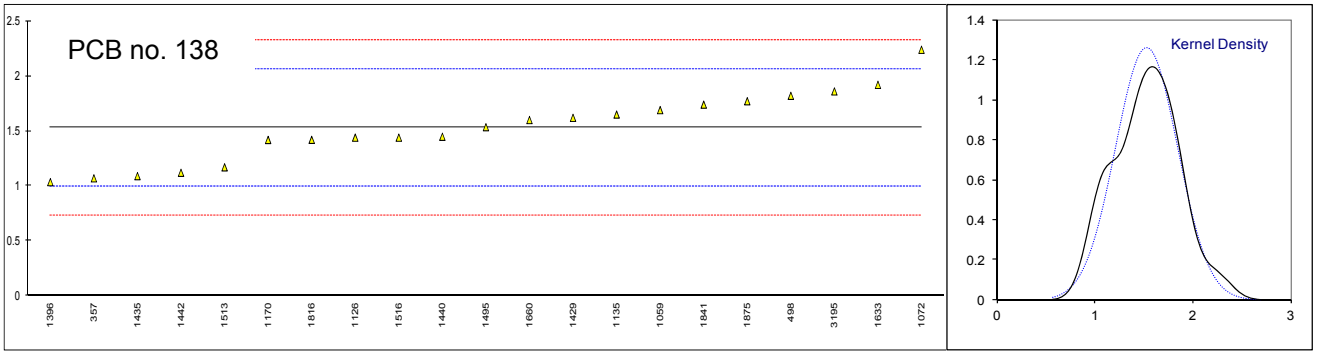

PCB no. 101
Kernel Density

Determination of PCB 118 and 138 on sample #16253; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)
341		-----		-----	-----		-----
343		-----		-----	-----		-----
357	EN12766-1	0.09		-----	1.07		-1.72
398		-----		-----	-----		-----
445		-----		-----	-----		-----
498	EN12766-1	<0,3		-----	1.82		1.10
511		-----		-----	-----		-----
614		-----		-----	-----		-----
840		-----		-----	-----		-----
912		-----		-----	-----		-----
1059		0.11		-----	1.69		0.61
1072	IEC61619	0.4405	D(0.01)	-----	2.2391		2.67
1126	EN12766-1	0.08		-----	1.44		-0.33
1135	In house	-----		-----	1.65		0.46
1170	EN12766-1	0.089		-----	1.419		-0.41
1201	EN12766-1	<0.5		-----	<0.5		<-3.86
1303		-----		-----	-----		-----
1304		-----		-----	-----		-----
1306		-----		-----	-----		-----
1352		-----		-----	-----		-----
1358		-----		-----	-----		-----
1367		-----		-----	-----		-----
1374		-----		-----	-----		-----
1396	IP462-1	-----		-----	1.03516		-1.85
1429		0.25		-----	1.62		0.34
1435		0.13		-----	1.09		-1.65
1440	IEC61619	0.096		-----	1.448		-0.30
1442	EN12766-1	0.06		-----	1.12		-1.53
1458		-----		-----	-----		-----
1495		-----		-----	1.5349		0.02
1505		-----		-----	-----		-----
1513	IEC61619	0.081		-----	1.170		-1.35
1516	IEC61619	0.10		-----	1.44		-0.33
1568		-----		-----	-----		-----
1633	EN12766-1	0.18		-----	1.92		1.47
1660	IEC61619	1.7	D(0.01)	-----	1.6		0.27
1743		-----		-----	-----		-----
1801		-----		-----	-----		-----
1816		<1		-----	1.42		-0.41
1841	IEC61619	0.07		-----	1.74		0.79
1875	In house	-----		-----	1.7711		0.91
1888		-----		-----	-----		-----
2122		-----		-----	-----		-----
3132		-----		-----	-----		-----
3195		<0.20		-----	1.86		1.25
4091		-----		-----	-----		-----
6089		-----		-----	-----		-----
9311		-----		-----	-----		-----
	normality	not OK			OK		
	n	12			21		
	outliers	2			0		
	mean (n)	(0.1113)			1.5284		
	st.dev. (n)	(0.05382)			0.31609		
	R(calc.)	(0.1507)			0.8850		
	R(EN12766-1:00)	(0.0339))			0.7451		
	Compare R(Horwitz)	(0.0694)			0.6424		

Lab 1072 reported possibly a false positive test result for PCB no. 118?
Lab 1201 reported possibly a false negative test result for PCB no. 138?
Lab 1660 reported possibly a false positive test result for PCB no. 118?

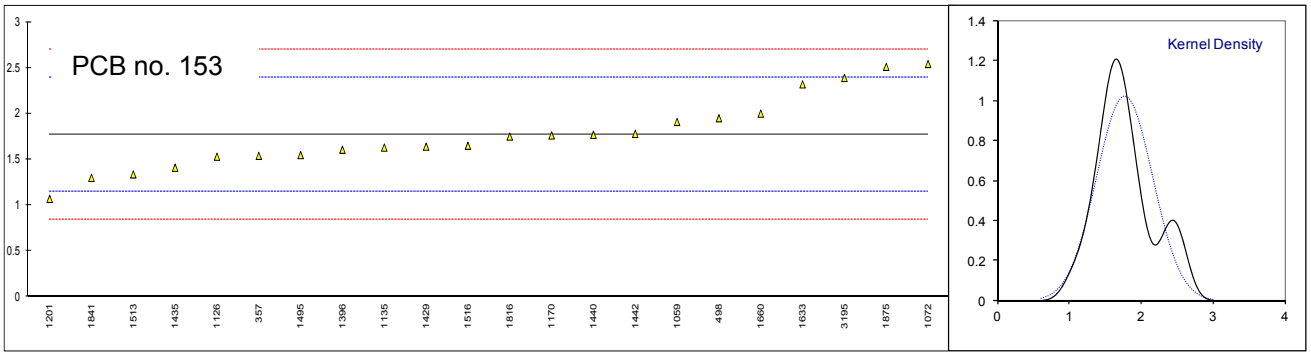

PCB no. 118
1442
1841
1126
1513
1170
357
1440
1516
1059
1435
1633
1429
1072
1660
Kernel Density

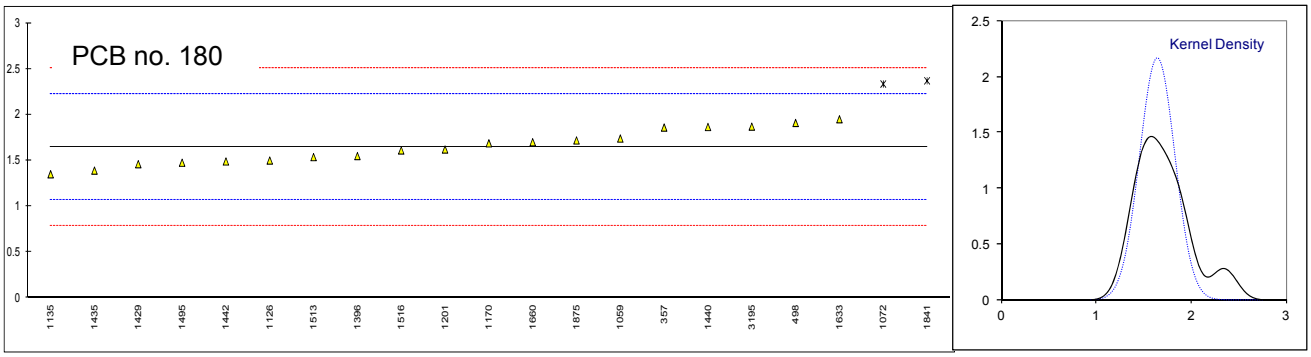

PCB no. 138
1396
357
1435
1442
1513
1170
1816
1126
1516
1440
1495
1660
1429
1135
1059
1841
1875
498
3195
1633
1072
Kernel Density

Determination of PCB 153 and 180 on sample #16253; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)
341		-----		-----	-----		-----
343		-----		-----	-----		-----
357	EN12766-1	1.54		-0.74	1.86	C	0.74
398		-----		-----	-----		-----
445		-----		-----	-----		-----
498	EN12766-1	1.95		0.58	1.91		0.92
511		-----		-----	-----		-----
614		-----		-----	-----		-----
840		-----		-----	-----		-----
912		-----		-----	-----		-----
1059		1.91		0.45	1.74		0.33
1072	IEC61619	2.5422		2.49	2.3350	R(0.05)	2.40
1126	EN12766-1	1.53		-0.78	1.50		-0.51
1135	In house	1.63		-0.45	1.35		-1.03
1170	EN12766-1	1.763		-0.02	1.688		0.14
1201	EN12766-1	1.07		-2.26	1.62		-0.09
1303		-----		-----	-----		-----
1304		-----		-----	-----		-----
1306		-----		-----	-----		-----
1352		-----		-----	-----		-----
1358		-----		-----	-----		-----
1367		-----		-----	-----		-----
1374		-----		-----	-----		-----
1396	IP462-1	1.60646		-0.53	1.54882		-0.34
1429		1.64		-0.42	1.46		-0.65
1435		1.41		-1.16	1.39		-0.89
1440	IEC61619	1.770		0.00	1.866		0.76
1442	EN12766-1	1.78		0.03	1.49		-0.55
1458		-----		-----	-----		-----
1495		1.5482		-0.72	1.476		-0.59
1505		-----		-----	-----		-----
1513	IEC61619	1.337		-1.40	1.537		-0.38
1516	IEC61619	1.65		-0.39	1.61		-0.13
1568		-----		-----	-----		-----
1633	EN12766-1	2.32		1.78	1.95		1.06
1660	IEC61619	2.0		0.74	1.7		0.19
1743		-----		-----	-----		-----
1801		-----		-----	-----		-----
1816		1.75		-0.07	-----	W	-----
1841	IEC61619	1.30		-1.52	2.37	R(0.05)	2.52
1875	In house	2.5108		2.39	1.7192		0.25
1888		-----		-----	-----		-----
2122		-----		-----	-----		-----
3132		-----		-----	-----		-----
3195		2.39		2.00	1.87		0.78
4091		-----		-----	-----		-----
6089		-----		-----	-----		-----
9311		-----		-----	-----		-----
	normality	OK			OK		
	n	22			19		
	outliers	0			2		
	mean (n)	1.7703			1.6466		
	st.dev. (n)	0.39053			0.18423		
	R(calc.)	1.0935			0.5159		
	R(EN12766-1:00)	0.8665			0.8044		
	Compare R(Horwitz)	0.7278			0.6843		

Lab 357 first reported for PCB no. 180: 2.84
Lab 1841 first reported for PCB no. 180: 2.94

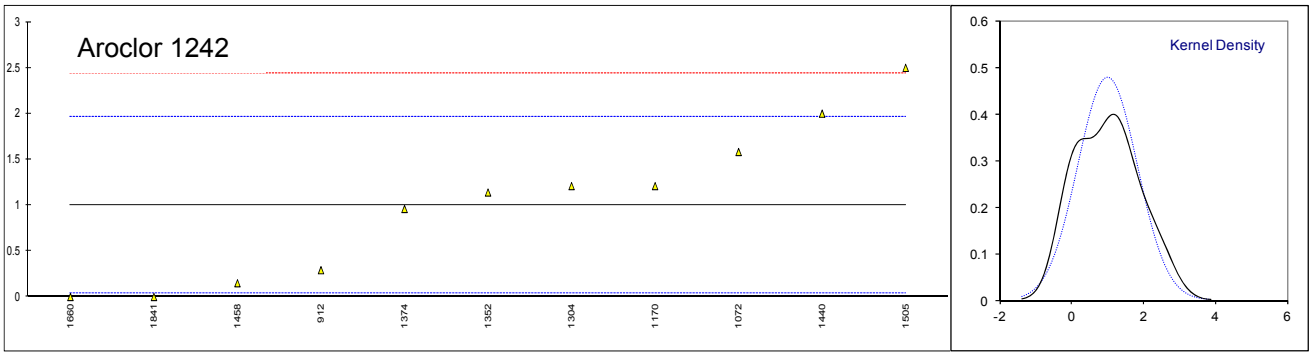

PCB no. 153
Kernel Density

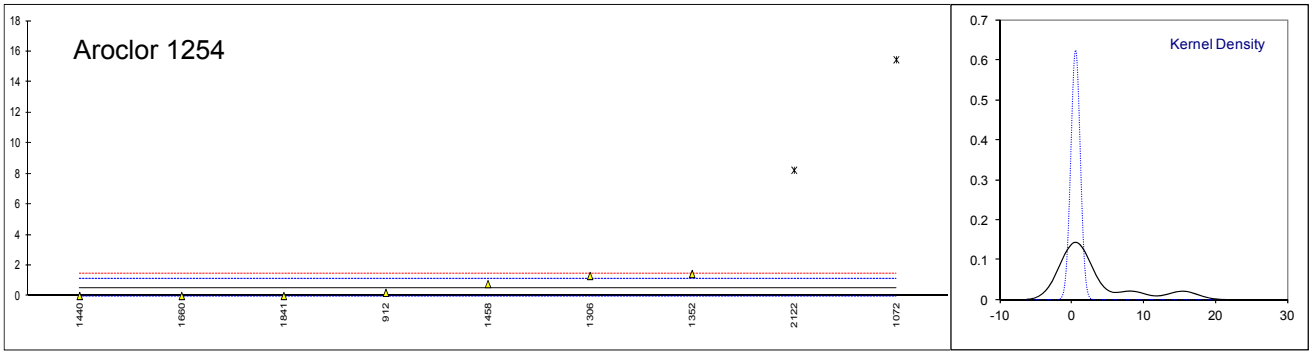

PCB no. 180
Kernel Density

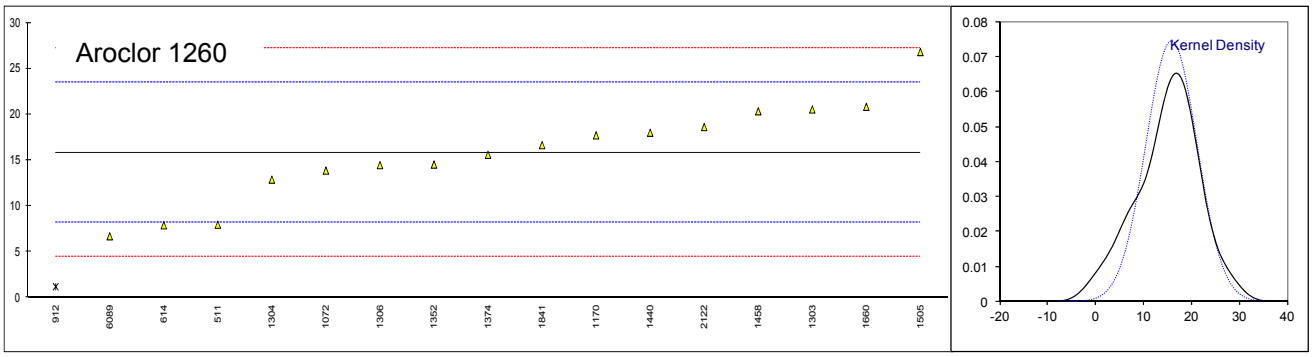
Determination of Aroclor 1242, 1254 and 1260 on sample #16253; results in mg/kg.

lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)
341		-----		-----	-----		-----	-----		-----
343		-----		-----	-----		-----	-----		-----
357		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
445		-----		-----	-----		-----	-----		-----
498		-----		-----	-----		-----	-----		-----
511		<1		-----	<1		-----	7.97		-2.07
614	D4059	<2		-----	<2		-----	7.9		-2.09
840		-----		-----	-----		-----	-----		-----
912	D4059	0.292		-1.48	0.204		-1.10	1.197	C, D(0.05)	-3.85
1059		-----		-----	-----		-----	-----		-----
1072	In house	1.582		1.20	15.466	G(0.05)	49.99	13.871		-0.52
1126		-----		-----	-----		-----	-----		-----
1135		-----		-----	-----		-----	-----		-----
1170	D4059	1.21		0.43	<2.0		-----	17.71		0.49
1201		-----		-----	-----		-----	-----		-----
1303	D4059	<2		-----	<2		-----	20.542		1.24
1304	INH-127	1.210		0.43	<0.50		-----	12.882		-0.78
1306	In house	-----		-----	1.31		2.60	14.47		-0.36
1352	In house	1.14		0.28	1.44		3.03	14.53		-0.34
1358		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1374	D4059	0.96		-0.09	<0.3		-----	15.59		-0.07
1396		-----		-----	-----		-----	-----		-----
1429		-----		-----	-----		-----	-----		-----
1435		-----		-----	-----		-----	-----		-----
1440	In house	2.00		2.07	0.00		-1.79	18.00		0.57
1442		-----		-----	-----		-----	-----		-----
1458	D4059	0.15		-1.78	0.78		0.83	20.341		1.19
1495		-----		-----	-----		-----	-----		-----
1505	D4059	2.5		3.12	<1		-----	26.8		2.89
1513		-----		-----	-----		-----	-----		-----
1516		-----		-----	-----		-----	-----		-----
1568		-----		-----	-----		-----	-----		-----
1633		-----		-----	-----		-----	-----		-----
1660	IEC61619	0		-2.09	0		-1.79	20.83		1.31
1743		-----		-----	-----		-----	-----		-----
1801		-----		-----	-----		-----	-----		-----
1816		-----		-----	-----		-----	-----		-----
1841	IEC61619	0.00		-2.09	0.00		-1.79	16.65		0.21
1875		-----		-----	-----		-----	-----		-----
1888		-----		-----	-----		-----	-----		-----
2122		-----		-----	8.22775	G(0.01)	25.76	18.62215		0.73
3132		-----		-----	-----		-----	-----		-----
3195		-----		-----	-----		-----	-----		-----
4091		-----		-----	-----		-----	-----		-----
6089	D4059	-----		-----	-----		-----	6.7		-2.41
9311		-----		-----	-----		-----	-----		-----
normality		OK			OK			OK		
n		11			7			16		
outliers		0			2			1		
mean (n)		1.0040			0.5334			15.8380		
st.dev. (n)		0.83197			0.63873			5.34634		
R(calc.)		2.3295			1.7885			14.9698		
R(D4059:00 (silicone))		1.3440			0.8364			10.6385		
Compare R(D4059:00 (packed))		1.0331			0.6429			8.1774		
Compare R(D4059:00 (megabore))		0.7924			0.4931			6.2719		

Lab 912 first reported: 1.609 for Aroclor 1260

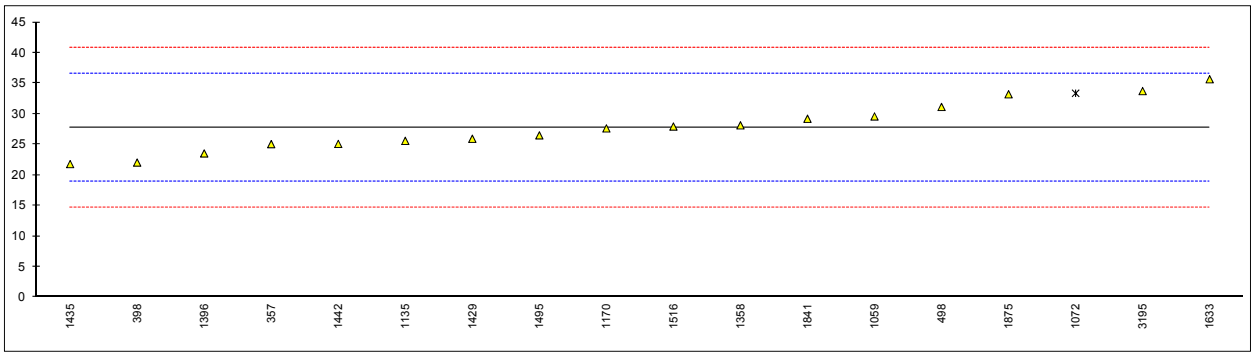

Aroclor 1242
1660
1841
1458
912
1374
1352
1304
1170
1072
1440
1505
Kernel Density

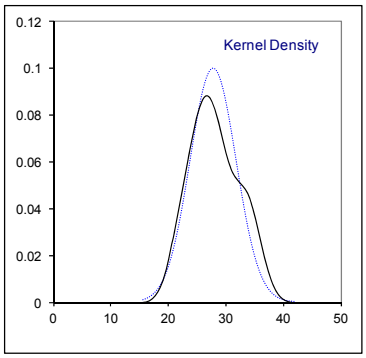

Aroclor 1254
1440
1660
1841
912
1458
1306
1352
2122
1072
Kernel Density


Aroclor 1260
912
6089
614
511
1304
1072
1306
1352
1374
1841
1170
1440
2122
1458
1303
1660
1505
Kernel Density

Determination of Total PCB, 5 times the sum of 6 congeners on sample #16253; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357	EN12766-2-B	25.07	C	-0.61	first reported: 29.97
398	EN12766-2-B	22.05		-1.30	
445		-----		-----	
498	EN12766-2-B	31.15		0.77	
511		-----		-----	
614		-----		-----	
840		-----		-----	
912		-----		-----	
1059	EN12766-2-B	29.6		0.42	
1072	EN12766-2-B	33.389	ex, E	1.28	ex: iis calculated: 44.736
1126		-----		-----	
1135	In house	25.6		-0.49	
1170	EN12766-2-B	27.64		-0.03	
1201		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358	IP462-2	28.14		0.09	
1367		-----		-----	
1374		-----		-----	
1396	IP462-2	23.5440		-0.96	
1429	EN12766-2-B	25.94		-0.42	
1435	IEC61619	21.80		-1.36	
1440		-----		-----	
1442	EN12766-2-B	25.1		-0.61	
1458		-----		-----	
1495	EN12766-2-B	26.5		-0.29	
1505		-----		-----	
1513		-----		-----	
1516	EN12766-2-B	27.95		0.04	
1568		-----		-----	
1633	EN12766-2-B	35.69		1.81	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841	EN12766-2-B	29.22		0.33	
1875	EN12766-2-B	33.2257		1.25	
1888		-----		-----	
2122		-----		-----	
3132		-----		-----	
3195	EN12766-2-B	33.75		1.37	
4091		-----		-----	
6089		-----		-----	
9311		-----		-----	
					iis calculated
	normality	OK			OK
	n	17			18
	outliers	0+1ex			0+1ex
	mean (n)	27.7629			27.7424
	st.dev. (n)	3.99703			3.70504
	R(calc.)	11.1917			10.3741
	R(EN12766-2B:01)	12.2618			12.2525

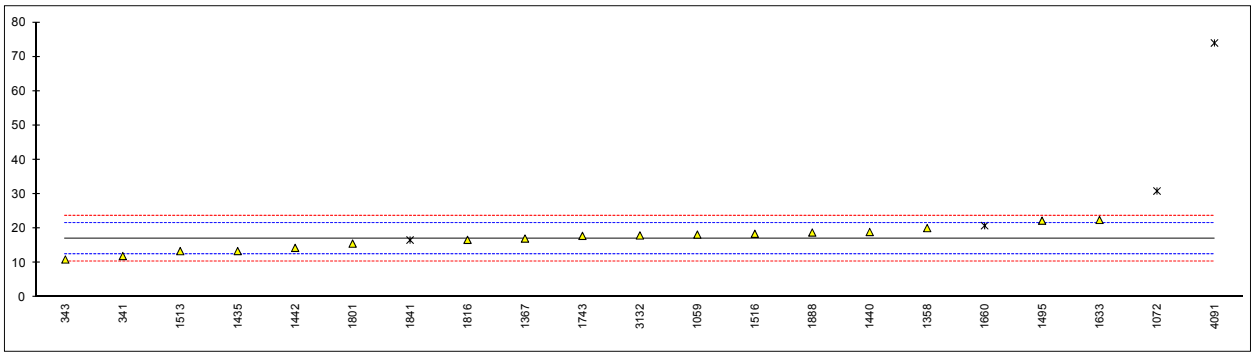

45
40
35
30
25
20
15
10
5
0
1435
398
1396
357
1442
1135
1429
1495
1170
1516
1358
1841
1059
498
1875
1072
3195
1633

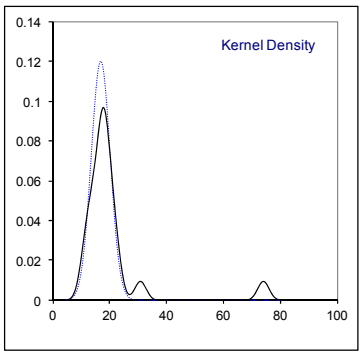

Kernel Density
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30
40
50

Determination of Total PCB, sum of all congeners on sample #16253; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	12		-2.24	
343	EN61619	11		-2.69	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		-----		-----	
614		-----		-----	
840		-----		-----	
912		-----		-----	
1059	EN12766-2-A	18.24		0.55	
1072	EN61619	30.920	ex	6.23	ex: is total of all Aroclors
1126		-----		-----	
1135		-----		-----	
1170		-----		-----	
1201		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1352		-----		-----	
1358	IP462-2	20.14		1.41	
1367	EN61619	17.09		0.04	
1374		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435	IEC61619	13.45		-1.59	
1440	EN61619	19.00		0.89	
1442	IEC61619	14.38		-1.17	
1458		-----		-----	
1495	EN12766-2-A	22.3		2.37	
1505		-----		-----	
1513		13.427		-1.60	
1516	IEC61619	18.47		0.66	
1568		-----		-----	
1633	EN61619	22.5		2.46	
1660	IEC61619	20.83	ex	1.71	ex: is total of all Aroclors
1743	EN61619	17.87		0.39	
1801	EN61619	15.62		-0.62	
1816	IEC61619	16.7		-0.14	
1841	IEC61619	16.65	ex	-0.16	ex: is identical as Aroclor 1260
1875		-----		-----	
1888	EN61619	18.851		0.83	
2122		-----		-----	
3132	EN61619	18		0.45	
3195		-----		-----	
4091	EN61619	74	R(0.01)	25.53	
6089		-----		-----	
9311		-----		-----	

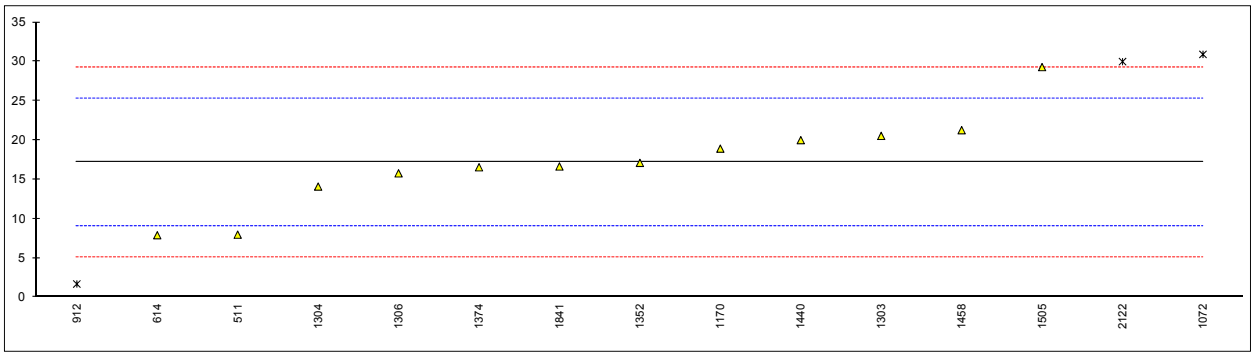
normality	OK
n	17
outliers	1+3ex
mean (n)	17.0022
st.dev. (n)	3.31961
R(calc.)	9.2949
R(EN61619:97)	6.2506

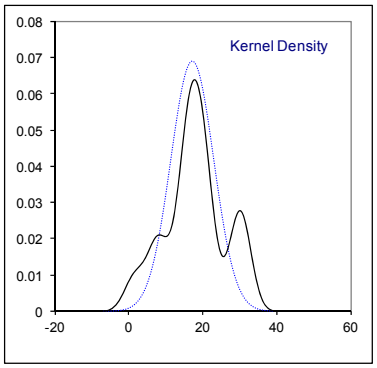

80
70
60
50
40
30
20
10
0
343
341
1513
1435
1442
1801
1841
1816
1367
1743
3132
1059
1516
1888
1440
1358
1660
1495
1633
1072
4091


Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60
80
100

Determination of Total PCB, sum of all Aroclors on sample #16253; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
398		-----		-----	
445		-----		-----	
498		-----		-----	
511		7.97		-2.28	
614	D4059	7.9		-2.30	
840		-----		-----	
912		2.105	ex	-3.83	ex: outlier in Aroclor1260
1059		-----		-----	
1072	In house	30.92	ex	3.40	ex: outlier in Aroclor1254
1126		-----		-----	
1135		-----		-----	
1170	D4059	18.92		0.43	
1201		-----		-----	
1303		20.542		0.83	
1304		14.092		-0.76	
1306	In house	15.78		-0.35	
1352		17.11		-0.02	
1358		-----		-----	
1367		-----		-----	
1374	D4059	16.55		-0.15	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1440	In house	20.00		0.70	
1442		-----		-----	
1458		21.271		1.01	
1495		-----		-----	
1505	D4059	29.3		3.00	
1513		-----		-----	
1516		-----		-----	
1568		-----		-----	
1633		-----		-----	
1660		-----		-----	
1743		-----		-----	
1801		-----		-----	
1816		-----		-----	
1841		16.65		-0.13	
1875		-----		-----	
1888		-----		-----	
2122		30	ex	3.18	ex: outlier in Aroclor1254
3132		-----		-----	
3195		-----		-----	
4091		-----		-----	
6089		-----		-----	
9311		-----		-----	
					iis calculated
normality		suspect			OK
n		12			14
outliers		0+3ex			0+3ex
mean (n)		17.1737			16.6868
st.dev. (n)		5.79211			6.13177
R(calc.)		16.2179			17.1690
R(D4059:00 (silicone))		11.3046			11.0633
Compare R(D4059:00 (packed))		8.6893			8.5039
Compare R(D4059:00 (megabore))		6.6646			6.5224


35
30
25
20
15
10
5
0
912
614
511
1304
1306
1374
1841
1352
1170
1440
1303
1458
1505
2122
1072


Kernel Density
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-20
0
20
40
60

APPENDIX 2

Number of participating laboratories per country

6 labs in	AUSTRALIA
2 labs in	BELGIUM
1 lab in	FINLAND
3 labs in	FRANCE
3 labs in	GERMANY
2 labs in	GREECE
1 lab in	INDIA
2 labs in	ITALY
1 lab in	LUXEMBOURG
1 lab in	MEXICO
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NORWAY
1 lab in	PERU
2 labs in	PORTUGAL
2 labs in	SLOVENIA
2 labs in	SOUTH AFRICA
7 labs in	SPAIN
6 labs in	UNITED KINGDOM
1 lab in	VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

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