

Results of Proficiency Test  
PCB in (Mineral) Oil  
October 2012

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 2012/2013, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.

In this interlaboratory study, 46 laboratories from 17 different countries have participated. See appendix 2 for the number of participating laboratories per country. In this report the results of the 2012 interlaboratory study on PCB analysis are presented and discussed.

## **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 #12147) that was donated by one of the participating laboratories. 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 QUALITY SYSTEM**

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system on ISO guide 43, ILAC-G13:2007 and ISO17043:2010. 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 January 2010 (iis-protocol, version 3.2).

### **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, being contaminated waste oil (positive on PCBs) was donated by a third party laboratory. After ultrasonic homogenisation, 46 subsamples were transferred to 8 mL amber glass vials, all labelled #12147.

The homogeneity of the subsamples #12147 was checked by determination of the organic chloride content in accordance with UOP779-08 on four stratified randomly selected samples:

	Organic chloride in mg/kg
sample #12147-1	7.9
sample #12147-2	8.2
sample #12147-3	8.9
sample #12147-4	8.8

Table 1: homogeneity test results of subsample #12147

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:

	#12147
r (samples)	1.0
reference method	UOP779:08
$0.3 \times R_{(\text{reference method})}$	2.1

Table 2: evaluation of the observed repeatability

The repeatability of the results of homogeneity test is in good agreement with 0.3 times the reproducibility as required by UOP779:08. Therefore, homogeneity of the samples was assumed. To each of the participating laboratories one vial of 8 mL (labelled #12147) was sent on October 24, 2012.

## 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 via Aroclors) on the sample.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website ([www.iisnl.com](http://www.iisnl.com)).

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

### 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 January 2010 (iis-protocol, version 3.2).

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. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the 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 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 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 (see appendix 3; nos.13 and 14).

### 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:

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

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 the fit-for-useness of the reported test result.

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 eleven participants, reported results after the final reporting date. Five participants did not report any results at all. Not all participants were able to report results for all tests. In total 41 participating laboratories reported 204 numerical results. Observed were 10 outlying results, which is 4.9% 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.

The abbreviations, used in these tables, are listed in appendix 3.

A not normal distribution was observed for PCB 101, PCB 180 and Aroclor 1242. The statistical evaluations of these two sets of test results 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. For the results on sample #12147, the following was concluded:

#### TOX

No significant conclusions were drawn as only four test results were reported.

#### Individual PCBs:

This determination was problematic for six congeners.

For the evaluation of the individual congeners method EN12766-1:00 / IP462-1/01 was used. In the methods IEC61619:97 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 seven statistical outliers were observed. The calculated reproducibilities of six congeners are not in agreement with requirements of EN12766-1:00 / IP462-1/01.

All test results (except one) of laboratory 1660 appeared to be statistical outliers. Therefore it was decided not to use any of the test results of this laboratory for the statistical evaluation.

#### Total PCB:

The determination of total PCB content was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of IEC 61619:97.

Several laboratories used IP426 or EN12766 as test method. In method EN12766-2:01 / IP462-2/02 total PCB content is calculated according:  $PCB_{Total} = 5 * \sum_{(n=6)} \text{congeners}$ . The total PCB results of laboratories 1358 and 1551 were excluded prior to the statistical analysis, because the factor five obviously was not applied.

Indiv. Aroclors:

The determination of the individual Aroclors was somewhat problematic. No significant conclusions were drawn for Aroclor 1242 as the reported test results were near or below the detection limit. No statistical outliers were observed for Aroclor 1254 and Aroclor 1260. However, the calculated reproducibility for each of the Aroclors is not in agreement with the requirements of ASTM D4059:10. The test results for Aroclor 1254 and Aroclor 1260 reported by laboratory 1463 were excluded because the (equal) test results were not in agreement with the reported total Aroclor content.

Total Aroclor:

This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D4059:10.

Summary:

All participants agreed that sample #12147 was positive on PCBs. The assigned value for the – by iis - calculated sums of the 6 PCB congeners 28, 52, 101, 138, 153 and 180 is 3.72 mg/kg. From this sum, a total concentration of 18.6 mg PCB/kg was estimated acc. to EN12766-2:01 / IP462-2/02. ( $PCB_{Total} = 5 * \sum_{(n=6)} \text{congeners}$ ). For the determination of the total Aroclors an average of 12.7 mg PCB/kg was found. From the data on total organic chloride (TOX) an average concentration of 12.2 mg/kg was calculated. From this concentration a total content of 21.4 mg PCB/kg was estimated using an average Cl content of 57%, assuming the presence of equal amounts of Aroclor 1260 and Aroclor 1254. 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.

	#12147
total PCB content, estimated from 6 congeners, in mg/kg	18.6
total PCB content, using IEC 61619:97 method, in mg/kg	15.1
estimated total PCB content using Aroclor method, in mg/kg	12.7
total PCB content, estimated for TOX homogeneity data, in mg/kg	21.4

Table 3: Comparison of estimations of total PCB content in sample #12147.

The total PCB content as determined by IEC61619:97 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	12.2	n.a	(10.0)
PCB no. 28	mg/kg	6	0.02	0.02	(-0.10)
PCB no. 52	mg/kg	20	0.37	0.25	0.16
PCB no. 101	mg/kg	20	0.95	0.92	0.46
PCB no. 118	mg/kg	16	0.47	0.37	0.22
PCB no. 138	mg/kg	20	0.97	0.70	0.47
PCB no. 153	mg/kg	20	0.88	0.48	0.42
PCB no. 180	mg/kg	20	0.53	0.39	0.24
Total PCB	mg/kg	19	15.1	11.1	5.77
Aroclor 1242	mg/kg	3	1.33	3.85	(1.66)
Aroclor 1254	mg/kg	12	7.94	7.13	6.34
Aroclor 1260	mg/kg	12	6.02	7.00	5.15
Total Aroclor	mg/kg	14	12.7	7.22	9.03

table 4: Performance of the group of participating laboratories on sample #12147

() 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 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 OCTOBER 2012 PROFICIENCY TEST WITH PREVIOUS PTS.

	<i>October 2012</i>	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>
Number of reporting labs	41	38	34	29
Number of results reported	204	195	186	329
Statistical outliers	10	4	15	8
Percentage outliers	4.9%	2.0%	8.1%	3.6%

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:

<i>Determination</i>	<i>October 2012</i>	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>
TOX	n.e.	n.e.	n.e.	n.e.
PCB (all)	--	+/-	-	--
Aroclor (all)	-	+/-	--	+/-

Table 6: comparison of observed precisions against standard requirements

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 #12147; results in mg/kg.

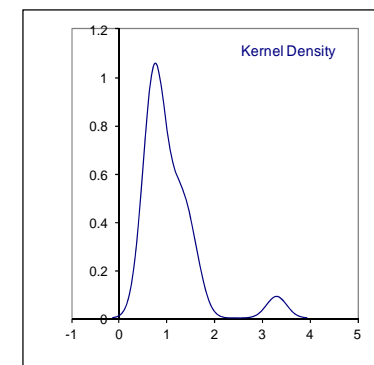
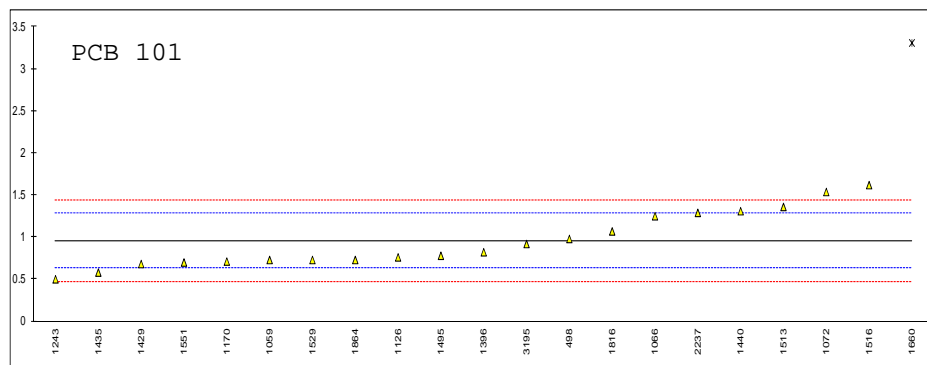
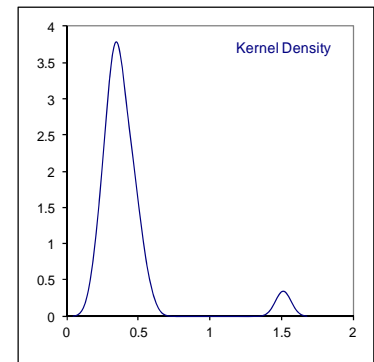
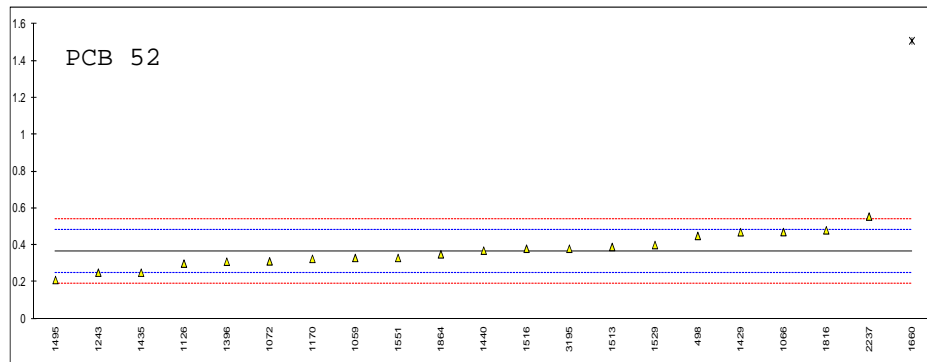
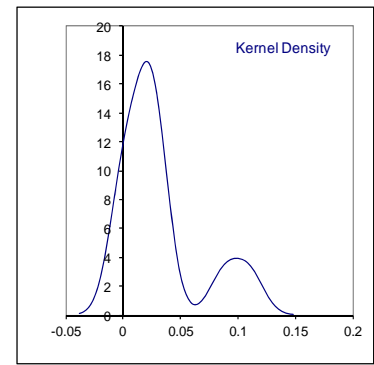
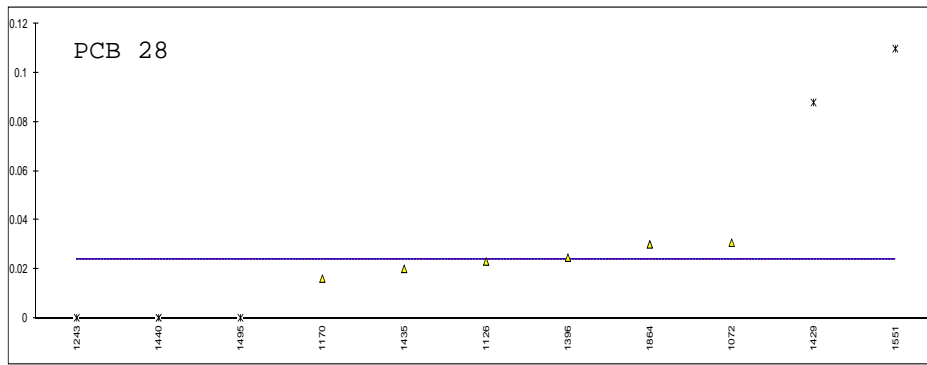
lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
398		----		----	
445		----		----	
498		----		----	
614		----		----	
902		----		----	
1059		----		----	
1066	UOP779	12		----	
1072		----		----	
1126	NEN7424	11.9		----	
1170		----		----	
1201		----		----	
1243		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1338		----		----	
1352		----		----	
1358		----		----	
1367		----		----	
1375		----		----	
1383		----		----	
1396		----		----	
1429	IP510	12.7		----	
1435		----		----	
1440		----		----	
1458		----		----	
1463		----		----	
1479		----		----	
1495		----		----	
1513		----		----	
1516		----		----	
1526		----		----	
1529		----		----	
1551		----		----	
1555		----		----	
1568		----		----	
1660		----		----	
1704		----		----	
1801		----		----	
1816		----		----	
1864	EPA5050/9056	73	C, G(0.01)	----	first reported:54.6
2160		----		----	
2237		----		----	
3195		----		----	
	normality	not OK			
	n	3			
	outliers	1			
	mean (n)	12.20			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(UOP779:08)	(9.98)			

Determination of PCB 28, 52 and 101 on sample #12147; results in mg/kg.

lab	method	No.28	mark	z(targ)	No.52	mark	z(targ)	No.101	mark	z(targ)	Remarks
341		----		----	----		----	----		----	
343		----		----	----		----	----		----	
398		----		----	----		----	----		----	
445		----		----	----		----	----		----	
498	EN12766B	<0.30		----	0.45		1.46	0.98		0.16	
614		----		----	----		----	----		----	
902		----		----	----		----	----		----	
1059	EN12766A	<0.2		----	0.33		-0.62	0.73		-1.37	
1066	EN12766	<0.2		----	0.4708		1.83	1.2503		1.82	
1072	EN61619	0.0307		----	0.3115		-0.94	1.5383		3.59	
1126	EN12766A	0.023		----	0.299		-1.15	0.761		-1.18	
1170	EN12766Mod.B	0.016		----	0.325		-0.70	0.711		-1.49	
1201		----		----	----		----	----		----	
1243	EN12766B	0	ex	----	0.25		-2.00	0.50		-2.78	
1303		----		----	----		----	----		----	
1304		----		----	----		----	----		----	
1306		----		----	----		----	----		----	
1338		----		----	----		----	----		----	
1352		----		----	----		----	----		----	
1358		----		----	----		----	----		----	
1367		----		----	----		----	----		----	
1375		----		----	----		----	----		----	
1383		----		----	----		----	----		----	
1396	IP462B	0.02460		----	0.30959		-0.97	0.82218		-0.80	
1429	EN12766B	0.088	DG(0.01)	----	0.470		1.81	0.682		-1.66	
1435	EN12766	0.02		----	0.25		-2.00	0.58		-2.29	
1440	IEC61619A	0	ex	----	0.37		0.08	1.31		2.19	
1458		----		----	----		----	----		----	
1463		----		----	----		----	----		----	
1479		----		----	----		----	----		----	
1495	EN12766B	0	ex	----	0.21		-2.70	0.78		-1.06	
1513	IEC61619A	<0.02		----	0.39		0.42	1.36		2.50	
1516	IEC61619A	<0.1		----	0.38		0.25	1.62	C	4.09	
1526		----		----	----		----	----		----	
1529	EN12766	<0.10		----	0.40		0.60	0.73		-1.37	
1551	IP462	0.11	DG(0.01)	----	0.33		-0.62	0.70		-1.55	
1555		----		----	----		----	----		----	
1568		----		----	----		----	----		----	
1660	EN12766A	<0.02		----	1.51	G(0.01)	19.85	3.31	G(0.01)	14.46	
1704		----	W	----	----	W	----	----	W	----	
1801		----		----	----		----	----		----	
1816		----		----	0.48		1.98	1.07		0.72	
1864	EN12766A	0.03		----	0.35		-0.27	0.73		-1.37	
2160		----		----	----		----	----		----	
2237	EN12766B	<0.1		----	0.555		3.29	1.29		2.07	
3195	EN12766	<0.1		----	0.38		0.25	0.92		-0.20	
	normality	OK			OK			not OK			
	n	6			20			20			
	outliers	2			1			1			
	mean (n)	0.024			0.366			0.953			
	st.dev. (n)	0.0057			0.0877			0.3302			
	R(calc.)	0.016			0.246			0.925			
	R(EN12766-1:99)	(-0.100)			0.161			0.456			

C = corrected result, first reported result lab 1516: PCB 101: 1.75  
W = withdrawn, first reported results by lab 1704: PCB 28: 0.0130  
PCB 52: 0.1104  
PCB 101: 0.5824

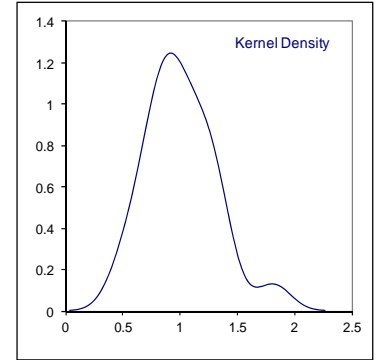
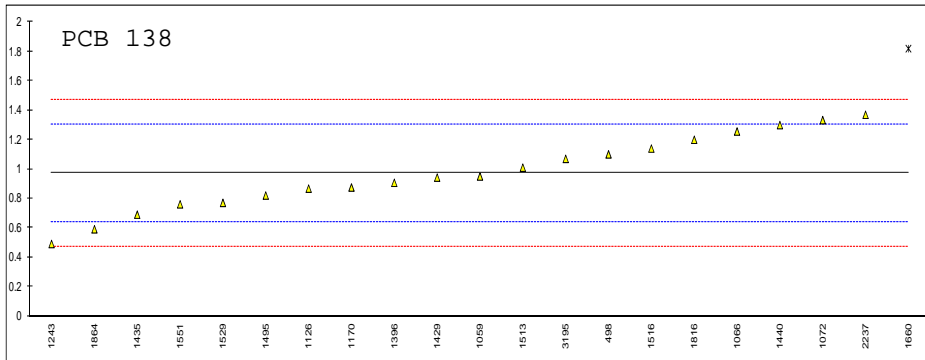
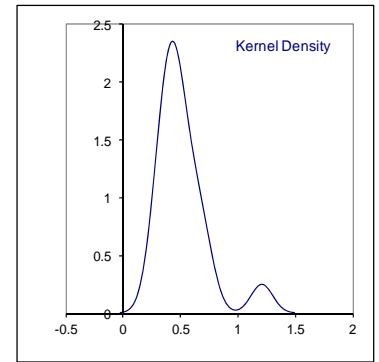
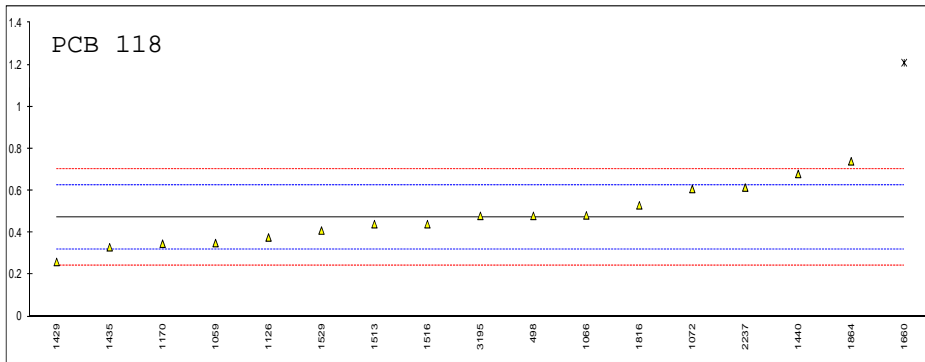
ex = excluded, zero is not a real value



Determination of PCB 118 and 138 on sample #12147; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		----		----	----		----	
343		----		----	----		----	
398		----		----	----		----	
445		----		----	----		----	
498	EN12766B	0.48		0.09	1.10		0.77	
614		----		----	----		----	
902		----		----	----		----	
1059	EN12766A	0.35		-1.60	0.95		-0.13	
1066	EN12766	0.482		0.12	1.2562		1.71	
1072	EN61619	0.6079		1.75	1.3334		2.17	
1126	EN12766A	0.377		-1.25	0.867		-0.63	
1170	EN12766Mod.B	0.347		-1.64	0.875		-0.58	
1201		----		----	----		----	
1243		----		----	0.49		-2.90	
1303		----		----	----		----	
1304		----		----	----		----	
1306		----		----	----		----	
1338		----		----	----		----	
1352		----		----	----		----	
1358		----		----	----		----	
1367		----		----	----		----	
1375		----		----	----		----	
1383		----		----	----		----	
1396	IP462B	----		----	0.90682		-0.39	
1429	EN12766B	0.260		-2.77	0.942		-0.18	
1435	EN12766	0.33		-1.86	0.69		-1.70	
1440	IEC61619A	0.68		2.69	1.30		1.97	
1458		----		----	----		----	
1463		----		----	----		----	
1479		----		----	----		----	
1495		----		----	0.82		-0.91	
1513	IEC61619A	0.44	C	-0.43	1.01		0.23	
1516	IEC61619A	0.44		-0.43	1.14		1.01	
1526		----		----	----		----	
1529	EN12766	0.41		-0.82	0.77		-1.21	
1551		----		----	0.76		-1.27	
1555		----		----	----		----	
1568		----		----	----		----	
1660	EN12766A	1.21	G(0.01)	9.58	1.82	G(0.05)	5.10	
1704		----	W	----	----	W	----	
1801		----		----	----		----	
1816	IEC61619	0.53		0.74	1.20		1.37	
1864	EN12766A	0.74		3.47	0.59	C	-2.30	
2160		----		----	----		----	
2237	EN12766B	0.615		1.84	1.37		2.39	
3195	EN12766	0.48		0.09	1.07		0.59	
	normality	OK			OK			
	n	16			20			
	outliers	1			1			
	mean (n)	0.473			0.972			
	st.dev. (n)	0.1337			0.2490			
	R(calc.)	0.374			0.697			
	R(EN12766-1:99)	0.215			0.466			

C = corrected result, first reported result lab 1513: PCB 101: 0.83  
 C = corrected result, first reported result lab 1864: PCB 138: 0.46  
 W = withdrawn, first reported results for lab 1704: PCB 118: 0.0735  
 PCB 138: 1.4379



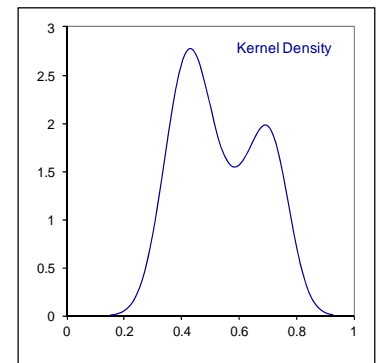
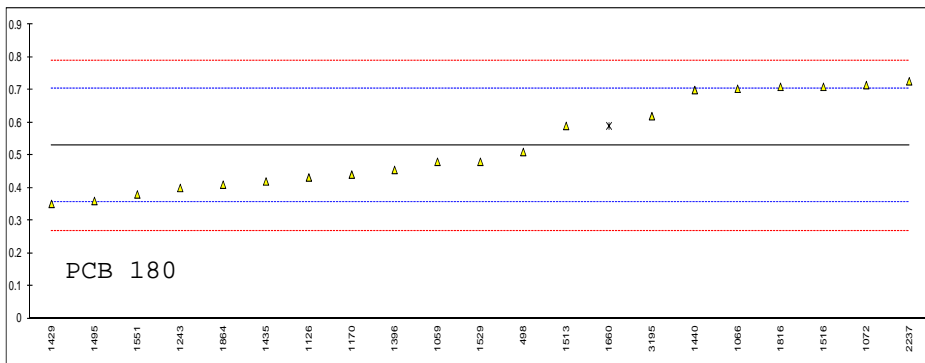
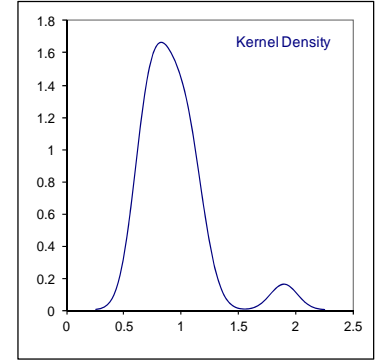
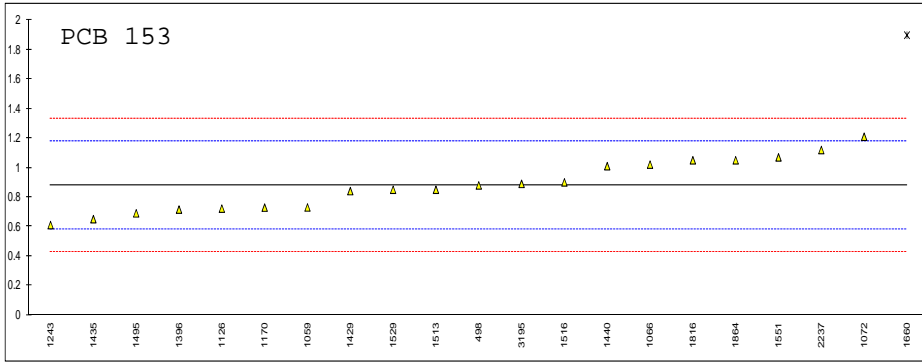
Determination of PCB 153 and 180 on sample #12147; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		----		----	----		----	
343		----		----	----		----	
398		----		----	----		----	
445		----		----	----		----	
498	EN12766B	0.88		0.00	0.51		-0.23	
614		----		----	----		----	
902		----		----	----		----	
1059	EN12766A	0.73		-1.00	0.48		-0.57	
1066	EN12766	1.0211		0.95	0.7041		2.00	
1072	EN61619	1.2099		2.21	0.7152		2.13	
1126	EN12766A	0.722		-1.05	0.432		-1.12	
1170	EN12766Mod.B	0.729		-1.00	0.441		-1.02	
1201		----		----	----		----	
1243	EN12766B	0.61		-1.80	0.40		-1.49	
1303		----		----	----		----	
1304		----		----	----		----	
1306		----		----	----		----	
1338		----		----	----		----	
1352		----		----	----		----	
1358		----		----	----		----	
1367		----		----	----		----	
1375		----		----	----		----	
1383		----		----	----		----	
1396	IP462B	0.71582		-1.09	0.45516		-0.86	
1429	EN12766B	0.841		-0.26	0.351	C	-2.05	
1435	EN12766	0.65		-1.53	0.42		-1.26	
1440	IEC61619A	1.01		0.87	0.70		1.95	
1458		----		----	----		----	
1463		----		----	----		----	
1479		----		----	----		----	
1495	EN12766B	0.69		-1.26	0.36		-1.95	
1513	IEC61619A	0.85		-0.20	0.59		0.69	
1516	IEC61619A	0.90		0.14	0.71		2.07	
1526		----		----	----		----	
1529	EN12766	0.85		-0.20	0.48		-0.57	
1551	IP462	1.07		1.27	0.38		-1.72	
1555		----		----	----		----	
1568		----		----	----		----	
1660	EN12766A	1.90	G(0.01)	6.81	0.59	ex	0.69	
1704		----	W	----	----	W	----	
1801		----		----	----		----	
1816	IEC61619	1.05		1.14	0.71		2.07	
1864	EN12766A	1.05		1.14	0.41		-1.38	
2160		----		----	----		----	
2237	EN12766B	1.12		1.61	0.727		2.26	
3195	EN12766	0.89		0.07	0.62		1.04	
	normality	OK			not OK			
	n	20			20			
	outliers	1			0			
	mean (n)	0.879			0.530			
	st.dev. (n)	0.1717			0.1384			
	R(calc.)	0.481			0.388			
	R(EN12766-1:99)	0.419			0.244			

C = corrected result, first reported result lab 1429: PCB 180: 0.267  
 W = withdrawn, first reported results for lab 1704: PCB 153: 1.1883  
 PCB 180: 1.0191

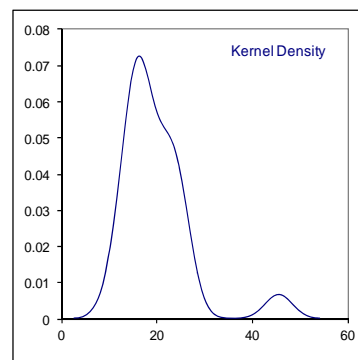
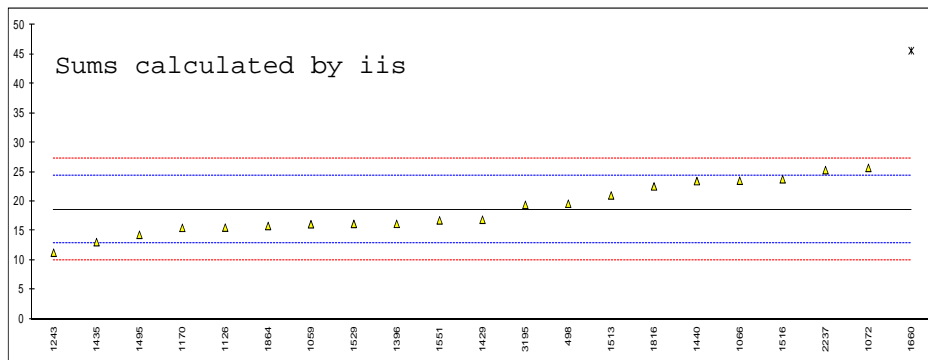
ex = excluded, see §4.1





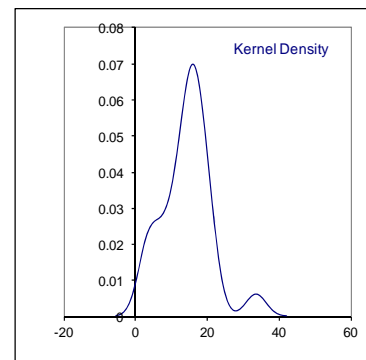
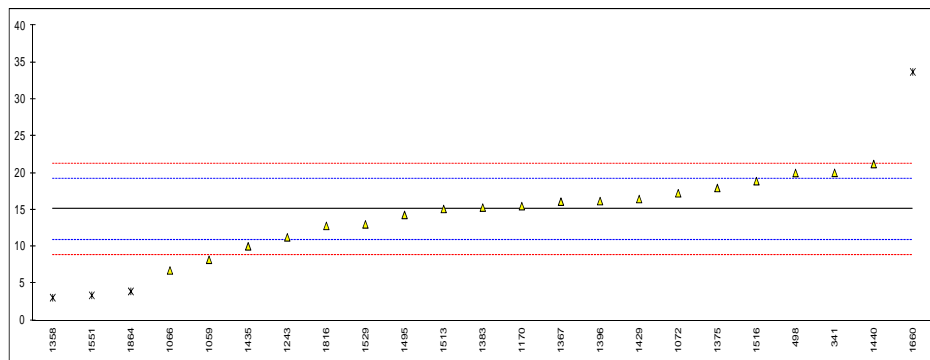
Total PCB by summation of the six congeners on sample #12147; results in mg/kg.

lab	method	sum of 6	mark	z(target)	5 x (sum of 6)	mark	z(target)	remarks
341		----		----	----		----	
343		----		----	----		----	
398		----		----	----		----	
445		----		----	----		----	
498	calc by iis	3.92		----	19.6		0.35	
614		----		----	----		----	
902		----		----	----		----	
1059	calc by iis	3.22		----	16.1		-0.86	
1066	calc by iis	4.7025		----	23.5125		1.71	
1072	calc by iis	5.14		----	25.70		2.47	
1126	calc by iis	3.104		----	15.52		-1.06	
1170	calc by iis	3.097		----	15.485		-1.08	
1201		----		----	----		----	
1243	calc by iis	2.25		----	11.25		-2.55	
1303		----		----	----		----	
1304		----		----	----		----	
1306		----		----	----		----	
1338		----		----	----		----	
1352		----		----	----		----	
1358		----		----	----		----	
1367		----		----	----		----	
1375		----		----	----		----	
1383		----		----	----		----	
1396	calc by iis	3.23417		----	16.17085		-0.84	
1429	calc by iis	3.374		----	16.87		-0.60	
1435	calc by iis	2.61		----	13.050		-1.92	
1440	calc by iis	4.69		----	23.45		1.69	
1458		----		----	----		----	
1463		----		----	----		----	
1479		----		----	----		----	
1495		2.86		----	14.30		-1.49	
1513	calc by iis	4.2		----	21.0		0.84	
1516	calc by iis	4.75		----	23.75		1.79	
1526		----		----	----		----	
1529	calc by iis	3.23		----	16.15		-0.85	
1551	calc by iis	3.35		----	16.75		-0.64	
1555		----		----	----		----	
1568		----		----	----		----	
1660	calc by iis	9.13	G(0.01)	----	45.65	G(0.01)	9.39	
1704		----		----	----		----	
1801		----		----	----		----	
1816		4.51		----	22.55		1.38	
1864	calc by iis	3.16		----	15.80		-0.97	
2160		----		----	----		----	
2237	calc by iis	5.062		----	25.31		2.33	
3195	calc by iis	3.88		----	19.4		0.28	
	normality	not OK			not OK			
	n	20			20			
	outliers	1			1			
	mean (n)	3.7172			18.5859			
	st.dev. (n)	0.85535			4.27676			
	R(calc.)	2.3950			11.9749			
	R(EN12766-2:01)	n.a			8.0670			



Determination of Total PCB on sample #12147; results in mg/kg.

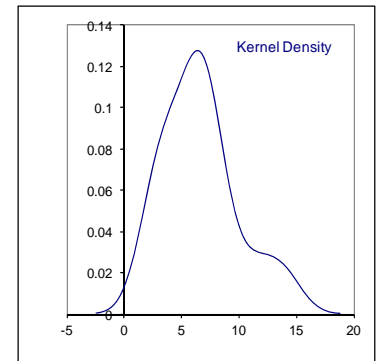
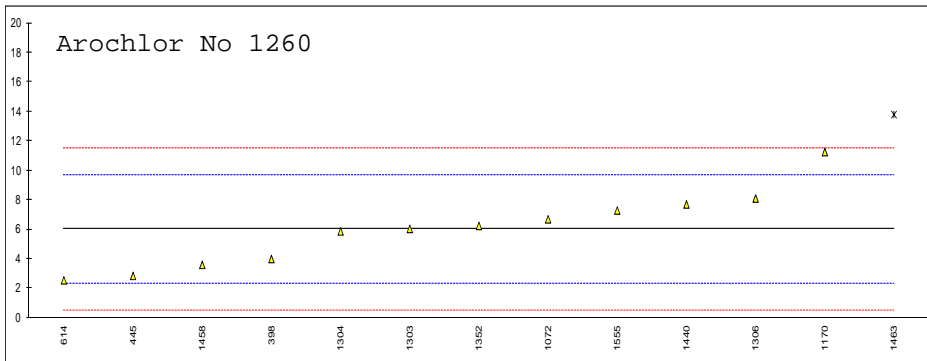
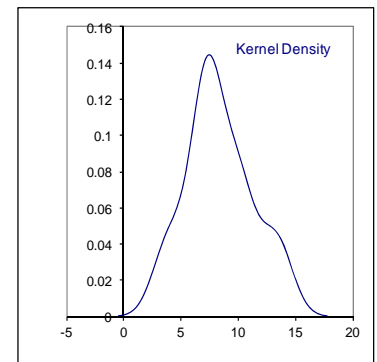
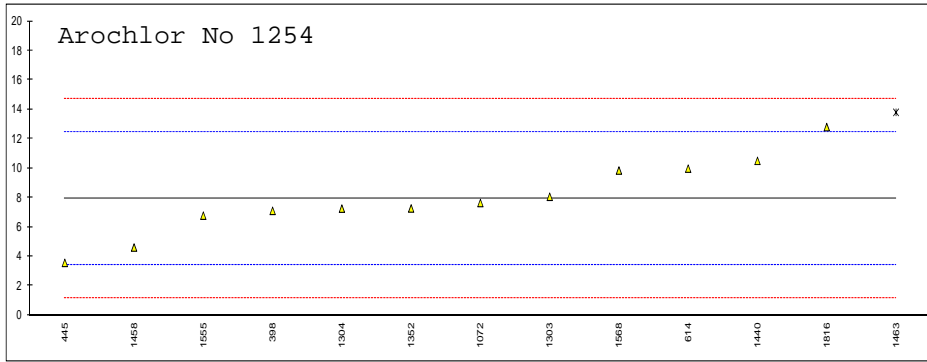
lab	method	value	mark	z(targ)	remarks
341	IEC61619	20		2.40	
343		----		----	
398		----		----	
445		----		----	
498	IEC61619B	19.98		2.39	
614		----		----	
902		----		----	
1059	EN12766A	8.2		-3.33	
1066	IEC61619	6.75		-4.04	
1072	EN61619	17.2353		1.06	
1126		----		----	
1170	EN12766B	15.49		0.21	
1201		----		----	
1243	IEC61619B	11.25		-1.85	
1303		----		----	
1304		----		----	
1306		----		----	
1338		----		----	
1352		----		----	
1358	IP462	3.037382	ex	-5.84	result excluded, see § 4.1
1367	IEC61619	16.1		0.50	
1375	IEC61619	17.95		1.40	
1383	IP462B	15.29		0.11	
1396	IP462B	16.17085		0.54	
1429	EN12766B	16.45		0.67	
1435	IEC61619	10.02		-2.45	
1440	IEC61619A	21.2		2.98	
1458		----		----	
1463		----		----	
1479		----		----	
1495	IEC61619	14.29		-0.37	
1513	IEC61619A	15.1		0.02	
1516	IEC61619A	18.88		1.85	
1526		----		----	
1529	EN61619	13		-1.00	
1551	IP426	3.36	ex	-5.68	result excluded, see § 4.1
1555		----		----	
1568		----		----	
1660	IEC61619	33.72	G(0.05)	9.06	
1704		----		----	
1801		----		----	
1816	IEC61619	12.8		-1.10	
1864	IEC61619A	3.9	G(0.05)	-5.42	first reported: 3.77
2160		----		----	
2237		----		----	
3195		----		----	
normality		OK			
n		19			
outliers		2			
mean (n)		15.061			
st.dev. (n)		3.9753			
R(calc.)		11.131			
R(IEC61619:97)		5.765			



Determination of Aroclor 1242, 1254 and 1260 on sample #12147; results in mg/kg.

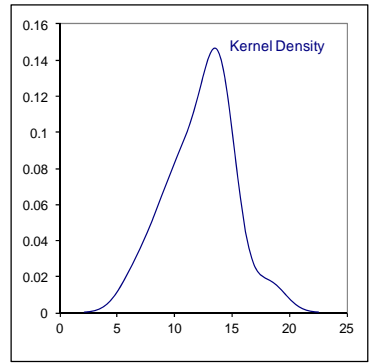
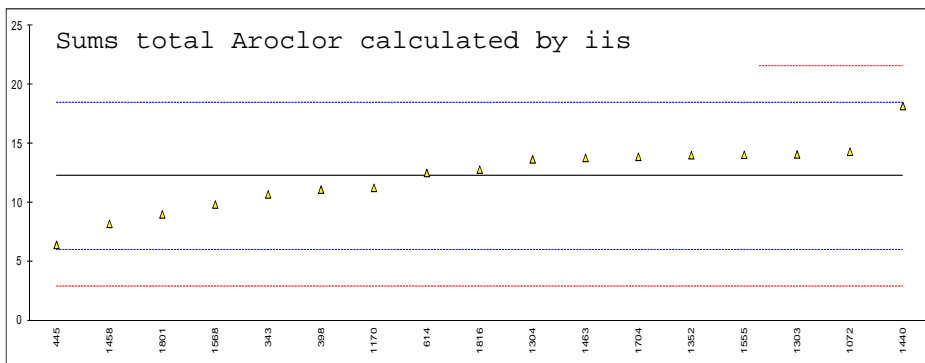
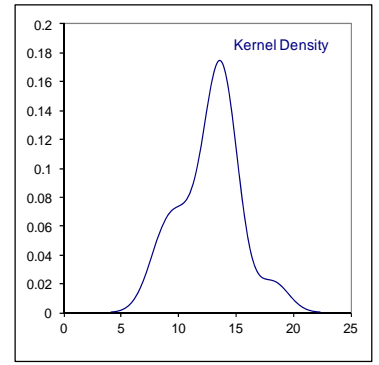
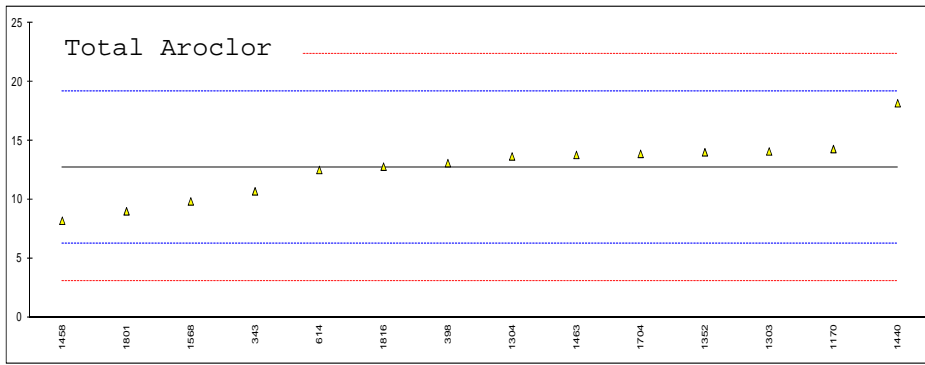
lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)	Remarks
341		----		----	----		----	----		----	
343		----		----	----		----	----		----	
398	D4059	<2		----	7.1		-0.37	4.0		-1.10	
445	IEC61619	<2		----	3.56		-1.94	2.85		-1.72	
498		----		----	----		----	----		----	
614	D4059	<2		----	9.97		0.89	2.55		-1.89	
902		----		----	----		----	----		----	
1059		----		----	----		----	----		----	
1066		----		----	----		----	----		----	
1072	D4059	2.92		----	7.63		-0.14	6.69		0.37	
1126		----		----	----		----	----		----	
1170	D4059Mod	----		----	----		----	11.25	C	2.85	
1201		----		----	----		----	----		----	
1243		----		----	----		----	----		----	
1303	in house	n.d.		----	8.058		0.05	6.041		0.01	
1304	INH-127	0.55		----	7.25		-0.31	5.87		-0.08	
1306		----		----	----		----	8.105958		1.14	
1338		----		----	----		----	----		----	
1352	INH-1767/IEC61619	0.524		----	7.259		-0.30	6.246		0.13	
1358		----		----	----		----	----		----	
1367		----		----	----		----	----		----	
1375		----		----	----		----	----		----	
1383		----		----	----		----	----		----	
1396		----		----	----		----	----		----	
1429		----		----	----		----	----		----	
1435		----		----	----		----	----		----	
1440	in house	<2		----	10.5		1.13	7.7		0.92	
1458	D4059	0	ex (1)	----	4.6		-1.48	3.6		-1.31	
1463		----		----	13.8	ex (2)	2.59	13.8	ex (2)	4.23	
1479		----		----	----		----	----		----	
1495		----		----	----		----	----		----	
1513		----		----	----		----	----		----	
1516		----		----	----		----	----		----	
1526		----		----	----		----	<5		----	
1529		----		----	----		----	----		----	
1551		----		----	----		----	----		----	
1555		----		----	6.77		-0.52	7.29		0.69	
1568	D4059	n.d.		----	9.840		0.84	n.d.		----	
1660		----		----	----		----	----		----	
1704		----		----	----		----	----		----	
1801		----		----	----		----	----		----	
1816		----		----	12.8		2.14	----		----	
1864		----		----	----		----	----		----	
2160		----		----	----		----	----		----	
2237		----		----	----		----	----		----	
3195		----		----	----		----	----		----	
	normality	not OK			OK			OK			
	n	3			12			12			
	outliers	0			0			0			
	mean (n)	1.331			7.945			6.016			
	st.dev. (n)	1.3759			2.5472			2.4989			
	R(calc.)	3.852			7.132			6.997			
	R(D4059:10)	(1.661)			6.341			5.147			

C = corrected result, first reported result lab 1170: Aroclor 1260: 13.98  
 ex (1) = result excluded, zero is not a real value (lab 1458)  
 ex (2) = result excluded, see §4.1 (lab 1463)



Determination of the Total Aroclor on sample #12147; results in mg/kg.

lab	method	value	mark	z(targ)	calculation by iis	mark	z(targ)	remarks
341		----		----	----		----	
343	EN61619	10.7		-0.63	10.7		----	
398	D4059	13.1		0.12	11.1		----	
445		----		----	6.41		----	
498		----		----	----		----	
614	D4059	12.52		-0.06	12.52		----	
902		----		----	----		----	
1059		----		----	----		----	
1066		----		----	----		----	
1072		----		----	17.24		----	
1126		----		----	----		----	
1170	D4059Mod.	14.3		0.49	11.25		----	
1201		----		----	----		----	
1243		----		----	----		----	
1303	in house	14.099		0.43	14.099		----	
1304	INH-127	13.67		0.29	13.67		----	
1306		----		----	----		----	
1338		----		----	----		----	
1352	D4059	14.029		0.40	14.029		----	
1358		----		----	----		----	
1367		----		----	----		----	
1375		----		----	----		----	
1383		----		----	----		----	
1396		----		----	----		----	
1429		----		----	----		----	
1435		----		----	----		----	
1440	in house	18.2		1.70	18.2		----	
1458	D4059	8.2		-1.40	8.2		----	
1463	D4059	13.8		0.33	13.8		----	
1479		----		----	----		----	
1495		----		----	----		----	
1513		----		----	----		----	
1516		----		----	----		----	
1526		----		----	<5		----	
1529		----		----	----		----	
1551		----		----	----		----	
1555		----		----	14.06		----	
1568	D4059	9.840		-0.89	9.84		----	
1660		----		----	----		----	
1704	IEC61619	13.8922		0.36	13.8922		----	
1801	IEC61619	9.00		-1.16	9.00		----	
1816	IEC61619	12.8		0.02	12.8		----	
1864		----		----	----		----	
2160		----		----	----		----	
2237		----		----	----		----	
3195		----		----	----		----	
	normality	OK			OK			
	n	14			17			
	outliers	0			0			
	mean (n)	12.725			12.4006			
	st.dev. (n)	2.5783			3.05032			
	R(calc.)	7.219			8.5409			
	R(D4059:10)	9.028			(8.9559)			



## **APPENDIX 2**

### **Number of participating laboratories per country**

5 labs in AUSTRALIA  
1 lab in BELGIUM  
1 lab in CANADA  
2 labs in FRANCE  
3 labs in GERMANY  
1 lab in INDIA  
1 lab in IRELAND  
3 labs in ITALY  
1 lab in MEXICO  
1 lab in NORWAY  
2 labs in PORTUGAL  
2 labs in SLOVENIA  
1 lab in SOUTH AFRICA  
8 labs in SPAIN  
4 labs in THE NETHERLANDS  
3 labs in TURKEY  
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
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

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