Results of Proficiency Test
PCB in (Mineral) Oil
November 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing L. Dijkstra
Corrector: dr. R. Visser & ing. R.J. Starink

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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 2011/2012, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.
In this interlaboratory study, 42 laboratories from 19 different countries have participated, but not all laboratories reported results for all evaluated components.
See appendix 2 for the number of participating laboratories per country. In this report the results of the proficiency test 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. In this study it was decided to send one sample of waste (mineral) oil contaminated with PCB that was donated by one of the participating laboratories.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO17043:2010. This ensures 100% confidentially of participant's data. Also 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 present 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 heavily contaminated waste oil (positive on PCBs and containing also other chlorinated components) was donated by a third party laboratory.
After ultrasonic homogenisation 60, subsamples were transferred to 8 mL amber glass vials, all labelled #11106.

The homogeneity of the subsamples #11106 was checked by determination of the organic chloride content in accordance with UOP779-08 on eight stratified randomly selected samples:

	Organic chloride in mg/L
sample #11106-1	51.7
sample #11106-2	51.8
sample #11106-3	51.9
sample #11106-4	51.9
sample #11106-5	52.2
sample #11106-6	51.6
sample #11106-7	51.7
sample #11106-8	51.0

Table 1: homogeneity test results of subsample #11106

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:

	#11106
r (samples)	1.0
reference method	UOP779:08
0.3 x $R_{(reference\ method)}$	2.6

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 #11106) was sent on October 26, 2011.

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 Extractable Organo halogenic Compounds (EOX) and Poly Chlorinated Biphenyls (via seven individual PCBs, via the determination the total PCB content and via Aroclors) on the sample.
To get comparable results a detailed report form, on which the units were prescribed, was sent together with each sample. Also a letter of instructions and a SDS were added to the 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.

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

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.

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; nr.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:

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

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:

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

4 EVALUATION

In this proficiency test some problems were encountered during execution. In total seven 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 38 participating laboratories reported 195 numerical results. Observed were 4 outlying results, which is 2.0% 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.
Not all original data sets proved to have a normal distribution. For PCB 118 and Total PCB a not normal distribution was observed and therefore the statistical evaluations of these two sets of test results should be used with due care.
None of the laboratories reported test results for EOX .

For the results on sample #11106, the following was concluded:

Individual PCBs: This determination was problematic for three congeners.
For the evaluation of the individual congeners method EN12766-1:99 was used. In the methods IEC61619:97 and DIN51527:93 only the reproducibilities of the total PCB content are mentioned, while in EN12766-1:99 the reproducibilities for each individual congener are mentioned.
In total two statistical outliers were observed. The calculated reproducibilities for PCB 28, PCB 118 and PCB 138, after rejection of the statistical outliers are not in agreement with the requirements of EN12766-1:99. However, the calculated reproducibilities for PCB 52, PCB 101, PCB 153 and PCB 180 are in full agreement with the reproducibilities mentioned in EN12766-1:99.

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.

Indiv. Aroclors: The determination of the individual Aroclors was rather problematic. The majority of the laboratories agreed that Aroclor 1260 was the main component in sample #11106. One laboratory did report Arochlor 1262 in stead. This result was placed under Aroclor 1260 for evaluation. Nine laboratories reported also the presence of a small amount of Aroclor 1242. The presence and concentration of Aroclor 1254 is quite uncertain

as only four laboratories reported a positive result, six others reported a result near or below the detection limit and another six laboratories did not report this Aroclor at all.
No statistical outliers were observed for Arochlors in total. The calculated reproducibilities for Aroclor 1242 and Aroclor 1260 are respectively in agreement and not in agreement with the requirements of ASTMD4059:05e1.

Total Aroclor: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTMD4059:05e1.

Summary: All participants agreed that sample #11106 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 19.6 mg/kg. From this sum, a total concentration of 98.1 mg PCB/kg was estimated acc. to EN12766-B. ($PCB_{Total} = 5 * \sum_{(n=6)}$congeners).
For the determination of the total Aroclors an average of 81.0 mg PCB/kg was found.
From the homogeneity data on organic chloride (OX) an average concentration of 51.7 mg was calculated. From this concentration a total content of 82.1 mg PCB/kg was estimated using an average Cl content of 63% for Aroclor 1260. This content is in good agreement with the estimated total PCB content using the other methods.

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

	#11106
total PCB content, estimated from 6 congeners, in mg/kg	98.1
total PCB content, using IEC 61619:97 method, in mg/kg	79.5
estimated total PCB content using Aroclor method, in mg/kg	81.0
total PCB content, estimated for OX homogeneity data, in mg/kg	82.1

Table 3: Comparison of estimations of total PCB content in sample #11106.

The total PCB content as determined by IEC61619:97 is in good agreement with the total PCB content as determined by the Aroclor method. However, only five test results for total PCB content appeared to be calculated cfr EN12766 (5 times summation of 6 congeners). And four of these five test results were the highest of all reported test results. This may indicate that the EN12766 calculation method may overestimate the total PCB content in some cases.

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)
PCB no. 28	mg/kg	19	0.32	0.17	0.14
PCB no. 52	mg/kg	18	0.54	0.22	0.26
PCB no. 101	mg/kg	19	1.97	0.68	0.96
PCB no. 118	mg/kg	15	0.68	0.37	0.32
PCB no. 138	mg/kg	19	4.43	2.40	2.18
PCB no. 153	mg/kg	19	5.45	2.26	2.69
PCB no. 180	mg/kg	18	6.81	2.92	3.36
sum of 6 individual PCBs	mg/kg	18	19.6	6.57	n.a.
Total PCB	mg/kg	22	79.5	46.3	21.9
Aroclor 1242	mg/kg	9	6.21	5.01	5.27
Aroclor 1254	mg/kg	5	14.4	n.e	n.e
Aroclor 1260	mg/kg	15	70.0	40.3	32.4
Total Aroclor	mg/kg	10	81.0	30.2	36.2

table 4: Performance of the group of participating laboratories on sample #11106

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 NOVEMBER 2011 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2011	*November 2010*	*November 2009*	*November 2008*
Number of reporting labs	38	34	29	28
Number of results reported	195	186	329	197
Statistical outliers	4	15	8	8
Percentage outliers	2.0%	8.1%	3.6%	4.1%

Table 6: 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 2011*	*November 2010*	*November 2009*	*November 2008*
EOX	n.e	n.e.	n.e.	n.e.
PCB (all)	+/-	-	--	+/-
Aroclor (all)	+/-	--	+/-	+

Table 7: comparison of observed precisions against standard requirements

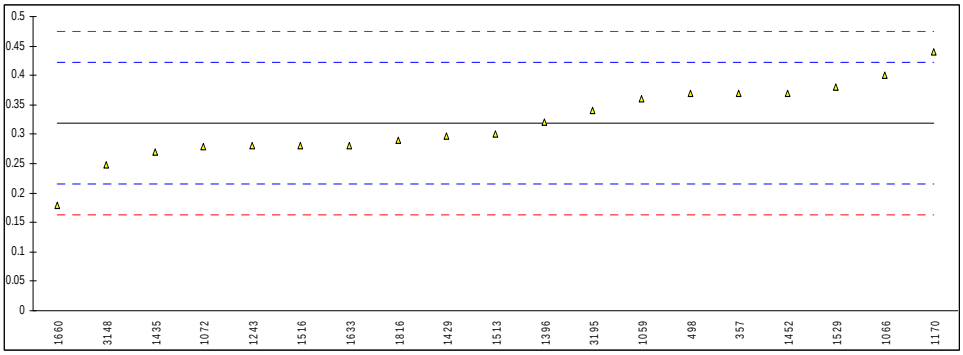
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

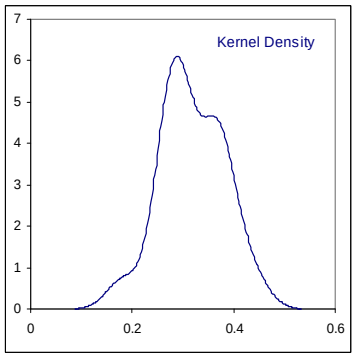
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

APPENDIX 1

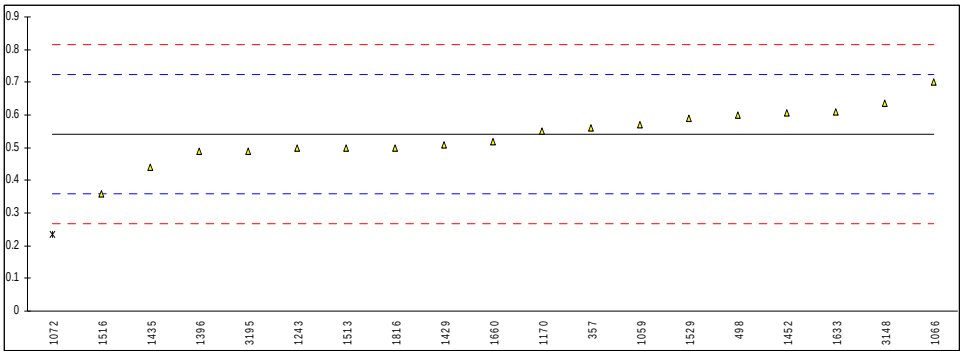
Determination of PCB 28, 52 and 101 on sample #11106; results in mg/kg.

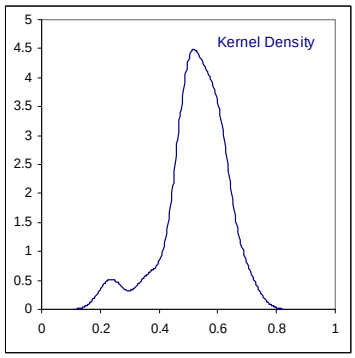
lab	method	No. 28	mark	z(targ)	No. 52	mark	z(targ)	No. 101	mark	z(targ)	Remarks
341		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
357	EN12766Mod-B	0.37		0.99	0.56		0.21	1.92		-0.16	
445		-----		-----	-----		-----	-----		-----	
498	EN12766	0.37		0.99	0.60		0.65	2.21		0.69	
614		-----		-----	-----		-----	-----		-----	
902		-----		-----	-----		-----	-----		-----	
1059	EN12766-A	0.36		0.80	0.57		0.32	2.10		0.37	
1066	EN12766	0.4		1.57	0.7		1.75	2.0		0.08	
1072	EN61619	0.2787		-0.77	0.2356	G(0.05)	-3.35	1.5282		-1.29	
1126		-----		-----	-----		-----	-----		-----	
1170	EN12766Mod.	0.44		2.34	0.55		0.10	2.04		0.19	
1243	EN12766-B	0.28		-0.75	0.50		-0.45	1.52		-1.32	
1245		-----		-----	-----		-----	-----		-----	
1303		-----		-----	-----		-----	-----		-----	
1304		-----		-----	-----		-----	-----		-----	
1306		-----		-----	-----		-----	-----		-----	
1338		-----		-----	-----		-----	-----		-----	
1352		-----		-----	-----		-----	-----		-----	
1358		-----		-----	-----		-----	-----		-----	
1367		-----		-----	-----		-----	-----		-----	
1375		-----		-----	-----		-----	-----		-----	
1383		-----		-----	-----		-----	-----		-----	
1396	IP462-B	0.32		0.03	0.49		-0.56	2.0		0.08	
1429	EN12766-B	0.297		-0.42	0.508		-0.36	1.685		-0.84	
1435	EN12766-A	0.27		-0.94	0.44		-1.10	1.99		0.05	
1452	EN12766-B	0.370		0.99	0.607		0.73	2.183		0.61	
1458		-----		-----	-----		-----	-----		-----	
1463		-----		-----	-----		-----	-----		-----	
1479		-----		-----	-----		-----	-----		-----	
1513	IEC61619-A	0.3		-0.36	0.5		-0.45	2.4		1.24	
1516	IEC61619-A	0.28		-0.75	0.36		-1.98	2.27		0.86	
1526		-----		-----	-----		-----	-----		-----	
1529	EN12766	0.38		1.18	0.59		0.54	1.77		-0.59	
1633	EN12766	0.28		-0.75	0.61		0.76	2.26		0.83	
1660	EN12766-A	0.18		-2.68	0.52		-0.23	1.75		-0.65	
1704		-----		-----	-----		-----	-----		-----	
1801		-----		-----	-----		-----	-----		-----	
1816	IEC61619	0.29		-0.55	0.50		-0.45	2.04		0.19	
2122		-----		-----	-----		-----	-----		-----	
3148	EN15318	0.248	C	-1.36	0.635		1.04	1.840		-0.39	first reported:0.495
3195	EN12766	0.34		0.41	0.49		-0.56	1.99		0.05	
	normality	OK			OK			OK			
	n	19			18			19			
	outliers	0			1			0			
	mean (n)	0.319			0.540			1.973			
	st.dev. (n)	0.0621			0.0786			0.2447			
	R(calc.)	0.174			0.220			0.685			
	R(EN12766-1:99)	0.145			0.255			0.965			





PCB 28





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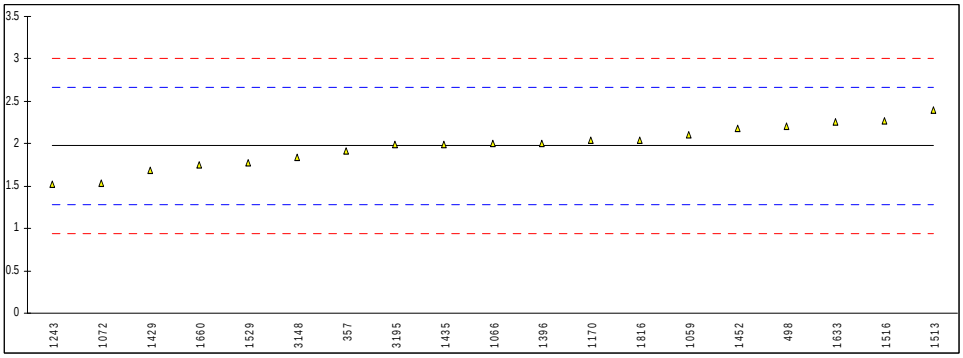
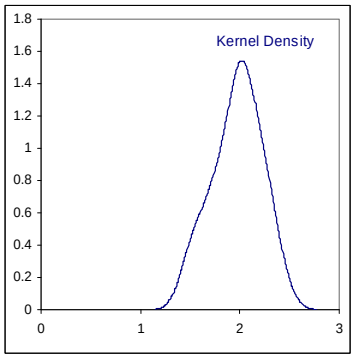
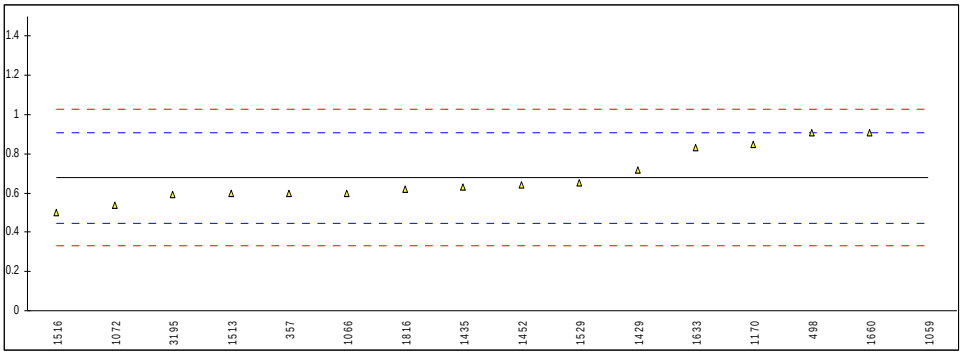


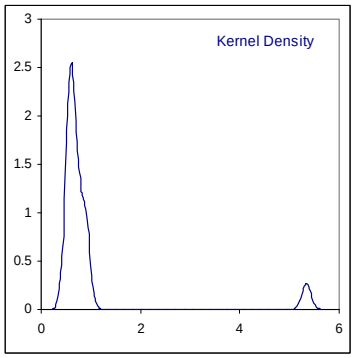


PCB 101

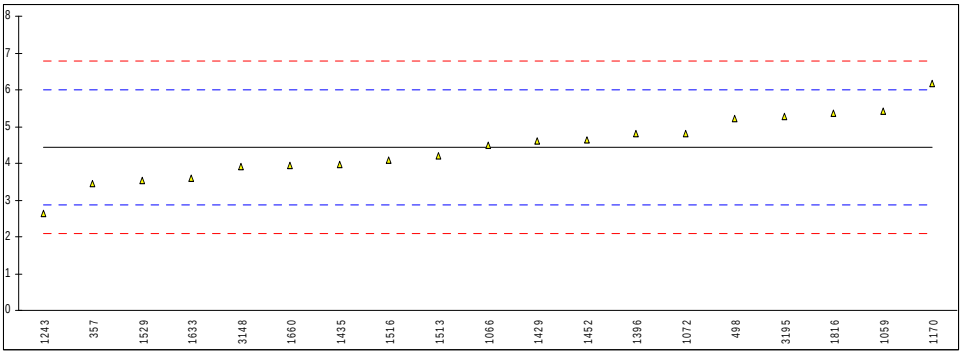
Determination of PCB 118 and 138 on sample #11106; results in mg/kg.

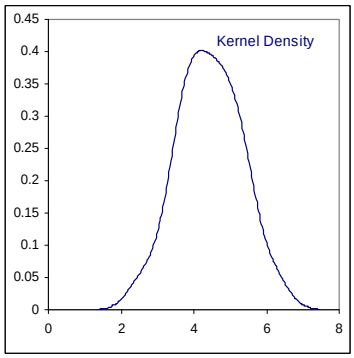
lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766Mod-B	0.60		-0.68	3.46		-1.25	
445		-----		-----	-----		-----	
498	EN12766	0.91		2.00	5.23		1.02	
614		-----		-----	-----		-----	
902		-----		-----	-----		-----	
1059	EN12766-A	5.34	G(0.01)	40.33	5.42		1.27	
1066	EN12766	0.6		-0.68	4.5		0.09	
1072	EN61619	0.5365		-1.23	4.8170		0.49	
1126		-----		-----	-----		-----	
1170	EN12766Mod.	0.85		1.48	6.18	C	2.24	first reported: 5.64
1243	EN12766-B	-----		-----	2.64	C	-2.30	first reported: 4.70
1245		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1375		-----		-----	-----		-----	
1383		-----		-----	-----		-----	
1396	IP462-B	-----		-----	4.81		0.49	
1429	EN12766-B	0.717		0.33	4.618		0.24	
1435	EN12766-A	0.63		-0.42	3.98		-0.58	
1452	EN12766-B	0.640		-0.34	4.647		0.28	
1458		-----		-----	-----		-----	
1463		-----		-----	-----		-----	
1479		-----		-----	-----		-----	
1513	IEC61619-A	0.6		-0.68	4.2		-0.30	
1516	IEC61619-A	0.50		-1.55	4.10		-0.42	
1526		-----		-----	-----		-----	
1529	EN12766	0.65		-0.25	3.54		-1.14	
1633	EN12766	0.83		1.31	3.58	C	-1.09	first reported: 3.10
1660	EN12766-A	0.91		2.00	3.93		-0.64	
1704		-----		-----	-----		-----	
1801		-----		-----	-----		-----	
1816	IEC61619	0.62		-0.51	5.35		1.18	
2122		-----		-----	-----		-----	
3148	EN15318	-----		-----	3.910		-0.67	
3195	EN12766	0.59		-0.77	5.28		1.09	
	normality	not OK			OK			
	n	15			19			
	outliers	1			0			
	mean (n)	0.679			4.431			
	st.dev. (n)	0.1329			0.8556			
	R(calc.)	0.372			2.396			
	R(EN12766-1:99)	0.324			2.183			





PCB 118

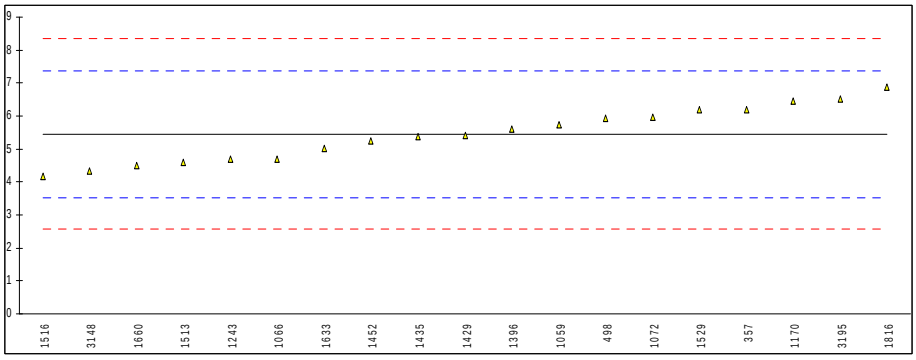


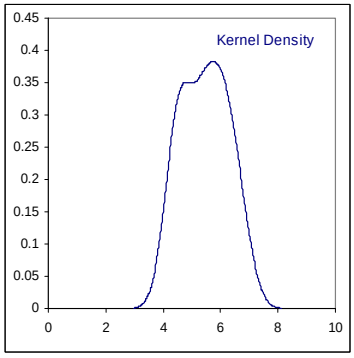


PCB 138

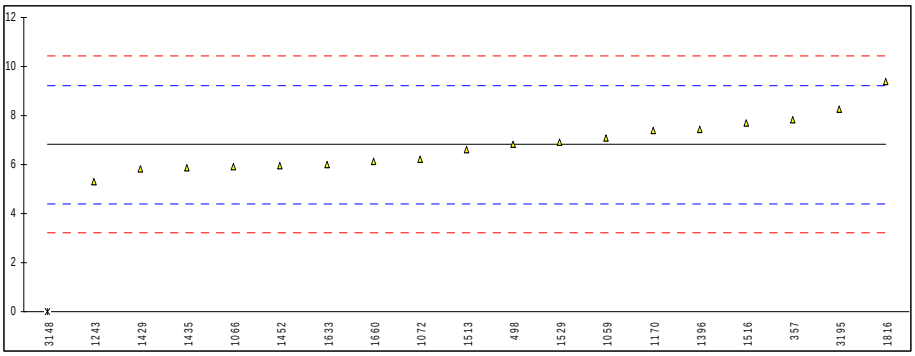
Determination of PCB 153 and 180 on sample #11106; results in mg/kg.

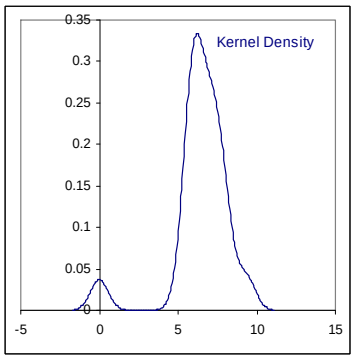
lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766Mod-B	6.20		0.78	7.82		0.84	
445		-----		-----	-----		-----	
498	EN12766	5.92		0.48	6.84		0.02	
614		-----		-----	-----		-----	
902		-----		-----	-----		-----	
1059	EN12766-A	5.73		0.29	7.10		0.24	
1066	EN12766	4.7		-0.79	5.90		-0.76	
1072	EN61619	5.9564		0.52	6.2110		-0.50	
1126		-----		-----	-----		-----	
1170	EN12766Mod.	6.47		1.06	7.38		0.47	
1243	EN12766-B	4.70	C	-0.79	5.3		-1.26	first reported: 2.64
1245		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1338		-----		-----	-----		-----	
1352		-----		-----	-----		-----	
1358		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1375		-----		-----	-----		-----	
1383		-----		-----	-----		-----	
1396	IP462-B	5.60		0.15	7.42		0.51	
1429	EN12766-B	5.42	C	-0.04	5.837		-0.81	first reported: 0.854
1435	EN12766-A	5.39		-0.07	5.89		-0.77	
1452	EN12766-B	5.265		-0.20	5.955		-0.71	
1458		-----		-----	-----		-----	
1463		-----		-----	-----		-----	
1479		-----		-----	-----		-----	
1513	IEC61619-A	4.6		-0.89	6.6		-0.18	
1516	IEC61619-A	4.19		-1.32	7.69		0.73	
1526		-----		-----	-----		-----	
1529	EN12766	6.19		0.77	6.9		0.07	
1633	EN12766	5.02		-0.45	5.98		-0.69	
1660	EN12766-A	4.51		-0.98	6.15		-0.55	
1704		-----		-----	-----		-----	
1801		-----		-----	-----		-----	
1816	IEC61619	6.89		1.49	9.38		2.14	
2122		-----		-----	-----		-----	
3148	EN15318	4.350		-1.15	0	ex, C	-5.67	first reported: 4.400; zero is not real value
3195	EN12766	6.53		1.12	8.28		1.22	
	normality	OK			OK			
	n	19			18			
	outliers	0			0			
	mean (n)	5.454			6.813			
	st.dev. (n)	0.8069			1.0434			
	R(calc.)	2.259			2.922			
	R(EN12766-1:99)	2.690			3.364			





PCB 153

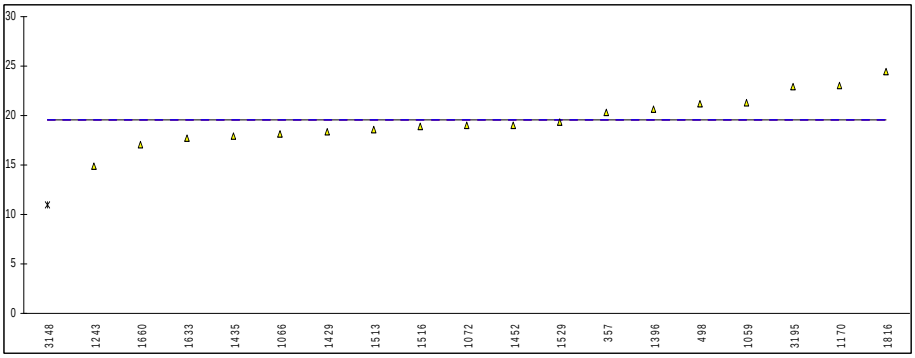


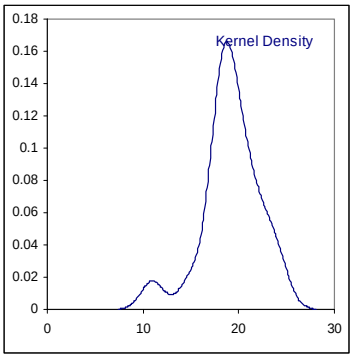


PCB 180

Summation of the 6 congeners 28, 52, 101, 138, 153 & 180 on sample #11106; results in mg/kg.

lab	method	sum of 6	5 x sum	mark	z(targ)	remarks
341		-----	------			
343		-----	------			
357	calc by iis	20.33	101.65			
445		-----	------			
498	calc by iis	21.17	105.85			
614		-----	------			
902		-----	------			
1059	calc by iis	21.28	106.4			
1066	calc by iis	18.2	91.0			
1072	calc by iis	19.03	95.1345			
1126		-----				
1170	calc by iis	23.06	115.3			
1243	calc by iis	14.94	74.7			
1245		-----	-----			
1303		-----	-----			
1304		-----	-----			
1306		-----	-----			
1338		-----	-----			
1352		-----	-----			
1358		-----	-----			
1367		-----	-----			
1375		-----	-----			
1383		-----	-----			
1396	calc by iis	20.64	103.20			
1429	calc by iis	18.37	91.825			
1435	calc by iis	17.96	89.80			
1452	calc by iis	19.03	95.135			
1458		-----	-----			
1463		-----	-----			
1479		-----	-----			
1513	calc by iis	18.6	93.0			
1516	calc by iis	18.89	94.45			
1526		-----	-----			
1529	calc by iis	19.37	96.85			
1633	calc by iis	17.73	88.65			
1660	calc by iis	17.04	85.20			
1704		-----	-----			
1801		-----	-----			
1816	calc by iis	24.45	122.25			
2122		-----	-----			
3148	calc by iis	10.980	54.915	G(0.05)		
3195	calc by iis	22.91	114.55			
	normality	OK	OK			
	n	18	18			
	outliers	1	1			
	mean (n)	19.611	98.053			
	st.dev. (n)	2.3453	11.7263			
	R(calc.)	6.567	32.834			
	R(IEC16169:97)	n.a.	26.513			

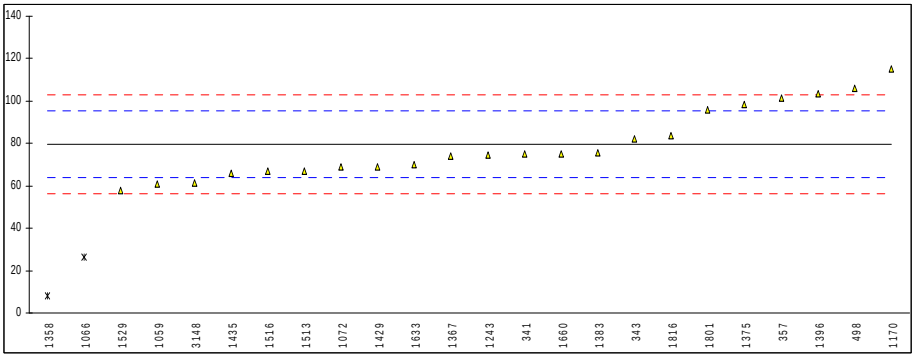


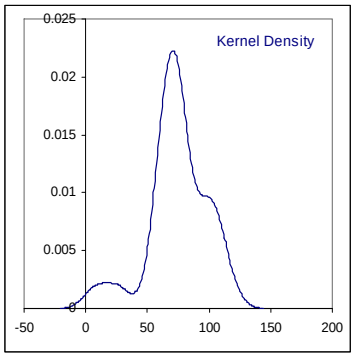


Determination of Total PCB on sample #11106; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341	EN61619	75.0		-0.58	
343	EN61619	82.2		0.34	
357	EN12766-B	101.6		2.83	
445		-----		-----	
498	EN12766B	106		3.39	
614		-----		-----	
902		-----		-----	
1059	EN12766-A	61.06		-2.36	
1066	IEC61619	26.3	DG(0.05)	-6.81	
1072	EN61619	68.9429		-1.35	
1126		-----		-----	
1170	EN12766Mod.	115.3	C	4.58	first reported: 112.6
1243	IEC61619	74.7	C	-0.62	first reported: 14.94
1245		-----		-----	
1303		-----		-----	
1304		-----		-----	
1306		-----		-----	
1338		-----		-----	
1352		-----		-----	
1358	IP462	8.1	DG(0.05)	-9.14	
1367	IEC61619	74.19		-0.68	
1375	IEC61619	98.6		2.44	
1383	IP462	75.75		-0.48	
1396	IP462	103.23		3.04	
1429	EN12766-B	68.995		-1.35	
1435	IEC61619	65.71		-1.77	
1452		-----		-----	
1458		-----		-----	
1463		-----		-----	
1479		-----		-----	
1513	IEC61619-A	67.0		-1.60	
1516	IEC61619-A	66.79		-1.63	
1526		-----		-----	
1529	EN61619	58		-2.75	
1633	IEC61619	70.2		-1.19	
1660	IEC61619	75.19		-0.55	
1704		-----		-----	
1801	IEC61619	95.8		2.08	
1816	IEC61619	83.7		0.54	
2122		-----		-----	
3148	EN15318	61.300		-2.33	
3195		-----		-----	

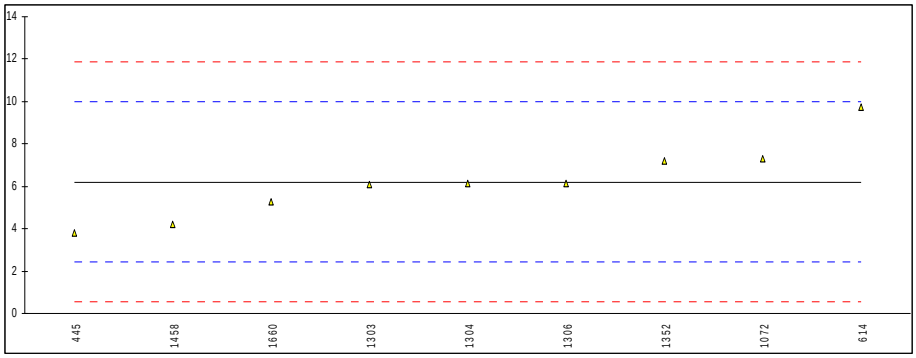
normality	not OK
n	22
outliers	2
mean (n)	79.512
st.dev. (n)	16.5396
R(calc.)	46.311
R(IEC61619:97)	21.878

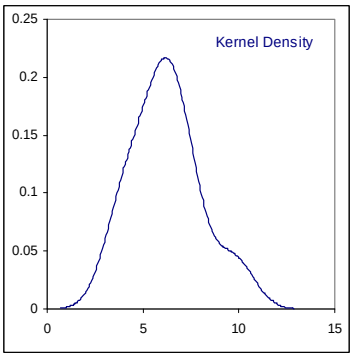




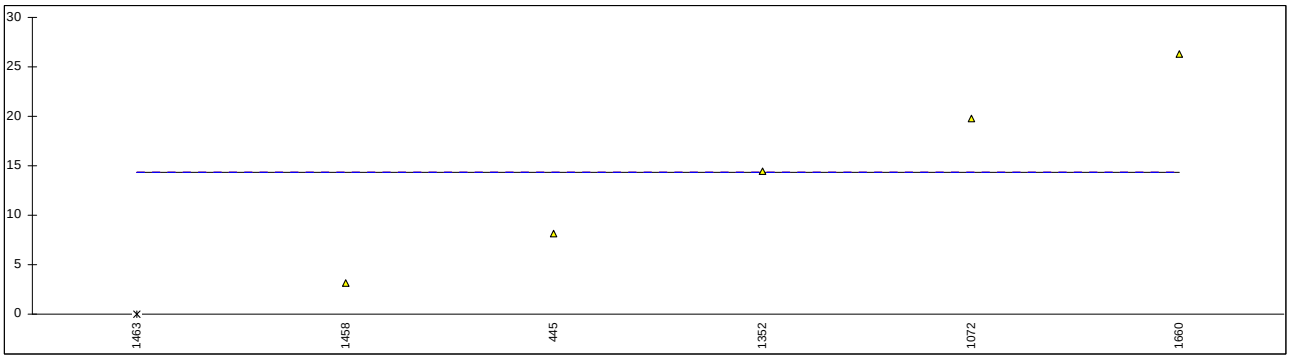
Determination of Aroclor 1242, 1254 and 1260 on sample #11106; results in mg/kg.

lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)	Remarks
341		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
357		-----		-----	-----		-----	-----		-----	
445	IEC61619	3.8		-1.28	8.2		-----	57.2		-1.11	
498		-----		-----	-----		-----	-----		-----	
614	D4059	9.76		1.88	<2		-----	62.46		-0.65	
902		-----		-----	-----		-----	-----		-----	
1059		-----		-----	-----		-----	-----		-----	
1066		-----		-----	-----		-----	-----		-----	
1072	D4059	7.3		0.58	19.8		-----	41.7		-2.45	
1126		-----		-----	-----		-----	-----		-----	
1170	D4059Mod	-----		-----	-----		-----	90.0		1.72	
1243		-----		-----	-----		-----	-----		-----	
1245	in house	-----		-----	-----		-----	26.4	ex	-3.77	Aroclor1262, see $4.1
1303	INH-0421	6.1		-0.06	n.d.		-----	67.6		-0.21	
1304	INH-127	6.12		-0.05	<0.50		-----	82.08		1.04	
1306	EPA600	6.1458		-0.04	-----		-----	74.4743		0.38	
1338	in house	-----		-----	-----		-----	69.3	C	-0.06	first reported:40.3
1352	INH-1767	7.22		0.54	14.41		-----	80.52		0.90	
1358		-----		-----	-----		-----	-----		-----	
1367		-----		-----	-----		-----	-----		-----	
1375		-----		-----	-----		-----	-----		-----	
1383		-----		-----	-----		-----	-----		-----	
1396		-----		-----	-----		-----	-----		-----	
1429		-----		-----	-----		-----	-----		-----	
1435		-----		-----	-----		-----	-----		-----	
1452		-----		-----	-----		-----	-----		-----	
1458	D4059	4.2		-1.07	3.2		-----	64.6		-0.47	
1463	D4059	<2		<-2.24	0	ex	-----	82.9		1.11	zero is not a real value
1479		-----		-----	-----		-----	-----		-----	
1513		-----		-----	-----		-----	-----		-----	
1516		-----		-----	-----		-----	-----		-----	
1526	EPA600	-----		-----	-----		-----	70		0.00	
1529		-----		-----	-----		-----	-----		-----	
1633		-----		-----	-----		-----	-----		-----	
1660	IEC61619	5.26		-0.51	26.31		-----	43.61		-2.28	
1704		-----		-----	-----		-----	-----		-----	
1801		-----		-----	-----		-----	-----		-----	
1816	IEC61619	-----		-----	-----		-----	79.1		0.78	
2122	in house	<5		-----	<5		-----	85	C	1.29	first reported:106
3148		-----		-----	-----		-----	-----		-----	
3195		-----		-----	-----		-----	-----		-----	
	normality	OK			n.a.			OK			
	n	9			5			15			
	outliers	0			0			0			
	mean (n)	6.21			14.38			70.04			
	st.dev. (n)	1.788			9.150			14.403			
	R(calc.)	5.01			25.62			40.33			
	R(D4059:05e1)	5.27			9.90			32.44			

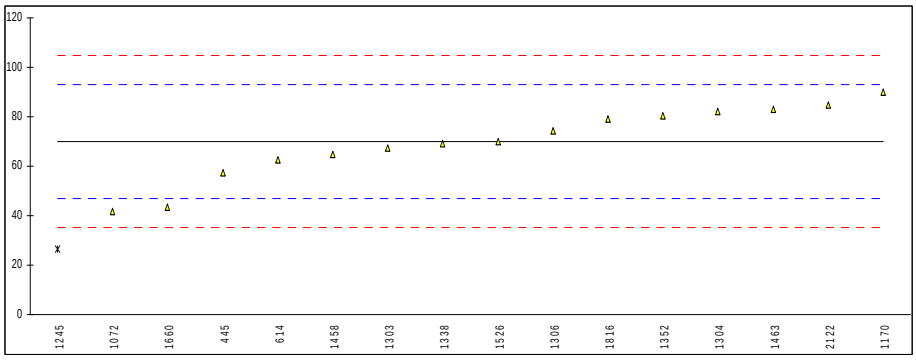


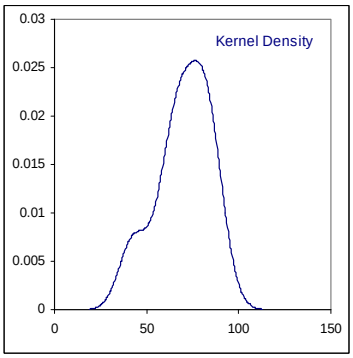


Arochlor 1242



Arochlor 1254

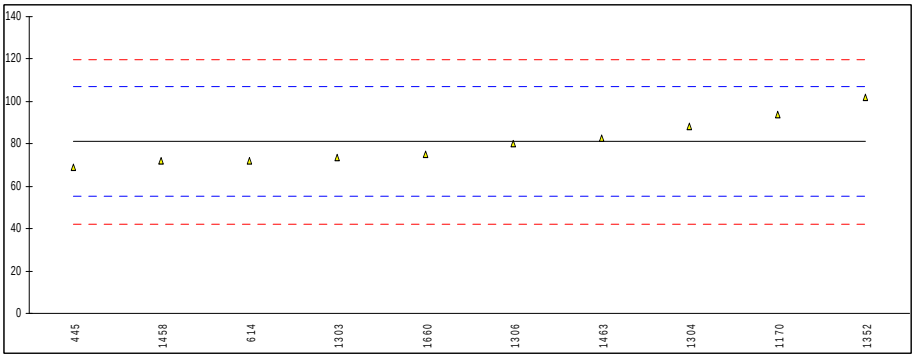


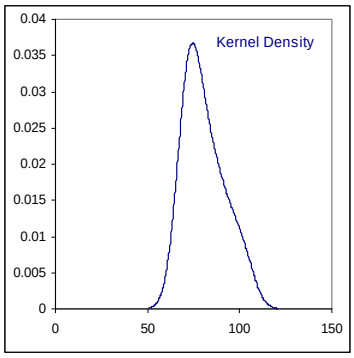


Arochlor 1260

Determination of the Total Aroclor on sample #11106; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		-----		-----	
343		-----		-----	
357		-----		-----	
445	IEC61619	69.2		-0.91	
498		-----		-----	
614	D4059	72.22		-0.68	
902		-----		-----	
1059		-----		-----	
1066		-----		-----	
1072		-----		-----	
1126		-----		-----	
1170	D4059Mod.	93.6		0.98	
1243		-----		-----	
1245		-----		-----	
1303	INH-0421	73.7		-0.56	
1304	INH-127	88.20		0.56	
1306	EPA600	80.3914		-0.04	
1338		-----		-----	
1352	INH-1767	102.15		1.64	
1358		-----		-----	
1367		-----		-----	
1375		-----		-----	
1383		-----		-----	
1396		-----		-----	
1429		-----		-----	
1435		-----		-----	
1452		-----		-----	
1458	D4059	72		-0.69	
1463	D4059	82.9		0.15	
1479		-----		-----	
1513		-----		-----	
1516		-----		-----	
1526		-----		-----	
1529		-----		-----	
1633		-----		-----	
1660	IEC61619	75.19		-0.45	
1704		-----		-----	
1801		-----		-----	
1816		-----		-----	
2122		-----		-----	
3148		-----		-----	
3195		-----		-----	
					With all available data (missing ones calculated by iis):
	normality	OK			OK
	n	10			15
	outliers	0			1
	mean (n)	80.96			78.73
	st.dev. (n)	10.782			10.055
	R(calc.)	30.19			28.15
	R(D4059:05e1)	36.16			35.42





APPENDIX 2

Number of participating laboratories per country

5 labs in AUSTRALIA
1 lab in BELGIUM
1 lab in CANADA
1 lab in FINLAND
1 lab in FRANCE
4 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in IRELAND
2 labs in ITALY
1 lab in NEW ZEALAND
1 lab in NORWAY
2 labs in PORTUGAL
2 labs in SLOVENIA
1 lab in SOUTH AFRICA
6 labs in SPAIN
3 labs in THE NETHERLANDS
1 lab 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

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, January 2010
2 prNEN 12766-2:2000.
3 ASTM E178-02
4 ASTM E1301-03
5 ISO 5725-86
6 ISO 5725, parts 1-6, 1994
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367/84
10 DIN 38402 T41/42
11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No4 January 2001
14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)