

Results of Proficiency Test
PCB in (Transformer) Oil
November 2010

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 (transformer) oil every year. During the annual proficiency testing program 2010/2011, it was decided to continue the proficiency test for the PCB analysis on (transformer) oil.

In this Interlaboratory study, 36 laboratories from 18 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 (transformer) 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% confidentiality 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 52 subsamples were transferred to 8 mL amber glass vials, all labelled #1087.

The homogeneity of the subsamples #1087 was checked by determination of the organic chloride content in accordance with UOP779-08 on seven stratified randomly selected samples:

	Organic chloride in mg/L
sample #1087-1	44
sample #1087-2	43
sample #1087-3	44
sample #1087-4	44
sample #1087-5	44
sample #1087-6	44
sample #1087-7	45

Table 1: homogeneity test results of subsample #1087

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:

	#1087
r (samples)	1.6
reference method	UOP779
$0.3 \times R_{(\text{reference method})}$	2.2

Table 2: evaluation of the observed repeatability

The repeatability of the results of homogeneity test was in full agreement with 0.3 times the reproducibility as required by UOP779. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories one vial of 8 mL (labelled #1087) was sent on October 20, 2010.

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.

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:

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

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 no problems were encountered during execution. All but two participants did report one or more test results.

In total 34 participating laboratories reported 186 numerical results. Observed were 15 outlying results, which is 8.1% of the numerical results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. 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 PCB 153 a not normal distribution was observed and therefore the statistical evaluations of these two congeners should be used with due care.

For the results on sample #1087, the following was concluded:

EOX: For this test only two test results were reported. Therefore no significant conclusion could be drawn.

Individual PCBs: This determination was somewhat problematic for all 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 PCBs are mentioned, and in EN12766-1:99 also reproducibilities for each individual congener are mentioned. All observed reproducibilities are somewhat larger than required by the reproducibilities of EN12766-1:99. Congener PCB 138 was most problematic of all, while in previous years PCB 118 was the most problematic congener. The difference between the observed and the required reproducibility is largest of all differences for this PCB 138.

Total PCB: This determination was problematic. Only one statistical outlier was observed, but the calculated reproducibility is not in agreement with the requirements of IEC 61619:97.

Indiv. Aroclors: The determination of the individual Aroclors was very problematic. In total three statistical outliers were observed. The majority of the laboratories agreed that Aroclor 1254 was the main component in sample #1087. None of the observed reproducibilities is in agreement with the requirements of ASTM D4059:05e1.

Total Aroclor: This determination was problematic. Only one statistical outlier was observed, but the calculated reproducibility is not in agreement with the requirements of ASTM D4059:05e1.

Summary: All participants agreed that sample #1078 was positive on PCBs. The assigned value for the –by iis- calculated sums of the 7 PCB congeners is 5.53 mg/kg (see page 18). From this value, a total concentration 27.7 mg PCB/kg was estimated acc. to EN12766-2:00. ($PCB_{Total} = 5 * \sum \text{congeners}$). For the Aroclors a total amount of 18.9 mg PCB/kg was calculated (see page 21). From the EOX content of 53.9 mg Cl/kg a total concentration of 100 mg PCB/kg was estimated using an average Cl content of 54% for Aroclor 1254. All estimates for total PCB are given in the next table.

	#1087
total PCB concentration, estimated from 7 congeners in mg/kg	27.7
total PCB concentration, estimated from EOX in mg/kg	(100)*
total PCB concentration, using IEC 61619:97 method in mg/kg	22.0
estimated total PCB content using Aroclor method in mg/kg	18.9

Table 3: Comparison of estimations of total PCB content in sample #1087

* only two test results were reported for EOX, which limits the reliability of this estimate

The EOX content present in sample #1078 obviously is not caused by the PCBs only. Also a significant amount of other chlorinated components must be present, thus making this a rather difficult sample for the PCB determination. Still, the results are not much different than the results in previous years when much easier materials were used with less other chlorinated components.

The total PCB content as determined by IEC61619 is slightly higher than the total PCB content as determined by the Aroclor method. The same was seen in previous PTs. However, the range of all estimates is quite acceptable in view of the required precision. And this is a good result in view of the difficult sample type (a heavily contaminated waste oil), that was used in this PT.

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)
Extractable Organic Halogens (EOX)	mg Cl/kg	2	53.9	n.a.	26.7
PCB no. 28	mg/kg	16	0.42	0.26	0.19
PCB no. 52	mg/kg	16	0.85	0.50	0.41
PCB no. 101	mg/kg	17	1.24	0.87	0.60
PCB no. 118	mg/kg	17	0.92	0.51	0.44
PCB no. 138	mg/kg	18	0.99	0.95	0.48
PCB no. 153	mg/kg	18	0.94	0.58	0.45
PCB no. 180	mg/kg	17	0.27	0.19	0.12
sum of 7 individual PCB	mg/kg	14	5.53	2.33	n.a.
Total PCB	mg/kg	21	22.00	10.93	7.50
Aroclor 1242	mg/kg	4	5.03	4.82	4.45
Aroclor 1254	mg/kg	11	15.39	20.80	10.41
Aroclor 1260	mg/kg	6	4.85	10.00	4.38
Total Aroclor	mg/kg	8	18.89	15.95	12.14

Table 4: Performance of the group of participating laboratories on sample #1087

Without further statistical calculations it can be concluded that for many components there is not a good compliance of the group of participating laboratories with the relevant standards. The problematic components have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2010 PROFICIENCY TEST WITH PREVIOUS PTS.

	<i>November 2010</i>	<i>November 2009</i>	<i>November 2008</i>	<i>November 2007</i>
Number of reporting labs	34	29	28	33
Number of results reported	186	329	197	217
Statistical outliers	15	8	8	18
Percentage outliers	8.1%	3.6%	4.1%	8.3%

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:

<i>Determination</i>	<i>November 2010</i>	<i>November 2009</i>	<i>November 2008</i>	<i>November 2007</i>
EOX	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

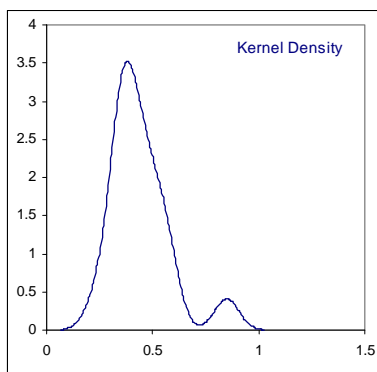
APPENDIX 1

Determination of Extractable Organic Chlorides on sample #1087; results in mg/kg.

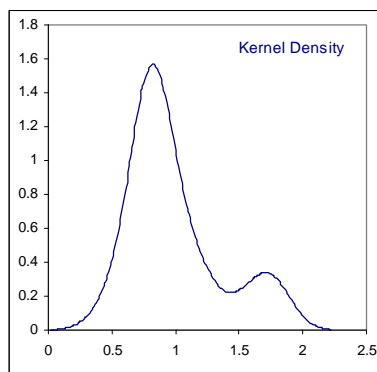
lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357		----		----	
445		----		----	
498		----		----	
614		----		----	
1059		----		----	
1066		----		----	
1072		----		----	
1104		----		----	
1126	NEN7425	55.7		----	
1170		----		----	
1243		----		----	
1245		----		----	
1285		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1338		----		----	
1350		----		----	
1352		----		----	
1367		----		----	
1371		----		----	
1375		----		----	
1435		----		----	
1513		----		----	
1516		----		----	
1526		----		----	
1529		----		----	
1633		----		----	
1660		----		----	
1801		----		----	
1816		----		----	
1826		52		----	
2160		----		----	
	normality	n.a.			
	n	0			
	outliers	0			
	mean (n)	53.9			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN12766-1)	26.7			

Determination of PCB 28, 52 and 101 on sample #1087; results in mg/kg.

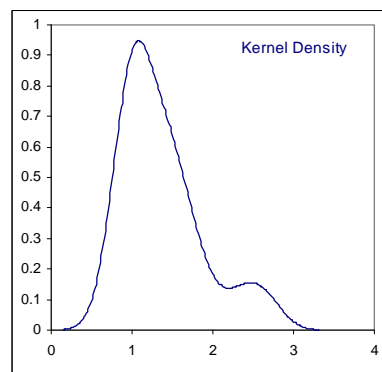
lab	method	No. 28	mark	z(targ)	No. 52	mark	z(targ)	No. 101	mark	z(targ)	Remarks
341		----		----			----			----	
343		----		----			----			----	
357	EN12766Bmod	0.42		0.06	0.76		-0.63	0.96		-1.29	
445		----		----			----			----	
498	EN12766	0.45		0.49	0.91		0.40	1.15		-0.40	
614		----		----			----			----	
1059	EN12766A	0.49		1.07	1.00		1.02	1.51		1.28	
1066	EN12766A	0.85	G(0.01)	6.28	1.55	G(0.01)	4.78	2.35	DG(0.05)	5.20	
1072	EN61619	0.5340		1.71	0.4896		-2.48	1.4011		0.77	
1104	EN12766	0.47		0.78	0.91		0.40	1.28		0.20	
1126	EN12766	0.376		-0.58	0.774		-0.53	1.213		-0.11	
1170	EN12766Bmod	0.56	C	2.08	0.93		0.54	1.13		-0.50	fr. 0.80
1243	EN12766B	0.36		-0.81	0.81		-0.28	0.97		-1.24	
1245		----		----			----			----	
1285		----		----			----			----	
1303		----		----			----			----	
1304		----		----			----			----	
1306		----		----			----			----	
1338		----		----			----			----	
1350		----		----			----			----	
1352		----		----			----			----	
1367		----		----			----			----	
1371	CMA/3/A	0.368		-0.70	0.761		-0.62	1.001		-1.10	
1375		----		----			----			----	
1435	EN12766A	0.36		-0.81	0.67		-1.24	0.82		-1.94	
1513	IEC61619	<0.2	false -?	<-3.13	0.8		-0.35	1.9		3.10	
1516	IEC61619	0.32		-1.39	1.76	DG(0.05)	6.22	1.69		2.12	
1526		----		----			----			----	
1529	EN12766	0.43		0.20	0.79		-0.42	0.94		-1.38	
1633	EN12766	0.24		-2.55	0.84		-0.08	1.57		1.56	
1660	IEC61619	0.58		2.37	1.19		2.32	2.64	DG(0.05)	6.55	
1801		----		----			----			----	
1816	IEC61619	0.36		-0.81	0.79		-0.42	1.49		1.18	
1826	EN12766	0.34		-1.10	1.2		2.38	0.94		-1.38	
2160	EN12766B	----		----	1.76	DG(0.05)	6.22	1.05		-0.87	
	normality	OK			OK			OK			
	n	16			16			17			
	outliers	1			3			2			
	mean (n)	0.416			0.852			1.236			
	st.dev. (n)	0.0933			0.1769			0.3094			
	R(calc.)	0.261			0.495			0.866			
	R(EN12766-1)	0.193			0.409			0.600			



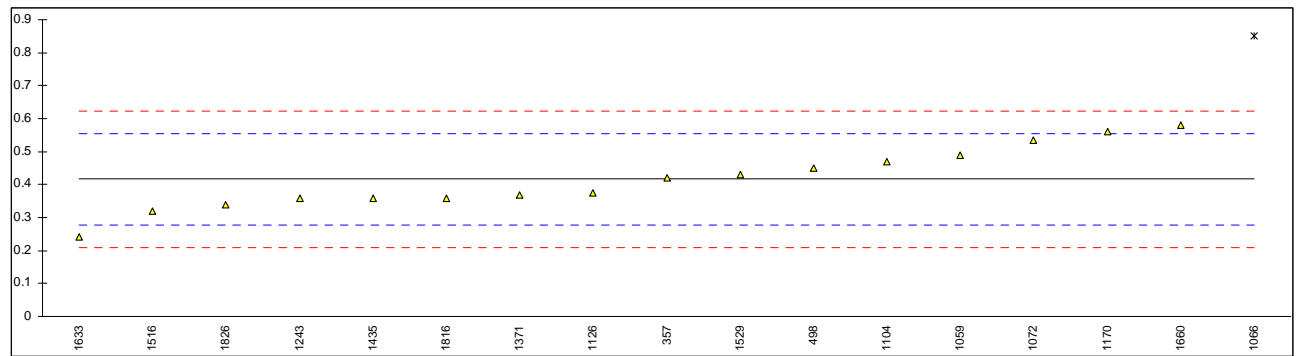
PCB 28



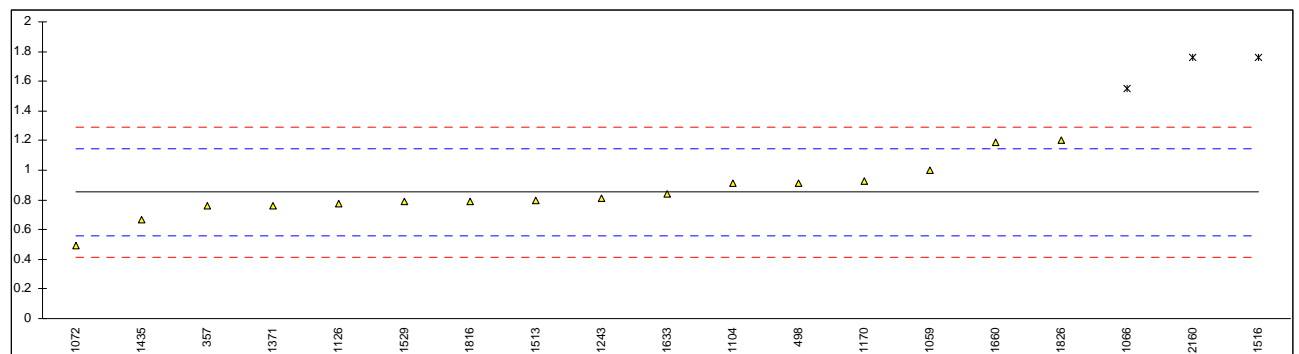
PCB52



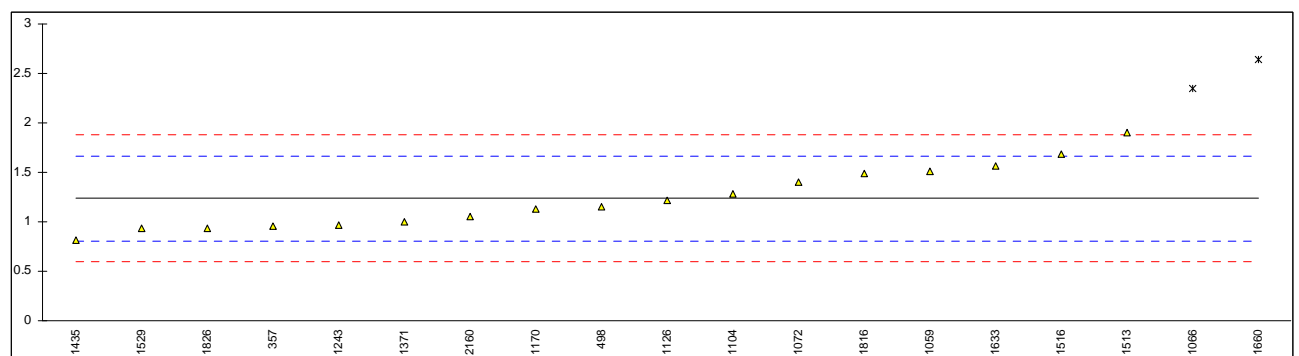
PCB101



PCB 28



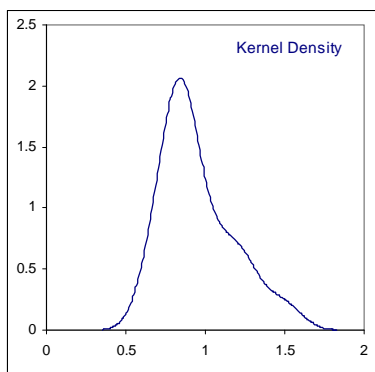
PCB 52



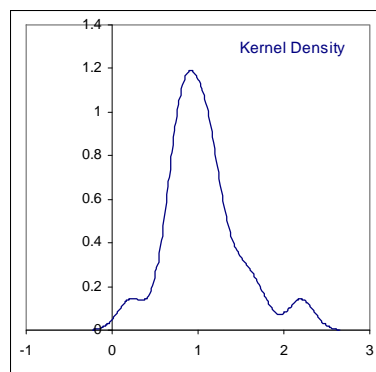
PCB 101

Determination of PCB 118 and 138 on sample #1087; results in mg/kg.

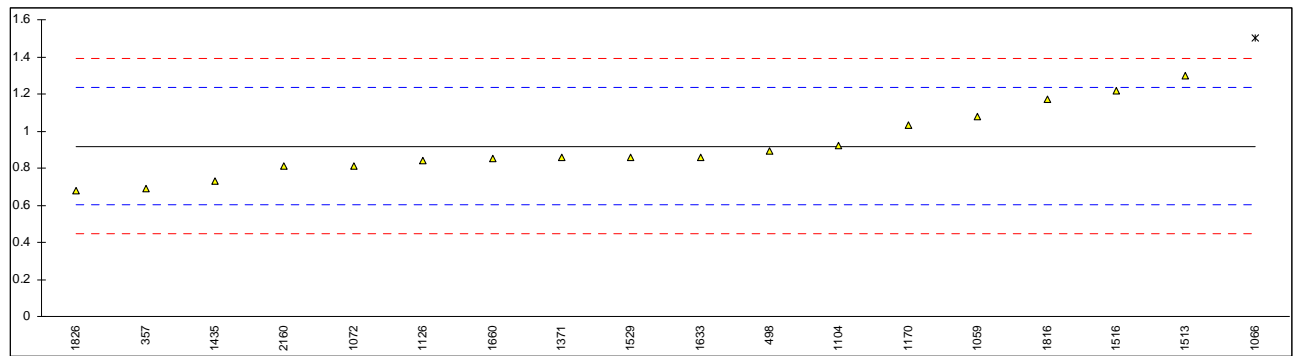
lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		----		----			----	
343		----		----			----	
357	EN12766Bmod	0.69		-1.44	0.73		-1.53	
445		----		----			----	
498	EN12766	0.89		-0.18	1.20		1.22	
614		----		----			----	
1059	EN12766A	1.08		1.03	0.22		-4.51	
1066	EN12766A	1.50	D(0.05)	3.69	1.70		4.14	
1072	IEC61619	0.8126		-0.67	1.0889		0.57	
1104	EN12766	0.92		0.01	1.16		0.99	
1126	EN12766	0.842		-0.48	1.090		0.58	
1170	EN12766Bmod	1.03		0.71	0.88		-0.65	
1243	EN12766B	----		----	0.75		-1.41	
1245		----		----			----	
1285		----		----			----	
1303		----		----			----	
1304		----		----			----	
1306		----		----			----	
1338		----		----			----	
1350		----		----			----	
1352		----		----			----	
1367		----		----			----	
1371	CMA/3/A	0.858		-0.38	0.800		-1.12	
1375		----		----			----	
1435	EN12766A	0.73		-1.19	0.85		-0.83	
1513	IEC61619	1.3		2.42	1.1		0.63	
1516	IEC61619	1.22		1.91	1.01		0.11	
1526		----		----			----	
1529	EN12766	0.86		-0.37	0.88		-0.65	
1633	EN12766	0.86		-0.37	2.20	C,G(0.05)	7.07	first reported 1.68
1660	IEC61619	0.85		-0.43	1.37		2.21	
1801		----		----			----	
1816	IEC61619	1.17		1.60	1.51		3.03	
1826	EN12766	0.68		-1.51	0.62		-2.17	
2160	EN12766B	0.81		-0.68	0.89		-0.59	
	normality	not OK			OK			
	n	17			18			
	outliers	1			1			
	mean (n)	0.918			0.992			
	st.dev. (n)	0.1816			0.3403			
	R(calc.)	0.508			0.953			
	R(EN12766-1)	0.442			0.479			



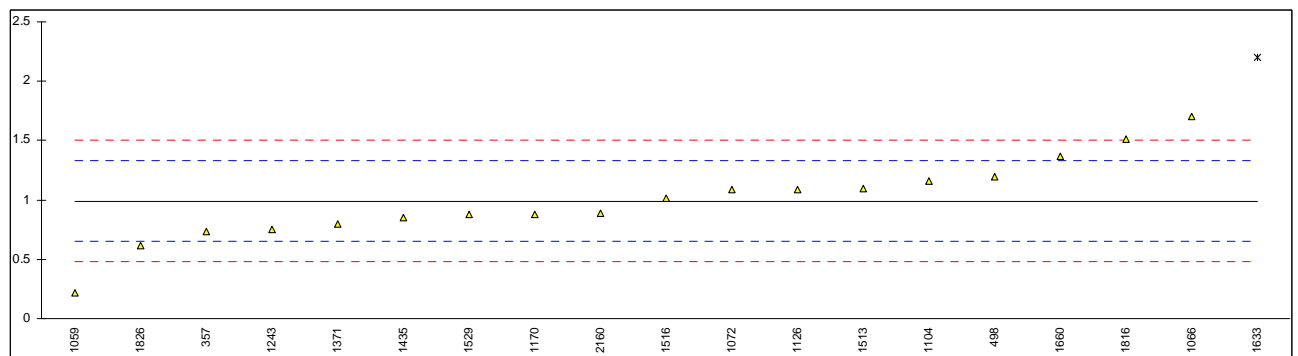
PCB 118



PCB138



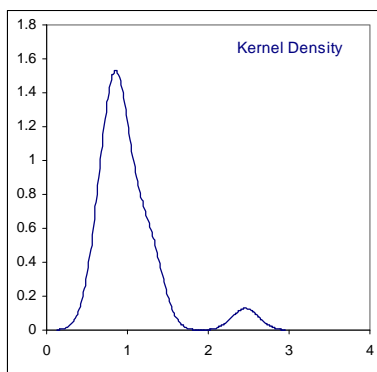
PCB 118



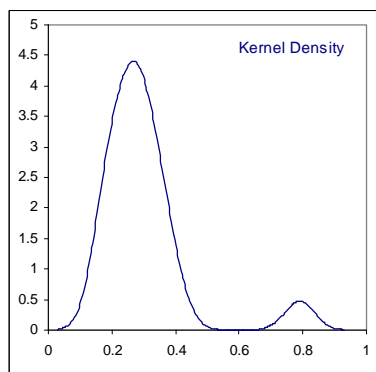
PCB 138

Determination of PCB 153 and 180 on sample #1087; results in mg/kg.

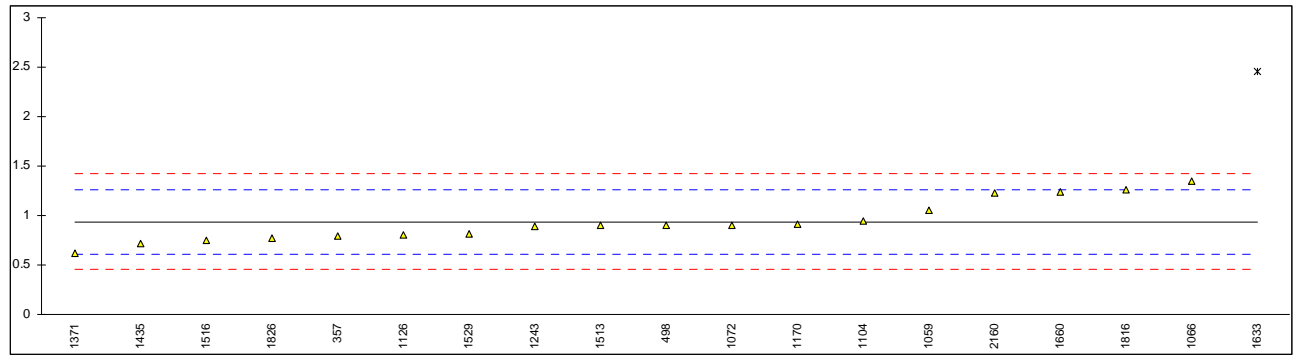
lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		----		----			----	
343		----		----			----	
357	EN12766Bmod	0.79		-0.91	0.20		-1.63	
445		----		----			----	
498	EN12766	0.90		-0.22	0.21		-1.39	
614		----		----			----	
1059	EN12766A	1.05		0.71	0.33		1.38	
1066	EN12766A	1.35		2.57	0.40		3.00	
1072	IEC61619	0.9059		-0.19	0.2590		-0.26	
1104	EN12766	0.95		0.09	0.18		-2.09	
1126	EN12766	0.808		-0.80	0.258		-0.28	
1170	EN12766Bmod	0.91		-0.16	0.27		-0.01	
1243	EN12766B	0.89		-0.29	0.33		1.38	
1245		----		----			----	
1285		----		----			----	
1303		----		----			----	
1304		----		----			----	
1306		----		----			----	
1338		----		----			----	
1350		----		----			----	
1352		----		----			----	
1367		----		----			----	
1371	CMA/3/A	0.616		-1.99	0.338		1.57	
1375		----		----			----	
1435	EN12766A	0.72		-1.34	0.17		-2.32	
1513	IEC61619	0.9		-0.22	0.3		0.69	
1516	IEC61619	0.75		-1.16	0.27		-0.01	
1526		----		----			----	
1529	EN12766	0.81		-0.78	0.28		0.22	
1633	EN12766	2.46	C,G(0.01)	9.46	0.79	C,G(0.01)	12.02	fr. 1.88 & 0.60
1660	IEC61619	1.24		1.89	0.25		-0.47	
1801		----		----			----	
1816	IEC61619	1.26		2.01	0.36		2.07	
1826	EN12766	0.77		-1.03	0.19		-1.86	
2160	EN12766B	1.23		1.82	----		----	
	normality	not OK			OK			
	n	18			17			
	outliers	1			1			
	mean (n)	0.936			0.270			
	st.dev. (n)	0.2086			0.0668			
	R(calc.)	0.584			0.187			
	R(EN12766-1)	0.451			0.121			



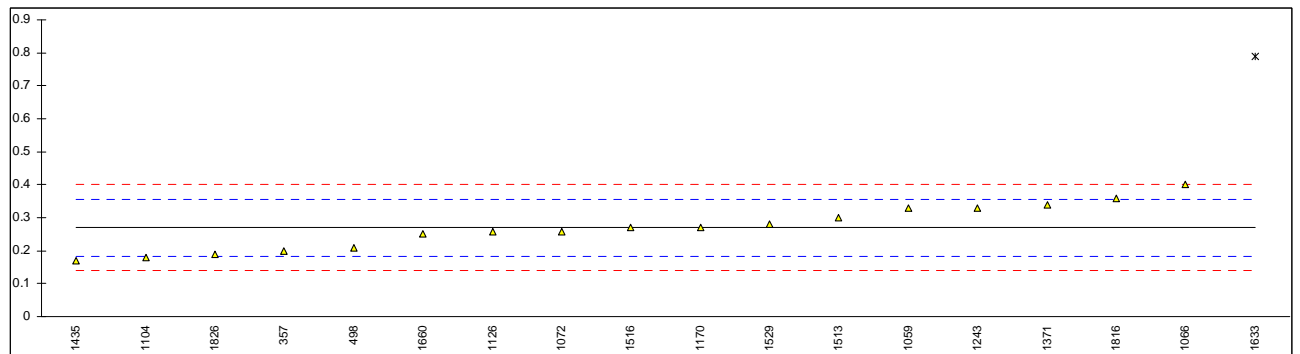
PCB 153



PCB180



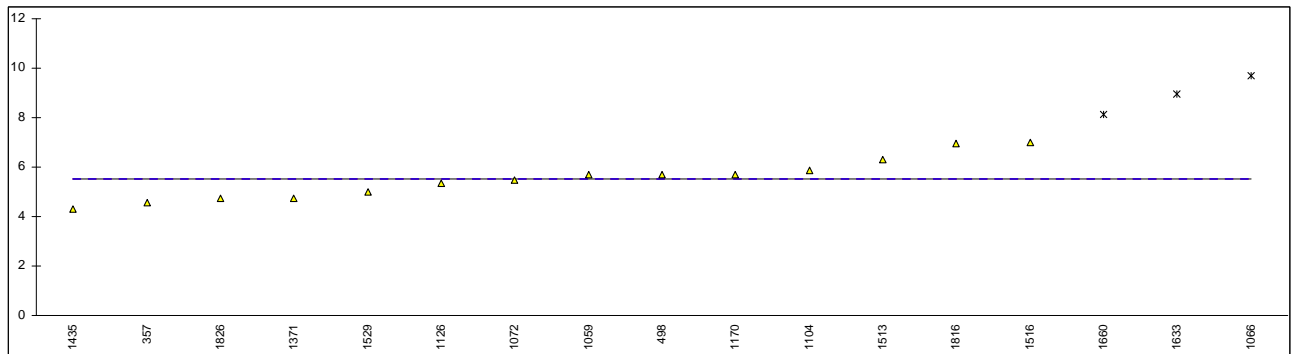
PCB 153



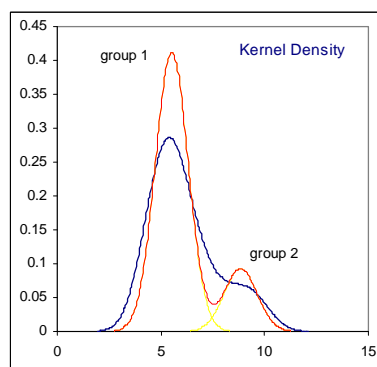
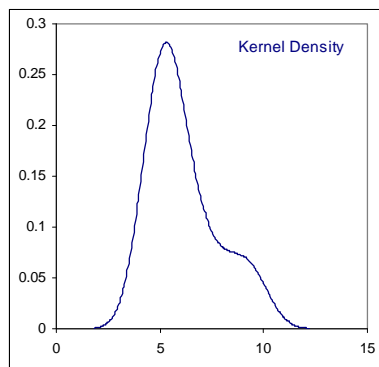
PCB 180

Summation of the 7 individual PCB on sample #1087; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357	calc by iis	4.55		----	
445		----		----	
498	calc by iis	5.71		----	
614		----		----	
1059	calc by iis	5.68		----	
1066	calc by iis	9.7		----	
1072	calc by iis	5.4911		----	
1104	calc by iis	5.87		----	
1126	calc by iis	5.361		----	
1170	calc by iis	5.71		----	
1243		----		----	
1245		----		----	
1285		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1338		----		----	
1350		----		----	
1352		----		----	
1367		----		----	
1371	calc by iis	4.742		----	
1375		----		----	
1435	calc by iis	4.32		----	
1513	calc by iis	6.3		----	
1516	calc by iis	7.02		----	
1526		----		----	
1529	calc by iis	4.99		----	
1633	calc by iis	8.96		----	
1660	calc by iis	8.12		----	
1801		----		----	
1816	calc by iis	6.94		----	
1826	calc by iis	4.74		----	
2160		----		----	
		<u>All data</u>		<u>Group 1</u>	<u>Group 2</u>
normality		not OK		OK	n.a.
n		17		14	3
outliers		0		0	0
mean (n)		6.130		5.530	8.927
st.dev. (n)		1.5561		0.8318	0.7905
R(calc.)		4.357		2.329	2.214

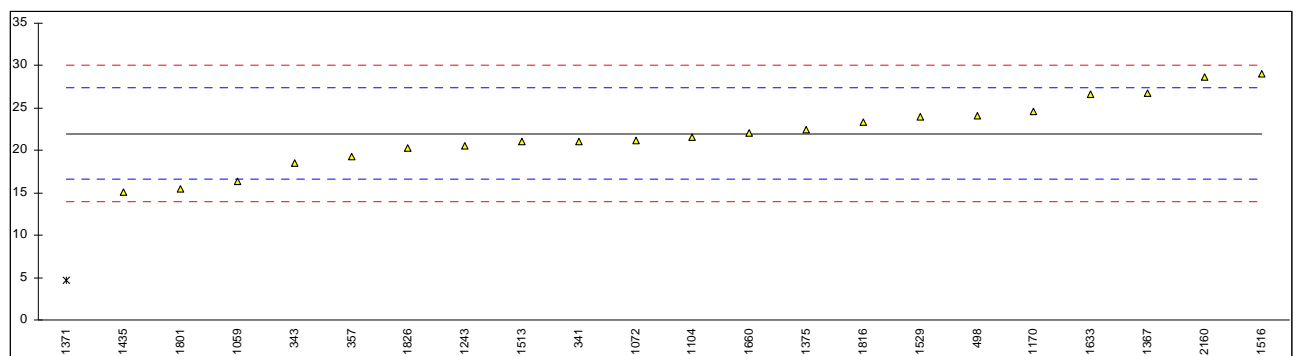
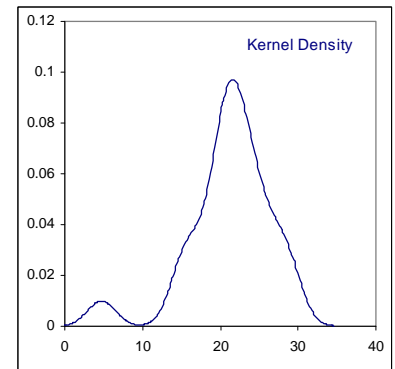


#1087



Determination of Total PCB on sample #1087; results in mg/kg.

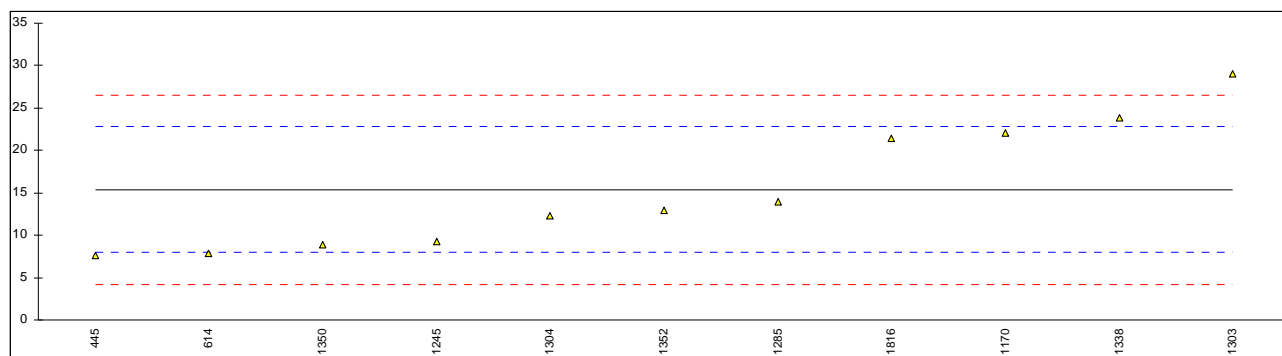
lab	method	value	mark	z(targ)	remarks
341	EN61619	21.0		-0.37	
343	EN61619	18.5		-1.31	
357	EN12766B	19.30		-1.01	
445		----		----	
498	EN12766B	24.06		0.77	
614		----		----	
1059	EN12766A	16.41		-2.09	
1066		----		----	
1072	EN61619	21.1260		-0.33	
1104	EN12766	21.6		-0.15	
1126		----		----	
1170	IEC61619mod	24.6		0.97	
1243	IEC61619	20.55		-0.54	
1245		----		----	
1285		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1338		----		----	
1350		----		----	
1352		----		----	
1367	IEC61619	26.8		1.79	
1371	CMA/3/A	4.742	G(0.01)	-6.44	reported sum of 7 individual PCBs
1375	IEC61619	22.5	C	0.19	first reported 9.1
1435	IEC61619	15.03		-2.60	
1513	IEC61619	21.0		-0.37	
1516	IEC61619	29.00		2.61	
1526		----		----	
1529	IEC61619	24		0.75	
1633	EN12766	26.6	C	1.72	first reported 24.0
1660	IEC61619	22.08		0.03	
1801	IEC61619	15.5		-2.43	
1816	IEC61619	23.3		0.49	
1826	EN12766	20.3	C	-0.63	first reported 4.74
2160	EN12766B	28.71		2.51	
	normality	OK			
	n	21			
	outliers	1			
	mean (n)	21.998			
	st.dev. (n)	3.9033			
	R(calc.)	10.929			
	R(IEC61619:97)	7.500			



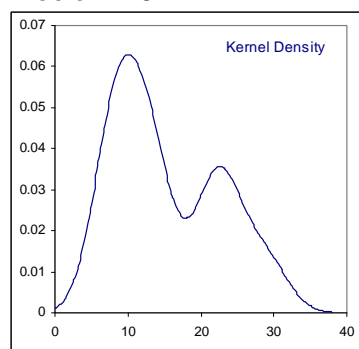
#1087

Determination of Aroclor 1242, 1254 and 1260 on sample #1087; results in mg/kg.

lab	method	1242	mark	z(targ)	1254	mark	z(targ)	1260	mark	z(targ)	Remarks
341		----		----	----		----	----		----	
343		----		----	----		----	----		----	
357		----		----	----		----	----		----	
445	IEC61619	4.45		-0.36	7.65		-2.08	1.59		-2.08	
498		----		----	----		----	----		----	
614	D4059	3.17		-1.16	7.86		-2.03	1.98		-1.84	
1059		----		----	----		----	----		----	
1066		----		----	----		----	----		----	
1072		----		----	----		----	----		----	
1104		----		----	----		----	----		----	
1126		----		----	----		----	----		----	
1170	D4059mod	----		----	22.09		1.80	----		----	
1243		----		----	----		----	----		----	
1245	in house	----		----	9.29		-1.64	----		----	
1285	D4059	0	ex (zero)	-3.13	13.95		-0.39	8.64		2.42	
1303	in house	34	DG(0.05)	18.03	29		3.66	----		----	
1304	in house	5.21	C	0.11	12.35		-0.82	3.26		-1.02	fr. <1
1306		----		----	----		----	10		3.29	
1338	in house	22.3	DG(0.05)	10.75	23.9	C	2.29	19.9	C,G(0.05)	9.62	fr. 37.2, 27.2 & 23.1
1350	D4059	----		----	8.93		-1.74	----		----	
1352	CH-03	7.28		1.40	12.91		-0.67	3.64		-0.77	
1367		----		----	----		----	----		----	
1371		----		----	----		----	----		----	
1375		----		----	----		----	----		----	
1435		----		----	----		----	----		----	
1513		----		----	----		----	----		----	
1516		----		----	----		----	----		----	
1526	EPA 600/4	----		----	----		----	<5		----	
1529		----		----	----		----	----		----	
1633		----		----	----		----	----		----	
1660		----		----	----		----	----		----	
1801		----		----	----		----	----		----	
1816	IEC61619	----		----	21.4		1.61	----		----	
1826		----		----	----		----	----		----	
2160		----		----	----		----	----		----	
	normality	n.a.			OK			OK			
	n	4			11			6			
	outliers	2			0			1			
	mean (n)	5.027			15.394			4.852			
	st.dev. (n)	1.7215			7.4275			3.5705			
	R(calc.)	4.820			20.797			9.997			
	R(D4059:05e1)	4.499			10.414			4.380			

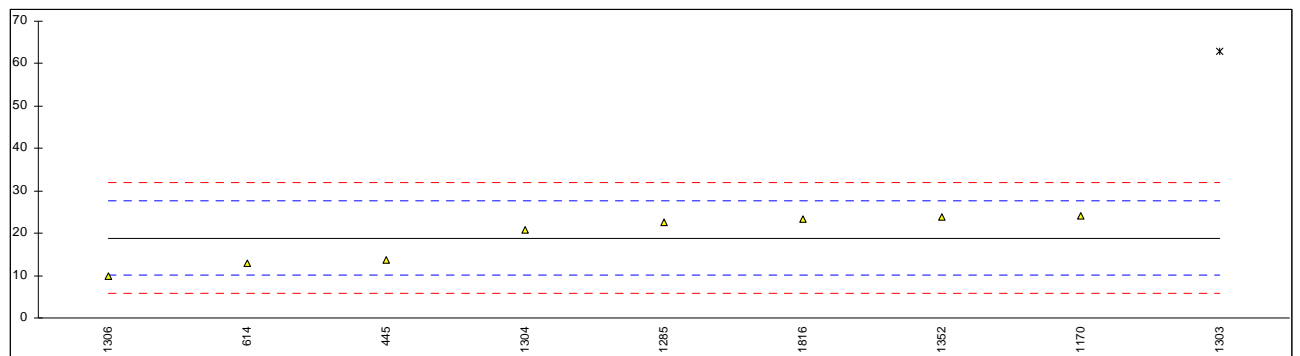


Aroclor 1254

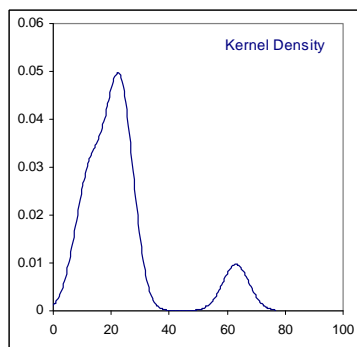


Determination of the Total Aroclor on sample #1087; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357		----		----	
445	IEC61619	13.71		-1.19	
498		----		----	
614	D4059	13.01		-1.36	
1059		----		----	
1066		----		----	
1072		----		----	
1104		----		----	
1126		----		----	
1170	D4059	23.97		1.17	
1243		----		----	
1245		----		----	
1285	D4059	22.59		0.85	
1303	in house	63	G(0.01)	10.17	
1304	in house	20.72	C	0.42	first reported 15.61
1306	EPA 600/4	10		-2.05	
1338		----		----	
1350		----		----	
1352	CH-03	23.83		1.14	
1367		----		----	
1371		----		----	
1375		----		----	
1435		----		----	
1513		----		----	
1516		----		----	
1526		----		----	
1529		----		----	
1633		----		----	
1660		----		----	
1801		----		----	
1816	IEC61619	23.3		1.02	
1826		----		----	
2160		----		----	
normality		OK			
n		8			
outliers		1			
mean (n)		18.891			
st.dev. (n)		5.6963			
R(calc.)		15.950			
R(D4059:05e1)		12.142			



#1087



APPENDIX 2

Number of participating laboratories per country

4 labs in AUSTRALIA

2 labs in BELGIUM

1 lab in FINLAND

1 lab in FRANCE

2 labs in GERMANY

1 lab in GREECE

2 labs in ITALY

1 lab in LUXEMBOURG

1 lab in MEXICO

1 lab in NEW ZEALAND

1 lab in NORWAY

2 labs in PORTUGAL

1 lab in SLOVENIA

2 labs in SOUTH AFRICA

6 labs in SPAIN

4 labs in THE NETHERLANDS

1 lab in TURKEY

3 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, November 2008
- 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/>)