Results of Proficiency Test
PCB in Transformer Oil
November 2009

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 2009/2010, it was decided to continue the round robin for the PCB analysis on Transformer Oil.
In this international Interlaboratory study, 30 laboratories from 17 different countries have participated, but not all laboratories reported results for all evaluated components.
See appendix 2 for a list of participating laboratories per country. In this report the results of the PCB analysis of the Transformer Oil proficiency test 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 two samples of used transformer oil that were donated by one of the participating laboratories for PCB analysis.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43 ands ILAC-G13:2007. 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 November 2008 (iis-protocol, version 3.1).

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 of 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 two different samples were used. The necessary bulk materials for the samples contaminated oil with PCB were donated by a third party laboratory.
After homogenisation, 41, resp. 44 subsamples were transferred to 8 mL amber glass vials, labelled #0990, resp. #0991.

The homogeneity of the subsamples #0990 and #0991 was checked by determination of the organic chloride in accordance with UOP779-08 on 2 x 4 stratified random selected samples:

	Organic chloride in mg/L
sample #0990-1	113
sample #0990-2	112
sample #0990-3	113
sample #0990-4	112
sample #0991-1	7
sample #0991-2	6
sample #0991-3	7
sample #0991-4	6

Table 1: homogeneity test results of subsamples #0990 and #0991

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	#0990	#0991
r (samples)	1.6	1.6
reference method	UOP779	UOP779
$0.3xR_{(reference\ method)}$	5.7	1.6

Table 2: evaluation of repeatabilities

The repeatabilities of the results of homogeneity test were in agreement with 0.3 times the reproducibilities as required by UOP779. Therefore, homogeneity of the samples was assumed.
To each of the participating laboratories two vials of 8 mL (labelled #0990, resp. #0991) were sent on October 26, 2009.

2.5 STABILITY OF THE SAMPLES

The stability of the transformer 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 individual PCB and via Aroclor) 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 November 2008 (iis-protocol, version 3.1).

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 G(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by G(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:

$z_{(target)}$ = (result - average of PT) / 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 one participants did send in one or more test results.
In total 29 participants reported 329 numerical results. Observed were 8 outlying results, which is 3.6 % 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.
All original data sets proved to have a normal distribution.

For the results on sample #0990, the following was concluded:

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

Individual PCBs: This determination may be 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 118 was most problematic of all as in previous years, because the difference between the observed and the required reproducibility is largest of all differences.

Total PCB: This determination is very problematic. Only one statistical outlier was observed, but the calculated reproducibilities are both not at all in agreement with the requirements of IEC 61619:97.

Indiv. Aroclors: The determination of the individual Aroclors is not problematic. At least two false negative results were reported. The majority of the laboratories agreed that Aroclor 1260 was the main component in sample #0990 and Aroclor 1254 in sample #0991. All observed reproducibilities are in full agreement with the requirements of EN12766 and/or ASTMD4059:05e1, except for the observed reproducibility on Aroclor 1254 in sample #0991.

Total Aroclor: This determination is not problematic for sample #0990, but it is rather problematic for sample #0991 (due to the above mentioned problems with Arochlor 1254).
Only the calculated reproducibility for sample #0990 is in good agreement with the requirements of ASTMD4059:05e1.

Summary: All participants agreed that both samples were positive on PCB.
The sum of the 7 assigned values of the congeners is 14.87 mg/kg for #0990 and 3.97 for #0991. From these sums, total concentrations of 74, resp 20 PCB/kg were estimated for the two samples, acc. to EN12766-2:00. ($PCB_{Total} = 5 * \sum_{congeners}$). For the Aroclors total amounts of 53.42, resp. 13.01 mg/kg were estimated. From the EOX content of 87.8, resp. 10.6 mg Cl/kg (n = 1!) a total concentration of 140, resp. 15 mg PCB/kg was estimated using Cl content of 63% from $C_{10}H_4Cl_6$ as PCB molecule in Aroclor 1260. All the total PCB conclusions are given in de the next table.

	#0990	#0991
total PCB concentration, estimated from 7 congeners in mg/kg	74	20
total PCB concentration, estimated from EOX in mg/kg	(140)*	(15)*
total PCB concentration, using IEC 61619:97 method in mg/kg	62	17
estimated total PCB content using Aroclor method in mg/kg	53	13

Table 3: Comparison of estimations of total PCB content in samples #0990 and #0991

* only one test result was reported for EOX, which limits the reliability of this estimate

The total PCB content as determined by IEC61619 is slightly higher than the total PCB content as determined by the Arochlor method. However, the range of all estimates 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)
Extractable Organic Halogens (EOX)	mg Cl/kg	1	87.8	n.a.	n.a.
PCB no. 28	mg/kg	10	0.11	0.18	0.04
PCB no. 52	mg/kg	10	0.15	0.15	0.06
PCB no. 101	mg/kg	15	1.51	1.03	0.73
PCB no. 118	mg/kg	8	0.21	0.25	0.09
PCB no. 138	mg/kg	16	3.59	2.89	1.77
PCB no. 153	mg/kg	15	4.37	2.81	2.15
PCB no. 180	mg/kg	16	4.59	3.73	2.26
sum of 7 individual PCB	mg/kg	16	14.87	3.75	n.a.
Total PCB	mg/kg	15	62.24	44.32	17.56
Aroclor 1242	mg/kg	4	1.15	1.46	1.49
Aroclor 1254	mg/kg	3	n.a.	n.a.	n.a.
Aroclor 1260	mg/kg	11	50.92	27.15	25.54
Total Aroclor	mg/kg	11	53.42	26.99	26.48

Table 4: Performance of the group of participating laboratories on sample #0990

Parameter	unit	n	average	2.8 * sd	R(lit)
Extractable Organic Halogens (EOX)	mg Cl/kg	1	10.6	n.a.	n.a.
PCB no. 28	mg/kg	5	0.11	0.20	0.04
PCB no. 52	mg/kg	16	0.35	0.38	0.16
PCB no. 101	mg/kg	15	0.90	1.11	0.43
PCB no. 118	mg/kg	14	0.57	1.08	0.27
PCB no. 138	mg/kg	14	0.78	0.77	0.38
PCB no. 153	mg/kg	14	0.79	0.80	0.38
PCB no. 180	mg/kg	16	0.48	0.52	0.23
sum of 7 individual PCBs	mg/kg	16	3.97	1.50	4.19
Total PCB	mg/kg	16	16.75	19.50	6.19
Aroclor 1242	mg/kg	2	n.a.	n.a.	n.a.
Aroclor 1254	mg/kg	10	10.47	22.88	7.80
Aroclor 1260	mg/kg	8	5.62	4.18	4.89
Total Aroclors	mg/kg	11	13.01	21.27	9.18

Table 5: Performance of the group of participating laboratories on sample #0991

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 2009 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2009	*November 2008*	*November 2007*	*November 2006*
Number of reporting labs	29	28	33	38
Number of results reported	329	197	217	285
Statistical outliers	8	8	18	30
Percentage outliers	3.6%	4.1%	8.3%	10.5%

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 proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	*November 2009*	*November 2008*	*November 2007*	*November 2006*
EOX	n.a.	n.a.	-	++
PCB (all)	--	+/-	+/-	--
Aroclor (all)	+/-	+	--	--

Table 7: comparison against standards

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
n.d.: not determined

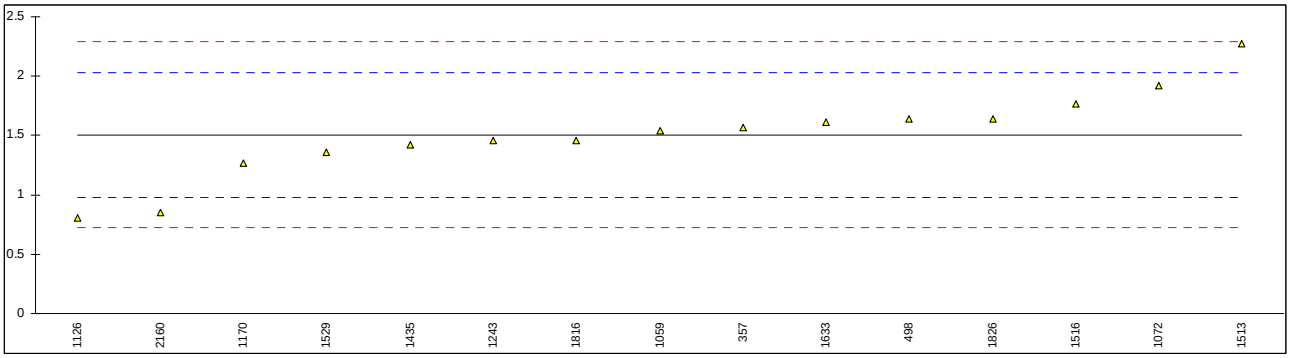
APPENDIX 1

Determination of Extractable Organic Chlorides on samples #0990 and #0991; results in mg/kg.

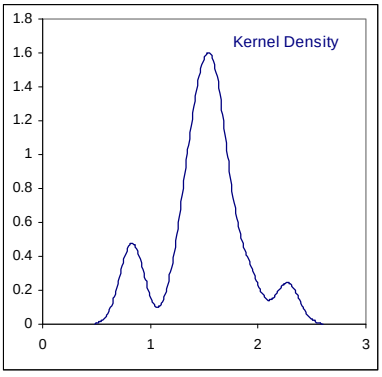
lab	method	#0990	#0991	remarks
341		-----	-----	
343		-----	-----	
357		-----	-----	
445		-----	-----	
498		-----	-----	
614		-----	-----	
963		-----	-----	
1059		-----	-----	
1072		-----	-----	
1126	NEN7425	87.8	10.6	
1170		-----	-----	
1243		-----	-----	
1245		-----	-----	
1258		-----	-----	
1303		-----	-----	
1304		-----	-----	
1306		-----	-----	
1435		-----	-----	
1505		-----	-----	
1513		-----	-----	
1516		-----	-----	
1526		-----	-----	rep. 152.79 mg/kg, resp. <100 mg/kg
1529		-----	-----	
1633		-----	-----	
1660		-----	-----	
1801		-----	-----	
1816		-----	-----	
1826		-----	-----	
2122		-----	-----	
2160		-----	-----	
	normality	n.a.	n.a.	
	n	1	1	
	outliers	n.a.	n.a.	
	mean (n)	n.a.	n.a.	
	st.dev. (n)	n.a.	n.a.	
	R(calc.)	n.a.	n.a.	
	R(Staatscourant246:93)	n.a.	n.a.	

Determination of PCB 28, 52 and 101 on sample #0990; 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	EN12766B	<0.1		-----	0.09		-2.73	1.57		0.24	
445		-----		-----	-----		-----	-----		-----	
498	EN12766B	<0.1		-----	<0.1		<-2.28	1.64		0.51	
614		-----		-----	-----		-----	-----		-----	
963		-----		-----	-----		-----	-----		-----	
1059	EN12766A	0.03		-5.39	0.09		-2.73	1.54		0.13	
1072	EN61619	0.1388		1.60	0.2008		2.31	1.9231		1.59	
1126	EN12766A	-----		-----	-----		-----	0.81		-2.66	
1170	in house	0.22		6.82	0.26	C	5.00	1.27		-0.90	fr 0.52
1243	EN12766	0.05		-4.10	0.11		-1.82	1.46		-0.18	
1245		-----		-----	-----		-----	-----		-----	
1258		-----		-----	-----		-----	-----		-----	
1303		-----		-----	-----		-----	-----		-----	
1304		-----		-----	-----		-----	-----		-----	
1306		-----		-----	-----		-----	-----		-----	
1435	EN12766A	0.07		-2.82	0.13		-0.91	1.42		-0.33	
1505		-----		-----	-----		-----	-----		-----	
1513	IEC61619	0.08		-2.18	0.14		-0.46	2.27		2.92	
1516	IEC61619	0.21		6.18	0.18		1.36	1.77		1.01	
1526		-----		-----	-----		-----	-----		-----	
1529	EN12766A	0.11		-0.25	0.18		1.36	1.36		-0.56	
1633	EN12766	0.13		1.04	0.46	CG(0.01)	14.11	1.61		0.40	fr 0.67
1660	D4059	<0.2		-----	<0.2		-----	<0.2		<-4.99	false neg?
1801		-----		-----	-----		-----	-----		-----	
1816	IEC61619	-----		-----	n.d.	C	-----	1.46		-0.18	fr 0.50
1826	EN12766	0.10		-0.89	0.12		-1.37	1.64		0.51	
2122		-----		-----	-----		-----	-----		-----	
2160	EN12766B	2.68	G(0.01)	164.87	5.67	G(0.01)	251.23	0.85		-2.50	
	normality	OK			OK			OK			
	n	10			10			15			
	outliers	1			2			0			
	mean (n)	0.114			0.150			1.506			
	st.dev. (n)	0.0631			0.0544			0.3667			
	R(calc.)	0.177			0.152			1.027			
	R(EN12766-1)	0.044			0.062			0.734			

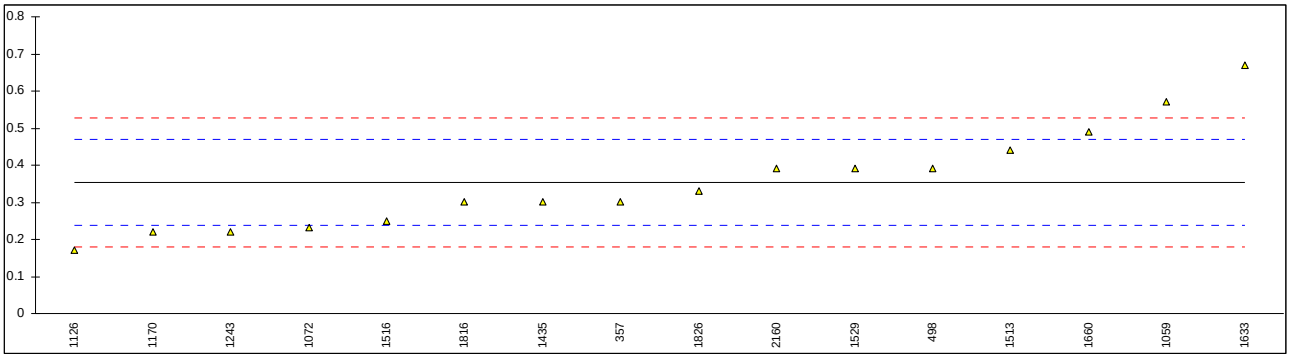


PCB 101

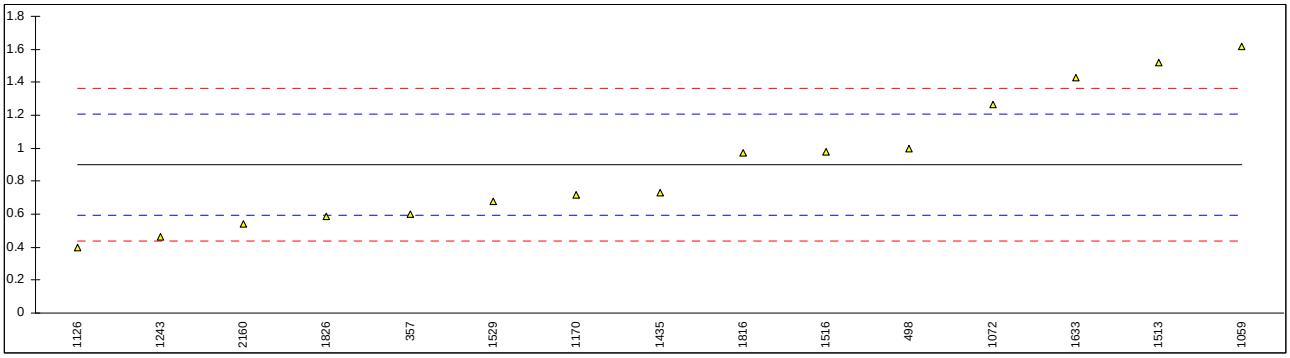


Determination of PCB 28, 52 and 101 on sample #0991; results in mg/kg.

lab	method	No. 28	mark	z(targ)	No. 52	mark	z(targ)	No. 101	mark	z(targ)	Remarks
341		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
357	EN12766B	<0.1		-----	0.30		-0.93	0.60		-1.94	
445		-----		-----	-----		-----	-----		-----	
498	EN12766B	<0.1		-----	0.39		0.62	1.00		0.64	
614		-----		-----	-----		-----	-----		-----	
963		-----		-----	-----		-----	-----		-----	
1059	EN12766A	0.03		-5.37	0.57		3.73	1.62		4.65	
1072	EN61619	<0.01		-7.42	0.2308		-2.12	1.2629		2.34	
1126	EN12766A	-----		-----	0.17		-3.17	0.40		-3.23	
1170	in house	0.043		-4.48	0.22		-2.31	0.72		-1.16	
1243	EN12766	-----		-----	0.22		-2.31	0.46		-2.84	
1245		-----		-----	-----		-----	-----		-----	
1258		-----		-----	-----		-----	-----		-----	
1303		-----		-----	-----		-----	-----		-----	
1304		-----		-----	-----		-----	-----		-----	
1306		-----		-----	-----		-----	-----		-----	
1435	EN12766A	n.d.		-----	0.30		-0.93	0.73		-1.10	
1505		-----		-----	-----		-----	-----		-----	
1513	IEC61619	<0.2		-----	0.44		1.49	1.52		4.01	
1516	IEC61619	<0.1		-----	0.25		-1.79	0.98		0.52	
1526		-----		-----	-----		-----	-----		-----	
1529	EN12766A	<0.10		-----	0.39		0.62	0.68		-1.42	
1633	EN12766	0.12		0.78	0.67		5.45	1.43		3.42	
1660	D4059	<0.2		-----	0.49		2.35	<0.2		<-4.52	false neg?
1801		-----		-----	-----		-----	-----		-----	
1816	IEC61619	n.d.		-----	0.30		-0.93	0.97		0.45	
1826	EN12766	0.17		4.20	0.33		-0.41	0.59		-2.00	
2122		-----		-----	-----		-----	-----		-----	
2160	EN12766B	0.18		4.88	0.39		0.62	0.54		-2.33	
	normality	OK			OK			OK			
	n	5			16			15			
	outliers	0			0			0			
	mean (n)	0.109			0.354			0.900			
	st.dev. (n)	0.0698			0.1370			0.3969			
	R(calc.)	0.195			0.383			1.111			
	R(EN12766-1)	0.041			0.162			0.433			



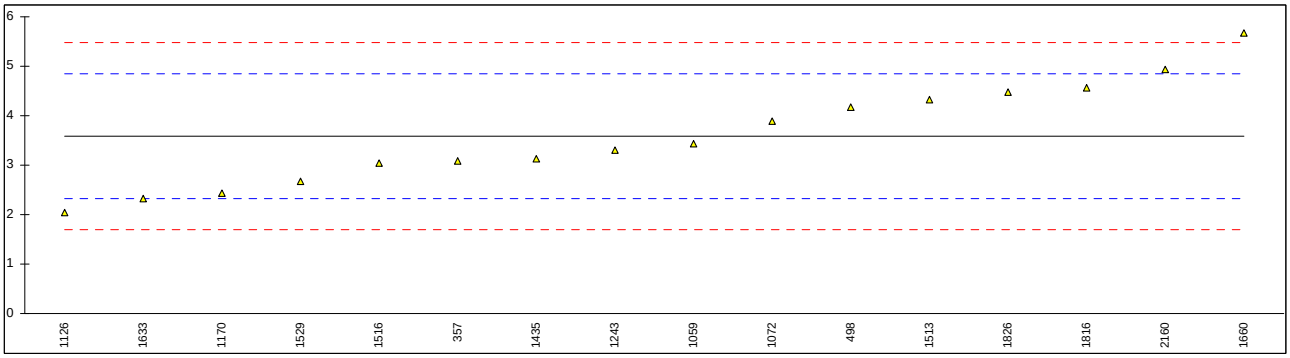
PCB 52



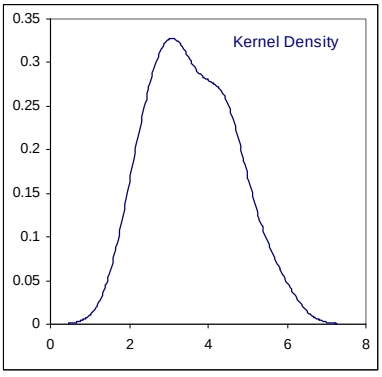
PCB 101

Determination of PCB 118 and 138 on sample #0990; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766B	0.20		-0.20	3.08		-0.81	
445		-----		-----	-----		-----	
498	EN12766B	0.26		1.68	4.17		0.91	
614		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1059	EN12766A	0.07		-4.27	3.43		-0.26	
1072	EN61619	<0.01		-6.46	3.8984		0.48	
1126	EN12766A	-----		-----	2.05		-2.44	
1170	in house	0.30	C	2.94	2.43		-1.84	fr 0.59
1243	EN12766	-----		-----	3.30		-0.46	
1245		-----		-----	-----		-----	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1435	EN12766A	0.13		-2.39	3.13		-0.73	
1505		-----		-----	-----		-----	
1513	IEC61619	0.33		3.88	4.32		1.15	
1516	IEC61619	0.15		-1.76	3.04		-0.88	
1526		-----		-----	-----		-----	
1529	EN12766A	0.21		0.12	2.67		-1.46	
1633	EN12766	1.03	CG(0.01)	25.81	2.32	C	-2.02	fr 1.39 resp. 0.87
1660	D4059	<0.2		-----	5.68		3.31	
1801		-----		-----	-----		-----	
1816	IEC61619	-----		-----	4.56		1.53	
1826	EN12766	4.76	G(0.01)	142.69	4.47		1.39	
2122		-----		-----	-----		-----	
2160	EN12766B	-----		-----	4.94		2.13	
	normality	OK			OK			
	n	8			16			
	outliers	2			0			
	mean (n)	0.206			3.593			
	st.dev. (n)	0.0883			1.0306			
	R(calc.)	0.247			2.886			
	R(EN12766-1)	0.089			1.768			

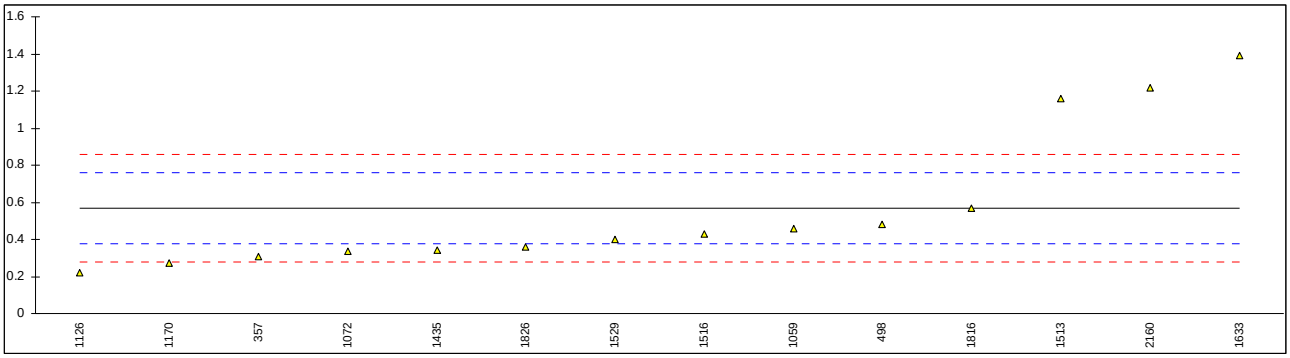


PCB 138

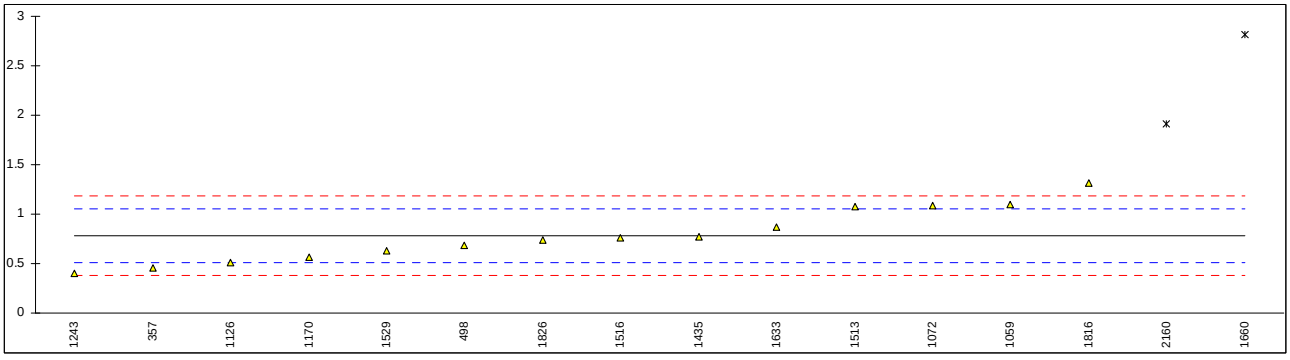


Determination of PCB 118 and 138 on sample #0991; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766B	0.31		-2.69	0.46		-2.41	
445		-----		-----	-----		-----	
498	EN12766B	0.48		-0.91	0.68		-0.77	
614		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1059	EN12766A	0.46		-1.12	1.10		2.36	
1072	EN61619	0.3364		-2.41	1.0848		2.25	
1126	EN12766A	0.22		-3.63	0.51		-2.04	
1170	in house	0.27		-3.10	0.57		-1.59	
1243	EN12766	-----		-----	0.40		-2.86	
1245		-----		-----	-----		-----	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1435	EN12766A	0.34		-2.37	0.77		-0.10	
1505		-----		-----	-----		-----	
1513	IEC61619	1.16		6.18	1.08		2.21	
1516	IEC61619	0.43		-1.44	0.76		-0.17	
1526		-----		-----	-----		-----	
1529	EN12766A	0.40		-1.75	0.63		-1.14	
1633	EN12766	1.39	C	8.58	0.87	C	0.65	fr 1.03 resp. 2.32
1660	D4059	<0.2		<-3.83	2.82	G(0.01)	15.20	false negative?
1801		-----		-----	-----		-----	
1816	IEC61619	0.57		0.03	1.31		3.93	
1826	EN12766	0.36		-2.17	0.74		-0.32	
2122		-----		-----	-----		-----	
2160	EN12766B	1.22	C	6.80	1.91	G(0.05)	8.41	fr 0.06
	normality	not OK			OK			
	n	14			14			
	outliers	0			2			
	mean (n)	0.568			0.783			
	st.dev. (n)	0.3866			0.2735			
	R(calc.)	1.083			0.766			
	R(EN12766-1)	0.268			0.375			



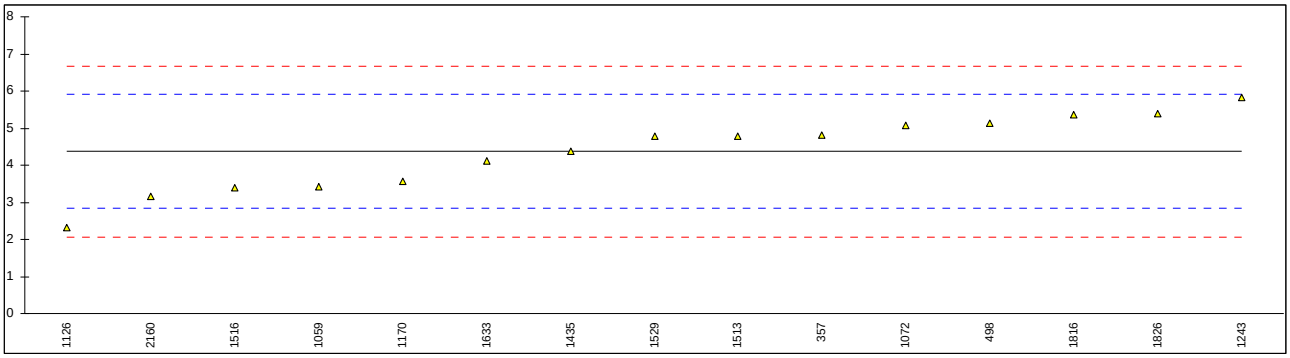
PCB 118



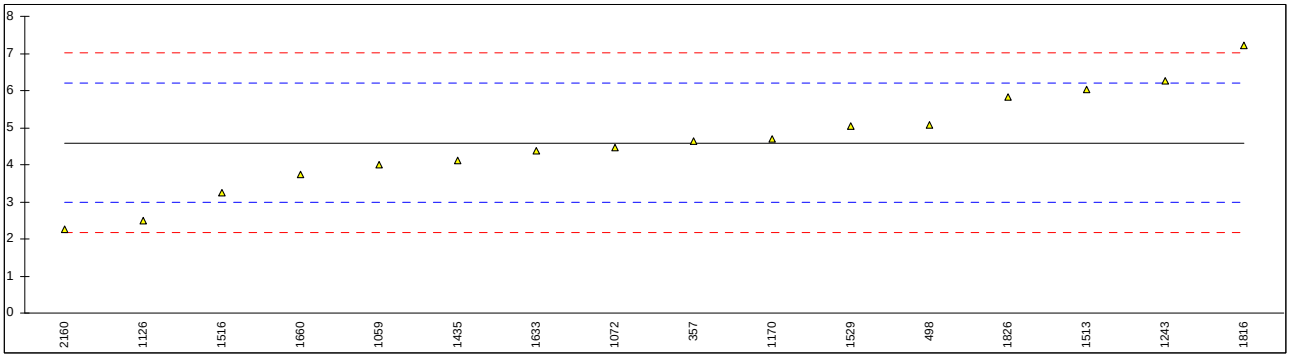
PCB 138

Determination of PCB 153 and 180 on sample #0990; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766B	4.81		0.57	4.63		0.05	
445		-----		-----	-----		-----	
498	EN12766B	5.14		1.00	5.08		0.61	
614		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1059	EN12766A	3.43		-1.22	4.00		-0.73	
1072	EN61619	5.0602		0.90	4.4754		-0.14	
1126	EN12766A	2.32		-2.67	2.48		-2.61	
1170	in house	3.56		-1.05	4.69		0.13	
1243	EN12766	5.84		1.91	6.25		2.06	
1245		-----		-----	-----		-----	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1435	EN12766A	4.39		0.03	4.11		-0.59	
1505		-----		-----	-----		-----	
1513	IEC61619	4.79		0.55	6.02		1.77	
1516	IEC61619	3.38		-1.29	3.25		-1.66	
1526		-----		-----	-----		-----	
1529	EN12766A	4.79		0.55	5.03		0.55	
1633	EN12766	4.13	C	-0.31	4.37	C	-0.27	fr 1.15 resp. 0.66
1660	D4059	<0.2		<-5.42	3.73		-1.06	false negative?
1801		-----		-----	-----		-----	
1816	IEC61619	5.36		1.29	7.21		3.25	
1826	EN12766	5.38		1.32	5.82		1.53	
2122		-----		-----	-----		-----	
2160	EN12766B	3.15		-1.59	2.26		-2.88	
	normality	OK			OK			
	n	15			16			
	outliers	0			0			
	mean (n)	4.369			4.588			
	st.dev. (n)	1.0016			1.3336			
	R(calc.)	2.805			3.734			
	R(EN12766-1)	2.152			2.261			



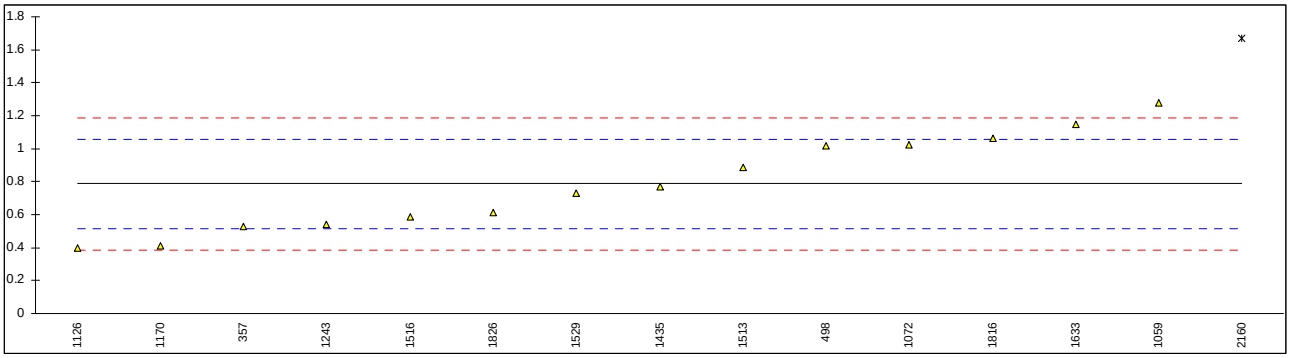
PCB 153



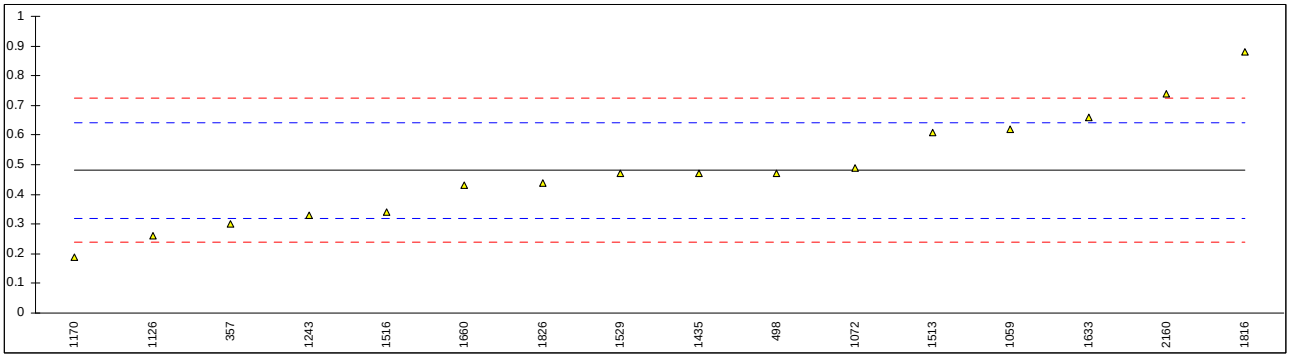
PCB 180

Determination of PCB 153 and 180 on sample #0991; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357	EN12766B	0.53		-1.90	0.30		-2.25	
445		-----		-----	-----		-----	
498	EN12766B	1.02		1.74	0.47		-0.14	
614		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1059	EN12766A	1.28		3.67	0.62		1.72	
1072	EN61619	1.0263		1.78	0.4882		0.09	
1126	EN12766A	0.40		-2.87	0.26		-2.74	
1170	in house	0.41		-2.80	0.19		-3.61	
1243	EN12766	0.54		-1.83	0.33		-1.88	
1245		-----		-----	-----		-----	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1435	EN12766A	0.77		-0.12	0.47		-0.14	
1505		-----		-----	-----		-----	
1513	IEC61619	0.89		0.77	0.61		1.60	
1516	IEC61619	0.59		-1.46	0.34		-1.75	
1526		-----		-----	-----		-----	
1529	EN12766A	0.73		-0.42	0.47		-0.14	
1633	EN12766	1.15	C	2.70	0.66	C	2.22	fr 4.13 resp. 4.37
1660	D4059	<0.2		<-4.36	0.43		-0.63	false negative?
1801		-----		-----	-----		-----	
1816	IEC61619	1.06		2.04	0.88		4.95	
1826	EN12766	0.61		-1.31	0.44		-0.51	
2122		-----		-----	-----		-----	
2160	EN12766B	1.67	G(0.05)	6.57	0.74		3.21	
	normality	OK			OK			
	n	14			16			
	outliers	1			0			
	mean (n)	0.786			0.481			
	st.dev. (n)	0.2861			0.1840			
	R(calc.)	0.801			0.515			
	R(EN12766-1)	0.377			0.226			



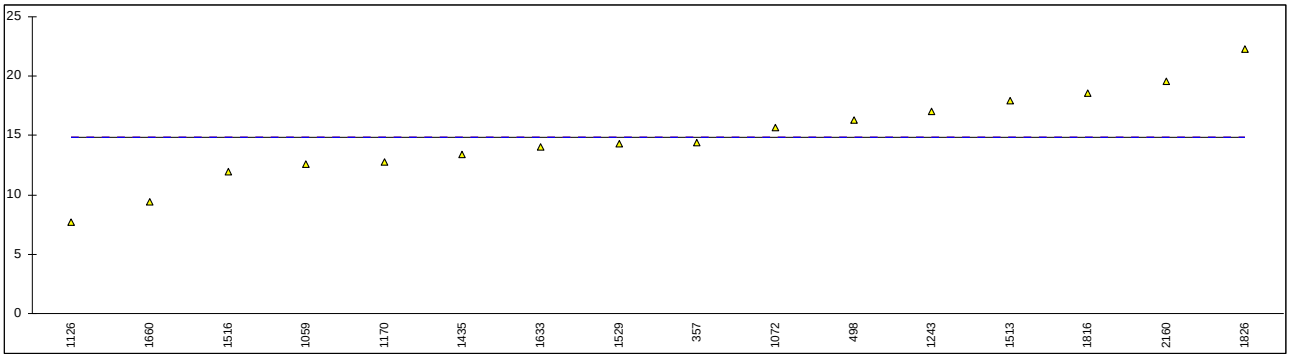
PCB 153



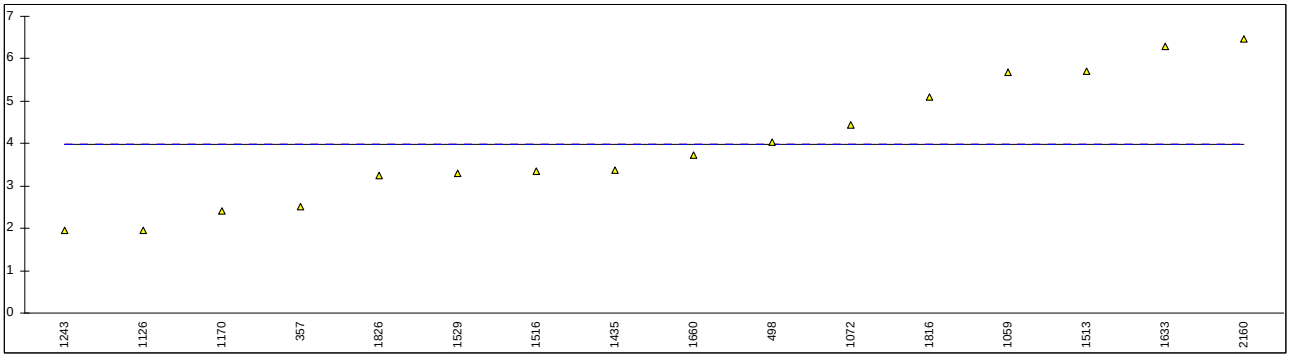
PCB 180

Summation of the 7 individual PCB on samples #0990 and #0991; results in mg/kg.

lab	method	#0990	mark	#0991	mark	remarks
341		-----		-----		
343		-----		-----		
357	calc. by iis	14.38		2.50		
445		-----		-----		
498	calc. by iis	16.29		4.04		
614		-----		-----		
963		-----		-----		
1059	calc. by iis	12.59		5.68		
1072	calc. by iis	15.6967		4.4294		
1126	calc. by iis	7.66		1.96		
1170	IEC61619	12.73		2.4		
1243	calc. by iis	17.01		1.95		
1245		-----		-----		
1258		-----		-----		
1303		-----		-----		
1304		-----		-----		
1306		-----		-----		
1435	calc. by iis	13.38		3.38		
1505		-----		-----		
1513	calc. by iis	17.95		5.7		
1516	calc. by iis	11.98		3.35		
1526		-----		-----		
1529	calc. by iis	14.35		3.30		
1633	calc. by iis	14.05		6.29		
1660	calc. by iis	9.41		3.74		
1801		-----		-----		
1816	calc. by iis	18.59		5.09		
1826	calc. by iis	22.29		3.24		
2122		-----		-----		
2160	calc. by iis	19.55		6.47		
	normality	OK		OK		
	n	16		16		
	outliers	0		0		
	mean (n)	14.869		3.970		
	st.dev. (n)	3.7496		1.4952		
	R(calc.)	10.499		4.186		



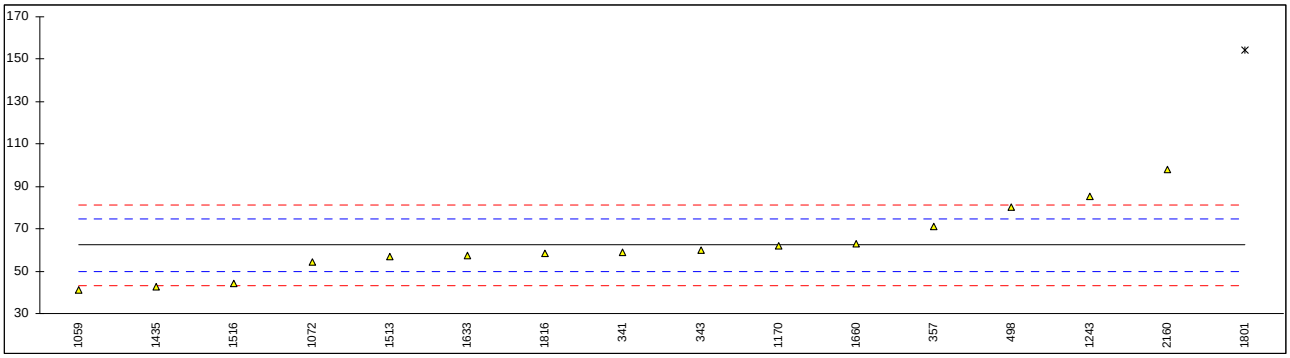
#0990



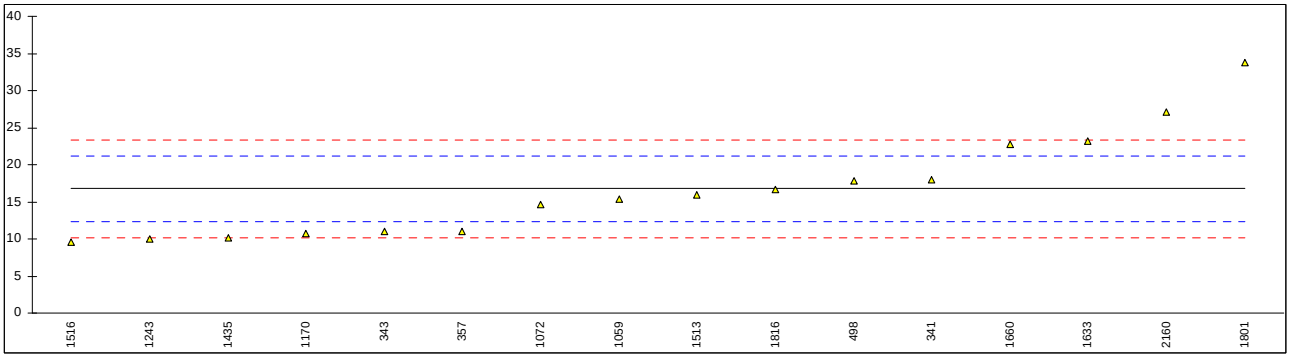
#0991

Determination of the Total PCB on samples #0990 and #0991; results in mg/kg.

lab	method	#0990	mark	z(targ)	#0991	mark	z(targ)	remarks
341	EN61619	58.9		-0.53	17.9		0.52	
343	EN61619	60		-0.36	11		-2.60	
357	EN12766	71.28		1.44	11.02		-2.59	
445		-----		-----	-----		-----	
498	EN61619	80.15		2.86	17.8		0.48	
614		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1059	EN61619	41.08		-3.37	15.37		-0.62	
1072	EN61619	54.1130		-1.30	14.6669		-0.94	
1126		-----		-----	-----		-----	
1170	EN61619	62.15		-0.01	10.77		-2.71	
1243	EN61619	85.5		3.71	10		-3.05	
1245		-----		-----	-----		-----	
1258		-----		-----	-----		-----	
1303		-----		-----	-----		-----	
1304		-----		-----	-----		-----	
1306		-----		-----	-----		-----	
1435	EN61619	42.70		-3.12	10.20		-2.96	
1505		-----		-----	-----		-----	
1513	EN61619	57		-0.84	16		-0.34	
1516	EN61619	44.25		-2.87	9.61		-3.23	
1526		-----		-----	-----		-----	
1529		-----		-----	-----		-----	
1633	EN61619	57.3		-0.79	23.2		2.92	
1660	EN61619	63.1		0.14	22.8		2.74	
1801	EN61619	154.4	G(0.01)	14.70	33.8		7.72	
1816	EN61619	58.3		-0.63	16.7		-0.02	
1826		-----		-----	-----		-----	
2122		-----		-----	-----		-----	
2160	EN12766	97.75		5.66	27.15		4.71	
	normality	OK			OK			
	n	15			16			
	outliers	1			0			
	mean (n)	62.238			16.749			
	st.dev. (n)	15.8295			6.9637			
	R(calc.)	44.323			19.498			
	R(IEC61619:97)	17.560			6.187			



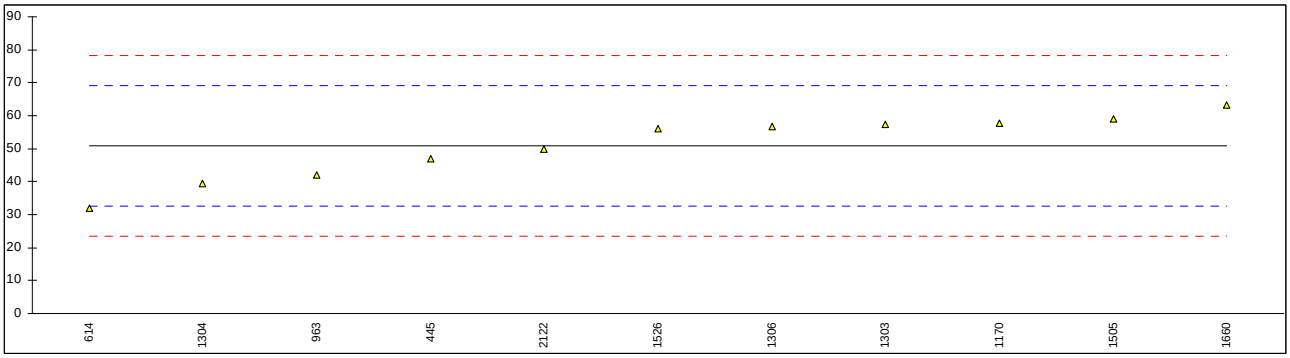
#0990



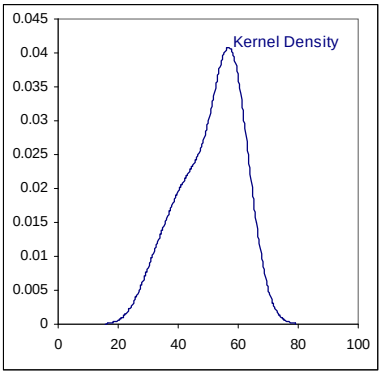
#0991

Determination of Aroclor 1242, 1254 and 1260 on sample #0990; results in mg/kg.

lab	method	1242	mark	z(targ)	1254	mark	z(targ)	1260	mark	z(targ)	Remarks
341		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
357		-----		-----	-----		-----	-----		-----	
445	IEC61619	0.66		-0.92	13.58		-----	46.98		-0.43	
498		-----		-----	-----		-----	-----		-----	
614	D4059	<2		-----	<2		-----	32.04		-2.07	
963	D4059	0.75		-0.75	4.25		-----	42.00		-0.98	
1059		-----		-----	-----		-----	-----		-----	
1072		-----		-----	-----		-----	-----		-----	
1126		-----		-----	-----		-----	-----		-----	
1170	in house	-----		-----	-----		-----	57.67		0.74	
1243		-----		-----	-----		-----	-----		-----	
1245		-----		-----	-----		-----	-----		-----	rep. 13 mg/kg 1262
1258		-----		-----	-----		-----	-----		-----	
1303	in house	1.7		1.03	n.d.		-----	57.3		0.70	
1304	in house	1.49		0.64	1.38		-----	39.46		-1.26	
1306	EPA600/4-81	-----		-----	-----		-----	56.63		0.63	
1435		-----		-----	-----		-----	-----		-----	
1505	D4059	<1		-----	<1		-----	58.9		0.88	
1513		-----		-----	-----		-----	-----		-----	
1516		-----		-----	-----		-----	-----		-----	
1526	EPA600/4-81	-----		-----	-----		-----	56		0.56	
1529		-----		-----	-----		-----	-----		-----	
1633		-----		-----	-----		-----	-----		-----	
1660	D4059	<2		-----	<2		-----	63.1		1.34	
1801		-----		-----	-----		-----	-----		-----	
1816		-----		-----	-----		-----	-----		-----	
1826		-----		-----	-----		-----	-----		-----	
2122	in house	-----		-----	<5		-----	50		-0.10	
2160		-----		-----	-----		-----	-----		-----	
	normality	n.a.			n.a.			OK			
	n	4			3			11			
	outliers	0			0			0			
	mean (n)	1.15			n.a.			50.92			
	st.dev. (n)	0.52			n.a.			9.698			
	R(calc.)	1.462			n.a.			27.15			
	R(D4059:05)	1.488			n.a.			25.54			

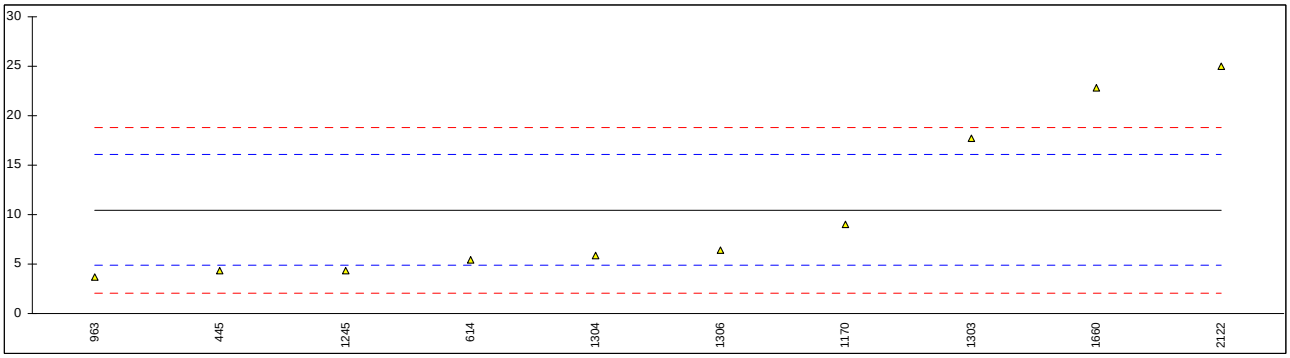


Aroclor 1260

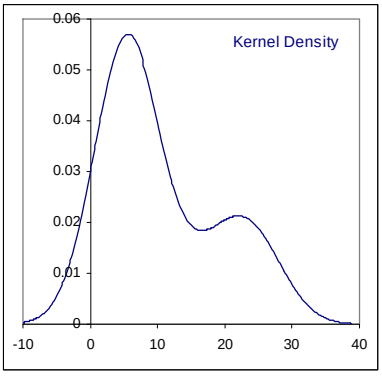


Determination of Aroclor 1242, 1254 and 1260 on sample #0991; results in mg/kg.

lab	method	1242	mark	z(targ)	1254	mark	z(targ)	1260	mark	z(targ)	Remarks
341		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
357		-----		-----	-----		-----	-----		-----	
445	IEC61619	0.42		-----	4.34		-2.20	2.78		-1.63	
498		-----		-----	-----		-----	-----		-----	
614	D4059	<2		-----	5.40		-1.82	6.40		0.45	
963	D4059	0.5		-----	3.75		-2.41	37.50	G(0.01)	18.25	
1059		-----		-----	-----		-----	-----		-----	
1072		-----		-----	-----		-----	-----		-----	
1126		-----		-----	-----		-----	-----		-----	
1170	in house	-----		-----	9.01		-0.52	-----		-----	
1243		-----		-----	-----		-----	-----		-----	
1245		-----		-----	4.36		-2.19	-----		-----	
1258		-----		-----	-----		-----	-----		-----	
1303	in house	n.d.		-----	17.7		2.60	5.9		0.16	
1304	in house	<0.2		-----	5.87		-1.65	5.54		-0.05	
1306	EPA600/4-81	-----		-----	6.42		-1.45	8.14		1.44	
1435		-----		-----	-----		-----	-----		-----	
1505	D4059	<1		-----	<1	false-?	<-3.40	5.8		0.10	
1513		-----		-----	-----		-----	-----		-----	
1516		-----		-----	-----		-----	-----		-----	
1526	EPA600/4-81	-----		-----	-----		-----	5.41		-0.12	
1529		-----		-----	-----		-----	-----		-----	
1633		-----		-----	-----		-----	-----		-----	
1660	D4059	<2		-----	22.8		4.43	<2		<-2.07	false negative?
1801		-----		-----	-----		-----	-----		-----	
1816		-----		-----	-----		-----	-----		-----	
1826		-----		-----	-----		-----	-----		-----	
2122	in house	-----		-----	25		5.22	5		-0.36	
2160		-----		-----	-----		-----	-----		-----	
	normality	n.a.			not OK			OK			
	n	2			10			8			
	outliers	0			0			1			
	mean (n)	n.a.			10.47			5.62			
	st.dev. (n)	n.a.			8.171			1.491			
	R(calc.)	n.a.			22.88			4.18			
	R(D4059:05)	n.a.			7.80			4.89			

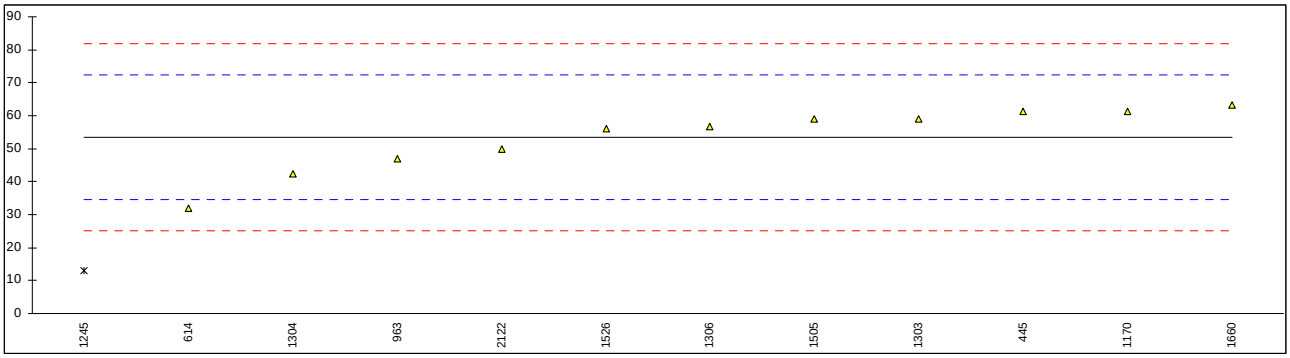


Aroclor 1254

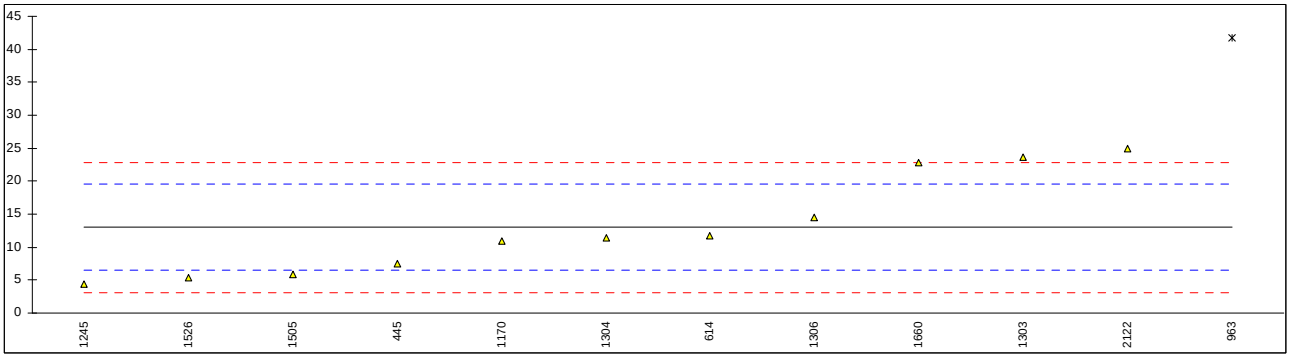


Determination of the Total Aroclor on samples #0990 and #0991; results in mg/kg.

lab	method	#0990	mark	z(targ)	#0991	mark	z(targ)	Remarks
341		-----		-----	-----		-----	
343		-----		-----	-----		-----	
357		-----		-----	-----		-----	
445	calc. by iis	61.22		0.82	7.54		-1.67	
498		-----		-----	-----		-----	
614	D4059	32.04		-2.26	11.80		-0.37	
963	calc. by iis	47.00		-0.68	41.75	G(0.05)	8.76	
1059		-----		-----	-----		-----	
1072		-----		-----	-----		-----	
1126		-----		-----	-----		-----	
1170	in house	61.4		0.84	10.88		-0.65	
1243		-----		-----	-----		-----	
1245	calc. by iis	13	G(0.05)	-4.27	4.36		-2.64	
1258		-----		-----	-----		-----	
1303	in house	59.0		0.59	23.6		3.23	
1304	in house	42.33		-1.17	11.41		-0.49	
1306	EPA600/4-81	56.63		0.34	14.56		0.47	
1435		-----		-----	-----		-----	
1505	D4059	58.9		0.58	5.8		-2.20	
1513		-----		-----	-----		-----	
1516		-----		-----	-----		-----	
1526	calc. by iis	56		0.27	5.41		-2.32	
1529		-----		-----	-----		-----	
1633		-----		-----	-----		-----	
1660	D4059	63.1		1.02	22.8		2.98	
1801		-----		-----	-----		-----	
1816		-----		-----	-----		-----	
1826		-----		-----	-----		-----	
2122	calc. by iis	50		-0.36	25		3.65	
2160		-----		-----	-----		-----	
	normality	OK			OK			
	n	11			11			
	outliers	1			1			
	mean (n)	53.42			13.01			
	st.dev. (n)	9.640			7.597			
	R(calc.)	26.99			21.27			
	R(IEC61619:97)	26.48			9.18			



#0990



#0991

APPENDIX 2

Number of participating laboratories per country

3 labs in	AUSTRALIA
1 lab in	BELGIUM
1 lab in	ESTONIA
1 lab in	FINLAND
2 labs in	GERMANY
2 labs in	GREECE
1 lab in	ITALY
1 lab in	NEW ZEALAND
1 lab in	NORWAY
2 labs in	PORTUGAL
1 lab in	SAUDI ARABIA
1 lab in	SLOVENIA
1 lab in	SOUTH AFRICA
6 labs in	SPAIN
3 labs in	THE NETHERLANDS
1 lab in	TURKEY
2 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
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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/)