

Results of Proficiency Test
PCB in (Mineral) Oil
November 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis13L06

January 2014

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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 2013/2014, it was decided to continue the proficiency test for the PCB analysis on (mineral) oil.

In this interlaboratory study, 45 laboratories from 20 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of the 2013 proficiency test on PCB are presented and discussed. This report is also electronically available through the iis internet site <http://www.iisnl.com>.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. It was decided to send one 8 ml vial with mineral oil contaminated with PCB (labelled #13208) that was donated by one of the participating laboratories. Sample analyses for fit-for-use and homogeneity testing were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

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

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

In this proficiency test only one sample was used. The necessary bulk material for the sample, being contaminated mineral oil (positive on PCBs) was donated by a third party laboratory. After ultrasonic homogenisation, 57 subsamples were transferred to 8 mL amber glass vials, all labelled #13208.

The homogeneity of the subsamples #13208 was checked by determination of the organic chloride content in accordance with UOP779-08 on seven stratified randomly selected samples:

	Organic chloride in mg/kg
sample #13208-1	27
sample #13208-2	28
sample #13208-3	28
sample #13208-4	28
sample #13208-5	28
sample #13208-6	28
sample #13208-7	26

Table 1: homogeneity test results of subsample #13208

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:

	#13208
r (samples)	2.2
reference method	Horwitz
$0.3 \times R_{(\text{reference method})}$	2.3

Table 2: evaluation of the observed repeatability

The repeatability of the test results is in agreement with 0.3 times the estimated reproducibility calculated using the Horwitz equation. Therefore, homogeneity of the samples was assumed. To each of the participating laboratories one vial of 8 mL (labelled #13208) was sent on October 23, 2013.

2.5 STABILITY OF THE SAMPLES

The stability of the oil, packed in the brown glass vials, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Total Organo halogenic Compounds (TOX) and Poly Chlorinated Biphenyls (via seven individual PCBs, via the determination of the total PCB content and via Aroclors) on the sample.

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

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

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1. Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>...' were not used in the statistical evaluation.

First the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test some no problems were encountered during execution. In total eleven participants, reported results after the final reporting date and one participant did not report any results at all. Not all participants were able to report results for all tests. In total 44 participating laboratories reported 254 numerical results. Observed were 6 outlying results, which is 2.4% 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 (see appendix 1). The abbreviations, used in these tables, are listed in appendix 3.

A not normal distribution was observed for PCB 28 and total PCB's (reported). The statistical evaluations of these two sets of test results should be used with due care. For the statistical evaluation of the individual PCBs the method EN12766-1:00 was used, this method is equal to IP462-1:01. For the results on sample #13208, the following was concluded:

TOX This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Individual PCBs: This determination was problematic for five of the seven congeners. For the evaluation of the individual congeners method EN12766-1:00 / IP462-1:01 was used. In the methods IEC61619:98 and DIN51527:93 only the reproducibilities of the total PCB content are mentioned, while in EN12766-1:00 / IP462-1:01 the reproducibilities for each individual congener are mentioned. In total only three statistical outliers were observed. The calculated reproducibilities of five congeners are not in agreement with requirements of EN12766-1:00 / IP462-1:01. Only for PCB no.118 the calculated reproducibility is in full agreement. For PCB no.28 no significant conclusions were drawn as the consensus value was near or below the detection limit.

Individual Aroclors: The determination of the individual Aroclors was problematic. In total two statistical outliers were observed. The calculated reproducibility of each Aroclor after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4059:10 (silicone).

Total PCB: For the determination of the total PCB content, several test methods are available. As the spread of the group of participants does not meet the precision data of the reference method, it was decided in the 2013 proficiency test for PCB to report the total PCB content and how the total content was determined and/or calculated.

After evaluation of the results it appeared that the reported test results for total PCB can be divided into three groups. The total PCB content may be reported as the sum of all PCB congeners according EN12766-2 method A or EN61619. It may be reported as 5 times of the sum of 6 PCB congeners according EN12766-2 method B. Finally, it may also be reported as the sum of all Aroclors according ASTM D4059. Based on the information given by the participants on the report form on "How was the total PCB content determined" the following evaluation was made.

Total PCB's, "sum of all PCB congeners"

This determination and/or calculation of total PCB content was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12766-2 method A:2001 and EN61619:98.

Total PCB's, "5 times of sum 6 PCB congeners"

This determination and/or calculation of total PCB content was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN12766-2 method B:2001. The consensus value of the group was in agreement with the consensus value found with the individual summation of the six congeners calculated by iis (41.4 vs 39.5 mg/kg).

Total PCB's, "sum of all Aroclors"

This determination and/or calculation of total PCB content was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D4059:10 (silicone). However, the spread of the group is not in agreement with ASTM D4059:10 (packed or megabore). The consensus value of the group was in agreement with the consensus value found with the individual summation of the Aroclors calculated by iis (27.6 vs 29.4 mg/kg).

Summary:

All participants agreed that sample #13208 was positive on PCBs. From the data on total organic chloride (TOX) an average concentration of 30.2 mg/kg was calculated. From this concentration a total content of 53.0 mg PCB/kg was estimated using an average Cl content of 57%, assuming the presence of equal amounts of Aroclor 1260 and Aroclor 1254. This content is somewhat higher than the estimated total PCB content using the other methods.

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

	#13208
total PCB content, estimated from 6 congeners, in mg/kg	39.5 - 41.4
total PCB content, using EN12766-2 method A, in mg/kg	31.2
estimated total PCB content using Aroclor method, in mg/kg	27.6 – 29.4
total PCB content, estimated for TOX data, in mg/kg	53.0

Table 3: Comparison of estimations of total PCB content in sample #13208.

The total PCB content as determined by EN12766-2, method A (or IEC61619:98) is in good agreement with the total PCB content as determined by the Aroclor method. The range of all four above estimates for total PCB content is quite acceptable in view of the required precision.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu IEC, EN, or ASTM standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
TOX	mg/kg	5	30.2	16.3	8.1
PCB no. 28	mg/kg	12	0.08	0.10	(0.02)
PCB no. 52	mg/kg	25	0.80	0.58	0.38
PCB no. 101	mg/kg	26	1.67	1.65	0.82
PCB no. 118	mg/kg	21	1.01	0.49	0.49
PCB no. 138	mg/kg	26	2.04	1.38	1.00
PCB no. 153	mg/kg	26	1.97	1.45	0.96
PCB no. 180	mg/kg	26	1.21	0.67	0.59
Aroclor 1242	mg/kg	8	2.75	5.04	2.86
Aroclor 1254	mg/kg	16	14.07	14.64	9.74
Aroclor 1260	mg/kg	17	13.20	10.59	9.28
Total PCB reported	mg/kg	38	33.23	23.90	n.e.

table 4: Performance of the group of participating laboratories on sample #13208

() Values between brackets should be used with due care, see also paragraph 4.1

Without further statistical calculations it can be concluded that for many components there is 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 2013 PROFICIENCY TEST WITH PREVIOUS PTS.

	<i>November 2013</i>	<i>October 2012</i>	<i>November 2011</i>	<i>November 2010</i>
Number of reporting labs	44	41	38	34
Number of results reported	254	204	195	186
Statistical outliers	6	10	4	15
Percentage outliers	2.4%	4.9%	2.0%	8.1%

Table 5: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

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

<i>Determination</i>	<i>November 2013</i>	<i>October 2012</i>	<i>November 2011</i>	<i>November 2010</i>
TOX	--	n.e.	n.e	n.e.
PCB (individual)	--	--	+/-	-
Aroclor (individual)	--	-	+/-	--

Table 6: comparison of observed precisions against standard requirements

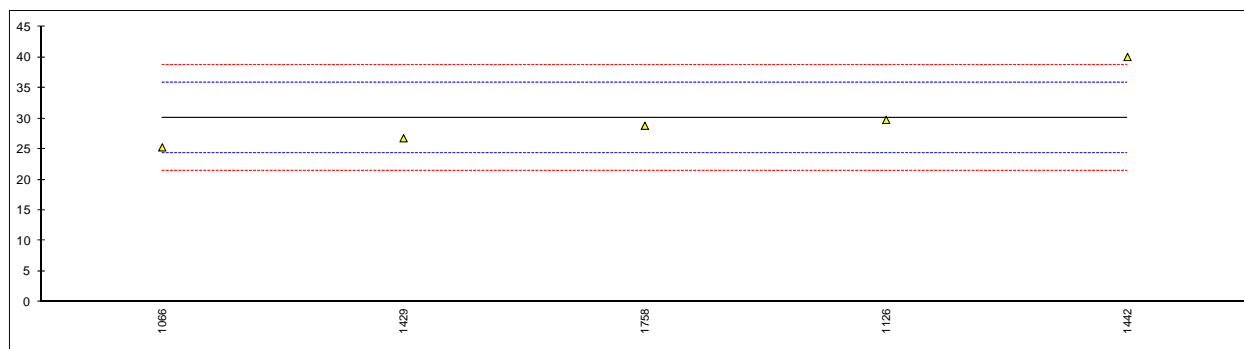
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Total Organohalogenic Compounds (TOX) on sample #13208; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357		----		----	
398		----		----	
445		----		----	
498		----		----	
614		----		----	
1059		----		----	
1066	UOP779	25.3		-1.68	
1072		----		----	
1126	EN7425	29.8		-0.13	
1170		----		----	
1201		----		----	
1243		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1352		----		----	
1358		----		----	
1367		----		----	
1374		----		----	
1383		----		----	
1396		----		----	
1429	D7359	26.8		-1.17	
1435		----		----	
1440		----		----	
1442	ISO10304/ISO14582	40.1		3.44	
1458		----		----	
1495		----		----	
1505		----		----	
1513		----		----	
1529		----		----	
1548		----		----	
1568		----		----	
1660		----		----	
1690		----		----	
1743		----		----	
1758	in house	28.84		-0.46	
1763		----		----	
1765		----		----	
1801		----		----	
1816		----		----	
2122		----		----	
2493		----		----	
3195		----		----	
normality		OK			
n		5			
outliers		0			
mean (n)		30.17			
st.dev. (n)		5.822			
R(calc.)		16.30			
R(Horwitz)		8.09			



Determination of PCB 28, 52 and 101 on sample #13208; results in mg/kg.

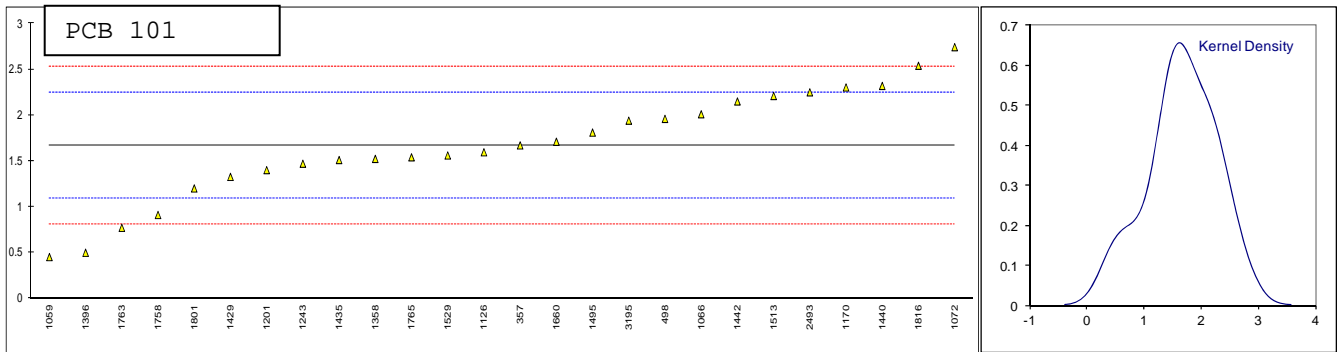
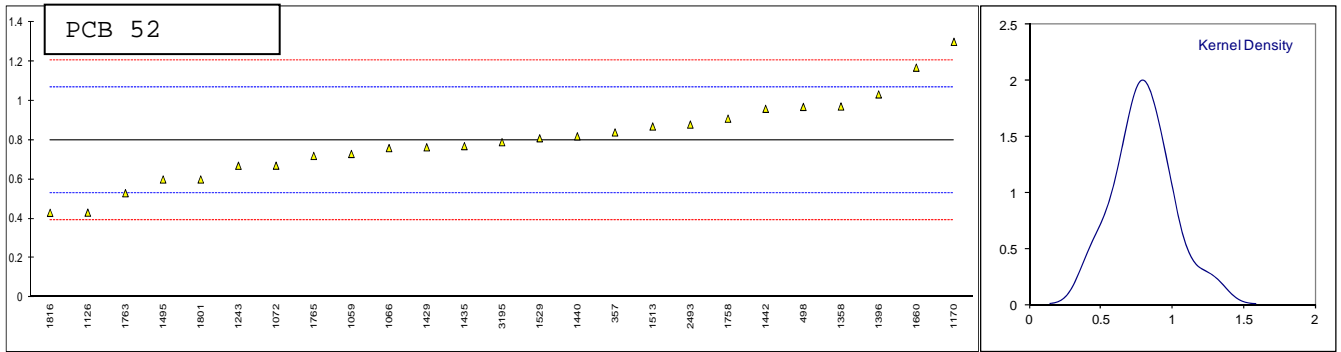
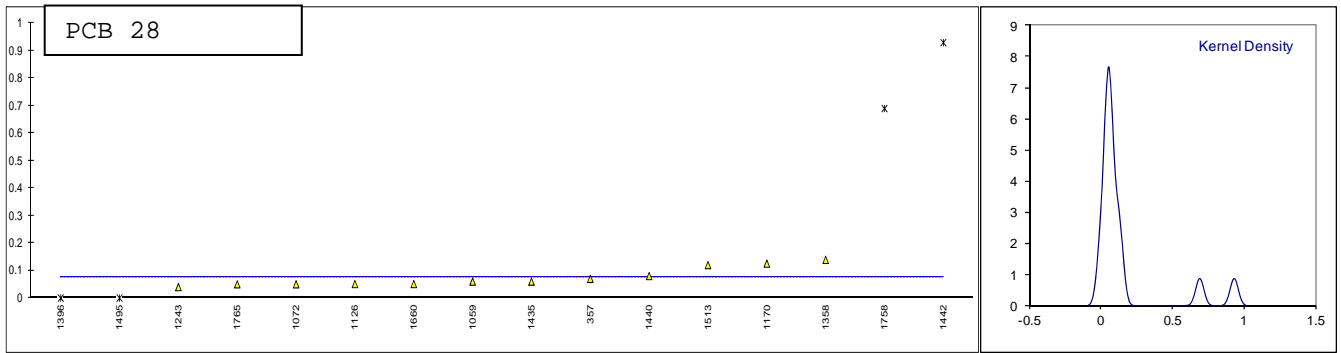
lab	method	No.28	mark	z(targ)	No.52	mark	z(targ)	No.101	mark	z(targ)	Remarks
341		----		----	----		----	----		----	
343		----		----	----		----	----		----	
357	EN12766-B	0.07		----	0.84		0.29	1.67		0.01	
398		----		----	----		----	----		----	
445		----		----	----		----	----		----	
498	EN12766-B	<0.30		----	0.97		1.25	1.96		1.00	
614		----		----	----		----	----		----	
1059	EN12766-1	0.06		----	0.73		-0.52	0.45		-4.18	
1066	EN12766-A	<0.2		----	0.76		-0.30	2.01		1.17	
1072	EN61619	0.0502		----	0.6706		-0.96	2.7432		3.69	
1126	EN12766	0.051		----	0.431		-2.72	1.595		-0.25	
1170	EN12766-B	0.1255		----	1.3015		3.70	2.3032		2.18	
1201	EN12766	<0.5		----	<0.5		----	1.4		-0.92	
1243	EN12766-B	0.04		----	0.67		-0.96	1.47		-0.68	
1303		----		----	----		----	----		----	
1304		----		----	----		----	----		----	
1306		----		----	----		----	----		----	
1352		----		----	----		----	----		----	
1358	IP462-2-A	0.139	C	----	0.972	C	1.27	1.523	C	-0.50	
1367		----		----	----		----	----		----	
1374		----		----	----		----	----		----	
1383		----		----	----		----	----		----	
1396	IP462-B	0	ex	----	1.03363		1.72	0.497439		-4.02	
1429	EN12766-B	<0.2		----	0.764		-0.27	1.326		-1.17	
1435	EN12766-A	0.06		----	0.77		-0.22	1.51		-0.54	
1440	EN12766-A	0.08		----	0.82		0.15	2.32		2.24	
1442	EN12766-A	0.93	C,G(0.01)	----	0.96		1.18	2.15		1.66	
1458		----		----	----		----	----		----	
1495	EN12766-B	0	ex	----	0.60		-1.48	1.81		0.49	
1505		----		----	----		----	----		----	
1513	IEC61619-A	0.12		----	0.87		0.52	2.21		1.86	
1529	EN12766-1	<0.10		----	0.81		0.07	1.56		-0.37	
1548		----		----	----		----	----		----	
1568		----		----	----		----	----		----	
1660	IEC61619	0.051		----	1.17		2.73	1.71		0.14	
1690		----		----	----		----	----		----	
1743		----		----	----		----	----		----	
1758	EN12766Mod-B	0.69	C,G(0.01)	----	0.91		0.81	0.91		-2.60	
1763	EN12766-B	<0.2		----	0.53		-1.99	0.77		-3.08	
1765	EN61619	0.05		----	0.72		-0.59	1.54		-0.44	
1801	IEC61619	n.d.		----	0.6		-1.48	1.2		-1.61	
1816	IEC61619	----		----	0.43		-2.73	2.54		3.00	
2122		----		----	----		----	----		----	
2493	EN12766-B	<0.5		----	0.88		0.59	2.25		2.00	
3195	EN12766	<0.1		----	0.79		-0.07	1.94		0.93	
	normality	not OK			OK			OK			
	n	12			25			26			
	outliers	2	+ 2 excl		0			0			
	mean (n)	0.075			0.800			1.668			
	st.dev. (n)	0.0341			0.2069			0.5889			
	R(calc.)	0.095			0.579			1.649			
	R(EN12766-1:00)	(0.016)			0.380			0.815			

Lab 1358: first reported 1.37, 1.99, 1.22

Lab 1442: first reported 0.60

Lab 1758: first reported 0.37

ex = excluded, zero is not a real value

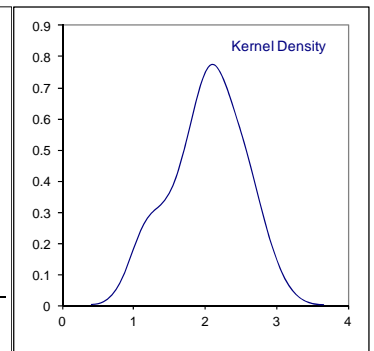
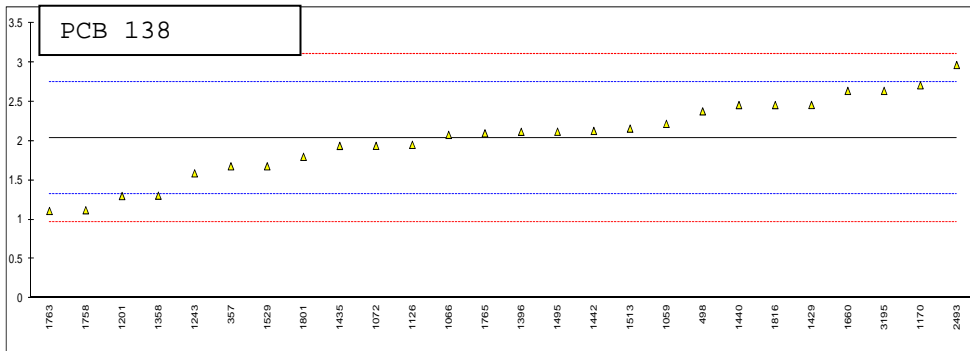
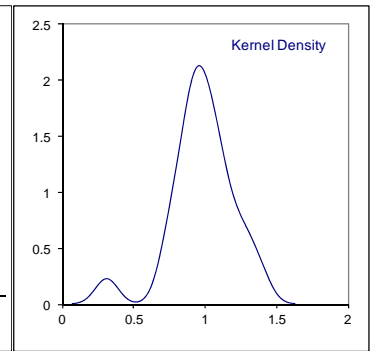
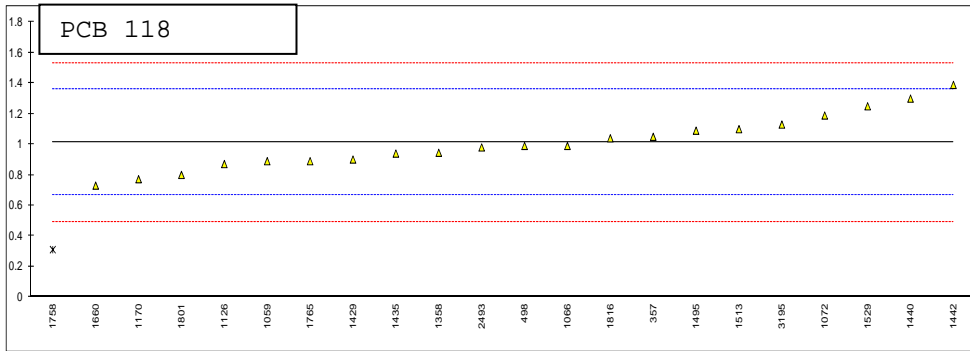


Determination of PCB 118 and 138 on sample #13208; results in mg/kg.

lab	method	No.118	mark	z(targ)	No.138	mark	z(targ)	Remarks
341		----		----	----		----	
343		----		----	----		----	
357	EN12766-B	1.05		0.22	1.68		-1.01	
398		----		----	----		----	
445		----		----	----		----	
498	EN12766-B	0.99		-0.12	2.38		0.95	
614		----		----	----		----	
1059	EN12766-1	0.89		-0.70	2.22		0.50	
1066	EN12766-A	0.99		-0.12	2.08		0.11	
1072	EN61619	1.1895		1.03	1.9403		-0.28	
1126	EN12766	0.871		-0.81	1.953		-0.25	
1170	EN12766-B	0.7719		-1.38	2.7120		1.87	
1201	EN12766	<0.5		<-2.94	1.3		-2.07	
1243		----		----	1.59		-1.26	
1303		----		----	----		----	
1304		----		----	----		----	
1306		----		----	----		----	
1352		----		----	----		----	
1358	IP462-2-A	0.945	C	-0.38	1.305	C	-2.06	
1367		----		----	----		----	
1374		----		----	----		----	
1383		----		----	----		----	
1396		----		----	2.11859		0.22	
1429	EN12766-B	0.901		-0.64	2.461		1.17	
1435	EN12766-A	0.94		-0.41	1.94		-0.28	
1440	EN12766-A	1.30		1.66	2.46		1.17	
1442	EN12766-A	1.39		2.18	2.13		0.25	
1458		----		----	----		----	
1495	EN12766-B	1.09		0.45	2.12		0.22	
1505		----		----	----		----	
1513	IEC61619-A	1.10		0.51	2.16		0.33	
1529	EN12766-1	1.25		1.38	1.68		-1.01	
1548		----		----	----		----	
1568		----		----	----		----	
1660	IEC61619	0.73		-1.62	2.64		1.67	
1690		----		----	----		----	
1743		----		----	----		----	
1758	EN12766Mod-B	0.31	C,G(0.05)	-4.04	1.12		-2.57	
1763		----		----	1.11		-2.60	
1765	EN61619	0.89		-0.70	2.10		0.16	
1801	IEC61619	0.8		-1.22	1.8		-0.67	
1816	IEC61619	1.04		0.17	2.46		1.17	
2122		----		----	----		----	
2493	EN12766-B	0.98		-0.18	2.97		2.59	
3195	EN12766	1.13		0.68	2.64		1.67	
	normality	OK			OK			
	n	21			26			
	outliers	1			0			
	mean (n)	1.011			2.041			
	st.dev. (n)	0.1732			0.4940			
	R(calc.)	0.485			1.383			
	R(EN12766-1:00)	0.486			1.002			

Lab 1358: first reported 1.84, 2.38

Lab 1758: first reported 0.25

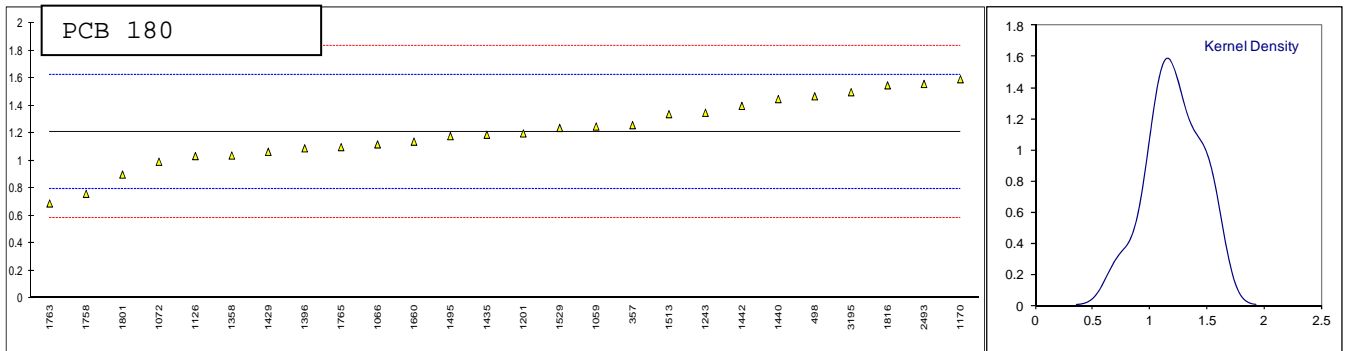
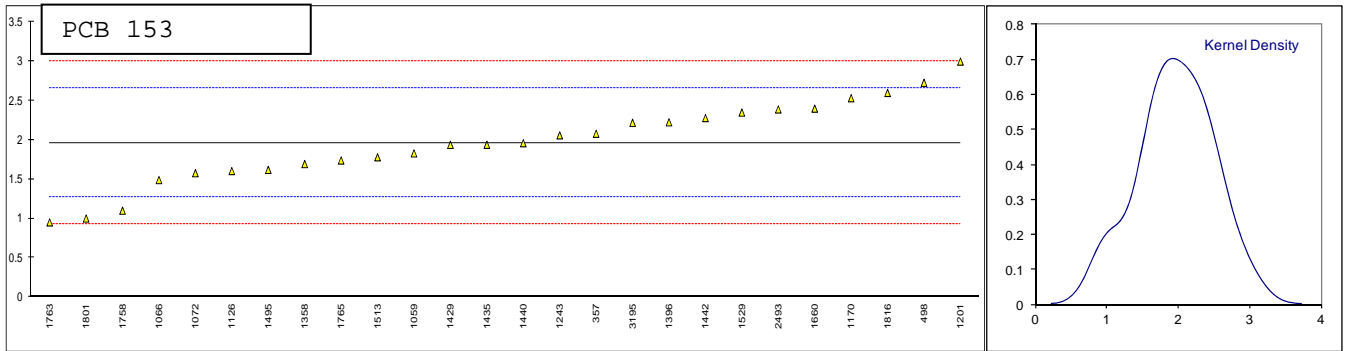


Determination of PCB 153 and 180 on sample #13208; results in mg/kg.

lab	method	No.153	mark	z(targ)	No.180	mark	z(targ)	Remarks
341		----		----	----		----	
343		----		----	----		----	
357	EN12766-B	2.08		0.33	1.26		0.24	
398		----		----	----		----	
445		----		----	----		----	
498	EN12766-B	2.73		2.22	1.47		1.24	
614		----		----	----		----	
1059	EN12766-1	1.83		-0.39	1.25		0.19	
1066	EN12766-A	1.49		-1.38	1.12		-0.43	
1072	EN61619	1.5776		-1.13	0.9936		-1.04	
1126	EN12766	1.605		-1.05	1.035		-0.84	
1170	EN12766-B	2.5334		1.65	1.5942		1.84	
1201	EN12766	3.0		3.00	1.2		-0.05	
1243	EN12766-B	2.06		0.28	1.35		0.67	
1303		----		----	----		----	
1304		----		----	----		----	
1306		----		----	----		----	
1352		----		----	----		----	
1358	IP462-2-A	1.694	C	-0.79	1.039	C	-0.82	
1367		----		----	----		----	
1374		----		----	----		----	
1383		----		----	----		----	
1396	IP462-B	2.22631		0.76	1.09124		-0.57	
1429	EN12766-B	1.939		-0.08	1.066		-0.69	
1435	EN12766-A	1.94		-0.07	1.19		-0.10	
1440	EN12766-A	1.96		-0.02	1.45		1.15	
1442	EN12766-A	2.28		0.91	1.40		0.91	
1458		----		----	----		----	
1495	EN12766-B	1.62		-1.00	1.18		-0.15	
1505		----		----	----		----	
1513	IEC61619-A	1.78		-0.54	1.34		0.62	
1529	EN12766-1	2.35		1.12	1.24		0.14	
1548		----		----	----		----	
1568		----		----	----		----	
1660	IEC61619	2.40		1.26	1.14		-0.34	
1690		----		----	----		----	
1743		----		----	----		----	
1758	EN12766Mod-B	1.10		-2.51	0.76	C	-2.15	
1763	EN12766-B	0.95		-2.95	0.69		-2.49	
1765	EN61619	1.74		-0.65	1.10		-0.53	
1801	IEC61619	1.0		-2.80	0.9		-1.48	
1816	IEC61619	2.60		1.84	1.55		1.62	
2122		----		----	----		----	
2493	EN12766-B	2.39		1.23	1.56		1.67	
3195	EN12766	2.22		0.74	1.50		1.39	
	normality	OK			OK			
	n	26			26			
	outliers	0			0			
	mean (n)	1.965			1.210			
	st.dev. (n)	0.5164			0.2375			
	R(calc.)	1.446			0.665			
	R(EN12766-1:00)	0.964			0.585			

Lab 1358: first reported 0.84, 1.51

Lab 1758: first reported 0.76



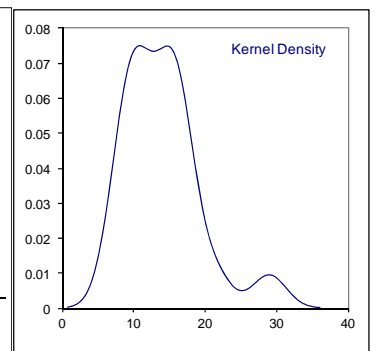
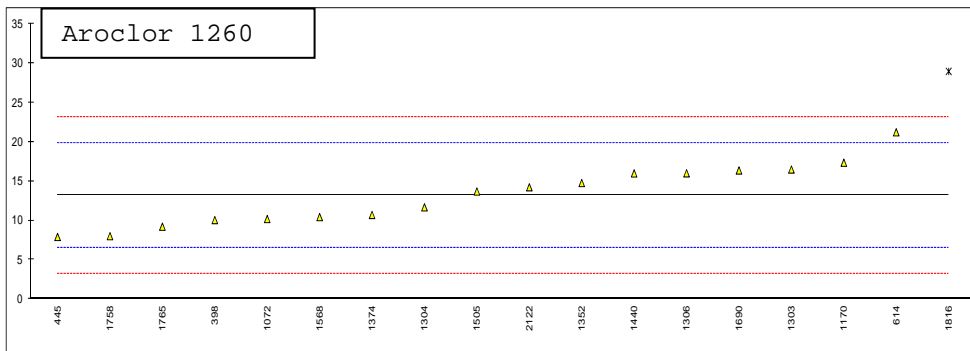
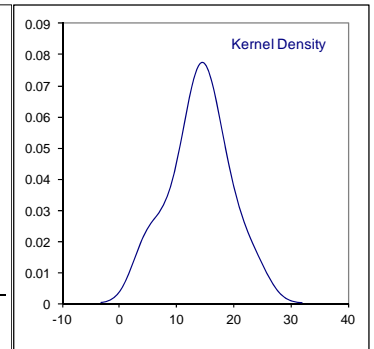
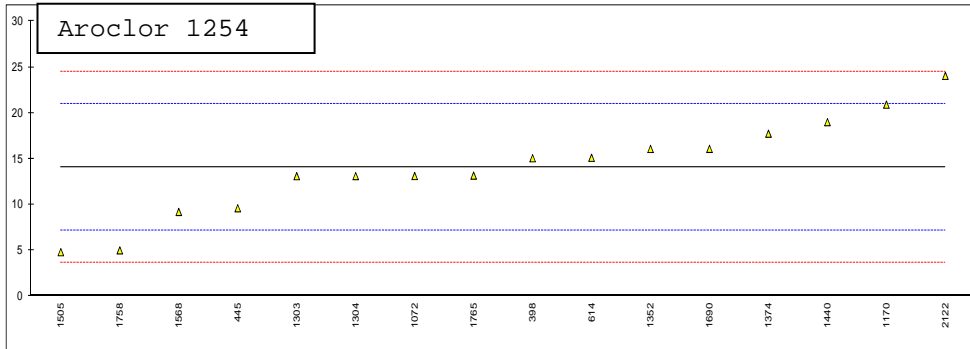
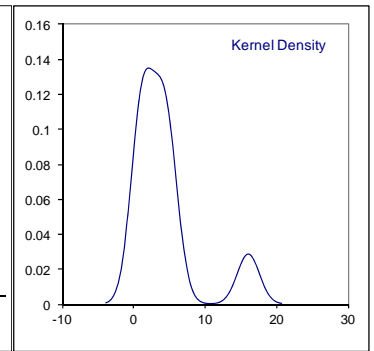
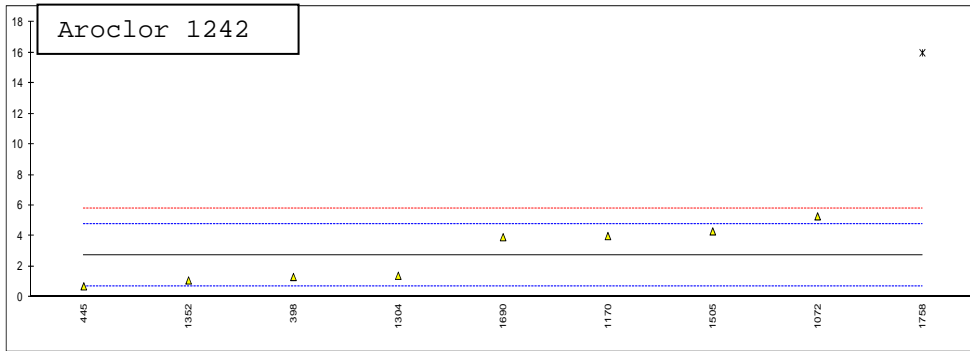
Determination of Aroclor 1242, 1254 and 1260 on sample #13208; results in mg/kg.

lab	method	No. 1242	mark	z(targ)	No. 1254	mark	z(targ)	No. 1260	mark	z(targ)	Remarks
341		----		----	----		----	----		----	
343		----		----	----		----	----		----	
357		----		----	----		----	----		----	
398	D4059	1.31		-1.41	15.06		0.28	10.05		-0.95	
445	D4059	0.7		-2.00	9.6		-1.29	7.9		-1.60	
498		----		----	----		----	----		----	
614	D4059	<2		----	15.11		0.30	21.24		2.43	
1059		----		----	----		----	----		----	
1066		----		----	----		----	----		----	
1072	D4059	5.285		2.48	13.116		-0.27	10.188		-0.91	
1126		----		----	----		----	----		----	
1170	D4059	3.9911		1.22	20.9122		1.97	17.3648		1.26	
1201		----		----	----		----	----		----	
1243		----		----	----		----	----		----	
1303	in house	<1		----	13.1		-0.28	16.5		1.00	
1304	INH-127	1.39		-1.33	13.11	C	-0.28	11.68		-0.46	
1306		----		----	----		----	16.0149		0.85	
1352	INH-1767	1.084		-1.63	16.073		0.58	14.771		0.47	
1358		----		----	----		----	----		----	
1367		----		----	----		----	----		----	
1374	D4059	n.d.		----	17.74		1.06	10.71		-0.75	
1383		----		----	----		----	----		----	
1396		----		----	----		----	----		----	
1429		----		----	----		----	----		----	
1435		----		----	----		----	----		----	
1440	in house	<2		----	19		1.42	16		0.85	
1442		----		----	----		----	----		----	
1458		----		----	----		----	----		----	
1495		----		----	----		----	----		----	
1505	D4059	4.3	C	1.52	4.8	C	-2.67	13.7	C	0.15	
1513		----		----	----		----	----		----	
1529		----		----	----		----	----		----	
1548		----		----	----		----	----		----	
1568	D4059	n.d.		----	9.196		-1.40	10.436		-0.83	
1660		----		----	----		----	----		----	
1690	D4059	3.92		1.15	16.08		0.58	16.38		0.96	
1743		----		----	----		----	----		----	
1758	EN61619	16	C,G(0.01)	12.98	5		-2.61	8		-1.57	
1763		----		----	----		----	----		----	
1765		----		----	13.16		-0.26	9.21		-1.20	
1801		----		----	----		----	----		----	
1816		----		----	----		----	29.0	G(0.05)	4.77	
2122		----		----	24.06155		2.87	14.23205		0.31	
2493		----		----	----		----	----		----	
3195		----		----	----		----	----		----	
	normality	OK			OK			OK			
	n	8			16			17			
	outliers	1			0			1			
	mean (n)	2.748			14.070			13.199			
	st.dev. (n)	1.7984			5.2286			3.7810			
	R(calc.)	5.035			14.640			10.587			Column
	R(D4059:10)	2.860			9.735			9.279			type
	Compare										Silicone
	R(D4059:10)	2.198			7.483			7.132			Packed
	R(D4059:10)	1.686			5.739			5.470			Megabore

Lab 1304: first reported 0.00

Lab 1505: first reported 5.5, <1.0, 16.3

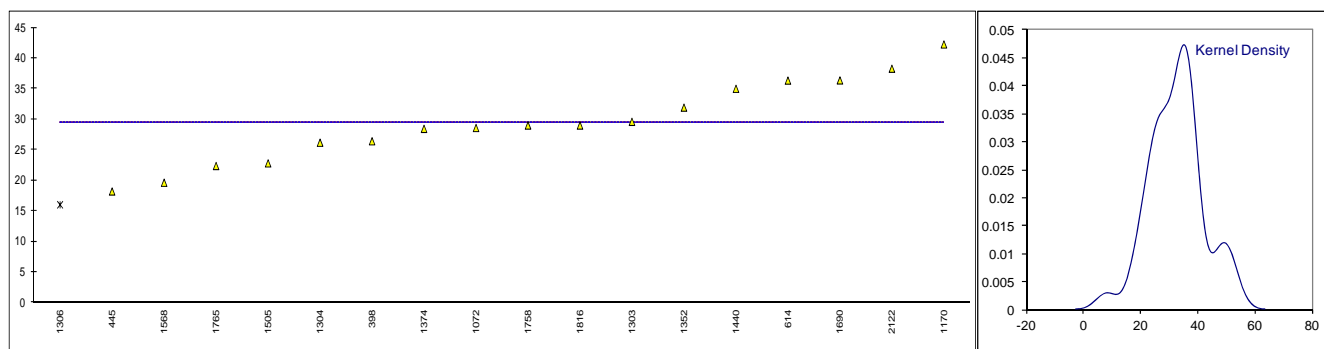
Lab 1758: first reported 15



Determination of Total PCB (reported) on sample #13208; results in mg/kg.

lab	method	value	Mark	z(targ)	remarks
341	EN61619	36		----	
343	EN61619	33		----	
357	EN12766-B	38.00		----	
398	D4059	26.42		----	
445		18.3		----	
498	EN12766B	48.86		----	
614		36.35		----	
1059	EN12766-1/2	24.4		----	
1066		37.3		----	
1072	EN61619	25.589		----	
1126		----		----	
1170		52.85		----	
1201		8.0	G(0.01)	----	
1243		35.9		----	
1303	in house	29.6		----	
1304		26.18	C	----	First reported 13.06
1306		----		----	
1352		31.928		----	
1358		37.95	C	----	First reported 145.02
1367	IEC61619	34.45		----	
1374	D4059	28.45		----	
1383	IP462	37.59		----	
1396		34.836		----	
1429	EN12766B	37.78		----	
1435	IEC61619	27.32		----	
1440	EN12766A	35		----	
1442		49.3	C	----	First reported 47.6
1458		----		----	
1495	EN12766B	36.69		----	
1505	D4059	22.8	C	----	First reported 21.8
1513		----		----	
1529	EN12766-1	30		----	
1548	IEC61619	37.2		----	
1568		19.632		----	
1660	IEC61619	32.05		----	
1690	D4059	36.60		----	
1743	IEC61619	26.7		----	
1758		24.4		----	
1763		20.25		----	
1765	EN61619	24.58		----	
1801		----		----	
1816	IEC61619	32.8		----	
2122		----		----	
2493		50.25	C	----	First reported 55.13
3195	IEC61619	45.45		----	

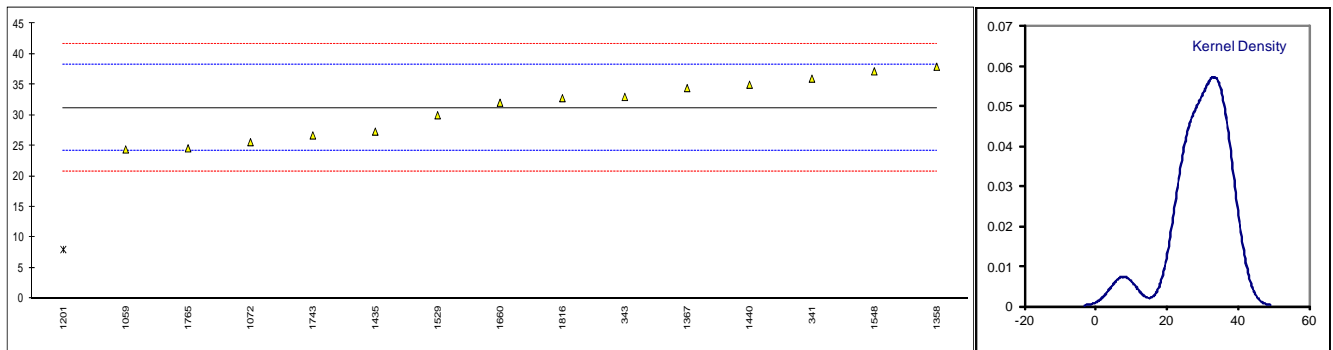
normality not OK
n 38
outliers 1
mean (n) 33.230
st.dev. (n) 8.5365
R(calc.) 23.902
R(lit) n.a.



Evaluation of Total PCB, only "sum of all PCB congeners" on sample #13208; results in mg/kg.

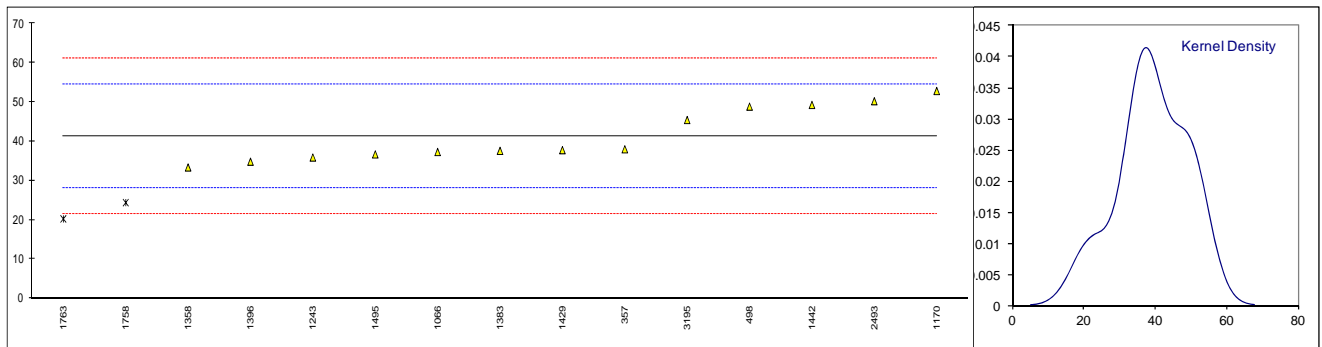
lab	method	value	mark	z(target)	remarks
341	EN61619	36		1.37	
343	EN61619	33		0.51	
357		----		----	
398		----		----	
445		----		----	
498		----		----	
614		----		----	
1059	EN12766-1/2	24.4		-1.95	
1066		----		----	
1072	EN61619	25.589		-1.61	
1126		----		----	
1170		----		----	
1201		8.0	G(0.01)	-6.63	probably sum of 7 congeners only?
1243		----		----	
1303		----		----	
1304		----		----	
1306		----		----	
1352		----		----	
1358	IP462-2	37.95	C	1.92	first reported 145.02
1367	IEC61619	34.45		0.92	
1374		----		----	
1383		----		----	
1396		----		----	
1429		----		----	
1435	IEC61619	27.32		-1.11	
1440	EN12766A	35		1.08	
1442		----		----	
1458		----		----	
1495		----		----	
1505		----		----	
1513		----		----	
1529	EN12766-1	30		-0.35	
1548	IEC61619	37.2		1.71	
1568		----		----	
1660	IEC61619	32.05		0.24	
1690		----		----	
1743	IEC61619	26.7		-1.29	
1758		----		----	
1763		----		----	
1765	EN61619	24.58		-1.90	
1801		----		----	
1816	IEC61619	32.8		0.45	
2122		----		----	
2493		----		----	
3195		----		----	

normality OK
n 14
outliers 1
mean (n) 31.217
st.dev. (n) 4.7589
R(calc.) 13.325
R(EN61619:98) 9.804 R(EN12766-2:A) = 9.804



Evaluation of Total PCB, only "5*sum of 6 PCB congeners" on sample #13208; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357	EN12766-B	38.00		-0.51	
398		----		----	
445		----		----	
498	EN12766-B	48.86	E	1.13	calculated by iis: 47.55
614		----		----	
1059		----		----	
1066		37.3		-0.62	
1072		----		----	
1126		----		----	
1170		52.85		1.73	
1201		----		----	
1243		35.9		-0.83	
1303		----		----	
1304		----		----	
1306		----		----	
1352		----		----	
1358		33.36		-1.22	
1367		----		----	
1374		----		----	
1383	IP462	37.59		-0.58	
1396		34.836		-0.99	
1429	EN12766-B	37.78		-0.55	
1435		----		----	
1440		----		----	
1442		49.3	C	1.20	first reported 47.6
1458		----		----	
1495	EN12766-B	36.69		-0.71	
1505		----		----	
1513		----		----	
1529		----		----	
1548		----		----	
1568		----		----	
1660		----		----	
1690		----		----	
1743		----		----	
1758	EN12766-B	24.4	ex	-2.57	is total for originally reported test results.
1763	EN12766-B	20.25	G(0.05)	-3.20	
1765		----		----	
1801		----		----	
1816		----		----	
2122		----		----	
2493	EN12766-B	50.25	C	1.34	first reported 55.13
3195	IEC61619	45.45		0.61	
Normality		not OK			
n		13			
outliers		1	+1 excl		
mean (n)		41.397			
st.dev. (n)		6.8316			
R(calc.)		19.128			
R(EN12766-2:B)		18.494			

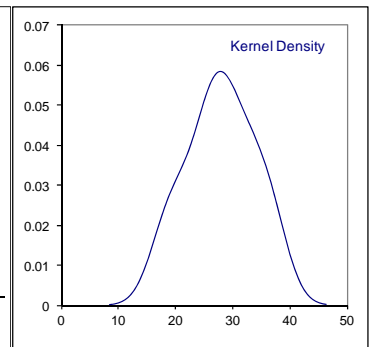
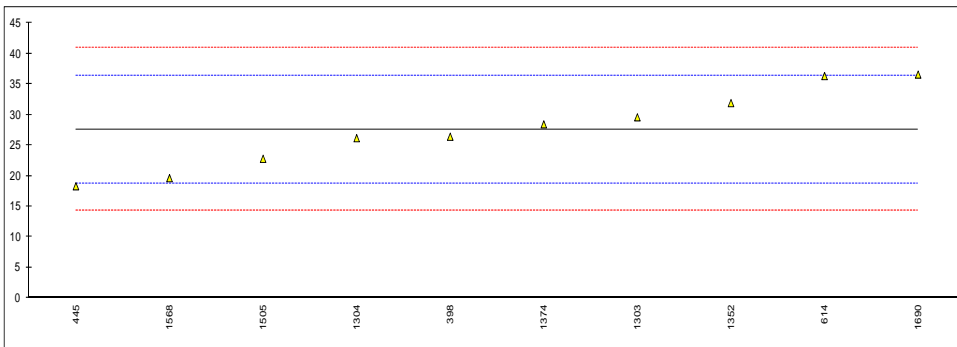


Evaluation of Total PCB, only "sum of all Aroclors" on sample #13208; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
341		----		----	
343		----		----	
357		----		----	
398	D4059	26.42		-0.27	
445		18.3		-2.10	
498		----		----	
614		36.35		1.97	
1059		----		----	
1066		----		----	
1072		----		----	
1126		----		----	
1170		----		----	
1201		----		----	
1243		----		----	
1303	in house	29.6		0.45	
1304		26.18	C	-0.33	first reported 13.06
1306		----		----	
1352		31.928		0.97	
1358		----		----	
1367		----		----	
1374	D4059	28.45		0.19	
1383		----		----	
1396		----		----	
1429		----		----	
1435		----		----	
1440		----		----	
1442		----		----	
1458		----		----	
1495		----		----	
1505	D4059	22.8	C	-1.09	first reported 21.8
1513		----		----	
1529		----		----	
1548		----		----	
1568		19.632		-1.80	
1660		----		----	
1690	D4059	36.60		2.02	
1743		----		----	
1758		----		----	
1763		----		----	
1765		----		----	
1801		----		----	
1816		----		----	
2122		----		----	
2493		----		----	
3195		----		----	

normality OK
n 10
outliers 0
mean (n) 27.626
st.dev. (n) 6.3020
R(calc.) 17.646
R(D4059:10) 16.309

Compare R(D4059:10 – megabore) = 9.615
Compare R(D4059:10 – packed) = 12.412



APPENDIX 2

Total PCB by summation of the reported results by iis on sample #13208; results in mg/kg.

lab	method	Sum of 6 congeners	mark	5 x (sum of 6 congeners)	mark	sum of Aroclors	
341		----		----		----	
343		----		----		----	
357	calc by iis	7.6		38		----	
398		----		----		26.42	
445		----		----		18.2	
498	calc by iis	9.51		47.55		----	
614		----		----		36.35	
1059	calc by iis	6.54		32.7		----	
1066	calc by iis	7.46		37.3		----	
1072	calc by iis	7.9755		39.8775		28.589	
1126	calc by iis	6.67		33.35		----	
1170	calc by iis	10.5698		52.849		42.2681	
1201	calc by iis	6.9		34.5		----	
1243	calc by iis	7.18		35.9		----	
1303		----		----		29.6	
1304		----		----		26.18	
1306		----		----		16.0149	G(0.05)
1352		----		----		31.928	
1358	calc by iis	6.672		33.36		----	
1367		----		----		----	
1374		----		----		28.45	
1383		----		----		----	
1396	calc by iis	6.967209		34.836045		----	
1429	calc by iis	7.556		37.78		----	
1435	calc by iis	7.41		37.05		----	
1440	calc by iis	9.09		45.45		35	
1442	calc by iis	9.85		49.25		----	
1458		----		----		----	
1495	calc by iis	7.33		36.65		----	
1505		----		----		22.8	
1513	calc by iis	8.48		42.4		----	
1529	calc by iis	7.64		38.2		----	
1548		----		----		----	
1568		----		----		19.632	
1660	calc by iis	9.111		45.555		----	
1690		----		----		36.38	
1743		----		----		----	
1758	calc by iis	5.49		27.45		29	
1763	calc by iis	4.05	G(0.05)	20.25	G(0.05)	----	
1765	calc by iis	7.25		36.25		22.37	
1801	calc by iis	5.5		27.5		----	
1816	calc by iis	9.58		47.9		29	
2122		----		----		38.2936	
2493	calc by iis	10.05		50.25		----	
3195	calc by iis	9.09		45.45		----	
	normality	OK		OK		OK	
	n	25		25		17	
	outliers	1		1		1	
	mean (n)	7.90		39.49		29.44	
	st.dev. (n)	1.383		6.914		6.682	
	R(calc.)	3.87		19.36		18.71	
	R(EN12766-2-B:01)	---		17.62		---	
	R(D4059:10-silicone)	---		---		16.94	

APPENDIX 3

Number of participating laboratories per country

6 labs in AUSTRALIA
1 lab in BELGIUM
1 lab in CROATIA
1 lab in FINLAND
2 labs in FRANCE
4 labs in GERMANY
1 lab in GREECE
1 lab in HUNGARY
2 labs in ITALY
1 lab in MEXICO
1 lab in MONTENEGRO
1 lab in NORWAY
1 lab in PORTUGAL
1 lab in SERBIA
2 labs in SLOVENIA
1 lab in SOUTH AFRICA
6 labs in SPAIN
4 labs in THE NETHERLANDS
1 lab in U.S.A.
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/>)