

# Results of Proficiency Test Brominated Flame retardants September 2013

Organised by: Institute for Interlaboratory Studies  
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## 1 INTRODUCTION

Worldwide, many consumer products with plastic parts are produced that contain brominated compounds as flame retardants. These brominated compounds are exceptionally effective at fire prevention.

Since the 1990s scientists have questioned the safety of the Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyls Ethers (PBDE), that bioaccumulate in blood, breast milk and fat tissues. As of June 1, 2006 the State of California began prohibiting the manufacture, distribution, and processing of flame retardant products containing pentabromodiphenyl ether (penta-BDE) and octabromodiphenyl ether (octa-BDE). The European Union decided to ban the use of both PBB and PBDE in electric and electronic devices. This ban was formalised in the RoHS Directive, and an upper limit of 1000 mg/kg for the sum of PBB and PBDE was set.

In February 2009, the Institute for Reference Materials and Measurements (IRMM) released the first certified reference materials (CRMs) to help analytical laboratories better detect these two classes of flame retardants. The certification study of 2007 was followed by an interlaboratory study in 2011. The test material used in this exercise was the earlier produced CRM. To avoid easy recognition by participants, the material was relabelled before use. Twenty-five laboratories from 15 countries participated in the exercise, from which 23 reported results.

A proficiency testing scheme (laboratory-evaluating interlaboratory study) for the determination of PBB and PBDE was started by the Institute for Interlaboratory Studies in 2009.

On request of several participants it was decided to continue the interlaboratory study for the determination of PBB and PBDE. In the interlaboratory study of September 2013, 79 laboratories from 21 different countries participated (See appendix 3). In this report, the results of the 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. It was decided to send 2 different plastic samples which are clearly positive on a number of brominated flame retardants and labelled #13152 and #13153 respectively. Participants were also requested to report some details of the methods used.

### 2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also [www.RVA.nl](http://www.RVA.nl)). 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 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). This protocol can be downloaded via the FAQ page of the iis website <http://www.iisnl.com>.

## 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

Two different samples were selected. The first material (#13152) was a black coloured Poly Vinyl Chloride (PVC) granulates fortified with Deca-Brominated Diphenyl Ether (deca-BDE). The second material (#13153) was a pink salmon coloured Poly Propylene that was also positive on deca-BDE.

Sample #13152 was divided over 120 subsamples of approx. 5 grams and sample #13153 was divided over 100 subsamples of approx. 2 grams.

The homogeneity of subsample #13152 was checked by determination of Deca-BDE content on 3 stratified randomly selected subsamples. And the homogeneity of subsample #13153 was checked by determination of Deca-BDE content on 7 stratified randomly selected subsamples

	Deca-BDE #13152 in mg/kg
Sample 1	4728
Sample 2	4915
Sample 3	4769

Table 1: test results of the homogeneity study on the subsamples #13152

	Deca-BDE #13153 in mg/kg
Sample 1	4348
Sample 2	4380
Sample 3	4448
Sample 4	4844
Sample 5	4420
Sample 6	4736
Sample 7	4756

Table 2: test results of the homogeneity study on the subsamples #13153

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibilities of the target methods, in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	Deca-BDE #13152 in mg/kg	Deca-BDE #13153 in mg/kg
r (observed)	275	531
reference	see § 4 and ref. 17	see § 4 and ref. 17
0.3 x R (reference)	1009	942

Table 3: evaluation of the observed repeatability against the requirement

Both calculated repeatabilities were in agreement with 0.3 times the assigned target reproducibility. Therefore, homogeneity of the subsamples of #13152 and #13153 was assumed.

To each of the participating laboratories one set of samples, (1\* sample #13152 and 1\* sample #13153) was sent on August 14, 2013.

## 2.5 ANALYSIS

The participants were requested to determine on both samples: octa-PBB, nona-PBB, deca-PBB, octa-BDE, nona-BDE and deca-BDE. It was explicitly requested to treat the samples as if they were routine samples and to report the analytical results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such results can't be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also, a letter of instructions was added to the package.

The laboratories were requested to complete the report form with some details of the methods used.

## 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report.

The laboratories are represented by the code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment.

Shortly 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 results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, 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. Before further calculations, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. In the case of an abnormal distribution, the statistical evaluation should be used with care.

In accordance to 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, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and 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 significant consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them 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 4; nos.14 and 15).

### 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, 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 target reproducibility (preferably taken from a standardized test method) by division with 2.8.

The z-scores were calculated in accordance with:

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

The  $z_{(\text{target})}$  scores are listed in the result tables in appendix 1.

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. See also appendix 4; ref. 16.

Absolute values for  $z < 2$  are very common and absolute values for  $z > 3$  are very rare. Therefore 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 interlaboratory study, no problems were encountered with the dispatch of the samples. Sixteen participants reported results after the final reporting date and three participants did not report any results at all. Not all laboratories were able to report all analytes requested. Finally, 76 of the 79 participants submitted analysis results. The 76 reporting laboratories reported 362 numerical test results. Observed were 45 outlying results, which is 11% of all reported numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A not-Gaussian data distribution was found on sample #13152 for nona-BDE. Therefore, the statistical evaluation on this component should be used with due care.

For the determination of PBB and PBDE, the IEC62321 method is considered to be the official EC test method. Regretfully this method does not (yet) mention precision data.

Normally, when no (suitable) reproducibility requirements from a test method are available, target requirements are estimated from the Horwitz equation.

However, from the IMEP-26 results (ref. 17) it was clear that earlier target standard deviations of 3 – 12% were not realistic for non-expert laboratories and that a realistic PT target is 25% of the assigned value. This made the reproducibility requirements estimated by using the Horwitz equation to be unrealistically small.

Therefore, the target requirements were taken from the findings of interlaboratory study IMEP-26. In the IMEP-26 report the results of an interlaboratory study are presented on the determination of the sum of polybrominated biphenyls (PBB), the sum of polybrominated diphenylethers (PBDE) and several individual brominated diphenylethers (ref. 17).

#### 4.1 EVALUATION PER COMPONENT AND PER SAMPLE

In this section, the results are discussed per component and per sample.

The participants were requested to report octa-, nona-, and deca-PBB and octa-, nona-, and deca-BDE. None of the participants, except laboratory 2156, detected octa-, nona-, and deca-PBB in any of the samples (#13152 and #13153).

The participants were also requested to report the analytical details of the methods. The analytical details are listed in Appendix 2.

Five of the six test results reported by laboratory 2558 appeared to be statistical outliers. Because all test results of one laboratory are correlated, the remaining test result of laboratory 2558 (for deca-BDE) was excluded manually prior to the statistical analysis.

**Octa-BDE:** A number of laboratories may have problems with this component. The majority of the participants reported the absence of Octa-BDE or a small concentration that is near or below the LOD. For sample #13152, the reported test results vary from 0.8 mg/kg to 633 mg/kg. For sample #13153, the reported test results vary from 1.6 mg/kg to 3128 mg/kg. Therefore no further conclusions were drawn.

**Nona-BDE:** Some analytical problems were observed. For each of the samples #13152 and #13153, seven statistical outliers were observed. The calculated reproducibilities after rejection of the seven outliers for each of the samples are not in agreement with the target reproducibility. The reported test results vary over a large range for samples #13152 and #13153, respectively 3.0 - 4783 and 2.3 – 3122.

**Deca-BDE:** Analytical problems were observed for a number of individual laboratories. For sample #13152, the calculated reproducibility after rejection of seven statistical outliers is in agreement with the target reproducibility. However, the reported test results vary over a large range: 4.3 – 121156 mg/kg. Also for sample #13153 the calculated reproducibility, after rejection of nine (!) statistical outliers is in agreement with the target reproducibility. However, the reported range of the test results is 6.0 - 13328 mg/kg.

#### 4.2 PERFORMANCE EVALUATION OF 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 calculated reproducibilities and the target reproducibilities derived from the literature standards are compared in the next tables.

Analytes	unit	n	Average	2.8 * sd	R (target)
Octa-BDE #13152	mg/kg	25	17	21	(12)*
Nona-BDE #13152	mg/kg	65	197	210	138
Deca-BDE #13152	mg/kg	69	4264	2486	2985

Table 4: performance overview for sample #13152



Analytes	unit	n	Average	2.8 * sd	R (target)
Octa-BDE #13153	mg/kg	27	20	29	(14)*
Nona-BDE #13153	mg/kg	65	306	285	214
Deca-BDE #13153	mg/kg	65	4417	1704	3092

Table 5: performance overview for sample #13153

\* values between brackets are for concentration near or below the limit of detection.

The observed reproducibilities for nona-BDE are larger than the target reproducibility requirements and therefore it was concluded that the determination of nona-BDE at low concentration levels in the evaluated materials is somewhat problematic.

## 5 COMPARISON WITH PREVIOUS PROFICIENCY TESTS

The evolution of the relative reproducibilities for PBDE as observed in this proficiency scheme and the comparison with the findings in the previous iis' PTs is visualized in the next table.

	Hexa-BDE	Hepta-BDE	Octa-BDE	Nona-BDE	Deca-BDE
2009	---	---	60 – 102%	60 – 110%	36 – 103%
2010	---	---	97 – 108%	104 - 112%	29 – 39%
2011	78%	43%	70%	43 – 65%	57 – 69%
2012	---	---	115%	113 - 144%	43 - 45%
2013	---	---	---	93 – 106%	39 – 58%

Table 6: comparison of the relative PBDE reproducibilities in the previous iis' PTs and in the present round

## 6 DISCUSSION

For the determination of PBB and PBDE, the IEC62321 method is considered to be the official EC test method. In this proficiency test the majority of the participants used a version of IEC62321. Surprisingly not all laboratories that reported to have performed IEC62321 may have followed the guidelines for the sample preparation of this method.

Several laboratories that reported to have performed IEC62321 answered the question “To what particle size was the sample reduced prior to analysis?” with “as received”, “<1mm”, “1mm”, “<2mm” or “2mm”, while the correct procedure should be grinding to <500 µm. Cryogenic grinding is strongly recommended.

On the question “What extraction solvent was used?” reported all participants to have used Toluene, except two laboratories. From these two laboratories reported one laboratory to have used Dichloromethane and another laboratory a mixture of Toluene and THF.

Other parts of the determination that may need attention are the possible congener degradation. IEC62321 mentions in Annex A.8 the necessary QC to check the congener degradation. Therefore the participants were requested to check the recovery. From the laboratories that answered this question with “YES”, almost all reported a recovery between 80% and 120%.

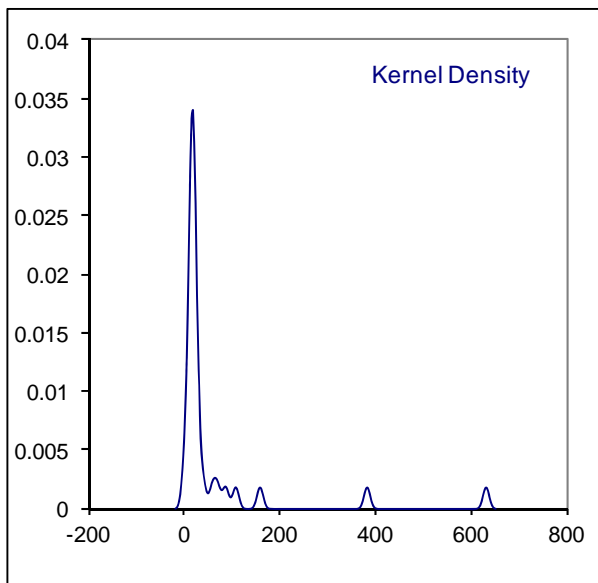
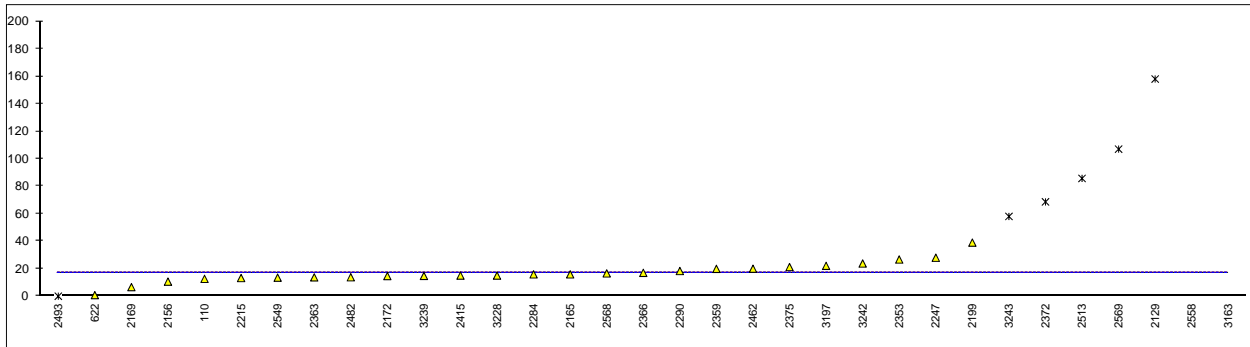
## APPENDIX 1

Determination of Octabromo diphenyl ether on sample #13152; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	INH-029	12.63554		----	
324	IEC62321	<5.0		----	
339	in house	<25		----	
551		n.d.		----	
601	IEC62321	<5		----	
605	IEC62321	<5		----	
607	IEC62321	<5		----	
622	IEC62321	0.8		----	
632		----		----	
826	IEC62321	<5		----	
840	IEC62321	n.d.		----	
1213	IEC62321	<50		----	
2115		----		----	
2129	IEC62321	158	C,G(0.01)	----	first reported:135
2132	in house	n.d.		----	
2139	IEC62321	n.d.		----	
2156	IEC62321	10.6		----	
2165	IEC62321	15.9		----	
2169	IEC62321	6.62		----	
2172	IEC62321	14.66		----	
2173	IEC62321	n.d.		----	
2176	in house	n.d.		----	
2199	IEC62321	39	C,G(0.05)	----	first reported:142
2201	IEC62321	<5		----	
2202	in house	n.d.		----	
2212	in house	<100		----	
2215	IEC62321	13.21		----	
2216	IEC62321	<50		----	
2230	IEC62321	<10		----	
2247	IEC62321	27.99		----	
2256	IEC62321	n.d.		----	
2284	in house	15.9		----	
2290	IEC62321	18.314		----	
2309	IEC62321	<50	C	----	first reported:87
2316	IEC62321	<50	C	----	first reported: 80
2350	IEC62321	n.d.		----	
2353	IEC62321	26.7		----	
2355	IEC62321	<5		----	
2357	IEC62321	n.d.		----	
2359	IEC62321	19.9		----	
2363	IEC62321	13.7		----	
2365	IEC62321	n.d.		----	
2366	IEC62321	17		----	
2370	IEC62321	n.d.		----	
2372	IEC62321	68.62	G(0.01)	----	
2375	EPA3550C	21.21		----	
2386	IEC62321	<50		----	
2403	IEC62321	n.d.		----	
2415	IEC62321	15.0		----	
2438	EN62321	<50		----	
2462	IEC62321	20.0		----	
2479	IEC62321	n.d.		----	
2482	in house	13.78		----	
2488		----		----	
2492		----		----	
2493		0	ex	----	result excluded, zero is not a real value
2494	IEC62321	n.d.		----	
2500	IEC62321	n.d.		----	
2513	IEC62321	85.7	G(0.01)	----	
2549	IEC62321	13.50		----	
2558	IEC62321	383	G(0.01)	----	
2568	IEC62321	16.6		----	
2569	IEC62321	107	G(0.01)	----	
3100	IEC62321	n.d.		----	
3146	in house	<100		----	
3153	IEC62321	<20		----	
3163	in house	633	G(0.01)	----	
3172	IEC62321	n.d.		----	
3182	IEC62321	<5		----	
3185	IEC62321	n.d.		----	
3190		----		----	

3197	IEC62321	22.1	----
3214	IEC62321	n.d.	----
3218	IEC62321	n.d.	----
3225		----	----
3228	IEC62321	15	----
3239	in house	14.76	----
3242	IEC62321	23.78	----
3243	in house	58	G(0.01) ----
normality		OK	
n		25	
outliers		7	+1 excl.
mean (n)		17.15	
st.dev. (n)		7.378	
R(calc.)		20.66	
R(target)		12.00	

Compare Horwitz:12.27



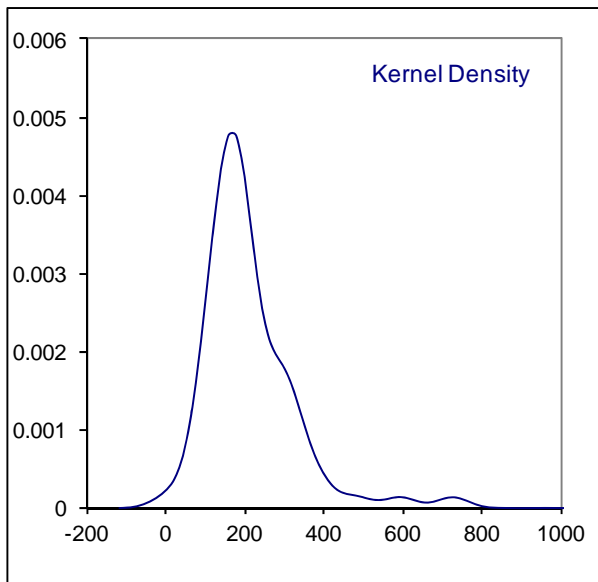
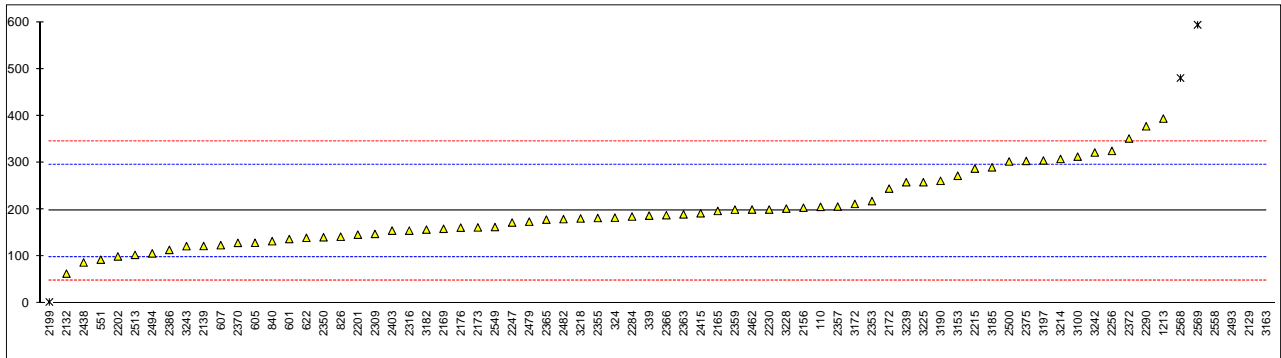
## Determination Nonabromo diphenyl ether on sample #13152; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
110	INH-029	205.8264		0.18	
324	IEC62321	182.7		-0.29	
339	in house	187		-0.20	
551		93.096		-2.11	
601	IEC62321	137.16		-1.21	
605	IEC62321	129.11		-1.38	
607	IEC62321	124.08		-1.48	
622	IEC62321	139.7		-1.16	
632		-----		-----	
826	IEC62321	142.16		-1.11	
840	IEC62321	132.6		-1.31	
1213	IEC62321	393.8	C	4.00	first reported: 662.0
2115		-----		-----	
2129	IEC62321	2867	C,G(0.01)	54.22	first reported:990
2132	in house	63.43		-2.71	
2139	IEC62321	122.35		-1.52	
2156	IEC62321	204		0.14	
2165	IEC62321	197		0.00	
2169	IEC62321	159		-0.77	
2172	IEC62321	244.6		0.97	
2173	IEC62321	161.9		-0.71	
2176	in house	161.43		-0.72	
2199	IEC62321	3.012	G(0.05)	-3.94	
2201	IEC62321	146.4		-1.03	
2202	in house	100.0		-1.97	
2212		-----		-----	
2215	IEC62321	287.3		1.83	
2216		-----		-----	
2230	IEC62321	200		0.06	
2247	IEC62321	172.32		-0.50	
2256	IEC62321	325		2.60	
2284	in house	185.1		-0.24	
2290	IEC62321	377.435		3.67	
2309	IEC62321	148	C	-0.99	first reported: 652
2316	IEC62321	155	C	-0.85	first reported: 380
2350	IEC62321	141		-1.14	
2353	IEC62321	218		0.43	
2355	IEC62321	182		-0.30	
2357	IEC62321	206.4		0.19	
2359	IEC62321	199.8		0.06	
2363	IEC62321	189.8		-0.15	
2365	IEC62321	178.5		-0.37	
2366	IEC62321	188		-0.18	
2370	IEC62321	129		-1.38	
2372	IEC62321	351.4		3.14	
2375	EPA3550C	303.62		2.17	
2386	IEC62321	114		-1.68	
2403	IEC62321	155		-0.85	
2415	IEC62321	192.0	C	-0.10	first reported: 466.8
2438	EN62321	87.2		-2.23	
2462	IEC62321	200.0		0.06	
2479	IEC62321	174.2		-0.46	
2482	in house	179.6		-0.35	
2488		-----		-----	
2492		-----		-----	
2493		2674	G(0.01)	50.30	
2494	IEC62321	106.5		-1.84	
2500	IEC62321	302.5		2.14	
2513	IEC62321	103.3		-1.90	
2549	IEC62321	162.76		-0.69	
2558	IEC62321	726	G(0.01)	10.74	
2568	IEC62321	480.3	G(0.05)	5.75	
2569	IEC62321	593.67	G(0.01)	8.06	
3100	IEC62321	312.74		2.35	
3146	in house	<150		-----	
3153	IEC62321	272		1.52	
3163	in house	4783	G(0.01)	93.13	
3172	IEC62321	212		0.31	
3182	IEC62321	157.2		-0.81	
3185	IEC62321	290		1.89	
3190	IEC62321	261.2		1.30	
3197	IEC62321	304.6		2.19	
3214	IEC62321	307.9		2.25	
3218	IEC62321	181		-0.32	
3225	IEC62321	258.2		1.24	

3228	IEC62321	202	0.10
3239	in house	258.2	1.24
3242	IEC62321	321.58	2.53
3243	in house	122	-1.52

normality	not OK
n	65
outliers	7
mean (n)	196.96
st.dev. (n)	74.890
R(calc.)	209.69
R(target)	137.88

Compare Horwitz:69.00



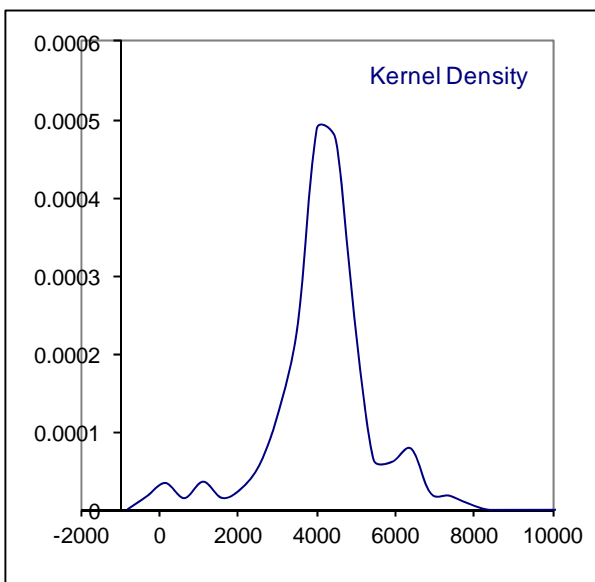
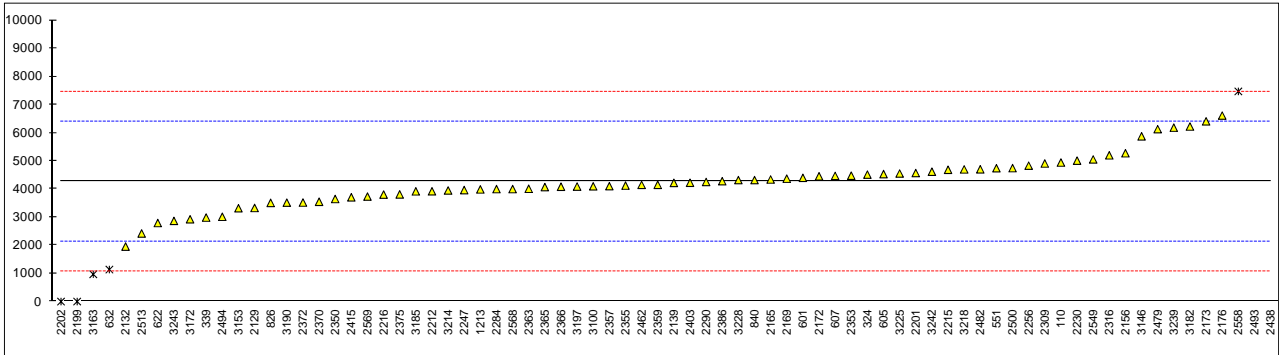
## Determination of Decabromo diphenyl ether on sample #13152; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	INH-029	4944.571		0.64	
324	IEC62321	4515		0.24	
339	in house	2989		-1.20	
551		4742.030		0.45	
601	IEC62321	4400.93		0.13	
605	IEC62321	4533.23		0.25	
607	IEC62321	4468.16		0.19	
622	IEC62321	2794.3		-1.38	
632	INH-107	1141.25	DG(0.05)	-2.93	
826	IEC62321	3509.75		-0.71	
840	IEC62321	4320.4		0.05	
1213	IEC62321	3989.8		-0.26	
2115		-----		-----	
2129	IEC62321	3330		-0.88	
2132	in house	1958.09		-2.16	
2139	IEC62321	4220.83		-0.04	
2156	IEC62321	5274		0.95	
2165	IEC62321	4341		0.07	
2169	IEC62321	4370		0.10	
2172	IEC62321	4456		0.18	
2173	IEC62321	6406.5		2.01	
2176	in house	6611.8		2.20	
2199	IEC62321	5.348	DG(0.01)	-3.99	
2201	IEC62321	4570.0		0.29	
2202	in house	4.323	DG(0.01)	-4.00	
2212	in house	3922		-0.32	
2215	IEC62321	4686		0.40	
2216	IEC62321	3805		-0.43	
2230	IEC62321	5010		0.70	
2247	IEC62321	3965.12		-0.28	
2256	IEC62321	4832		0.53	
2284	in house	3999.6		-0.25	
2290	IEC62321	4253.124		-0.01	
2309	IEC62321	4910		0.61	
2316	IEC62321	5200		0.88	
2350	IEC62321	3648		-0.58	
2353	IEC62321	4476		0.20	
2355	IEC62321	4125		-0.13	
2357	IEC62321	4104.6		-0.15	
2359	IEC62321	4151.7		-0.11	
2363	IEC62321	4011.0		-0.24	
2365	IEC62321	4071.7		-0.18	
2366	IEC62321	4084		-0.17	
2370	IEC62321	3550		-0.67	
2372	IEC62321	3523		-0.70	
2375	EPA3550C	3810.20		-0.43	
2386	IEC62321	4280		0.01	
2403	IEC62321	4225		-0.04	
2415	IEC62321	3709.0		-0.52	
2438	EN62321	121156	G(0.01)	109.65	
2462	IEC62321	4150.1		-0.11	
2479	IEC62321	6128.3		1.75	
2482	in house	4703		0.41	
2488		-----		-----	
2492		-----		-----	
2493		27542	G(0.01)	21.84	
2494	IEC62321	3017		-1.17	
2500	IEC62321	4745.1		0.45	
2513	IEC62321	2423.49		-1.73	
2549	IEC62321	5054.94		0.74	
2558	IEC62321	7470	G(0.05)	3.01	
2568	IEC62321	4000	C	-0.25	first reported: 4938
2569	IEC62321	3734.33		-0.50	
3100	IEC62321	4098.48		-0.16	
3146	in house	5874		1.51	
3153	IEC62321	3320		-0.89	
3163	in house	974	DG(0.05)	-3.09	
3172	IEC62321	2928		-1.25	
3182	IEC62321	6223.8	C	1.84	first reported:8454.5
3185	IEC62321	3920		-0.32	
3190	IEC62321	3519.0		-0.70	
3197	IEC62321	4090.2		-0.16	
3214	IEC62321	3950.5		-0.29	
3218	IEC62321	4700		0.41	
3225	IEC62321	4553.7		0.27	

3228	IEC62321	4320	0.05
3239	in house	6183	1.80
3242	IEC62321	4618	0.33
3243	in house	2870	-1.31

normality	OK
n	69
outliers	7
mean (n)	4264.09
st.dev. (n)	887.885
R(calc.)	2486.08
R(target)	2984.87

Compare Horwitz:542.95



## Determination of Octabromo diphenyl ether on sample #13153; results in mg/kg

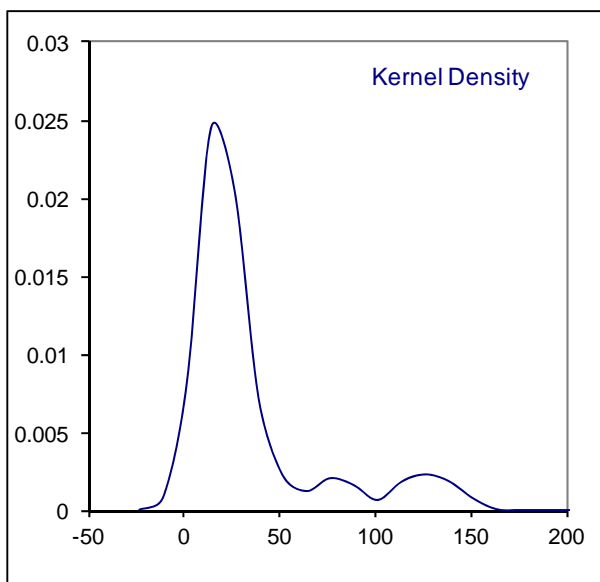
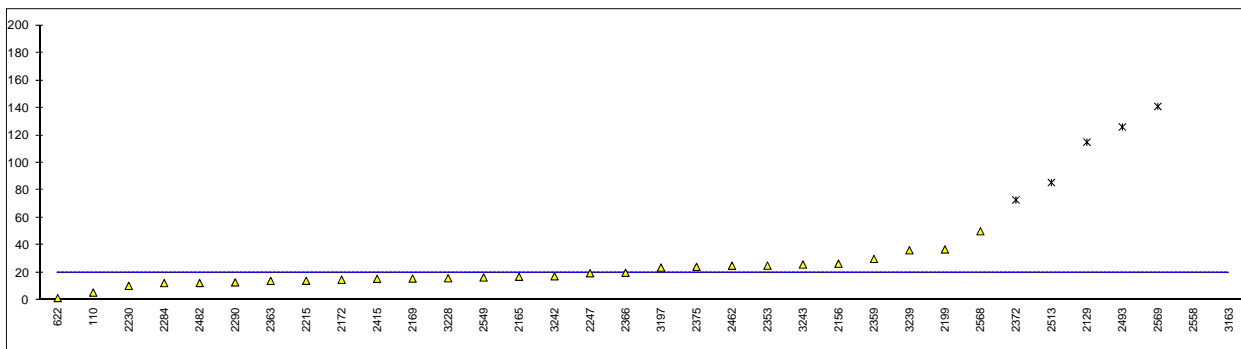
lab	method	value	mark	z(targ)	remarks
110	INH-029	5.59443		----	
324	IEC62321	<5.0		----	
339	in house	<25		----	
551		n.d.		----	
601	IEC62321	<5		----	
605	IEC62321	<5		----	
607	IEC62321	<5		----	
622	IEC62321	1.6		----	
632		----		----	
826	IEC62321	n.d		----	
840	IEC62321	n.d		----	
1213	IEC62321	<50		----	
2115		----		----	
2129	IEC62321	115	C,G(0.01)	----	first reported: 207
2132	in house	n.d.		----	
2139	IEC62321	n.d.		----	
2156	IEC62321	26.6		----	
2165	IEC62321	17.1		----	
2169	IEC62321	15.8		----	
2172	IEC62321	14.94		----	
2173	IEC62321	n.d.		----	
2176	in house	n.d.		----	
2199	IEC62321	37	C,(G0.05)	----	first reported: 133
2201	IEC62321	<5		----	
2202	in house	n.d		----	
2212	in house	<100		----	
2215	IEC62321	14.17		----	
2216		----		----	
2230	IEC62321	10.5		----	
2247	IEC62321	19.67		----	
2256	IEC62321	n.d.		----	
2284	in house	12.5		----	
2290	IEC62321	12.996		----	
2309	IEC62321	<50	C	----	first reported: 99
2316	IEC62321	<50	C	----	first reported: 85
2350	IEC62321	n.d		----	
2353	IEC62321	25.2		----	
2355	IEC62321	<5		----	
2357	IEC62321	n.d		----	
2359	IEC62321	30.1		----	
2363	IEC62321	14.1		----	
2365	IEC62321	n.d		----	
2366	IEC62321	20		----	
2370	IEC62321	n.d		----	
2372	IEC62321	72.97	G(0.01)	----	
2375	EPA3550C	24.32		----	
2386	IEC62321	<50		----	
2403	IEC62321	n.d		----	
2415	IEC62321	15.6		----	
2438	EN62321	<50		----	
2462	IEC62321	25.1		----	
2479	IEC62321	n.d		----	
2482	in house	12.53		----	
2488		----		----	
2492		----		----	
2493		126	G(0.05)	----	
2494	IEC62321	n.d		----	
2500	IEC62321	n.d.		----	
2513	IEC62321	85.6	G(0.01)	----	
2549	IEC62321	16.58		----	
2558	IEC62321	521	G(0.01)	----	
2568	IEC62321	50.2	G(0.05)	----	
2569	IEC62321	141	G(0.05)	----	
3100	IEC62321	n.d.		----	
3146	in house	<100		----	
3153	IEC62321	<20		----	
3163	in house	3128	G(0.01)	----	
3172	IEC62321	n.d		----	
3182	IEC62321	<5		----	
3185	IEC62321	n.d.		----	
3190		----		----	
3197	IEC62321	23.8		----	
3214	IEC62321	n.d.		----	
3218	IEC62321	n.d.		----	
3225		----		----	



3228	IEC62321	16	----
3239	in house	36.35	----
3242	IEC62321	17.43	----
3243	in house	26	----

normality	OK
n	27
outliers	7
mean (n)	20.06
st.dev. (n)	10.205
R(calc.)	28.57
R(target)	14.05

Compare Horwitz:14.02



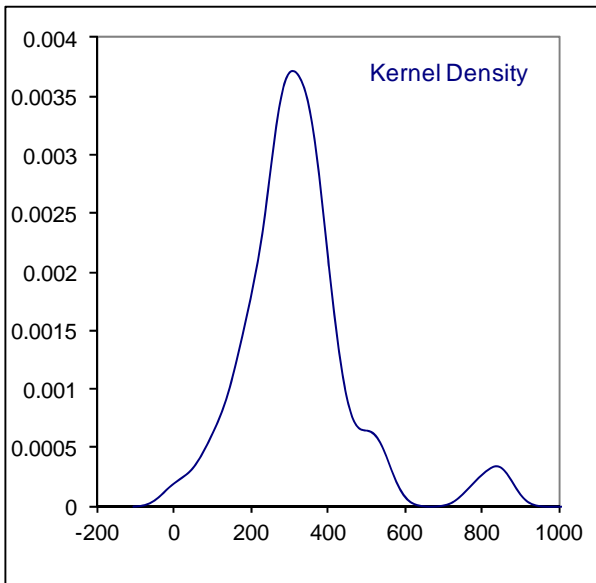
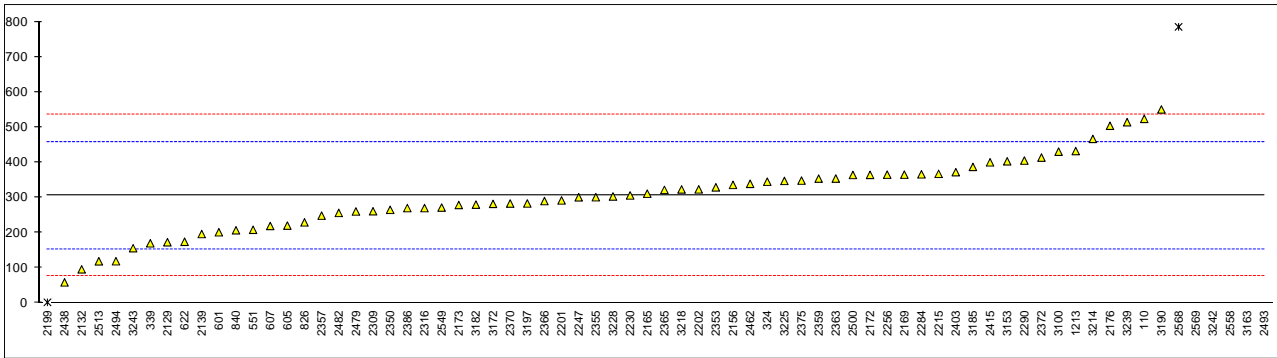
## Determination of Nonabromo diphenyl ether on sample #13153; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	INH-029	523.657		2.85	
324	IEC62321	345.1		0.51	
339	in house	170		-1.78	
551		208.384		-1.27	
601	IEC62321	201.38		-1.37	
605	IEC62321	220.23		-1.12	
607	IEC62321	219.15		-1.13	
622	IEC62321	174.4		-1.72	
632		-----		-----	
826	IEC62321	229.45		-1.00	
840	IEC62321	207.0		-1.29	
1213	IEC62321	432.1	C	1.65	first reported: 636.7
2115		-----		-----	
2129	IEC62321	173		-1.74	
2132	in house	95.89		-2.75	
2139	IEC62321	196.31		-1.43	
2156	IEC62321	336		0.40	
2165	IEC62321	311		0.07	
2169	IEC62321	365		0.77	
2172	IEC62321	364.4		0.77	
2173	IEC62321	278.8		-0.35	
2176	in house	503.9		2.59	
2199	IEC62321	2.284	G(0.05)	-3.97	
2201	IEC62321	291.9		-0.18	
2202	in house	323.2		0.23	
2212		-----		-----	
2215	IEC62321	367.3		0.80	
2216		-----		-----	
2230	IEC62321	306		0.00	
2247	IEC62321	300.77		-0.07	
2256	IEC62321	365		0.77	
2284	in house	366.0		0.79	
2290	IEC62321	405.112		1.30	
2309	IEC62321	261	C	-0.59	first reported: 488
2316	IEC62321	270	C	-0.47	first reported: 479
2350	IEC62321	265		-0.53	
2353	IEC62321	329		0.30	
2355	IEC62321	301		-0.06	
2357	IEC62321	248.6		-0.75	
2359	IEC62321	353.9		0.63	
2363	IEC62321	354.0		0.63	
2365	IEC62321	321.7		0.21	
2366	IEC62321	290		-0.21	
2370	IEC62321	283		-0.30	
2372	IEC62321	413.6		1.41	
2375	EPA3550C	348.25		0.56	
2386	IEC62321	270		-0.47	
2403	IEC62321	372		0.87	
2415	IEC62321	400.0		1.23	
2438	EN62321	58.9		-3.23	
2462	IEC62321	339.0		0.43	
2479	IEC62321	260.4		-0.59	
2482	in house	256.3		-0.65	
2488		-----		-----	
2492		-----		-----	
2493		3122	G(0.01)	36.84	
2494	IEC62321	119		-2.44	
2500	IEC62321	364.2		0.76	
2513	IEC62321	118.8		-2.45	
2549	IEC62321	271.48		-0.45	
2558	IEC62321	1310	G(0.01)	13.14	
2568	IEC62321	785	C,G(0.01)	6.27	first reported:551
2569	IEC62321	843	G(0.01)	7.03	
3100	IEC62321	430.45		1.63	
3146	in house	<150		-----	
3153	IEC62321	403		1.27	
3163	in house	1662	G(0.01)	17.74	
3172	IEC62321	282		-0.31	
3182	IEC62321	279.8		-0.34	
3185	IEC62321	387		1.06	
3190	IEC62321	550.3		3.20	
3197	IEC62321	283.2		-0.30	
3214	IEC62321	466.6		2.10	
3218	IEC62321	323		0.23	

3225	IEC62321	347.3		0.54
3228	IEC62321	303		-0.04
3239	in house	514.1		2.73
3242	IEC62321	852.62	G(0.05)	7.15
3243	in house	156		-1.96

normality	OK
n	65
outliers	7
mean (n)	305.77
st.dev. (n)	101.901
R(calc.)	285.32
R(target)	214.04

Compare Horwitz:100.26



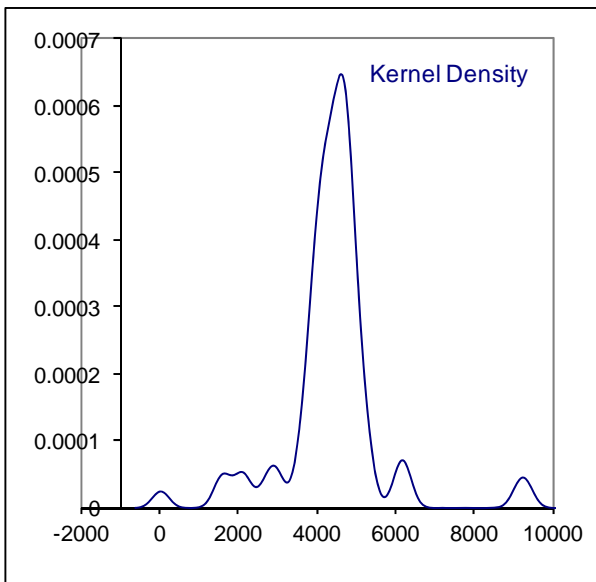
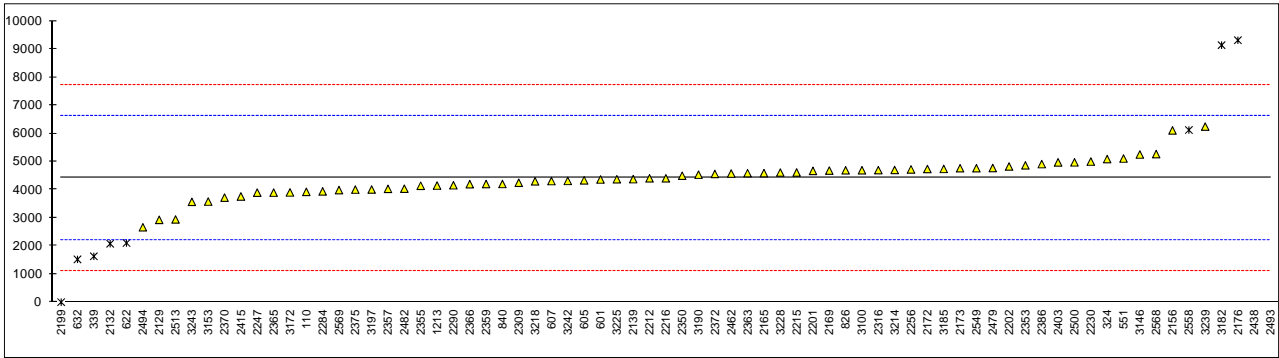
## Determination of Decabromo diphenyl ether on sample #13153; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	INH-029	3924.44		-0.45	
324	IEC62321	5089		0.61	
339	in house	1635	DG(0.01)	-2.52	
551		5109.581		0.63	
601	IEC62321	4363.49		-0.05	
605	IEC62321	4334.66		-0.07	
607	IEC62321	4308.28		-0.10	
622	IEC62321	2106.8	DG(0.01)	-2.09	
632	INH-107	1526.42	DG(0.01)	-2.62	
826	IEC62321	4691.06		0.25	
840	IEC62321	4204.5		-0.19	
1213	IEC62321	4147.2		-0.24	
2115		-----		-----	
2129	IEC62321	2930		-1.35	
2132	in house	2082.50	DG(0.01)	-2.11	
2139	IEC62321	4376.10		-0.04	
2156	IEC62321	6107		1.53	
2165	IEC62321	4589		0.16	
2169	IEC62321	4680		0.24	
2172	IEC62321	4736		0.29	
2173	IEC62321	4764.4		0.31	
2176	in house	9309.7	G(0.01)	4.43	
2199	IEC62321	5.994	G(0.05)	-3.99	
2201	IEC62321	4669.9		0.23	
2202	in house	4826		0.37	
2212	in house	4405		-0.01	
2215	IEC62321	4613		0.18	
2216	IEC62321	4405		-0.01	
2230	IEC62321	5000		0.53	
2247	IEC62321	3896.3		-0.47	
2256	IEC62321	4721		0.28	
2284	in house	3942.1		-0.43	
2290	IEC62321	4159.047		-0.23	
2309	IEC62321	4248		-0.15	
2316	IEC62321	4695		0.25	
2350	IEC62321	4498		0.07	
2353	IEC62321	4867		0.41	
2355	IEC62321	4140		-0.25	
2357	IEC62321	4030.4		-0.35	
2359	IEC62321	4203.3		-0.19	
2363	IEC62321	4587.0		0.15	
2365	IEC62321	3897.7		-0.47	
2366	IEC62321	4198		-0.20	
2370	IEC62321	3720		-0.63	
2372	IEC62321	4560		0.13	
2375	EPA3550C	4005.20		-0.37	
2386	IEC62321	4910		0.45	
2403	IEC62321	4971		0.50	
2415	IEC62321	3760.0		-0.59	
2438	EN62321	12056.5	G(0.01)	6.92	
2462	IEC62321	4575.2		0.14	
2479	IEC62321	4772.2		0.32	
2482	in house	4036		-0.34	
2488		-----		-----	
2492		-----		-----	
2493		13328	G(0.01)	8.07	
2494	IEC62321	2667		-1.58	
2500	IEC62321	4972.4		0.50	
2513	IEC62321	2944.208		-1.33	
2549	IEC62321	4764.49		0.31	
2558	IEC62321	6120	ex	1.54	result excluded, see §4.1
2568	IEC62321	5267	C	0.77	first reported: 5620
2569	IEC62321	3987.33		-0.39	
3100	IEC62321	4691.47		0.25	
3146	in house	5252		0.76	
3153	IEC62321	3580		-0.76	
3163	in house	n.d		-----	
3172	IEC62321	3904		-0.46	
3182	IEC62321	9135.3	G(0.01)	4.27	
3185	IEC62321	4739		0.29	
3190	IEC62321	4535.6		0.11	
3197	IEC62321	4008.4		-0.37	
3214	IEC62321	4702.5		0.26	
3218	IEC62321	4300		-0.11	
3225	IEC62321	4371.6		-0.04	

3228	IEC62321	4610	0.18
3239	in house	6242	1.65
3242	IEC62321	4313	-0.09
3243	in house	3570	-0.77

normality	OK	
n	65	
outliers	9	+1 excl.
mean (n)	4416.74	
st.dev. (n)	608.705	
R(calc.)	1704.37	
R(target)	3091.72	

Compare Horwitz:559.42



## APPENDIX 2

## Analytical details for sample #13152

Lab	cryogenic milled	max particle size	particle size checked	extraction solvent used	recovery checked	internal standard used
110	No	<1 mm	sieved through a sieve of 0.5 µm	Toluene	Yes, 97.3%	Decachlorobiphenyl
324	No			Toluene	No	13C-HxBDE and 13C-DecaBDE Wellington Laboratories
339	No	about 1 mm	visual	Toluene	No	Internal standard for injection PCB 180
551	Yes			Toluene		Decachlorobiphenyl
601	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 109%	
605	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 90%	
607	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 90%	
622	No	<1 mm		Toluene	No	
632	No			Toluene	No	None
826	Yes	<500 µm	sieved through a sieve of <500 µm	Toluene	No	
840	No	~1 mm		Toluene	Yes, 75-109%	Decachlorobiphenyl - Chemservice
1213	Yes	<500 µm		Toluene	Yes, 123%	
2115						
2129	Yes	<0.1 mm		Dichloromethane	Yes, 80-120%	PCB 209 - Riedel de Haen
2132	Yes	<500 µm		Toluene	No	PCB 209 - Dr. Ehrenstorfer
2139	Yes			Toluene	No	
2156	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	
2165	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	PCB-209
2169	Yes	<250 µm	visual	Toluene	Yes, 92.1%	
2172	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 92.1%	Anthrene D10 - Dr. E
2173	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 85%	Decachlorobiphenyl
2176	No	1 - 2 mm	estimate	Toluene	Yes, 106%	Decachlorobiphenyl - Accu standard CAS#2051-24-3
2199						
2201	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	None
2202	Yes	<500 µm		Toluene	No	None
2212	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	Decachlorobiphenyl
2215	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	Anthracene D10 - CIL
2216	Yes	<1 mm	sieved through a mill sieve	Toluene	Yes, 108%	CB-209
2230	Yes	1 < mm	sieved through a sieve of 1.0 mm	Toluene	Yes, 90.4%	Anthracene D10
2247	Yes	<75 µm	visual	Toluene	Yes, 94%	None
2256	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 85%	CB-209
2284	Yes	<1 mm		Toluene	Yes, 84.3%	
2290	No	<1 mm		Toluene	Yes, 80%	PCB 209, Accustand.
2309	Yes	<500 µm	sieved through a sieve of 250 µm	Toluene	Yes, 98%	Decachlorobiphenyl - Accustandard
2316	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 92%	CB209 decachlorobiphenyl
2350	Yes	<1 mm	sieved through a sieve of 1.0 mm	Toluene	Yes, 102%	Decachlorobiphenyl - Supelco
2353	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 102%	PCB209 CAS 10386-84-2 - Accustandard
2355	No	<500 µm	sieved through a sieve of 0.425 mm	Toluene	No	
2357	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	None

2359	Yes			Toluene	Yes, 94-108%	QCB / Mirex - Chemservice
2363	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 92%	decachlorobiphenyl - Chemservice
2365	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 101.6%	PCB209 - Accustandard
2366						
2370	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 79.4%	Decachlorobiphenyl - Accustandard
2372			visual			Decachlorobiphenyl - Supelco
2375		2*2mm	sieved through a Helias Digi met	Toluene		Decachlorobiphenyl - Dr. Ehrenstorfer
2386	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	<sup>13</sup> C <sub>12</sub> BDE209 and <sup>13</sup> C <sub>12</sub> BDE206 - Cambridge Isotope Lab
2403	No	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 99%	Decachlorobiphenyl
2415	No	<1 mm	sieved through a sieve of 5 mm	Toluene	No	None
2438	No	<2 mm	not measured	Toluene	Yes, 50%	Decachlorobiphenyl
2462	No	<1.5 mm	sieved through a sieve of 3 mm	Toluene	Yes, 92.3%	PCB209
2479	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 80%	Decachlorobiphenyl
2482	No			Toluene	Yes, 92%	
2488						
2492						
2493	No	< 1 mm		Toluene / THF	No	C13 PBBE209
2494	No	<1 mm	measuring with caliper	Toluene	No	None
2500	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	Yes, 88%	Anthracene D10
2513						
2549	No	as received		Toluene	Yes, >90%	None
2558	No	as received		Toluene	No	PCB209
2568	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	MBDE-MXE-Wellington
2569	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	PCB 209-Accustand.
3100	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 89%	CB 209 - Dr. E
3146						
3153	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	Decachlorobiphenyl - Sigma Aldrich
3163	No			none	No	Standard mix solution - Accu standard
3172	Yes	<500 µm		Toluene	Yes, 85%	PCB 209 - Accustandard, MBDE 209 - Wellington
3182	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	No	PCB, result based on external standard calc.
3185	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 103%	Decachlorobiphenyl - Accustandard
3190	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 85%	None
3197	No	0.5 mm	sieved through a sieve of 0.5 mm	Toluene	Yes, 80%	PCB 209 - Dr. Ehrenstorfer
3214	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 96%	Decachlorobiphenyl - Chemservice
3218	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 83%	PCB 209 - Dr. E
3225	Yes	<1 mm		Toluene	No	Decachlorobiphenyl (CB 209)
3228	No	<1 mm	sieve through a sieve of 1 mm	Toluene	No	Dibromoctafluoro biphenyl
3239	Yes	<1 mm		Toluene	Yes, 80%	Decachlorobiphenyl - Accustandard
3242	Yes	as received		Toluene	No	None
3243	Yes		sieved through a sieve of 2 mm	Toluene	Yes, 73%	PCB 209

## Analytical details for sample #13153

Lab	cryogenic milled	max particle size	particle size checked	extraction solvent used	recovery checked	internal standard used
110	Yes	<1 mm	sieved through a sieve of 0.5 µm	Toluene	Yes, 96.7%	Decachlorobiphenyl
324	No			Toluene	No	13C-HxBDE and 13C-DecaBDE Wellington Laboratories
339	No	about 1 mm	visual	Toluene	No	internal standard for injection PCB 180
551	Yes			Toluene		Decachlorobiphenyl
601	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 109%	
605	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 90%	
607	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 90%	
622	No	<1 mm		Toluene	No	
632	No			Toluene	No	none
826	Yes	<500 µm	sieved through a sieve of <500 µm	Toluene	No	
840	No	~1 mm		Toluene	Yes, 75-109%	Decachlorobiphenyl - Chemservice
1213	Yes	<500 µm		Toluene	Yes, 123%	
2115						
2129	Yes	<0.1 mm		Dichloromethane	Yes, 80-120 %	PCB 209 - Riedel de Haen
2132	Yes	<500 µm		Toluene	No	PCB 209 - Dr. Ehrenstorfer
2139	Yes			Toluene	No	
2156	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	
2165	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	PCB-209
2169	Yes	<250 µm	visual	Toluene	Yes, 87.9%	
2172	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 92.1%	Anthrene D10 - Dr. E
2173	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 85%	Decachlorobiphenyl
2176	No	1 - 2 mm	estimate	Toluene	Yes, 108%	Decachlorobiphenyl - Accu standard CAS#2051-24-3
2199						
2201	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	none
2202	Yes	<500 µm		Toluene	No	none
2212	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	Decachlorobiphenyl
2215	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	Anthracene D10 - CIL
2216	Yes	<1 mm	sieved through a mill sieve	Toluene	Yes, 108%	CB-209
2230	Yes	<1 mm	sieved through a sieve of 1.0 mm	Toluene	Yes, 90.4%	Anthracene D10
2247	Yes	<500 µm	visual	Toluene	Yes, 94%	None
2256	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 85%	CB-209
2284	No	<1 mm		Toluene	Yes, 84.3%	
2290	No	<1 mm		Toluene	Yes, 82%	PCB 209
2309	Yes	<500 µm	sieved through a sieve of 250 µm	Toluene	Yes, 98%	Decachlorobiphenyl - Accustandard
2316	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 95%	CB209 decachlorobiphenyl
2350	Yes	<1 mm	sieved through a sieve of 1.0 mm	Toluene	Yes, 102%	Decachlorobiphenyl - Supelco
2353	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 99%	PCB209 CAS 10386-84-2 - Accustandard
2355	No	<500 µm	sieved through a sieve of 0.425 mm	Toluene	No	
2357	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	none
2359	Yes			Toluene	Yes, 94-108%	QCB / Mirex - Chemservice



2363	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 92%	Decachlorobiphenyl - Chemservice
2365	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 101.6%	PCB209 - Accustandard
2366						
2370	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 85%	Decachlorobiphenyl - Accustandard
2372			visual			Decachlorobiphenyl - Supelco
2375		2*2, 1mm	sieved through a Helias Digi met	Toluene		
2386	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	No	<sup>13</sup> C <sub>12</sub> BDE209 and <sup>13</sup> C <sub>12</sub> BDE206 - Cambridge Isotope Laboratories
2403	No	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 99%	Decachlorobiphenyl
2415	No	<1 mm	sieved through a sieve of 5 mm	Toluene	No	none
2438	No	<2 mm	not measured	Toluene	Yes, 55%	Decachlorobiphenyl
2462	No	<1.5 mm	sieved through a sieve of 2 mm	Toluene	Yes, 92.3%	PCB209
2479	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 80%	Decachlorobiphenyl
2482	No			Toluene	Yes, 92%	
2488						
2492						
2493	No	<1 mm		Toluene / THF	No	C13 PBBE209
2494	No	<1 mm	measuring with caliper	Toluene	No	none
2500	Yes	<1 mm	sieved through a sieve of 1 mm	Toluene	Yes, 88%	Anthracene D10
2513						
2549	No	as received		Toluene	Yes, >90%	none
2558	No	as received		Toluene	No	PCB209
2568	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	MBDE-MXE - Wellington
2569	No	<1 mm	sieved through a sieve of 1 mm	Toluene	No	PCB 209 - Accustandard
3100	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 83%	CB 209 - Dr. E
3146						
3153	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	No	Decachlorobiphenyl - Sigma Aldrich
3163	No			none	No	Standard mix solution - Accustandard
3172	Yes	<500 µm		Toluene	Yes, 95%	PCB 209 - Accustandard, MBDE 209 - Wellington
3182	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	No	PCB, result based on external standard calc.
3185	No	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 103%	Decachlorobiphenyl - Accustandard
3190	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 85%	none
3197	No	0.5 mm	sieved through a sieve of 0.5 mm	Toluene	Yes, 80%	PCB 209 - Dr. Ehrenstorfer
3214	Yes	<500 µm	sieved through a sieve of 500 µm	Toluene	Yes, 96%	Decachlorobiphenyl - Chemservice
3218	Yes	<500 µm	sieved through a sieve of 0.5 mm	Toluene	Yes, 83%	PCB 209 - Dr. E
3225	Yes	<1 mm		Toluene	No	Decachlorobiphenyl (CB 209)
3228	No	<1 mm	sieve through a sieve of 1 mm	Toluene	No	dibromoctafluorobiphenyl
3239	Yes	<1 mm		Toluene	Yes, 80%	Decachlorobiphenyl - Accustandard
3242	Yes	as received		Toluene	No	none
3243	Yes		sieved through a sieve of 2 mm	Toluene	Yes, 73%	PCB 209

**APPENDIX 3****Number of participating laboratories per country**

1 lab in BELGIUM  
2 labs in BRAZIL  
1 lab in FRANCE  
6 labs in GERMANY  
6 labs in HONG KONG  
1 lab in HUNGARY  
5 labs in INDIA  
2 labs in INDONESIA  
2 labs in ITALY  
2 labs in JAPAN  
4 labs in KOREA  
4 labs in MALAYSIA  
22 labs in P.R. of CHINA  
1 lab in PHILIPPINES  
2 labs in SINGAPORE  
5 labs in TAIWAN R.O.C.  
2 labs in THAILAND  
1 lab in THE NETHERLANDS  
3 labs in TURKEY  
3 labs in U.S.A.  
4 labs in VIETNAM

## APPENDIX 4

### 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
n.a.	= not applicable
n.d.	= not detected
IMEP	= International Measurement Evaluation Programme

### Literature:

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