Institute for
Interlaboratory Studies

Results of Proficiency Test Total Brominated Flame Retardants in Polymers September 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis22P07

October 2022

CONTENTS

1 INTRODUCTION

Since the 1990s scientists have questioned the safety of the Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyl Ethers (PBDE) because it may bio-accumulate in blood, breast milk and fat tissues. The European Union decided to ban the use of both PBB and PBDE in electrical and electronic devices. This ban was formalized in the RoHS Directive 2011/65/EU on the restriction of the use of certain hazardous substances in electrical and electronic equipment and an upper limit of 1000 mg/kg PBB or PBDE was set. Hexabromocyclododecane (HBCDD) was listed in the Annex XIV of REACH and hence is subject to Authorization. HBCDD is persistent, bio-accumulative and toxic to water-living organisms and slowly banned worldwide.

Since 2009 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Poly Brominated Diphenyl Ethers (PBDE). The scope was extended with Hexabromocyclododecane (HBCDD) and Poly Brominated Biphenyls (PBB) over the years. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of total Brominated Flame Retardants in Polymers.

In this interlaboratory study 88 laboratories in 24 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Total Brominated Flame Retardants in Polymers proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different samples made of PVC of approximately 3 grams each labelled #22705 and #22706 respectively.
The participants were requested to report rounded and unrounded test results. The unrounded test 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/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 organization 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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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

For the first sample a batch of pink PVC blocks was selected which was artificially fortified with some PBDE. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #22705.
The homogeneity of the subsamples was checked by the determination of Deca-BDE according to an in house method on 8 stratified randomly selected subsamples.

	Deca-BDE in mg/kg
sample #22705-1	819
sample #22705-2	788
sample #22705-3	776
sample #22705-4	785
sample #22705-5	764
sample #22705-6	746
sample #22705-7	791
sample #22705-8	759

Table 1: homogeneity test results of subsamples #22705

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Deca-BDE in mg/kg
r (observed)	63
reference test method	IEC62321-6:15
0.3 x R (reference test method)	165

Table 2: evaluation of the repeatability of subsamples #22705

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of red PVC rings was selected which was artificially fortified with Hexabromocyclododecane (HBCDD). After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #22706.
The homogeneity of the subsamples was checked by the determination of HBCDD according to an in house method on 8 stratified randomly selected subsamples.

	HBCDD in mg/kg
sample #22706-1	2737
sample #22706-2	2874
sample #22706-3	2812
sample #22706-4	2682
sample #22706-5	2821
sample #22706-6	2842
sample #22706-7	2630
sample #22706-8	2852

Table 3: homogeneity test results of subsamples #22706

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	HBCDD in mg/kg
r (observed)	247
reference test method	IMEP-26:11
0.3 x R (reference test method)	584

Table 4: evaluation of the repeatability of subsamples #22706

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two PVC samples labelled #22705 and #22706 respectively were sent on August 24, 2022.

2.5 ANALYZES

The participants were requested to determine on both samples the total content of the following Brominated Flame Retardants: Octabromobiphenyl (Octa-BB), Nonabromobiphenyl (Nona-BB), Decabromobiphenyl (Deca-BB), Octabromodiphenylether (Octa-BDE), Nonabromodiphenylether (Nona-BDE), Decabromodiphenylether (Deca-BDE), Hexabromocyclododecane (HBCDD) and Other Brominated Flame Retardant(s).
It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization 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 June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test 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, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them 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 test results are plotted. The corresponding laboratory numbers are on 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 reference test method. 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. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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 proficiency test some problems were encountered with the dispatch of the samples. Eight participants reported test results after the final reporting date and eight other participants did not report any test results. Not all participants were able to report all tests requested.
In total 80 participants reported 180 numerical test results. Observed were 8 outlying test results, which is 4.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of PBB and PBDE the IEC62321-6 method is considered to be the official IEC test method. The 2015 version of IEC62321 does mention precision data for PBDE and these have been used for the evaluation of Nona-BDE and Deca-BDE. Unfortunately, no official test method exists for the determination of HBCDD. In iis PTs when no (suitable) reproducibility requirement from a test method is available the target reproducibility is estimated from the Horwitz equation. Fortunately, an Interlaboratory

Comparison report is available: IMEP-26 Determination of Brominated Flame Retardants in plastic (see lit. 13). Although HBCDD is not mentioned in IMEP-26 the relative target standard deviation for Brominated Flame Retardants mentioned in IMEP-26 is used for the evaluation of HBCDD in this report.

sample #22705

Nona-BDE: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC62321-6:2015.

Deca-BDE: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IEC62321-6:2015.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Brominated Flame Retardants mentioned in paragraph 2.5. Therefore, no z-scores are calculated. The reported results are given in appendix 2.

sample #22706

HBCDD: This determination was not problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IMEP-26:2011.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Brominated Flame Retardants mentioned in paragraph 2.5. Therefore, no z-scores are calculated. The reported results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference methods are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Nona-BDE	mg/kg	45	42.2	51.9	42.7
Deca-BDE	mg/kg	78	772	459	544

Table 5: reproducibility of test on sample #22705

Component	unit	n	average	2.8 * sd	R(lit)
HBCDD	mg/kg	49	2768	961	1937

Table 6: reproducibilities of tests on sample #22706

Without further statistical calculations it can be concluded that for the determination of Deca-BDE or HBCDD there is a good compliance with the reference test methods.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2022 WITH PREVIOUS PTS

	September 2022	September 2021	September 2020	August 2019	September 2018
Number of reporting laboratories	80	73	84	67	77
Number of test results	180	183	193	168	256
Number of statistical outliers	8	9	8	8	9
Percentage of statistical outliers	4.4%	4.9%	4.1%	4.8%	3.5%

Table 7: 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 test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	September 2022	September 2021	September 2020	August 2019	2018 -2009	Target*)
Nona-BDE	44%	47%	45%	43%	15-51%	25-34%
Deca-BDE	21%	18%	24%	24%	10-37%	25-34%
Deca-BB	n.e.	n.e.	n.e.	n.e.	22%	25%
HBCDD	12%	25%	23%	(151%)	17-49%	25%

Table 8: development of the uncertainties over the years

*) The target of IEC62321-6:2015 is dependent on concentration. Targets calculated at 50 – 10000 mg/kg respectively.

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method IEC62321-6 with GC/MS is used by most of the reporting participants. Several participants used a different test method for the determination of HBCDD than for the determination of Deca-BDE and Nona-BDE.

For this PT some analytical details were requested which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- A majority (about 90%) of the reporting participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- About 20% of the reporting participants used the sample as received and about 80% did further cut or further grind the samples prior to analysis.
- About 55% used less than 0.5 grams of sample intake and about 45% used a sample intake of 0.5 to 1 gram.
- To release the components from the sample about 40% used ultrasonic and about 35% of used Soxhlet.
- About 90% of all the participants used Toluene or a Toluene mixture as solvent to release the analytes.

- The extraction time used differs from 15 minutes to 12 hours. About 30% used an extraction time between 120 and 180 minutes, about 40% used an extraction time less than 120 minutes.
- An extraction temperature of 60 or 70 °C is mostly reported.

For Deca-BDE and HBCDD the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no further analysis has been performed to the effect of the analytical details.

5 DISCUSSION

The material of both samples in this PT (sample #22705 and sample #22706) was PVC. To extract the requested components (components mentioned in paragraph 2.5) from a polymer the extraction solvent, the extraction conditions and the contact surface area are important. In the PT of 2022 on Total Brominated Flame Retardants in Polymers it appeared that none of the requested analytical details were dominant as for Deca-BDE and HBCDD the calculated reproducibility is in agreement with the requirements of the reference test methods.

According to the RoHS Directive 2011/65/EU, electrical and electronic equipment are not allowed to contain more than 1000 mg/kg PBB or PBDE (see paragraph 1).
When the results of this interlaboratory study were compared with respect to the above regulation it is noticed that eleven of the reporting laboratories would have rejected sample #22705 based on the test results of Deca-BDE.

For HBCDD no upper limit of HBCDD is defined in governmental regulations yet. Therefore, based on the test results of HBCDD no conclusions could be drawn with respect to acceptation or rejection of sample #22706.

6 CONCLUSION

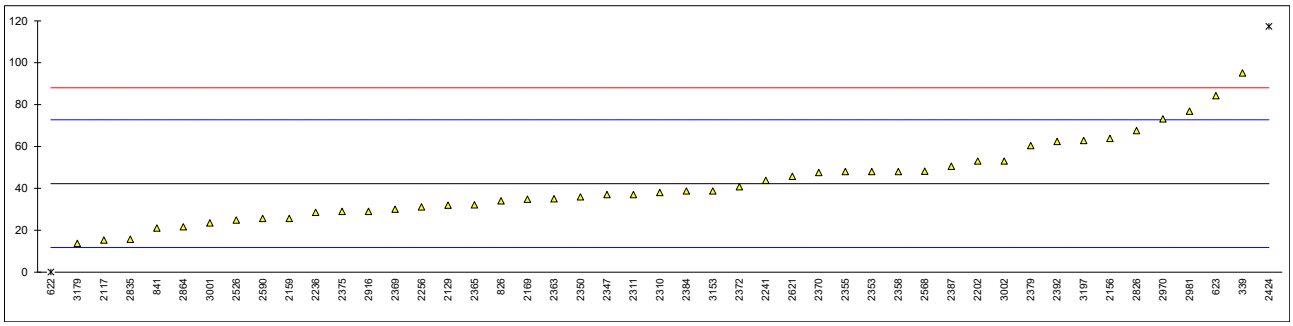
Although it can be concluded that most of the participants have no problem with the determination on PBDE and HBCDD in PVC, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

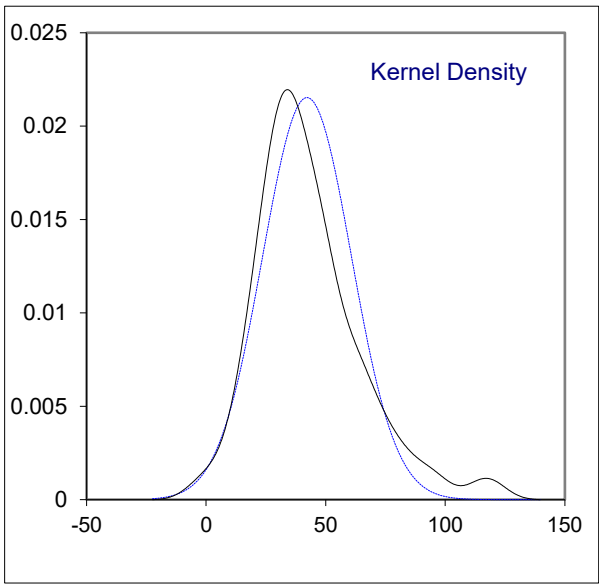
APPENDIX 1

Determination of Nonabromodiphenylether (Nona-BDE) on sample #22705; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	95.1		3.47	
523		-----		-----	
551		-----		-----	
622	IEC62321-6 - GC/MS	0	R(0.05)	-2.77	
623	IEC62321-6 - GC/MS	84.21		2.75	
826	IEC62321-6 - GC/MS	34		-0.54	
841	IEC62321-6	21		-1.39	
2115		-----		-----	
2117	In house	15.3		-1.77	
2121		-----		-----	
2129	In house	32		-0.67	
2139		-----		-----	
2156	IEC62321-6 - GC/MS	63.8	C	1.41	first reported 95
2159	In house	25.60		-1.09	
2165	IEC62321-6 - GC/MS	not detected		-----	
2169	IEC62321-6 - GC/MS	34.8		-0.49	
2177		-----		-----	
2179	IEC62321-6 - GC/MS	not detected		-----	
2184		not applicable		-----	
2201	IEC62321-6 - HPLC-PDA/UV	not detected		-----	
2202	IEC62321-6 - GC/MS	53		0.71	
2216	IEC62321-6 - GC/MS	Not Detected		-----	
2236	In house	28.47		-0.90	
2241	IEC62321-6 - GC/MS	43.83		0.10	
2247	IEC62321-6	not detected		-----	
2255	ISO17881-1	Not detected		-----	
2256	ISO17881-1	31.14		-0.73	
2265		-----		-----	
2267		-----		-----	
2289		-----		-----	
2293		-----		-----	
2295		-----		-----	
2297	IEC62321-6 - GC/MS	Not detected		-----	
2310	IEC62321-6 - GC/MS	38		-0.28	
2311	IEC62321-6 - GC/MS	37.0		-0.34	
2316	IEC62321-6 - GC/MS	Not Detected		-----	
2320	In house	<5		-----	
2347	IEC62321-6 - GC/MS	37		-0.34	
2350	IEC62321-6 - GC/MS	35.9		-0.42	
2352		-----		-----	
2353	IEC62321-6 - GC/MS	48.03		0.38	
2355	IEC62321-6/QC/T944	48		0.38	
2358	IEC62321-6 - GC/MS	48.03		0.38	
2363	IEC62321-6 - GC/MS	35		-0.47	
2365	IEC62321-6 - GC/MS	32.13		-0.66	
2366	IEC62321-6 - GC/MS	<5		-----	
2369	IEC62321-6	30		-0.80	
2370	IEC62321-6 - GC/MS	47.6		0.35	
2372	IEC62321-6 - GC/MS	40.76		-0.10	
2375	IEC62321-6 - GC/MS	29		-0.87	
2379	IEC62321-6 - GC/MS	60.3994		1.19	
2382	IEC62321-6 - GC/MS	＜10		-----	
2384	IEC62321-6 - GC/MS	38.71		-0.23	
2386	IEC62321-6 - GC/MS	<25		-----	
2387	IEC62321-6 - GC/MS	50.55		0.55	
2392	IEC62321-6 - GC/MS	62.3959		1.32	
2410		-----		-----	
2424	IEC62321-6 - GC/MS	117.38	R(0.05)	4.93	
2481		not analysed		-----	
2500	IEC62321-6 - GC/MS	nd		-----	
2504	IEC62321-6	Not detected		-----	
2526	IEC62321-6	24.84		-1.14	
2568	IEC62321-6 - GC/MS	48.14		0.39	
2590	In house	25.592		-1.09	
2612		-----		-----	
2621	IEC62321-6 - GC/MS	45.66		0.22	
2643		-----		-----	
2674		not applicable		-----	
2826	IEC62321-6 - GC/MS	67.5672		1.66	
2835	IEC62321-6 - GC/MS	15.6385		-1.74	
2864	IEC62321-6 - GC/MS	21.64		-1.35	
2916	IEC62321-6 - GC/MS	29		-0.87	
2917		-----		-----	
2970	IEC62321-6 - GC/MS	73.19		2.03	

lab	method	value	mark	z(targ)	remarks
2981	IEC62321-6 - GC/MS	76.79	C	2.27	first reported 101.6
2993		not analysed		-----	
2995		-----		-----	
3001	ISO17881-1	23.46		-1.23	
3002	IEC62321-6 - GC/MS	53.02		0.71	
3100	IEC62321-6 - GC/MS	<100		-----	
3153	IEC62321-6 - GC/MS	38.71		-0.23	
3163		-----		-----	
3172		-----		-----	
3179	IEC62321-6 - GC/MS	13.7		-1.87	
3182		-----		-----	
3185		-----		-----	
3197	IEC62321-6 - GC/MS	62.8		1.35	
3246	IEC62321-6 - GC/MS	not detected		-----	
	normality	OK			
	n	45			
	outliers	2			
	mean (n)	42.233			
	st.dev. (n)	18.5377	RSD = 44%		
	R(calc.)	51.906			
	st.dev.(IEC62321-6:15)	15.2479			
	R(IEC62321-6:15)	42.694			

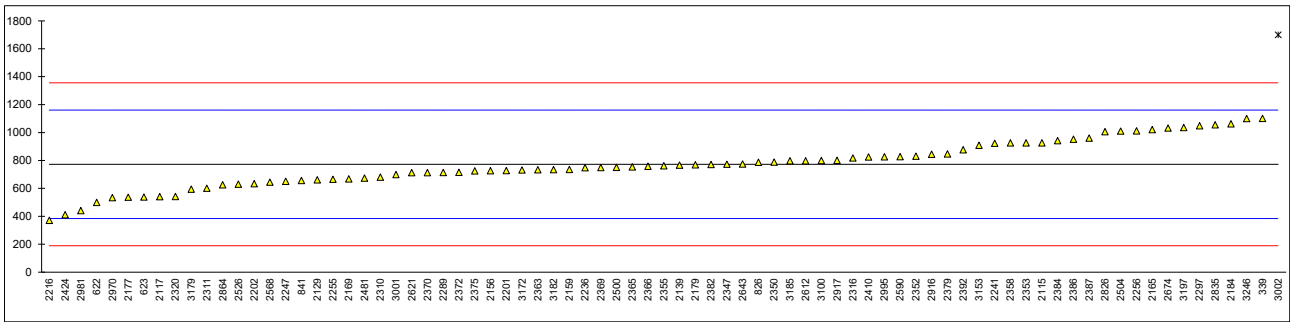


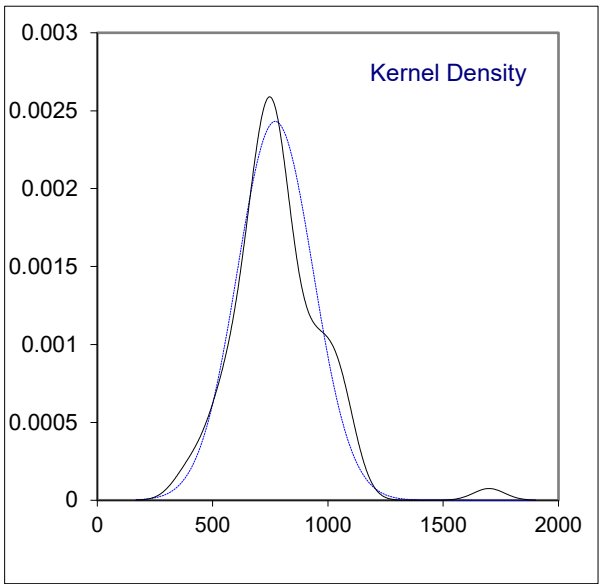


Determination of Decabromodiphenylether (Deca-BDE) on sample #22705; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	1101		1.69	
523		-----		-----	
551		-----		-----	
622	IEC62321-6 - GC/MS	499.04		-1.41	
623	IEC62321-6 - GC/MS	537.19		-1.21	
826	IEC62321-6 - GC/MS	786		0.07	
841	IEC62321-6	656		-0.60	
2115	ISO17881-1:16	925.89		0.79	
2117	In house	540.55		-1.19	
2121		-----		-----	
2129	In house	660		-0.58	
2139	IEC62321-6 - GC/MS	765.92		-0.03	
2156	IEC62321-6 - GC/MS	726		-0.24	
2159	In house	735.04		-0.19	
2165	IEC62321-6 - GC/MS	1020.0		1.28	
2169	IEC62321-6 - GC/MS	668.2		-0.53	
2177	IEC62321-6 - GC/MS	536.757		-1.21	
2179	IEC62321-6 - GC/MS	768.02		-0.02	
2184	IEC62321-6 - GC/MS	1062		1.49	
2201	IEC62321-6 - HPLC-PDA/UV	726.9		-0.23	
2202	IEC62321-6 - GC/MS	633		-0.72	
2216	IEC62321-6 - GC/MS	371		-2.06	
2236	In house	747.76		-0.13	
2241	IEC62321-6 - GC/MS	922.29		0.77	
2247	IEC62321-6	650.45		-0.63	
2255	ISO17881-1	665.0		-0.55	
2256	ISO17881-1	1012.16		1.24	
2265		-----		-----	
2267		-----		-----	
2289	IEC62321-6 - HPLC-PDA/UV	714		-0.30	
2293		-----		-----	
2295		-----		-----	
2297	IEC62321-6 - GC/MS	1047.3		1.42	
2310	IEC62321-6 - GC/MS	680		-0.47	
2311	IEC62321-6 - GC/MS	600.0		-0.89	
2316	IEC62321-6 - GC/MS	817.1		0.23	
2320	In house	542		-1.18	
2347	IEC62321-6 - GC/MS	772		0.00	
2350	IEC62321-6 - GC/MS	787.6		0.08	
2352	GB/T39560.6	830		0.30	
2353	IEC62321-6 - GC/MS	925.44		0.79	
2355	IEC62321-6/QC/T944	762		-0.05	
2358	IEC62321-6 - GC/MS	925.44		0.79	
2363	IEC62321-6 - GC/MS	733		-0.20	
2365	IEC62321-6 - GC/MS	754.32		-0.09	
2366	IEC62321-6 - GC/MS	758		-0.07	
2369	IEC62321-6	748		-0.12	
2370	IEC62321-6 - GC/MS	713		-0.30	
2372	IEC62321-6 - GC/MS	715.1		-0.29	
2375	IEC62321-6 - GC/MS	725		-0.24	
2379	IEC62321-6 - GC/MS	846.3109		0.38	
2382	IEC62321-6 - GC/MS	770.2		-0.01	
2384	IEC62321-6 - GC/MS	940.99		0.87	
2386	IEC62321-6 - GC/MS	952		0.93	
2387	IEC62321-6 - GC/MS	959.14		0.96	
2392	IEC62321-6 - GC/MS	875.5226		0.53	
2410	IEC62321-6 - GC/MS	825		0.27	
2424	IEC62321-6 - GC/MS	411.12		-1.86	
2481	In house	673		-0.51	
2500	IEC62321-6 - GC/MS	750.0		-0.11	
2504	IEC62321-6	1009.91		1.22	
2526	IEC62321-6	630.22		-0.73	
2568	IEC62321-6 - GC/MS	644.03		-0.66	
2590	In house	826.305		0.28	
2612	IEC62321-6 - GC/MS	797.90		0.13	
2621	IEC62321-6 - GC/MS	712.02		-0.31	
2643	In house	774		0.01	
2674	IEC62321-6 - GC/MS	1031		1.33	
2826	IEC62321-6 - GC/MS	1005.5180		1.20	
2835	IEC62321-6 - GC/MS	1055.6615		1.46	
2864	IEC62321-6 - GC/MS	624.93		-0.76	
2916	IEC62321-6 - GC/MS	844		0.37	
2917	In house	801		0.15	
2970	IEC62321-6 - GC/MS	533.01		-1.23	

lab	method	value	mark	z(targ)	remarks
2981	IEC62321-6 - GC/MS	440.9	C	-1.70	first reported 592.4
2993		not analysed		-----	
2995	IEC62321-3-3	826	C	0.28	first reported 179
3001	ISO17881-1	698.3		-0.38	
3002	IEC62321-6 - GC/MS	1699.04	R(0.01)	4.77	
3100	IEC62321-6 - GC/MS	799.54		0.14	
3153	IEC62321-6 - GC/MS	908.79		0.70	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	731.31		-0.21	
3179	IEC62321-6 - GC/MS	594.3		-0.92	
3182	IEC62321-6 - GC/MS	734.49		-0.19	
3185	IEC62321-6 - GC/MS	796.86		0.13	
3197	IEC62321-6 - GC/MS	1036.0		1.36	
3246	IEC62321-6 - GC/MS	1100.2		1.69	
	normality	OK			
	n	78			
	outliers	1			
	mean (n)	772.115			
	st.dev. (n)	164.0830	RSD = 21%		
	R(calc.)	459.432			
	st.dev.(IEC62321-6:15)	194.3034			
	R(IEC62321-6:15)	544.050			

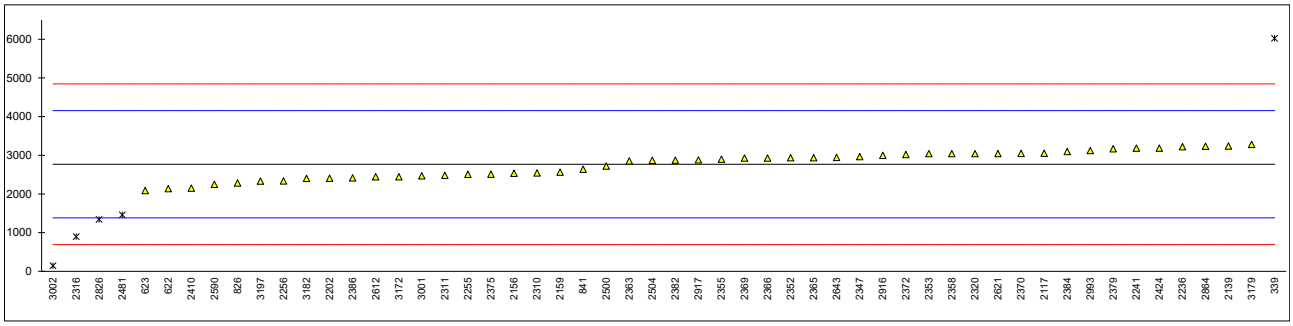


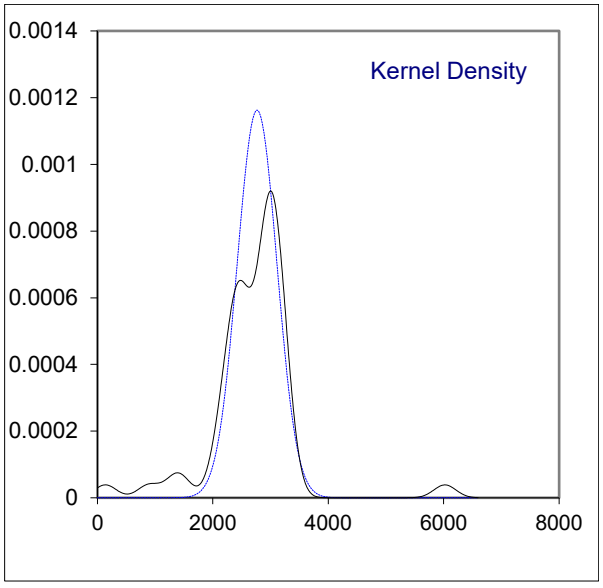


Determination of Hexabromocyclododecane (HBCDD) on sample #22706; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	6021.75	R(0.01)	4.70	
523		-----		-----	
551		-----		-----	
622	IEC62321-6 - GC/MS	2135.01		-0.91	
623	In house	2087.17		-0.98	
826	IEC62321-9	2283		-0.70	
841	IEC62321-6	2636.6		-0.19	
2115		-----		-----	
2117	In house	3053.90		0.41	
2121		-----		-----	
2129		-----		-----	
2139	IEC62321-6 - GC/MS	3237.63		0.68	
2156	IEC62321-6 - GC/MS	2535		-0.34	
2159	In house	2563.02		-0.30	
2165		-----		-----	
2169		-----		-----	
2177		-----		-----	
2179		not applicable		-----	
2184		not applicable		-----	
2201		not analyzed		-----	
2202	IEC62321-6 - GC/MS	2404		-0.53	
2216		-----		-----	
2236	In house	3220.67		0.65	
2241	IEC62321-6 - GC/MS	3182.72		0.60	
2247	IEC62321-6	Detected		-----	
2255	ISO17881-1	2505.0		-0.38	
2256	ISO17881-1	2332.27		-0.63	
2265		-----		-----	
2267		-----		-----	
2289		-----		-----	
2293		-----		-----	
2295		-----		-----	
2297		not determined		-----	
2310	IEC62321-6 - GC/MS	2540		-0.33	
2311	IEC62321-6 - GC/MS	2480.0		-0.42	
2316	In house	893.3	R(0.01)	-2.71	
2320	In house	3041		0.39	
2347	IEC62321-6 - GC/MS	2964		0.28	
2350		not analyzed		-----	
2352	GB/T39560.6	2935		0.24	
2353	IEC62321-6 - GC/MS	3038.74		0.39	
2355	EPA3550C	2898		0.19	
2358	IEC62321-6 - GC/MS	3038.74		0.39	
2363	IEC62321-9	2851		0.12	
2365	IEC62321-6 - GC/MS	2935.59		0.24	
2366	IEC62321-6 - GC/MS	2925		0.23	
2369	IEC62321-9	2925		0.23	
2370	GB/T29493.1	3050		0.41	
2372	IEC62321-6 - GC/MS	3016		0.36	
2375	IEC62321-6 - GC/MS	2512		-0.37	
2379	IEC62321-9	3164.8491		0.57	
2382	In house	2870.0		0.15	
2384		3094.98		0.47	
2386	IEC62321-6 - GC/MS	2410		-0.52	
2387		-----		-----	
2392		not determined		-----	
2410	IEC62321-6 - HPLC-PDA/UV	2150		-0.89	
2424	IEC62321-6 - GC/MS	3183.89		0.60	
2481	In house	1458	R(0.05)	-1.89	
2500	IEC62321-9	2718.8		-0.07	
2504	IEC62321-6 - GC/MS	2868.50		0.15	
2526		-----		-----	
2568		-----		-----	
2590	IEC62321-6 - GC/MS	2248.713		-0.75	
2612	IEC62321-6 - GC/MS	2444.06		-0.47	
2621	IEC62321-6 - GC/MS	3044.31		0.40	
2643	In house	2943		0.25	
2674		not applicable		-----	
2826	IEC62321-6 - GC/MS	1338.81	R(0.05)	-2.07	
2835		-----		-----	
2864	IEC62321-6 - GC/MS	3233.8	C	0.67	first reported 909.8
2916	IEC62321-6 - GC/MS	2999		0.33	
2917	In house	2880		0.16	
2970		not analyzed		-----	

lab	method	value	mark	z(targ)	remarks
2981		-----		-----	
2993		3120		0.51	
2995		-----		-----	
3001	ISO17881-1	2465		-0.44	
3002	IEC62321-6 - GC/MS	140.10	C,R(0.01)	-3.80	first reported <3
3100		-----		-----	
3153		-----		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	2445.1		-0.47	
3179	IEC62321-6 - GC/MS	3280.0		0.74	
3182	IEC62321-6 - GC/MS	2401.78		-0.53	
3185		-----		-----	
3197	IEC62321-6 - GC/MS	2328.9	C	-0.63	first reported 4555.6
3246		not applicable		-----	
	normality	OK			
	n	49			
	outliers	5			
	mean (n)	2767.771			
	st.dev. (n)	343.3199	RSD = 12%		
	R(calc.)	961.296			
	st.dev.(IMEP-26:11)	691.9426			
	R(IMEP-26:11)	1937.439			





APPENDIX 2

Abbreviations of components

Octa-BB = Octabromobiphenyl
Nona-BB = Nonabromobiphenyl
Deca-BB = Decabromobiphenyl
Octa-BDE = Octabromodiphenylether
Nona-BDE = Nonabromodiphenylether
Deca-BDE = Decabromodiphenylether
HBCDD = Hexabromocyclododecane
Other = Other Brominated Flame Retardant(s)

Other reported Brominated Flame Retardants in sample #22705; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
339	<1	<2	<10	15.3	<10	-----
523	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----
622	0	0	0	0	0	0
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
826	-----	-----	-----	-----	-----	-----
841	<5	<5	<5	<5	<5	<5
2115	-----	-----	-----	-----	-----	-----
2117	not determined	not determined	not determined	not determined	not determined	not determined
2121	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	-----	<5
2139	-----	-----	-----	-----	-----	-----
2156	<20	<20	<20	<20	<20	<20
2159	not determined	not determined	not determined	not determined	not determined	not determined
2165	not detected	not detected	not detected	not detected	-----	-----
2169	<10	<10	<10	<10	-----	-----
2177	-----	-----	-----	-----	-----	-----
2179	not detected	not detected	not detected	not detected	not applicable	not detected
2184	not applicable	not applicable	not applicable	not applicable	not applicable	not applicable
2201	not detected	not detected	not detected	not detected	not detected	not detected
2202	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2216	Not Detected	Not Detected	Not Detected	Not Detected	-----	-----
2236	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2241	< 10	< 10	< 10	20.53	< 10	< 10
2247	not detected	not detected	not detected	not detected	not detected	not detected
2255	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2256	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----
2267	-----	-----	-----	-----	-----	-----
2289	-----	-----	-----	-----	-----	-----
2293	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----
2297	Not detected	Not detected	Not detected	Not detected	Not determined	Not determined
2310	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2316	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	not analyzed	not analyzed
2352	-----	-----	-----	-----	-----	-----
2353	not detected	not detected	not detected	not detected	not detected	not detected
2355	<5	<5	<5	<5	<10	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	<5	<5	<5	<5	<5	<5
2365	＜5	＜5	＜5	＜5	＜20	-----
2366	<5	<5	<5	<5	<5	-----
2369	<5	<5	<5	<5	<10	-----
2370	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2375	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not Analyzed	Not Analyzed
2382	＜10	＜10	＜10	＜10	＜10	＜10
2384	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	<25	<25	<50	<25	<50	-----

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
2387	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected
2392	Not detected	Not detected	Not detected	Not detected	Not determined	Not determined
2410	-----	-----	-----	-----	-----	-----
2424	not detected	not detected	not detected	11.94	-----	-----
2481	not analysed	not analysed	not analysed	<LQ=2	<10	-----
2500	nd	nd	nd	nd	nd	-----
2504	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2526	Not Detected	Not Detected	Not Detected	-----	-----	-----
2568	<10	<10	<10	<10	-----	-----
2590	-----	-----	-----	-----	-----	-----
2612	-----	-----	-----	-----	-----	-----
2621	-----	-----	-----	-----	-----	-----
2643	-----	-----	-----	-----	-----	-----
2674	not detected	not detected	not detected	not detected	not applicable	-----
2826	ND	ND	ND	ND	ND	NA
2835	not detected	not detected	not detected	not detected	-----	-----
2864	not determined	not determined	not determined	not determined	not determined	not determined
2916	below	below	below	below	below	-----
2917	not detected	-----	not detected	not detected	not detected	not detected
2970	not detected	not detected	not detected	not detected	not analyzed	not analyzed
2981	not detected	not detected	not detected	not detected	-----	-----
2993	not analysed	not analysed	not analysed	not analysed	not detected	not analysed
2995	not detected	8.01	11	-----	-----	-----
3001	NOT	NOT	NOT	NOT	NOT	NOT DETECTED
3002	< 3	< 3	< 3	5.89	< 3	< 3
3100	<100	<100	<100	<100	--	--
3153	<20	<20	<20	<20	-----	-----
3163	-----	-----	-----	-----	-----	-----
3172	< 1	< 1	< 1	< 1	< 1	-----
3179	not detected	not detected	not detected	6.6	not detected	2.2
3182	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3185	not detected	not detected	not detected	not detected	-----	-----
3197	<20	<20	<20	<20	<20	-----
3246	not detected	not detected	not detected	not detected	not applicable	not applicable

Other reported Brominated Flame Retardants in sample #22706; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
339	<1	<2	<10	<1	<2	<10	-----
523	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----
622	0	0	0	0	0	0	-----
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
826	-----	-----	-----	-----	-----	-----	-----
841	<5	<5	<5	<5	<5	23.2	<5
2115	-----	-----	-----	-----	-----	-----	3.73
2117	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2121	-----	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5	<5
2139	-----	-----	-----	-----	-----	-----	-----
2156	<20	<20	<20	<20	<20	<20	<20
2159	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2165	not detected	not detected	not detected	not detected	not detected	not detected	-----
2169	<10	<10	<10	<10	<10	<10	-----
2177	-----	-----	-----	-----	-----	-----	-----
2179	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2184	not applicable	not applicable	not applicable	not applicable	not applicable	not detected	not applicable
2201	not detected	not detected	not detected	not detected	not detected	not analyzed	not analyzed
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2216	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2236	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2241	< 10	< 10	< 10	< 10	< 10	< 10	< 10
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2256	-----	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----	-----
2267	-----	-----	-----	-----	-----	-----	-----
2289	-----	-----	-----	-----	-----	-----	-----
2293	-----	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----	-----
2297	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not determined
2310	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	40.0	Not Detected
2316	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	<5	<5	<5	<5	<5	<5	<5
2347	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	not analyzed
2352	-----	-----	-----	-----	-----	-----	-----
2353	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2355	<5	<5	<5	<5	<5	<5	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<5	<5	<5	<5	<5	<5	<5
2365	＜5	＜5	＜5	＜5	＜5	＜5	-----
2366	<5	<5	<5	<5	<5	<5	-----
2369	<5	<5	<5	<5	<5	<5	-----
2370	<5	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2375	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not Analyzed
2382	＜10	＜10	＜10	＜10	＜10	＜10	＜10
2384	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	<25	<25	<50	<25	<25	<50	-----
2387	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2392	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not determined
2410	-----	-----	-----	-----	-----	-----	-----
2424	not detected	not detected	not detected	not detected	not detected	not detected	-----
2481	not analysed	not analysed	not analysed	<LQ=2	not analysed	<LQ=10	-----
2500	ND	ND	ND	ND	ND	ND	-----
2504	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2526	Not Detected	Not Detected	Not detected	-----	-----	28.12	-----
2568	<10	<10	<10	<10	<10	<10	-----
2590	-----	-----	-----	-----	-----	-----	-----
2612	-----	-----	-----	-----	-----	-----	-----
2621	-----	-----	-----	-----	-----	Not	-----
2643	-----	-----	-----	-----	-----	-----	-----
2674	not detected	not detected	not detected	not detected	not detected	not detected	-----
2826	ND	ND	ND	ND	ND	ND	NA
2835	not detected	not detected	not detected	not detected	not detected	not detected	-----
2864	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2916	below	below	below	below	below	below	128
2917	not detected	-----	not detected	not detected	-----	not detected	not detected
2970	not detected	not detected	not detected	not detected	not detected	not detected	not analyzed
2981	not detected	not detected	not detected	not detected	not detected	not detected	-----

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
2993	not analysed	not analysed	not analysed	not analysed	not analysed	not analysed	not analysed
2995	not detected	8.01	1.8	-----	-----	179	-----
3001	NOT	NOT	NOT	NOT	NOT	NOT	NOT
3002	< 3	< 3	< 3	< 3	< 3	35.0	< 3
3100	<100	<100	<100	<100	<100	<100	--
3153	<20	<20	<20	<20	<20	<20	-----
3163	-----	-----	-----	-----	-----	-----	-----
3172	< 1	< 1	< 1	< 1	-----	< 1	-----
3179	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3182	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3185	not detected	not detected	not detected	not detected	not detected	not detected	-----
3197	<20	<20	<20	<20	<20	<20	-----
3246	not detected	not detected	not detected	not detected	not detected	not detected	not applicable

lab 2115: 3.73 others, Mono-BDE 1.8 mg/kg and DiBB 1.93 mg/kg

APPENDIX 3 Analytical details

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (minutes)	extraction temp (°C)
339	No	Used as received	1g	Ultrasonic	toluene	60min	60°C
523	---	---		---			
551	---	---		---			
622	Yes	Further cut	0.1 gram	Soxhlet	Toluene	6 hours	
623	Yes	Further cut	2.2gr	Ultrasonic	Toluene	30/60	60
826	Yes	Further grinded	#22705 0.1 g/10 mL #22706 0.5 g/20 mL	Ultrasonic	#22705 Toluene #22706 THF	90 min	60 °C
841	Yes	Further cut	~0.1g	Soxhlet	Toluene	60	50
2115	No	Used as received	0.6g	Ultrasonic	Toluene	30 + 15 min	Room temp
2117	Yes	Used as received	0.2 g	Thermal Desorption	Toluol	120 min	90
2121	---	---		---			
2129	Yes	Crygonic grinded	1.5	Ultrasonic	Dichlormethane	30min	labnormtemp
2139	Yes	Further cut	0.2g	Ultrasonic	toluene hexane	2 hours	60 ℃
2156	Yes	Further cut	1g	Soxhlet	Toluene	120 minutes	-
2159	Yes	Further cut	0,3 gr	Ultrasonic	Toluene	120 minutes	70 °C
2165	Yes	Further cut	0.5g	Ultrasonic	Toluene.	3 hours.	60°C
2169	Yes	Further grinded	0.5 g	Ultrasonic	THF	60 min	
2177	Yes	Freeze grinded	0.1 g	Soxhlet	Toluene	120minutes	110℃
2179	No	---		---			
2184	Yes	Used as received	0.5 gram	Ultrasonic	Toluene	3 hours	70°C
2201	Yes	Further cut	0.1g	Soxhlet	Toluene	4 hours	Soxhlet
2202	Yes	Used as received	0.5g	Stirrer	THF/toluene/ hexane	12hrs	room temp
2216	Yes	Further grinded	1.2245 g	Soxhlet	Toluene	120	Reflux
2236	Yes	Further cut	0.1 gm / 0.5 gm	Ultrasonic	Toluene	60	60
2241	Yes	Further cut	0.5 grams	Ultrasonic	toluene	60	60
2247	Yes	Further cut	0.6gm	Ultrasonic	Toluene	60Min	60.0
2255	Yes	Further cut	0.5	ASE	Chlorobenzene-Methanol	30	135
2256	Yes	Further cut	1.0017g	Ultrasonic	Toluene	120 mins	70℃
2265	---	---		---			
2267	---	---		---			
2289	Yes	Further cut	0.1g	Soxhlet	toluene	2h	-
2293	---	---		---			
2295	---	---		---			
2297	Yes	Used as received	0.5	Ultrasonic	toluene	120	60
2310	Yes	Further cut	1	Ultrasonic	Toluene	60	60
2311	Yes	Further cut	0.1	Thermal Desorption	Toluene	60	60
2316	Yes	Further grinded	0.1 gram for Flame Retardant 0.3 gram For HBCDD	Soxhlet	Toluene	120 minutes	60° C
2320	Yes	Further cut	0.2g	Soxhlet	Toluene	2 hrs soxhlet 1hr Ultrasonic	boiling temp Toluene Soxhlet,50°C Ultrasonic.
2347	Yes	Further cut	0.1g	Soxhlet	toluene	Soxhlet 6h	
2350	Yes	Further cut	0.1g	Ultrasonic	Toluene	120min	50℃
2352	Yes	Further grinded	0.1g	Soxhlet	Toluene	240mins	
2353	Yes	Further grinded	0.1g	Soxhlet	toluene	120 minutes	20- 30 cycles
2355	Yes	Further cut	PBBPBDE:0.1g HBCDD:0.2g	Soxhlet	toluene	PBBPBDE:4h HBCDD:2h	PBBPBDE:280 HBCDD:60
2358	Yes	Further grinded	0.1g	Soxhlet	Toluene	120 minutes	20 - 30 cycles p hour
2363	Yes	Further cut	1g	Soxhlet	Toluene	240mins	NA
2365	Yes	Further grinded	0.1g	Soxhlet	Toluene	4-6h	/
2366	Yes	Further cut	0.1g	Soxhlet	toluene		
2369	---	---		---			
2370	Yes	Further grinded	IEC 62321-6:0.1g, GB/T 29493.1:0.15g	Ultrasonic	toluene	116mins 30mins	250℃, room temp
2372	Yes	Further grinded	0.5g	Ultrasonic	Toluene	1h	40
2375	Yes	Further cut	0,5gram	Ultrasonic	Toluene	60 minutes	60 °C
2379	Yes	Further grinded	0.1 g	Soxhlet	Toluene	360 min	-
2382	Yes	Further cut	0.5g	Ultrasonic	Toluene	1h	60℃
2384	Yes	Further grinded	0.1g	Ultrasonic	Toluene	60	60°C
2386	Yes	Used as received	1 g	Soxhlet	toluene	240 min	soxhlet

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (minutes)	extraction temp (°C)
2387	Yes	Further grinded	0.1	Soxhlet	Toluene	300minutes	Reflux temp
2392	Yes	Further grinded	0.1 g	Soxhlet	Toluene	150 Minutes	-
2410	No	Used as received	0.1 g	Ultrasonic	THF	60 min	70 °C
2424	Yes	Further cut	0.1 - 0.2	Ultrasonic	Toluene	180	60
2481	Yes	Further cut	0.25	Ultrasonic	toluene	60	60
2500	Yes	Used as received	1g	Ultrasonic			
2504	Yes	Further cut	0.1 gram	Ultrasonic	Toluene	120 minutes	70°C
2526	Yes	Further grinded	1 gram	Ultrasonic	Toluene	30 min	60°C
2568	---	---		---			
2590	Yes	Further grinded	0.5g	Soxhlet	DCM:toluene		
2612	Yes	Further cut		Ultrasonic	Toluol	60min	60°C
2621	Yes	Further cut	0.1	Soxhlet	toluene	360 minutes	110
2643	No	Further cut	0.5	Ultrasonic	Toluene	1hr	Room Temp.
2674	Yes	Further cut	2.0g	Ultrasonic	Toluene	3 hours	60℃
2826	Yes	Used as received	IEC 62321-6 0.1g IEC 62321-9: 0.5g	Soxhlet	Toluene	120mins 360mins	NA
2835	Yes	Further cut	PBB/ PBDE - 0.5g	ASE	PBB/ PBDE - Toulene	15 min	150°C
2864	Yes	Further cut	0.2 grams	Ultrasonic	Toluene	60	60
2916	No	Further grinded	0,1	Soxhlet	Toluene	150	111
2917	No	Used as received	0.25	Ultrasonic	Toluene	60 min	70 °C
2970	Yes	Further grinded	0.6g	Soxhlet	toluene	180min	-
2981	Yes		0.5g	Soxhlet	toluene	180min	25℃
2993	No	Used as received	0.1-0.4 g	Ultrasonic	acetone, hexane	30 minutes	room temp
2995	No	Further cut	#22705 : 0.5 mg #22706 : 0.5 mg	pyrolysis	the chemical analysis GC/MS	26 min	300°C
3001	No	Used as received	0.5	Ultrasonic	TOLUENE	120	70
3002	Yes	Further cut	0.25 g	Thermal Desorp.	THF/HEXANE	60 minutes	50°C
3100	Yes	Further cut	0.1005grams	Soxhlet	Toluene	6Hour	
3153	Yes	Further grinded	0.2 gram	Soxhlet	Toluene	4 hrs reflux	
3163	---	---		---			
3172	Yes	Further cut	1.5	Ultrasonic	toluene:acetone 1:1	30	25
3179	Yes	Used as received	1g each	Heatblock	Toluene	120	100
3182	Yes	Further grinded	0.1 g	Soxhlet	Toluene	6 hrs.	60
3185	Yes	Further grinded	0.2g	Soxhlet	Toluene	240 minutes	not appl.
3197	Yes	Further cut		Soxhlet	Toluene	120	reflux temp
3246	Yes	Used as received	0.1g	Ultrasonic	Toluene	30 min	N/A

APPENDIX 4

Number of participants per country

1 lab in AUSTRIA
1 lab in BANGLADESH
1 lab in BRAZIL
3 labs in FRANCE
8 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
5 labs in INDIA
2 labs in INDONESIA
4 labs in ITALY
5 labs in JAPAN
6 labs in KOREA, Republic of
3 labs in MALAYSIA
1 lab in MEXICO
19 labs in P.R. of CHINA
1 lab in SINGAPORE
1 lab in SRI LANKA
4 labs in TAIWAN
4 labs in THAILAND
2 labs in THE NETHERLANDS
1 lab in TUNISIA
5 labs in TURKEY
3 labs in U.S.A.
2 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test 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
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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