Results of Proficiency Test Total Brominated Flame Retardants in Polymers September 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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. As of June 1, 2006 the State of California began prohibiting the manufacture, distribution, and processing of flame retardant products, containing Pentabromodiphenylether (Penta-BDE) and Octabromodiphenylether (Octa-BDE). 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) has been under suspicion since 2008, when it was placed on the list of Substances of Very High Concern of the European Chemicals Agency. HBCDD is toxic to water-living organisms. It has been included in the EPA's List of Chemicals of Concern since 2010. In 2011 it was listed in the Annex XIV of REACH and hence is subject to Authorization. HBCDD is slowly banned worldwide.

Since 2009 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyl Ethers (PBDE) and the PT was extended with Hexabromocyclododecane (HBCDD) in 2015. During the annual proficiency testing program 2020/2021 it was decided to continue with the proficiency test for the analysis of total Brominated Flame Retardants in polymers. In this interlaboratory study 92 laboratories in 24 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Brominated Flame Retardants in Polymer 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 2 different polymer samples labelled #20655 and #20656 of approximately 3 grams each.
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 light grey PVC blocks, artificially fortified with Decabromodiphenylether (Deca-BDE), was selected. After homogenization small plastic bags were filled with approximately 3 grams and labelled #20655. 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 #20655-1	1423
sample #20655-2	1463
sample #20655-3	1396
sample #20655-4	1521
sample #20655-5	1440
sample #20655-6	1496
sample #20655-7	1450
sample #20655-8	1427

Table 1: homogeneity test results of subsamples #20655

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)	114
reference test method	IEC62321-6:15
0.3 x R (reference test method)	303

Table 2: evaluation of the repeatability of subsamples #20655

The calculated repeatability was 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 turquoise PVC blocks, artificially fortified with Hexabromocyclododecane (HBCDD), was selected. After homogenization small plastic bags were filled with approximately 3 grams and labelled #20656.
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 #20656-1	2090
sample #20656-2	2010
sample #20656-3	2147
sample #20656-4	2088
sample #20656-5	1986
sample #20656-6	2048
sample #20656-7	2110
sample #20656-8	2036

Table 3: homogeneity test results of subsamples #20656

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)	151
reference test method	IMEP-26:11
0.3 x R (reference test method)	434

Table 4: evaluation of the repeatability of subsamples #20656

To each of the participating laboratories one sample labelled #20655 and one sample labelled #20656 were sent on August 12, 2020.

2.5 ANALYZES

The participants were requested to determine on both samples the total of: Octabromobiphenyl (Octa-BB), Nonabromobiphenyl (Nona-BB), Decabromobiphenyl (Deca-BB), Octabromodiphenylether (Octa-BDE), Nonabromodiphenylether (Nona-BDE), Decabromodiphenylether (Deca-BDE) and Hexabromocyclododecane (HBCDD). It was also requested to report if the laboratory was accredited for the requested components that were determined 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 appendix 1 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 appendix 1. 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.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. 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 was projected over the Kernel Density Graph for reference.

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 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 of 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 interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. After this period still eight participants did not report any test results. Not all participants were able to report all elements requested.
Finally, the 84 reporting laboratories reported 193 numerical test results. Observed were 8 outlying test results, which is 4.1% of the statistically evaluated numerical test results.
In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the 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.

Unfortunately, no official test method exists for the determination of HBCDD. Normally, when no (suitable) reproducibility requirement from a test method is available, target requirements are estimated from the Horwitz equation. Fortunately, an Interlaboratory Comparison report is available: IMEP-26 Determination of Brominated Flame Retardants in plastic (ref. 17). 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.

For the determination of PBB and PBDE the IEC62321-6 method is considered to be the official EC 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.

Sample #20655

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

Deca-BDE: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of IEC62321-6:2015.

Sample #20656

HBCDD: This determination may be problematic for a number of laboratories. Seven statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of IMEP-26:2011.

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 official test method IEC62321-6:2015 and derived from the IMEP-26 results (ref. 17) are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Nona-BDE	mg/kg	58	69.5	87.3	61.4
Deca-BDE	mg/kg	79	1095	725	766

Table 5: reproducibilities of tests on sample #20655

Component	unit	n	average	2.8 * sd	R(lit)
HBCDD	mg/kg	48	2241	1448	1569

Table 6: reproducibility of test on sample #20656

Without further statistical calculations, it could be concluded that for the analyzes of Deca-BDE and HBCDD at this concentration level there is a good compliance of the group of participating laboratories with the reference test methods.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2020 WITH PREVIOUS PTS

	September 2020	August 2019	September 2018	September 2017	September 2016
Number of reporting laboratories	84	67	77	66	60
Number of test results	193	168	256	195	160
Number of statistical outliers	8	8	9	14	11
Percentage of statistical outliers	4.1%	4.8%	3.5%	7.2%	6.9%

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, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	September 2020	August 2019	September 2018	September 2017	2015 -2009	target **)
Nona-BDE	45%	43%	n.e.	38%	15-51%	25-34%
Deca-BDE	24%	24%	28%	16%	10-37%	25-34%
Deca-BB	n.e.	n.e.	22%	n.e.	n.e.	25%
HBCDD	23%	(151%)*	17%	39%	24-49%	25%

Table 8: comparison of the uncertainty with previous proficiency tests

*) no z-scores were calculated

**) Since 2017 a target range was mentioned for the uncertainty of IEC62321-6:2015 as this is concentration dependent (50-10000 mg/kg).

The uncertainties observed in this PT are in general in line with the uncertainties observed in previous PTs.

Sample #20656 was used before in Proficiency Test iis15P07 as #15153. It is observed that the average concentration of #20656 and #15153 are comparable. However, the uncertainty of HBCDD in the 2020 PT iis20P07 has improved when compared to the uncertainty in the 2015 PT iis15P07, see next table.

Component	unit	#20656			#15153		
		n	average	R(calc)	n	average	R(calc)
HBCDD	mg/kg	48	2241	1448	30	2457	3400

Table 9: comparison of sample #20656 with sample #15153

4.4 EVALUATION OF THE ANALYTICAL DETAILS

From the reported test methods it appeared that a majority of the participants used IEC62321-6-GC/MS for the determination of PBDE and HBCDD as test method. A number of laboratories used a different test method for the determination of HBCDD than for the determination of PBDE and PBB.

For this proficiency test some analytical details were requested and the reported details are given in appendix 3. Based on the answers given by the participants the following can be summarized:

- A vast majority (about 90%) mentioned that they are ISO/IEC17025 accredited to the determination the reported components.
- About 65% further cut the samples prior to analysis and about 35% used the samples as received.
- About 55% used 0.5 grams or less of sample intake and about 35% used 0.5 to 1 grams.
- To release the components from the sample about 55% used ultrasonic and about 40% of used Soxhlet.
- Toluene or a Toluene mixture was used as solvent to release the analytes by a majority of the participants.

- The extraction time used differs from 15 minutes to 16 hours. About 40% used an extraction time of 120 to 180 minutes, about 35% used an extraction time less than 120 minutes and about 20% used an extraction time over 180 minutes.

For Deca-BDE and HBCDD the calculated reproducibility is in full agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

The material of both samples in this PT (sample #20655 and sample #20656) 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 2020 on 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 full 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 §1 Introduction).
When the results of this interlaboratory study were compared with respect to the above regulation, it is noticed that 70% of the reporting laboratories would reject sample #20655 based on the test results of Deca-BDE.

For HBCDD no upper limit of HBCDD is defined yet in governmental regulations. Therefore, no significant conclusions were drawn with respect to acceptation or rejection of sample #20656 for containing to much HBCDD.

6 CONCLUSION

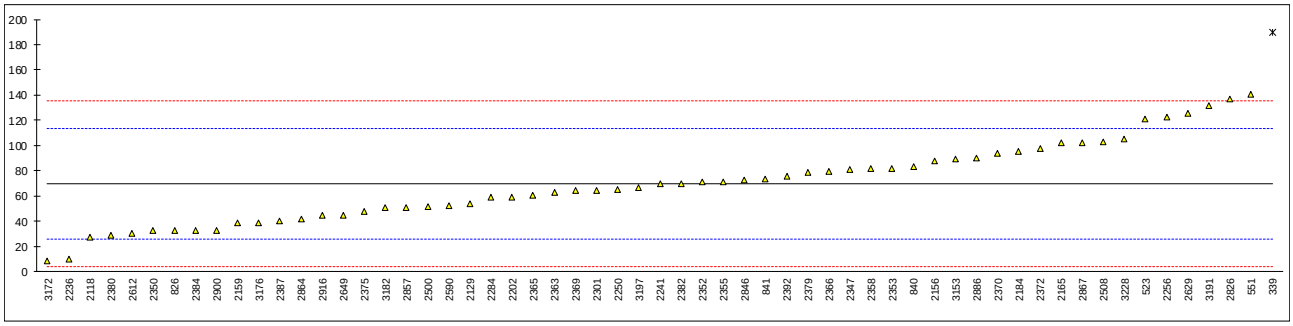
Although it can be concluded that most of the participants have no problem with the determination on PBDE and HBCDD in PVC, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis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

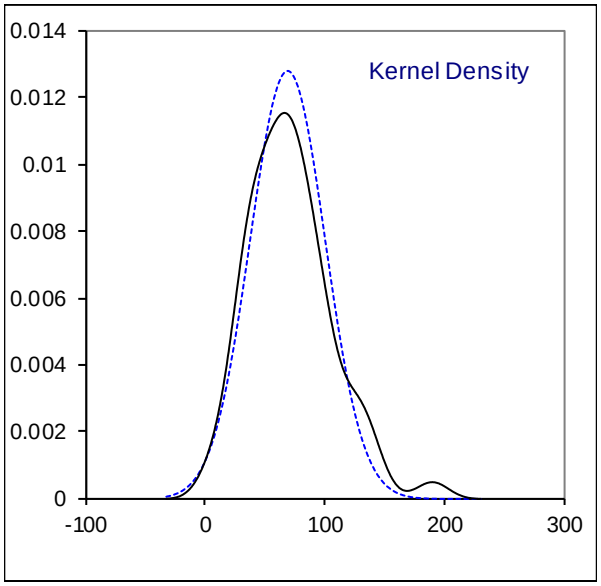
APPENDIX 1

Determination of Nonabromodiphenylether (Nona-BDE) on sample #20655; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	IEC62321-6Mod - GC/MS	190	R(0.05)	5.50	
523	IEC62321-6	121.25	C	2.36	first reported 205.47
551	IEC62321-6 - GC/MS	140.7454		3.25	
623		-----		-----	
826	IEC62321-6 - GC/MS	32.5		-1.69	
840	IEC62321-6 - GC/MS	83		0.62	
841	IEC62321-6 - GC/MS	73.19		0.17	
1842	In house	not analysed		-----	
2115		-----		-----	
2118	In house	27.29		-1.92	
2129	IEC62321-6 - GC/MS	54.0		-0.71	
2137		-----		-----	
2156	IEC62321-6 - GC/ECD	87.63		0.83	
2159	IEC62321-6 - GC/MS	38.8		-1.40	
2165	IEC62321-6 - GC/MS	102		1.48	
2184	IEC62321-6 - GC/MS	95		1.16	
2201	IEC62321-6 - GC/MS	not determined.[<50]		-----	
2202	IEC62321-6 - GC/MS	59		-0.48	
2212	In house	Not Applicable		-----	
2216	IEC62321-6 - GC/MS	none detected		-----	
2236	In house	10.21		-2.70	
2241	IEC62321-6 - GC/MS	69.6		0.01	
2247	IEC62321-6	not detected		-----	
2250	IEC62321-6Mod - GC/MS	65	C	-0.20	first reported 389
2256	IEC62321-6 - GC/MS	122.32		2.41	
2267		-----		-----	
2284	IEC62321-6 - GC/MS	59		-0.48	
2289		-----		-----	
2301	In house	64.2957		-0.24	
2310	IEC62321-6 - GC/MS	<50		-----	
2311	IEC62321-6 - GC/MS	<100		-----	
2316	IEC62321-6 - GC/MS	not detected		-----	
2347	IEC62321-6 - GC/MS	81		0.53	
2350	IEC62321-6 - GC/MS	32.5		-1.69	
2352	IEC62321-6 - GC/MS	71.2		0.08	
2353	IEC62321-6 - GC/MS	82.07		0.57	
2355	IEC62321-6 - GC/MS	71.5		0.09	
2358	IEC62321-6 - GC/MS	82.07		0.57	
2363	IEC62321-6 - GC/MS	63		-0.29	
2365	IEC62321-6 - GC/MS	60.9		-0.39	
2366	IEC62321-6 - GC/MS	79.67		0.47	
2369	IEC62321-6 - GC/MS	64		-0.25	
2370	IEC62321-6 - GC/MS	94.1		1.12	
2372	IEC62321-6 - GC/MS	97.66	C	1.29	first reported 1066.95
2375	ISO17881-1	48		-0.98	
2379	IEC62321-6 - GC/MS	78.9308		0.43	
2380	In house	29.05		-1.84	
2382	IEC62321-6 - GC/MS	70.0		0.02	
2384	IEC62321-6 - GC/MS	32.68		-1.68	
2386	IEC62321-6 - GC/MS	< 25		-----	
2387	IEC62321-6 - GC/MS	40.553		-1.32	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	75.6760	C	0.28	first reported not detected, f.r. as #20656
2481		-----		-----	
2500	IEC62321-6 - GC/MS	51.25		-0.83	
2508	In house	102.58		1.51	
2590	IEC62321-6 - GC/MS	52.081		-0.79	
2612	IEC62321-6 - GC/MS	30.3		-1.79	
2621		-----		-----	
2622		-----		-----	
2629	IEC62321-6 - GC/MS	125.6		2.56	
2649	ISO17881-1	45.0		-1.12	
2674	IEC62321-6 - GC/MS	N.A.		-----	
2809		-----		-----	
2826	IEC62321-6 - GC/MS	137.14		3.09	
2835	EPA3545A/IEC62321-6	Not Detected		-----	
2846	IEC62321-6 - GC/MS	72.33		0.13	
2857	IEC62321-6 - GC/MS	51		-0.84	
2864	IEC62321-6 - GC/MS	41.76		-1.26	
2867	IEC62321-6 - GC/MS	102		1.48	
2886	In house	90		0.94	
2900	IEC62321-6 - GC/MS	32.9		-1.67	
2914		-----		-----	
2916	In house	44.6		-1.13	

lab	method	value	mark	z(targ)	remarks
2930	IEC62321-6 - HPLC-PDA/UV	not detected		-----	
2931		not detected		-----	
2932		-----		-----	
3100	GB/T26125	--		-----	
3153		89.5		0.91	
3154		-----		-----	
3163		-----		-----	
3172	GB/T24279	8.605		-2.78	
3176	ISO17881-1	39.0		-1.39	
3182	IEC62321-6 - GC/MS	50.82		-0.85	
3183		-----		-----	
3185		-----		-----	
3191	IEC62321-6 - GC/MS	131.64		2.84	
3197	IEC62321-6 - GC/MS	66.5		-0.14	
3210		-----		-----	
3228	IEC62321-6 - GC/MS	105		1.62	
3237		-----		-----	
3239	IEC62321-6 - GC/MS	not detected (<50)		-----	
	normality	OK			
	n	58			
	outliers	1			
	mean (n)	69.465			
	st.dev. (n)	31.1890	RSD = 45%		
	R(calc.)	87.329			
	st.dev.(IEC62321-6:15)	21.9285			
	R(IEC62321-6:15)	61.400			

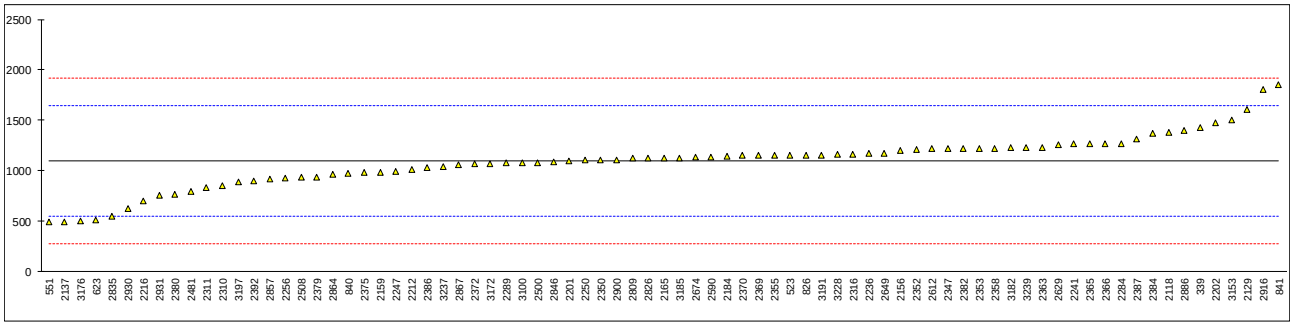


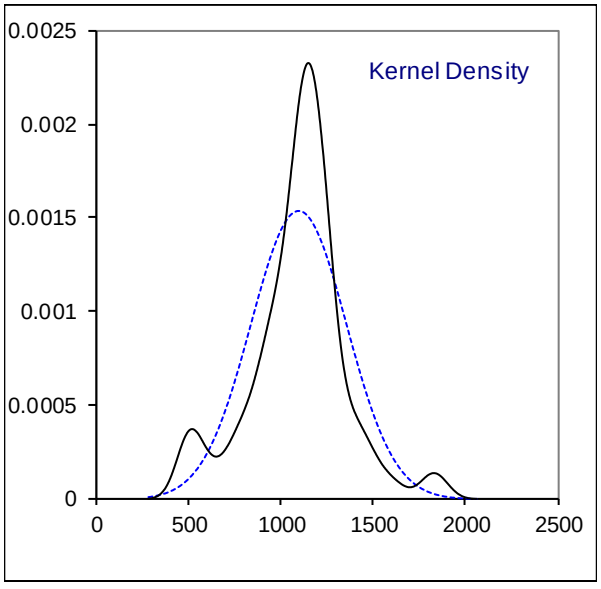


Determination of Decabromodiphenylether (Deca-BDE) on sample #20655; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	IEC62321-6Mod - GC/MS	1424		1.21	
523	IEC62321-6	1157.08	C	0.23	first reported 2233.15
551	IEC62321-6 - GC/MS	489.3434		-2.21	
623	IEC62321-6	514.3763	C	-2.12	first reported 514376.301
826	IEC62321-6 - GC/MS	1158		0.23	
840	IEC62321-6 - GC/MS	976		-0.43	
841	IEC62321-6 - GC/MS	1849.06		2.76	
1842	In house	not analysed		-----	
2115		-----		-----	
2118	In house	1384.03		1.06	
2129	IEC62321-6 - GC/MS	1609		1.88	
2137	IEC62321-6 - GC/MS	496.49		-2.19	
2156	IEC62321-6 - GC/ECD	1199.94		0.39	
2159	IEC62321-6 - GC/MS	985.3		-0.40	
2165	IEC62321-6 - GC/MS	1124		0.11	
2184	IEC62321-6 - GC/MS	1145		0.18	
2201	IEC62321-6 - GC/MS	1101.0		0.02	
2202	IEC62321-6 - GC/MS	1477		1.40	
2212	In house	1008		-0.32	
2216	IEC62321-6 - GC/MS	697.5		-1.45	
2236	In house	1169.24		0.27	
2241	IEC62321-6 - GC/MS	1265.0		0.62	
2247	IEC62321-6	997.00		-0.36	
2250	IEC62321-6Mod - GC/MS	1103		0.03	
2256	IEC62321-6 - GC/MS	926.37		-0.62	
2267		-----		-----	
2284	IEC62321-6 - GC/MS	1270		0.64	
2289	IEC62321-6 - HPLC-PDA/UV	1077		-0.06	
2301		-----		-----	
2310	IEC62321-6 - GC/MS	850		-0.89	
2311	IEC62321-6 - GC/MS	834		-0.95	
2316	IEC62321-6 - GC/MS	1162.8		0.25	
2347	IEC62321-6 - GC/MS	1220		0.46	
2350	IEC62321-6 - GC/MS	1106.0		0.04	
2352	IEC62321-6 - GC/MS	1209.1		0.42	
2353	IEC62321-6 - GC/MS	1223.91		0.47	
2355	IEC62321-6 - GC/MS	1156.5		0.23	
2358	IEC62321-6 - GC/MS	1223.91		0.47	
2363	IEC62321-6 - GC/MS	1234		0.51	
2365	IEC62321-6 - GC/MS	1265.1		0.62	
2366	IEC62321-6 - GC/MS	1266.36		0.63	
2369	IEC62321-6 - GC/MS	1154		0.22	
2370	IEC62321-6 - GC/MS	1150		0.20	
2372	IEC62321-6 - GC/MS	1066.95	C	-0.10	first reported not determined
2375	ISO17881-1	985		-0.40	
2379	IEC62321-6 - GC/MS	935.1721		-0.58	
2380	In house	769.74		-1.19	
2382	IEC62321-6 - GC/MS	1220.0		0.46	
2384	IEC62321-6 - GC/MS	1372.35		1.02	
2386	IEC62321-6 - GC/MS	1032		-0.23	
2387	IEC62321-6 - GC/MS	1311.319		0.79	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	900.1438	C	-0.71	first reported not detected, f.r. as #20656
2481	In house	794		-1.10	
2500	IEC62321-6 - GC/MS	1080.21		-0.05	
2508	In house	932.63		-0.59	
2590	IEC62321-6 - GC/MS	1133.091		0.14	
2612	IEC62321-6 - GC/MS	1217		0.45	
2621		-----		-----	
2622		-----		-----	
2629	IEC62321-6 - GC/MS	1255.9		0.59	
2649	ISO17881-1	1170	C	0.28	first reported 2170.0
2674	IEC62321-6 - GC/MS	1132		0.14	
2809	IEC62321-6 - GC/MS	1121.50		0.10	
2826	IEC62321-6 - GC/MS	1123.19		0.10	
2835	EPA3545A/IEC62321-6	545.3715		-2.01	
2846	IEC62321-6 - GC/MS	1087.13		-0.03	
2857	IEC62321-6 - GC/MS	921		-0.63	
2864	IEC62321-6 - GC/MS	963.97		-0.48	
2867	IEC62321-6 - GC/MS	1063		-0.12	
2886	In house	1400		1.12	
2900	IEC62321-6 - GC/MS	1106.1		0.04	
2914		-----		-----	
2916	In house	1805		2.60	

lab	method	value	mark	z(targ)	remarks
2930	IEC62321-6 - HPLC-PDA/UV	621		-1.73	
2931		759.29		-1.23	
2932		-----		-----	
3100	GB/T26125	1080		-0.05	
3153		1505		1.50	
3154		-----		-----	
3163		-----		-----	
3172	GB/T24279	1071.35		-0.08	
3176	ISO17881-1	500.0		-2.17	
3182	IEC62321-6 - GC/MS	1225.94		0.48	
3183		-----		-----	
3185	IEC62321-6 - GC/MS	1124.1		0.11	
3191	IEC62321-6 - GC/MS	1158.4		0.23	
3197	IEC62321-6 - GC/MS	885.6		-0.76	
3210		-----		-----	
3228	IEC62321-6 - GC/MS	1162		0.25	
3237	IEC62321-6 - GC/MS	1042		-0.19	
3239	IEC62321-6 - GC/MS	1229.58		0.49	
	normality	suspect			
	n	79			
	outliers	0			
	mean (n)	1094.512			
	st.dev. (n)	258.8330	RSD = 24%		
	R(calc.)	724.732			
	st.dev.(IEC62321-6:15)	273.3944			
	R(IEC62321-6:15)	765.504			

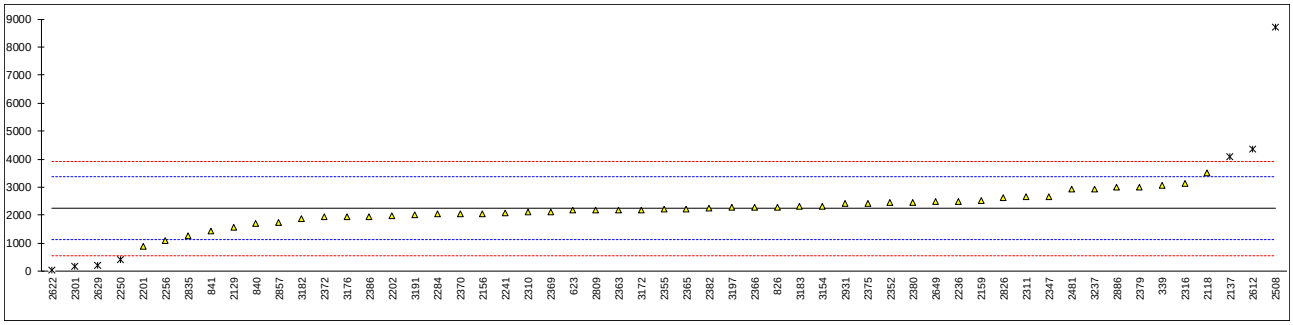


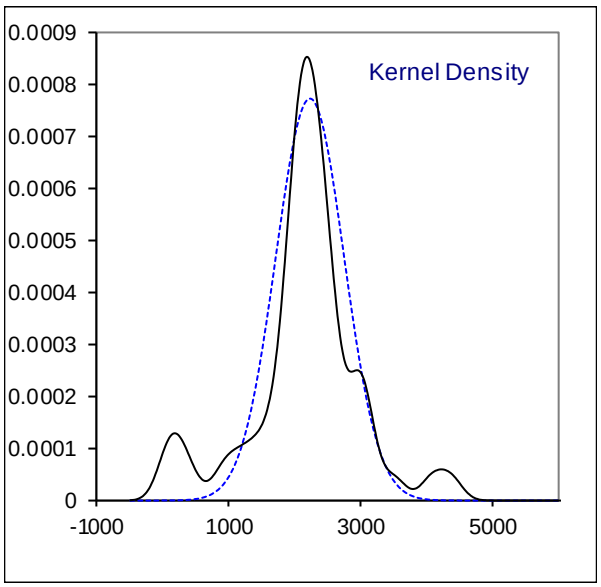


Determination of Hexabromocyclododecane (HBCDD) on sample #20656; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	IEC62321-6Mod - GC/MS	3072		1.48	
523		-----		-----	
551		-----		-----	
623	In house	2173.375		-0.12	
826	In house	2290		0.09	
840	IEC62321-6 - GC/MS	1705		-0.96	
841		1417.8		-1.47	
1842	In house	not analysed		-----	
2115		-----		-----	
2118	In house	3518.08		2.28	
2129	ISO17881-2 - LC/MS	1563		-1.21	
2137	IEC62321-6 - GC/MS	4090.07	R(0.05)	3.30	
2156	IEC62321-6 - GC/ECD	2058.87		-0.33	
2159	IEC62321-6 - GC/MS	2518.0		0.49	
2165	IEC62321-6 - GC/MS	Not applicable		-----	
2184	IEC62321-6 - GC/MS	not applicable		-----	
2201	IEC62321-6 - GC/MS	898.5		-2.40	
2202	In house	1970		-0.48	
2212	In house	Not Applicable		-----	
2216	IEC62321-6 - GC/MS	none detected		-----	possibly a false negative test result?
2236	In house	2484.15	C	0.43	first reported not detected
2241	IEC62321-6 - GC/MS	2089.6		-0.27	
2247	IEC62321-6	Detected		-----	
2250	In house	419	C,R(0.05)	-3.25	first reported 494
2256	IEC62321-6 - GC/MS	1083.37		-2.07	
2267		-----		-----	
2284	IEC62321-6 - GC/MS	2032		-0.37	
2289		-----		-----	
2301	In house	168.6831	R(0.05)	-3.70	
2310	IEC62321-6 - GC/MS	2100		-0.25	
2311	IEC62321-6 - GC/MS	2647		0.72	
2316	In house	3144	C	1.61	first reported 7023.7
2347	IEC62321-6 - GC/MS	2656		0.74	
2350	IEC62321-6 - GC/MS	NA		-----	
2352	IEC62321-6 - GC/MS	2440.0		0.35	
2353	IEC62321-6 - GC/MS	NA		-----	
2355	IEC62321-6 - GC/MS	2222.0		-0.03	
2358	IEC62321-6 - GC/MS	N/A		-----	
2363	IEC62321-6 - GC/MS	2190.5		-0.09	
2365	IEC62321-6 - GC/MS	2222.4		-0.03	
2366	IEC62321-6 - GC/MS	2288.23		0.08	
2369	IEC62321-6 - GC/MS	2124		-0.21	
2370	IEC62321-6 - GC/MS	2050		-0.34	
2372	IEC62321-6 - GC/MS	1933		-0.55	
2375		2427		0.33	
2379	IEC62321-6 - GC/MS	3009.1883		1.37	
2380	In house	2463.93		0.40	
2382	IEC62321-6 - GC/MS	2254.0	C	0.02	first reported <5
2384		-----		-----	
2386	IEC62321-9 - GC/MS	1961		-0.50	
2387		-----		-----	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	Not analysed	C	-----	first reported not detected
2481	In house	2927		1.22	
2500	IEC62321-6 - GC/MS	not applicable		-----	
2508		8697.57	R(0.01)	11.52	
2590		-----		-----	
2612	IEC62321-6 - GC/MS	4374	C,R(0.05)	3.81	first reported 7762
2621		-----		-----	
2622	In house	45.014	R(0.05)	-3.92	
2629	IEC62321-6	224.07	C,R(0.05)	-3.60	first reported 464.4
2649	ISO17881-1	2480	C	0.43	first reported 5088.0
2674	IEC62321-6 - GC/MS	N.A.		-----	
2809	IEC62321-6 - GC/MS	2178.50		-0.11	
2826	IEC62321-6 - GC/MS	2619.9515		0.68	
2835	EPA3545A&8270E	1267.09	C	-1.74	first reported not detected
2846	IEC62321-6 - GC/MS	not determined		-----	
2857	IEC62321-6 - GC/MS	1756		-0.87	
2864		-----		-----	
2867	IEC62321-6 - GC/MS	not applicable		-----	
2886	In house	3000		1.35	
2900	IEC62321-6 - GC/MS	not analysed		-----	
2914		-----		-----	
2916		-----		-----	
2930		<62	C, f-?	<-3.89	first reported not detected

lab	method	value	mark	z(targ)	remarks
2931		2418.68		0.32	
2932		-----		-----	
3100	GB/T26125	--		-----	
3153		not applicable		-----	
3154	IEC62321-6 - GC/MS	2328.7		0.16	
3163		-----		-----	
3172	GB/T24279	2191.4	C	-0.09	first reported 191.58
3176	ISO17881-1	1938.0		-0.54	
3182	IEC62321-6 - GC/MS	1890.80		-0.63	
3183		2327.75		0.15	
3185		-----		-----	
3191	IEC62321-6 - GC/MS	2023.1		-0.39	
3197	IEC62321-6 - GC/MS	2275		0.06	
3210		-----		-----	
3228	IEC62321-6 - GC/MS	N.A.		-----	
3237	IEC62321-6 - GC/MS	2946		1.26	
3239	IEC62321-6 - GC/MS	not analysed		-----	
	normality	OK			
	n	48			
	outliers	7			
	mean (n)	2241.124			
	st.dev. (n)	517.0380	RSD = 23%		
	R(calc.)	1447.706			
	st.dev.(IMEP-26:11)	560.2810			
	R(IMEP-26:11)	1568.787			





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 #20655; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
339	<1	<2	<10	2.03	<30	3.85 (TBBPA)
523	<5	<5	<5	<5	-----	-----
551	-----	-----	-----	11.5723	-----	-----
623	-----	-----	-----	-----	Not detected	-----
826	-----	-----	-----	-----	-----	-----
840	0	0	0	0	0	-----
841	< 5	< 5	< 5	< 5	< 5	< 5
1842	not analysed	not analysed	not analysed	not analysed	not analysed	2225
2115	-----	-----	-----	-----	-----	-----
2118	not detected	not detected	not detected	not detected	not detected	not detected
2129	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----
2156	< 20	< 20	< 20	< 20	< 20	< 20
2159	-----	-----	-----	-----	-----	-----
2165	<Reporting limit	<Reporting limit	<Reporting limit	<Reporting limit	Not applicable	<Reporting limit
2184	not detected	not detected	not detected	not detected	not applicable	not detected
2201	not detected	not detected	not detected	not detected	not detected	not detected
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2212	Not Applicable	Not Applicable	Not Applicable	<100	Not Applicable	<100
2216	none detected	none detected	none detected	none detected	none detected	none detected
2236	Not detected	Not detected	Not detected	Not detected	Not detected	-----
2241	<10	<10	<10	<10	<10	<10
2247	not detected	not detected	not detected	not detected	not detected	not detected
2250	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----
2267	-----	-----	-----	-----	-----	-----
2284	-----	-----	-----	-----	-----	-----
2289	<5	<5	<5	<5	-----	<5
2301	-----	-----	-----	-----	-----	-----
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
2347	<5	<5	<5	<5	<10	-----
2350	<5	<5	<5	<5	NA	NA
2352	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	NA	ND
2355	＜5	＜5	＜5	＜5	＜10	-----
2358	n.d.	n.d.	n.d.	n.d.	N/A	n.d.
2363	<5	<5	<5	<5	<5	<5
2365	<5	<5	<5	<5	<10	-----
2366	<5	<5	<5	<5	<5	not determined
2369	<5	<5	<5	<5	<10	<5
2370	<5	<5	<5	<5	<5	<5
2372	not determined	not determined	not determined	not determined C	not determined	not determined
2375	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2380	<5	<5	<5	-----	<5	-----
2382	＜5.0	＜5.0	＜5.0	＜5.0	＜5.0	＜5.0
2384	Not Detected [<5]	Not Detected [<5]	Not Detected [<5]	Not Detected [<5]	Not Detected [<5]	-----
2386	< 25	< 25	< 50	< 25	< 50	-----
2387	Not Detected [<5]	Not Detected [<5]	Not Detected [<5]	Not Detected [<5]	-----	-----
2390	-----	-----	-----	-----	-----	-----
2392	Not detected	Not detected	Not detected	Not detected	Not analysed C	Not analysed C
2481	-----	-----	-----	-----	-----	-----
2500	<50	<50	<50	<50	not applicable	not applicable
2508	-----	-----	-----	26.87	-----	-----

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
2590	-----	-----	-----	-----	-----	-----
2612	not detected	not detected	not detected	not detected	not detected	not detected
2621	-----	-----	-----	-----	-----	-----
2622	-----	-----	-----	-----	not detected	-----
2629	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	-----
2649	ND	ND	ND	ND	ND	ND
2674	<RL	<RL	<RL	<RL	N.A.	<RL
2809	-----	-----	-----	-----	-----	-----
2826	<100	<100	<100	<100	<50	-----
2835	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2846	-----	-----	-----	-----	-----	-----
2857	not determined	not determined	not determined	not determined	not determined	not analysed
2864	not determined	not determined	not determined	8.85	-----	-----
2867	not detected	not detected	not detected	not detected	not detected	not detected
2886	Not detected	Not detected	Not detected	Not detected	Not detected	-----
2900	not detected	not detected	not detected	not detected	not analysed	not analysed
2914	-----	-----	-----	-----	-----	-----
2916	-----	-----	136	64	-----	-----
2930	not detected	not detected	not detected	not detected	not detected	not detected
2931	not detected	not detected	not detected	not detected	not detected	not detected
2932	-----	-----	-----	-----	-----	-----
3100	<5	<5	<5	<5	--	--
3153	<20	<20	<20	<20	not applicable	not applicable
3154	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5	-----
3176	-----	-----	-----	-----	-----	-----
3182	< 5	< 5	< 5	< 5	< 5	< 5
3183	-----	-----	-----	-----	not detected	-----
3185	not detected	not detected	not detected	not detected	-----	-----
3191	-----	-----	-----	-----	-----	-----
3197	<20	<20	<20	<20	<20	-----
3210	-----	-----	-----	-----	-----	-----
3228	n.d.	n.d.	n.d.	n.d.	N.A.	n.d.
3237	-----	-----	-----	-----	-----	-----
3239	not detected (<50)	not detected (<50)	not detected (<50)	not detected (<50)	not analysed	not detected (<50)

Lab 2372: first reported 97.66 for Octa-BDE
Lab 2392: first reported not detected for both HBCDD and Other

Other reported Brominated Flame retardants in sample #20656; 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	3.09 (Penta-BDE)
523	<5	<5	<5	<5	<5	<5	-----
551	-----	-----	-----	-----	-----	-----	-----
623	-----	-----	-----	-----	-----	Not detected	-----
826	-----	-----	-----	-----	-----	-----	-----
840	0	0	0	0	12	152	-----
841	< 5	< 5	< 5	< 5	< 5	6.40	< 5
1842	not analysed	not analysed	not analysed	not analysed	not analysed	not analysed	2259
2115	-----	-----	-----	-----	-----	-----	-----
2118	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2129	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----
2156	< 20	< 20	< 20	< 20	< 20	< 20	< 20
2159	-----	-----	-----	-----	-----	-----	-----
2165	<Reporting limit	<Reporting limit	<Reporting limit	<Reporting limit	<Reporting limit	<Reporting limit	<Reporting limit
2184	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2201	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2212	Not Applicable	Not Applicable	Not Applicable	<100	Not Applicable	<100	<100
2216	none detected	none detected	none detected	none detected	none detected	none detected	none detected
2236	Not detected	Not detected	Not detected	Not detected	Not detected	7.53	-----
2241	<10	<10	<10	<10	<10	<10	<10
2247	not detected	not detected	not detected	not detected	not detected	92.70	not detected
2250	-----	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----	-----
2267	-----	-----	-----	-----	-----	-----	-----
2284	-----	-----	-----	-----	-----	-----	-----
2289	<5	<5	<5	<5	<5	<5	<5
2301	-----	-----	-----	-----	-----	-----	-----
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	<100	Not Detected
2316	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2347	<5	<5	<5	<5	<5	<5	-----

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
2350	<5	<5	<5	<5	<5	<5	NA
2352	-----	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	ND	ND	ND
2355	<5	<5	<5	<5	<5	<5	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<5	<5	<5	<5	<5	<5	<5
2365	<5	<5	<5	<5	<5	<5	-----
2366	<5	<5	<5	<5	<5	<5	not determined
2369	<5	<5	<5	<5	<5	<5	<5
2370	<5	<5	<5	<5	<5	<5	<5
2372	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2375	-----	-----	-----	-----	-----	21	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2380	<5	<5	<5	<5	<5	<5	-----
2382	＜5.0	＜5.0	＜5.0	＜5.0	<5 C	＜5.0	＜5.0
2384	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	-----
2386	< 25	< 25	< 50	< 25	< 25	< 50	-----
2387	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	Not Detect. [<5]	-----
2390	-----	-----	-----	-----	-----	-----	-----
2392	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not analysed
2481	-----	-----	-----	-----	-----	74.4	-----
2500	<50	<50	<50	<50	<50	<50	not applicable
2508	-----	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	46.479	-----
2612	not detected	not detected	not detected	not detected	not detected	29.4	not detected
2621	-----	-----	-----	-----	-----	-----	-----
2622	-----	-----	-----	-----	-----	-----	-----
2629	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	< 5 mg/kg	-----
2649	ND	ND	ND	ND	ND	ND	ND
2674	<RL	<RL	<RL	<RL	<RL	<RL	<RL
2809	-----	-----	-----	-----	-----	-----	-----
2826	<100	<100	<100	<100	<100	<100	-----
2835	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2846	-----	-----	-----	-----	-----	-----	-----
2857	not detected	not detected	not detected	not detected	not detected	not detected	not analysed
2864	not determined	not determined	not determined	not determined	N.D.	5.29	-----
2867	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2886	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	-----
2900	not detected	not detected	not detected	not detected	not detected	not detected	not analysed
2914	-----	-----	-----	-----	-----	-----	-----
2916	-----	-----	76.7	0.44	1.6	17.1	-----
2930	not detected	not detected	not detected	not detected	not detected	15	not detected
2931	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2932	-----	-----	-----	-----	-----	-----	-----
3100	<5	<5	<5	<5	<5	<5	--
3153	<20	<20	<20	<20	<20	<20	not applicable
3154	-----	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5	< 5	-----
3176	-----	-----	-----	-----	-----	12.0	-----
3182	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3183	-----	-----	-----	-----	-----	-----	-----
3185	not detected	not detected	not detected	not detected	not detected	not detected	-----
3191	-----	-----	-----	-----	-----	-----	-----
3197	<20	<20	<20	<20	<20	<20	-----
3210	-----	-----	-----	-----	-----	-----	-----
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	N.D.
3237	-----	-----	-----	-----	-----	-----	-----
3239	not detect. (<50)	not detect. (<50)	not detect. (<50)	not detect. (<50)	not detect. (<50)	not detec (<150)	not detect. (<50)

Lab 2382: first reported 2254 for Nona-BDE
Lab 2392: f.r. as #20655: first reported 75.6760 for Nona-BDE, 900.1438 for Deca-BDE and not detected for Other.

APPENDIX 3

Analytical details

lab	ISO17025 accredited	sample intake (g)	sample grinded or cut	release/extract technique	release solvent	extraction time (min)	extraction temp (°C)
339	No	1g	Used as received	Ultrasonic	Toluene	60	60
523	Yes	2 g	Further cut	Ultrasonic	Toluene	60 min	50
551	Yes	0.1g	Further cut	Soxhlet	Toluene.	120 minutes.	Toluene's boiling temp.
623	Yes	Deca-BDE = 0.1 gr ; HBCDD = 1 gr	Further cut	Ultrasonic	Toluene	Deca-BDE=30min, HBCDD=1hour	50
826	Yes		Further grinded	Ultrasonic	Toluene	60 mins	60
840	Yes	0.10	Further cut	Ultrasonic	Toluene	60	50
841	Yes	0.5036 grams	Further cut	Ultrasonic	toluene	1 hour	50
1842	Yes		Used as received	---			
2115	---		---	---			
2118	No	0.5 g	Used as received	Ultrasonic	toluene/MeOH (60/40)	120 minutes	80
2129	Yes	0,5g per sample	Further grinded	Ultrasonic	#20655: dichloromethane #20656: toluene	#20655: 30 min #20656: 60 min	#20655: room temp. #20656: 60
2137	Yes		Further cut	Ultrasonic	Toluene		40
2156	Yes	0.5 grams	Further cut	Ultrasonic	Toluene	120	60
2159	Yes	0,5	Further cut	Ultrasonic	Toluene, 10 ml	120	70
2165	Yes	0.5	Further cut	Ultrasonic	Toluene	180	60
2184	Yes	2 gram	Used as received	Ultrasonic	Toluene	3 hours	60
2201	Yes	0.1g	Further cut	Ultrasonic	Toluene	2h	70
2202	Yes	0.5g	Used as received	Stirrer	THF/Toluene/Hexane	4 hours	room temp.
2212	Yes	0.1g, 0.05g, 0.01g	Further cut	Ultrasonic	Toluene	60 minutes	60
2216	Yes	0.1 grams	Further grinded	Soxhlet	toluene	2 hour	reflux; not measured
2236	Yes	0.5 grams	Used as received	Ultrasonic	Toluene	60 minutes	70
2241	Yes	0.5 grams	Further cut	Ultrasonic	toluene	60	60
2247	Yes		Further cut	Ultrasonic	Toluene	60	60
2250	Yes	0,5	Used as received	Soxhlet	Toluene	120	200
2256	Yes	0.5028g	Further cut	Soxhlet	Toluene	2h	Soxhlet extract
2267	---		---	---			
2284	Yes	0.2g	Further grinded	Ultrasonic	Toluene	60 min	50
2289	Yes	0.1g	Further cut	Soxhlet	toluene	120mins	110
2301	No	1 gram	Used as received	Ultrasonic	Toluene	120 minutes	70
2310	Yes	O.1	Used as received	Ultrasonic	Toluene	30	60
2311	Yes	0.1 0.1g Flame ret.	Further cut	Soxhlet	Toluene	120	120
2316	Yes	0.3g for HBCDD	Further grinded	Soxhlet	Toluene	2 hours	70
2347	Yes	0.1g	Further cut	Soxhlet	Toluene	6hours	
2350	Yes	0.1g	Used as received	Ultrasonic	Toluene	120min	50
2352	Yes	0.1g	Further cut	Soxhlet	Toluene	240min	
2353	Yes	0.1g	Further grinded	Soxhlet	Toluene	2 hours	20-30 cycles per hour
2355	Yes	0.1	Further cut	Soxhlet, HBCDD = Ultrasonic	Toluene	PBBPBDE:16h HBCDD:60min	HBCDD:60
2358	Yes	0.1 g	Further grinded	Soxhlet	Toluene	2 hours	20-30 cycle per hour
2363	Yes	2g	Further grinded	Soxhlet	Methylbenzene	240min	60
2365	Yes	0.1g	Further grinded	Soxhlet	Toluene	180min	250
2366	Yes	0.1g	Further cut	Soxhlet	toluene	2h	270
2369	---		---	---			
2370	Yes		Further grinded	Soxhlet	toluene	1hr47mins	250
2372	Yes	0.5g	Further cut	Soxhlet	Toluene	70	250
2375	Yes	0,3 gram	Further cut	Ultrasonic	Toluene	60 minutes	60
2379	Yes	0.1 g	Further grinded	Soxhlet	Toluene	4 Hr	-
2380	Yes	0.3 g	Used as received	Ultrasonic	Toluene	60 min	60
2382	Yes	0.1g	Further cut	#20655 = Soxhlet, #20656 = Ultrasonic	10ml toluene	analyte : soxhlet 4h analyte : Ultrasonic 1h	60
2384	Yes	0.5g	Further grinded	Soxhlet	Toluene	960 minutes	reflux temp.
2386	Yes	1.0 g	Further grinded	Soxhlet	Toluene	4 h	Soxhlet conditions
2387	Yes	1g	Further grinded	Soxhlet	Toluene	960-1260	Reflux Temp.
2390	---		---	---			

lab	ISO17025 accredited	sample intake (g)	sample grinded or cut	release/extract technique	release solvent	extraction time (min)	extraction temp (°C)
2392	Yes	0.1 g	Further grinded	Soxhlet	Toluene	120 min	60
2481	Yes	250 ± 25 mg	Further cut	Ultrasonic	Toluene	60 min	60
2500	Yes	0.1 g	Further cut	Ultrasonic	toluene	120 min	70
2508	Yes	0,5	Used as received	Ultrasonic	Toluene	1X30 & 1x20	RT
2590	No	0.5 g	Further cut	Soxhlet	DCM:toluene	360 min	Not applicable
2612	Yes	0,1g	Further cut	Ultrasonic	Toluene	60 minutes	60
2621	---		---	---			
2622	No	0.55	Used as received	Ultrasonic	Isooctane	45 minutes	50
2629	Yes	0.5 g	Used as received	Ultrasonic	For PBDE: Toluene; For HBCDD: toluene	60 minutes	70
2649	Yes	20655: 1.5 gm 20656: 2.0 gm	Further cut	Ultrasonic	Tolune	2 hours	70
2674	Yes	0.5g	Used as received	Ultrasonic	Toluene	180mins	60
2809	Yes	0.2	Further cut	Ultrasonic	TOLUENE	120	70
2826	Yes	0.1g	Further grinded	Soxhlet	Toluene	150mins	
2835	Yes	HBCDD-0.2g PBB & PBDE-0.5g	Further cut	ASE	PBB & PBDE-Toluene HBCDD-Methylene Chloride	15 min	150
2846	Yes	0.746 ~ 0.769 g	Other	Ultrasonic	THF(tetrahydrofuran) and toluene	Over 60 min	30
2857	Yes	PBB&PBDE 0.1g HBCDD 0.2g	Further cut	Ultrasonic	PBB&PBDE Toluene:MeOH =9:1 HBCDD Toluene:MeOH =1:1	1H	60
2864	Yes	0.2g	Further cut	Soxhlet	toluene	180 min	60
2867	Yes	0.5g	Further cut	Ultrasonic	Toluene	180min	60℃
2886	No	1	Used as received	Ultrasonic	Cyclohexan + acetone	120	Not measured
2900	Yes	0.2 g	Further cut	Ultrasonic	Toluene	120	70
2914	---		---	---			
2916	No	0,1-0,2g	Further cut	Mechanical Shaking	Toluene	250	25°C
2930	Yes	0,1 g	Further cut	Ultrasonic	n-Propanol	15	40°C
2931	No		---	---			
2932	---		---	---			
3100	Yes	0.1g	Further cut	Soxhlet	Toluene		
3153	Yes	0.2g each	Further grinded	Soxhlet	Toluene	4 hours reflux	not applicable
3154	---		---	---			
3163	---		---	---			
3172	Yes	0.5	Used as received	Ultrasonic	Acetone-Toluene	45	25
3176	No	1	Used as received	Ultrasonic	Toluene	45	room temp. Lab used soxhlet to extract at 2.5
3182	Yes	0.3 grams	Further grinded	Soxhlet	Toluene	360 minutes	minutes/cycle
3183	No	130 mg	The weighed amount of sample was extracted in one piece	Ultrasonic	Tetrahydrofuran for extraction, Hexan for solving	60 minutes	60
3185	Yes	0.2g	Further cut	Soxhlet	Toluene	240mins	not applicable
3191	Yes	0.1 g	Further grinded	Soxhlet	Toluene.	150 min.	About 140
3197	Yes		Further cut	Soxhlet	Toluene	120 minutes	Reflux Temp
3210	---		---	---			
3228	Yes	0.5	Further cut	Ultrasonic	toluene	180	60
3237	Yes	0,25 gr	Further grinded	Soxhlet	Toluene	120 min	200
3239	Yes	3g was received. About 1.5g of sample was further grinded. 0.1g of grinded sample was used for extraction and analysis.	Further grinded	Soxhlet	Toluene	2 hours	115

APPENDIX 4

Number of participants per country

2 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in DENMARK
3 labs in FRANCE
10 labs in GERMANY
6 labs in HONG KONG
5 labs in INDIA
2 labs in INDONESIA
4 labs in ITALY
3 labs in MALAYSIA
1 lab in MEXICO
21 labs in P.R. of CHINA
1 lab in PAKISTAN
2 labs in SINGAPORE
5 labs in SOUTH KOREA
1 lab in SWITZERLAND
5 labs in TAIWAN
3 labs in THAILAND
2 labs in THE NETHERLANDS
5 labs in TURKEY
2 labs in U.S.A.
1 lab in UNITED KINGDOM
5 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
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
f.r.	= first reported
f+?	= possible false positive test result?
f-?	= possible false negative test result?

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