Results of Proficiency Test Total Brominated Flame Retardants in Polymers September 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing. A. Ouwerkerk
Report: iis21P07

November 2021

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. 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 2021/2022 it was decided to continue with the proficiency test for the analysis of total Brominated Flame Retardants in polymers.

In this interlaboratory study 82 laboratories in 23 different 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 2 different samples positive on some Brominated Flame Retardants, labelled #21705 and #21706 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 purple PVC rings, artificially fortified with Hexabromocyclododecane (HBCDD), was selected. After homogenization small plastic bags were filled with approximately 3 grams and labelled #21705.
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 #21705-1	702
sample #21705-2	668
sample #21705-3	684
sample #21705-4	653
sample #21705-5	662
sample #21705-6	662
sample #21705-7	651
sample #21705-8	683

Table 1: homogeneity test results of subsamples #21705

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)	49
reference test method	IMEP-26:11
0.3 x R (reference test method)	141

Table 2: evaluation of the repeatability of subsamples #21705

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 pink PVC blocks, artificially fortified with Decabromodiphenylether (Deca-BDE), was selected. After homogenization small plastic bags were filled with approximately 3 grams and labelled #21706.
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 #21706-1	3978
sample #21706-2	3994
sample #21706-3	4183
sample #21706-4	4122
sample #21706-5	4116
sample #21706-6	4065
sample #21706-7	3933
sample #21706-8	4078

Table 3: homogeneity test results of subsamples #21706

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)	236
reference test method	IEC62321-6:15
0.3 x R (reference test method)	840

Table 4: evaluation of the repeatability of subsamples #21706

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 one sample labelled #21705 and one sample labelled #21706 was sent on August 18, 2021.

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), Hexabromocyclododecane (HBCDD) and other Brominated Flame Retardant(s). 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 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 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.

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, 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

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. One participant reported test results after the final reporting date and nine other participants did not report any test results. Not all participants were able to report all tests requested.
In total 73 participants reported 183 numerical test results. Observed were 9 outlying test results, which is 4.9%. 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.

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. 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.

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 #21705

HBCDD: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full 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 were calculated for these components. The reported results can be found in appendix 2.

sample #21706

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. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 were calculated for these components. The reported results can be found 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 official test method IEC62321-6:2015 and derived from the IMEP-26 results are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
HBCDD	mg/kg	47	685	477	479

Table 5: reproducibility of test on sample #21705

Component	unit	n	average	2.8 * sd	R(lit)
Nona-BDE	mg/kg	58	158	206	122
Deca-BDE	mg/kg	69	3164	1619	2187

Table 6: reproducibilities of tests on sample #21706

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 2021 WITH PREVIOUS PTS

	September 2021	September 2020	August 2019	September 2018	September 2017
Number of reporting laboratories	73	84	67	77	66
Number of test results	183	193	168	256	195
Number of statistical outliers	9	8	8	9	14
Percentage of statistical outliers	4.9%	4.1%	4.8%	3.5%	7.2%

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 2021	September 2020	August 2019	September 2018	2017 -2009	Target*)
Nona-BDE	47%	45%	43%	n.e.	15-51%	25-34%
Deca-BDE	18%	24%	24%	28%	10-37%	25-34%
Deca-BB	n.e.	n.e.	n.e.	22%	n.e.	25%
HBCDD	25%	23%	(151%)	17%	24-49%	25%

Table 8: development of the uncertainties over the years

*) The target of IEC62321-6:2015 is dependent on concentration.

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

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method IEC62321-6-GC/MS is used by the majority of the reporting participants. A number of participants used a different test method for the determination of HBCDD than for the determination of PBDE and PBB.

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

- A vast majority (about 90%) of the reporting participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- About 15% of the reporting participants used the sample as received and about 80% did further cut or further grind the samples prior to analysis.
- About 57% used 0.5 grams or less of sample intake and about 30% used a sample intake of 0.5 to 1 gram.
- To release the components from the sample about 55% used ultrasonic and about 40% of used Soxhlet.

- Almost all participants (about 95%) used Toluene or a Toluene mixture as solvent to release the analytes.
- The extraction time used differs from 15 minutes to 21 hours. About 45% used an extraction time of 120 to 180 minutes, about 30% used an extraction time less than 120 minutes and about 25% 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 #21705 and sample #21706) 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 2021 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 (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 all of the reporting laboratories would reject sample #21706 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 #21705.

6 CONCLUSION

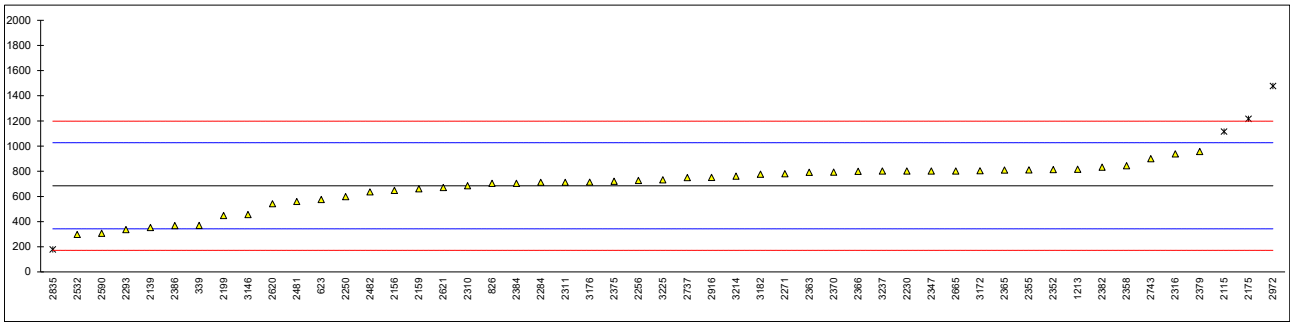
Although it can be concluded that most of the participants have no problem with the determination on PBDE and HBCDD in PVC, each participating laboratory will have to evaluate its performance in this study and make decisions about any necessary corrective actions. Therefore, participation on a regular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

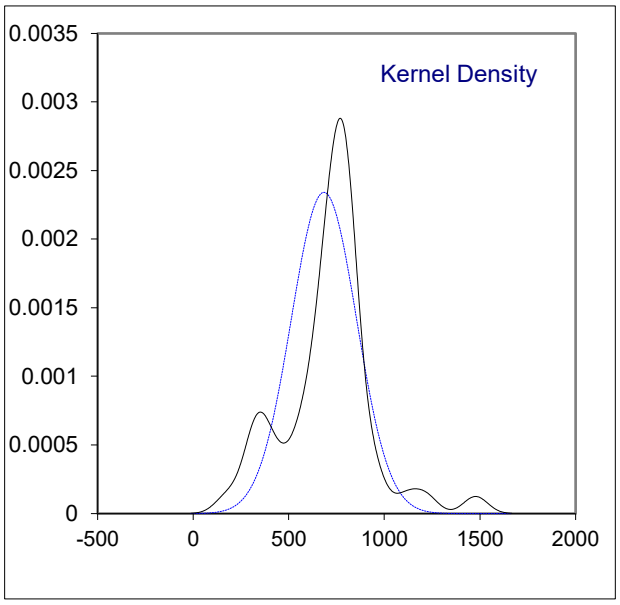
APPENDIX 1

Determination of Hexabromocyclododecane (HBCDD) on sample #21705; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339	IEC62321-6 - GC/MS	369		-1.84	
523	IEC62321-6 - GC/MS	Not analyzed	C	-----	first reported not detected
551		-----		-----	
623	In house	575.83		-0.64	
826	In house	704		0.11	
841		-----		-----	
1213	IEC62321-6 - GC/MS	815.33		0.76	
2115	In house	1115.2	R(0.05)	2.52	
2139	IEC62321-6 - GC/MS	352.66		-1.94	
2156	IEC62321-6 - GC/MS	647.23		-0.22	
2159	In house	660.19		-0.14	
2175	In house	1216.9	R(0.05)	3.11	
2176		-----		-----	
2184		-----		-----	
2199	IEC62321-6 - GC/MS	448.33		-1.38	
2201	IEC62321-6 - GC/MS	not applicable		-----	
2202	IEC62321-6 - GC/MS	N.D.		-----	
2230	In house	802		0.69	
2232		-----		-----	
2236		-----		-----	
2250	In house	598		-0.51	
2256	IEC62321-6 - GC/MS	727		0.25	
2265		-----		-----	
2271	In house	780.1		0.56	
2284	IEC62321-6 - GC/MS	712.2		0.16	
2293	IEC62321-6 - GC/MS	335.82		-2.04	
2310	IEC62321-6 - GC/MS	685		0.00	
2311	IEC62321-6 - GC/MS	712.25		0.16	
2316	In house	938.3		1.48	
2320		-----		-----	
2347	IEC62321-6 - GC/MS	802		0.69	
2350	IEC62321-6 - GC/MS	not analyzed		-----	
2352	IEC62321-6 - GC/MS	813.3		0.75	
2353	In house	NA		-----	
2355	IEC62321-6 - GC/MS	811.0		0.74	
2358	IEC62321-6 - GC/MS	843.5		0.93	
2363	IEC62321-6 - GC/MS	792.4		0.63	
2365	IEC62321-6 - GC/MS	809.6		0.73	
2366	IEC62321-6 - GC/MS	799		0.67	
2370	GB/T29493.1	794		0.64	
2375	IEC62321-6 - GC/MS	721		0.21	
2379	IEC62321-6 - GC/MS	957.1992		1.59	
2382	In house	832.1		0.86	
2384	IEC62321-6 - GC/MS	704.62		0.12	
2386	IEC62321-6 - GC/MS	368		-1.85	
2387		-----		-----	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	Not analyzed		-----	
2415		-----		-----	
2481	In house	560		-0.73	
2482	IEC62321-6 - HPLC/QQQ	636		-0.28	
2488	IEC62321-6 - GC/MS	n.d		-----	
2532	IEC62321-6 - GC/MS	297		-2.26	
2568		-----		-----	
2590	IEC62321-6 - GC/MS	306.36		-2.21	
2620	IEC62321-6 - GC/MS	542		-0.83	
2621	IEC62321-6 - GC/MS	671.412		-0.08	
2665	In house	802.2		0.69	
2674	IEC62321-6 - GC/MS	not applicable		-----	
2737	ISO17881-1	749.4		0.38	
2743	ISO17881-1	900.801116		1.26	
2826		-----		-----	
2835	IEC62321-6 - GC/MS	178.08	C,R(0.05)	-2.96	first reported 67.4
2864	IEC62321-6 - GC/MS	not determined		-----	
2910	IEC62321-6 - GC/MS	not applicable		-----	
2916	IEC62321-6 - GC/MS	751		0.39	
2930		-----		-----	
2970		-----		-----	
2972	IEC62321-6 - GC/MS	1477.88	R(0.05)	4.64	
2981		-----		-----	
3100	GB/T26125	not determined		-----	
3146	In house	456.13		-1.33	

lab	method	value	mark	z(targ)	remarks
3163		-----		-----	
3172	IEC62321-6 - GC/MS	804.345		0.70	
3176	IEC62321-6 - GC/MS	714.0		0.17	
3182	IEC62321-6 - GC/MS	776.58		0.54	
3185		-----		-----	
3210		-----		-----	
3214	IEC62321-6 - GC/MS	762.28		0.45	
3225	IEC62321-6 - GC/MS	732		0.28	
3237	IEC62321-6 - GC/MS	801.9		0.69	
	normality	OK			
	n	47			
	outliers	4			
	mean (n)	684.561			
	st.dev. (n)	170.5056	RSD = 25%		
	R(calc.)	477.416			
	st.dev.(IMEP-26:11)	171.1403			
	R(IMEP-26:11)	479.193			

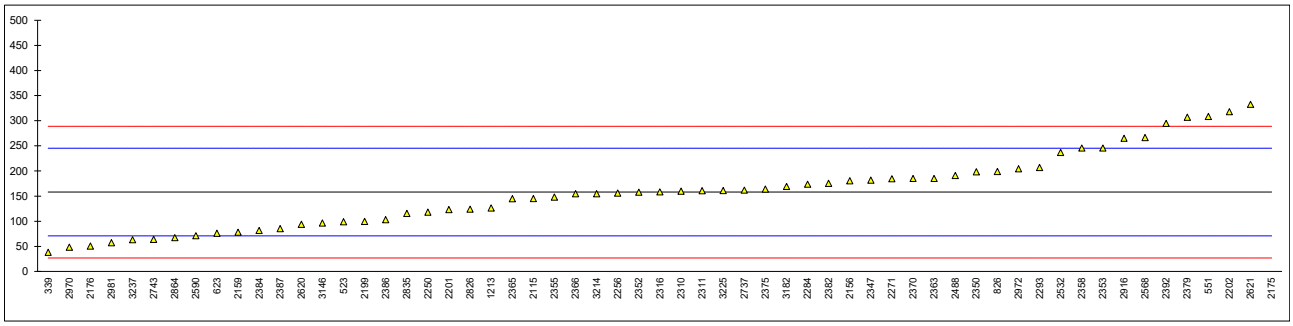


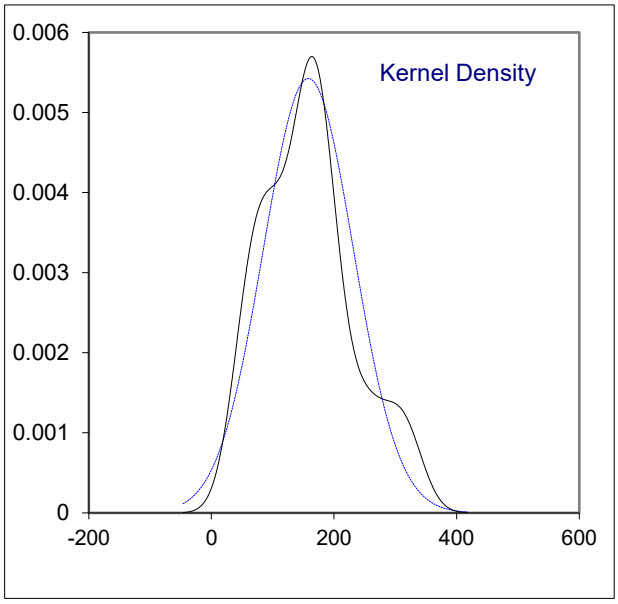


Determination of Nonabromodiphenylether (Nona-BDE) on sample #21706; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339	IEC62321-6 - GC/MS	38.2		-2.74	
523	IEC62321-6 - GC/MS	99.10		-1.35	
551	IEC62321-6 - GC/MS	308.626		3.45	
623	IEC62321-6 - GC/MS	75.99		-1.88	
826	IEC62321-6 - GC/MS	199		0.94	
841		-----		-----	
1213	IEC62321-6 - GC/MS	126.33		-0.73	
2115	In house	145.1		-0.30	
2139		-----		-----	
2156	IEC62321-6 - GC/MS	180.5		0.52	
2159	In house	78.04		-1.83	
2175	In house	8566.9	R(0.01)	192.65	
2176	IEC62321-6 - GC/MS	50.5		-2.46	
2184	IEC62321-6 - GC/MS	not applicable		-----	
2199	IEC62321-6 - GC/MS	99.5	C	-1.34	first reported <50
2201	IEC62321-6 - GC/MS	123	C	-0.80	first reported ND (<100)
2202	IEC62321-6 - GC/MS	318		3.67	
2230	IEC62321-6 - GC/MS	<5	f-?	<-3.51	possibly a false negative test result?
2232		-----		-----	
2236		-----		-----	
2250	IEC62321-6 - GC/MS	118	C	-0.92	first reported 402
2256	IEC62321-6 - GC/MS	156		-0.05	
2265		-----		-----	
2271	IEC62321-6 - GC/MS	184.5		0.61	
2284	IEC62321-6 - GC/MS	173.7		0.36	
2293	IEC62321-6 - GC/MS	206.8		1.12	
2310		160		0.05	
2311	IEC62321-6 - GC/MS	160.68		0.06	
2316	IEC62321-6 - GC/MS	158.3		0.01	
2320		-----		-----	
2347	IEC62321-6 - GC/MS	182		0.55	
2350	IEC62321-6 - GC/MS	198.3		0.92	
2352	IEC62321-6 - GC/MS	157.9		0.00	
2353	IEC62321-6 - GC/MS	245.68		2.01	
2355	IEC62321-6 - GC/MS	148.0		-0.23	
2358	IEC62321-6 - GC/MS	245.68		2.01	
2363	IEC62321-6 - GC/MS	185.2		0.62	
2365	IEC62321-6 - GC/MS	145.0		-0.30	
2366	IEC62321-6 - GC/MS	155		-0.07	
2370	IEC62321-6 - GC/MS	185		0.62	
2375	IEC62321-6 - GC/MS	164		0.14	
2379	IEC62321-6 - GC/MS	307.0931	C	3.42	first reported 424.782
2382	IEC62321-6 - GC/MS	175.3		0.40	
2384	IEC62321-6 - GC/MS	81.71		-1.75	
2386	IEC62321-6 - GC/MS	103		-1.26	
2387	IEC62321-6 - GC/MS	85.2		-1.67	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	294.7492		3.13	
2415		-----		-----	
2481	In house	Not analysed		-----	
2482		-----		-----	
2488	IEC62321-6 - GC/MS	191.1	C	0.76	first reported 19.1
2532	IEC62321-6 - GC/MS	237		1.81	
2568	IEC62321-6 - GC/MS	266.70	C	2.49	first reported 354.56
2590	IEC62321-6 - GC/MS	71.16		-1.99	
2620	IEC62321-6 - GC/MS	94		-1.47	
2621	IEC62321-6 - GC/MS	332.836		4.01	
2665	In house	not analyzed		-----	
2674	IEC62321-6 - GC/MS	not applicable		-----	
2737	ISO17881-1	161.8		0.09	
2743	ISO17881-1	64.1349791		-2.15	
2826	IEC62321-6 - GC/MS	124		-0.78	
2835	IEC62321-6 - GC/MS	115.335		-0.98	
2864		67.41		-2.08	
2910	IEC62321-6 - GC/MS	not applicable		-----	
2916	IEC62321-6 - GC/MS	265		2.45	
2930		-----		-----	
2970	IEC62321-6 - GC/MS	48.42		-2.51	
2972	IEC62321-6 - GC/MS	204.6		1.07	
2981	IEC62321-6 - GC/MS	57.26		-2.31	
3100	GB/T26125	not detected		-----	
3146	In house	96.42		-1.41	
3163		-----		-----	

lab	method	value	mark	z(targ)	remarks
3172		-----		-----	
3176		-----		-----	
3182	IEC62321-6 - GC/MS	169.14		0.26	
3185		-----		-----	
3210		-----		-----	
3214	IEC62321-6 - GC/MS	155		-0.07	
3225	IEC62321-6 - GC/MS	161	C	0.07	first reported 355
3237	IEC62321-6 - GC/MS	63.3		-2.17	
	normality	OK			
	n	58			
	outliers	1			
	mean (n)	158.005			
	st.dev. (n)	73.5694	RSD = 47%		
	R(calc.)	205.994			
	st.dev.(IEC62321-6:15)	43.6492			
	R(IEC62321-6:15)	122.218			

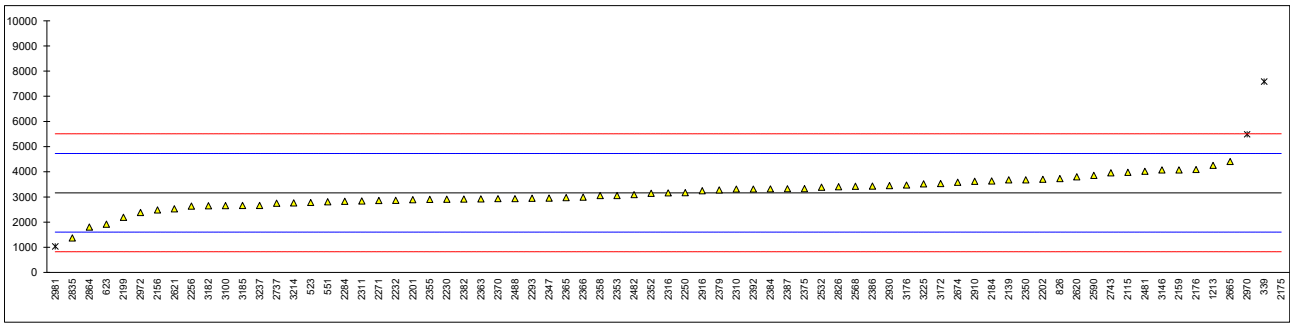


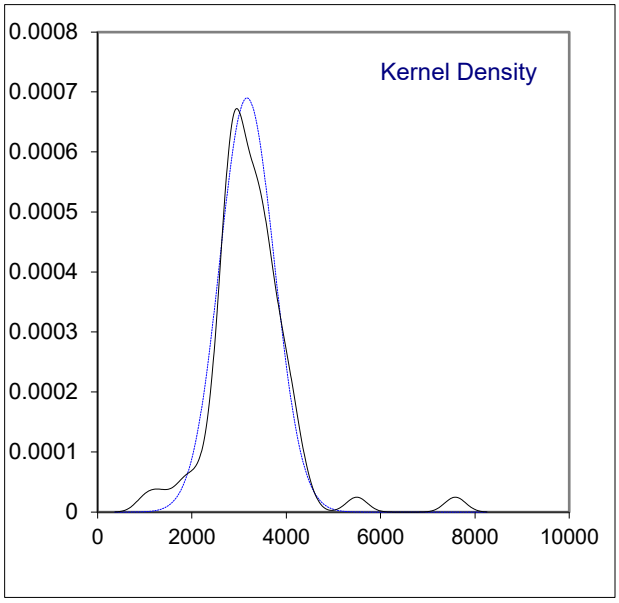


Determination of Decabromodiphenylether (Deca-BDE) on sample #21706; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339	IEC62321-6 - GC/MS	7580	R(0.01)	5.65	
523	IEC62321-6 - GC/MS	2782.29		-0.49	
551	IEC62321-6 - GC/MS	2808.137		-0.46	
623	IEC62321-6 - GC/MS	1917.94		-1.60	
826	IEC62321-6 - GC/MS	3735		0.73	
841		-----		-----	
1213	IEC62321-6 - GC/MS	4253.43		1.39	
2115	In house	3980	C	1.04	first reported 6649.6
2139	IEC62321-6 - GC/MS	3680.51		0.66	
2156	IEC62321-6 - GC/MS	2483.92		-0.87	
2159	In house	4075.69		1.17	
2175	In house	44637.6	R(0.01)	53.09	
2176	IEC62321-6 - GC/MS	4089.6		1.18	
2184	IEC62321-6 - GC/MS	3637		0.61	
2199	IEC62321-6 - GC/MS	2189.21		-1.25	
2201	IEC62321-6 - GC/MS	2892.8		-0.35	
2202	IEC62321-6 - GC/MS	3703		0.69	
2230	IEC62321-6 - GC/MS	2908		-0.33	
2232	IEC62321-6 - GC/MS	2867		-0.38	
2236		-----		-----	
2250	IEC62321-6 - GC/MS	3174		0.01	
2256	IEC62321-6 - GC/MS	2639		-0.67	
2265		-----		-----	
2271	IEC62321-6 - GC/MS	2863.5		-0.38	
2284	IEC62321-6 - GC/MS	2825.1		-0.43	
2293	IEC62321-6 - GC/MS	2949.0	C	-0.28	first reported 44079.37
2310		3315		0.19	
2311	IEC62321-6 - GC/MS	2841.476		-0.41	
2316	IEC62321-6 - GC/MS	3163.9		0.00	
2320		-----		-----	
2347	IEC62321-6 - GC/MS	2958		-0.26	
2350	IEC62321-6 - GC/MS	3681.9		0.66	
2352	IEC62321-6 - GC/MS	3144.2		-0.03	
2353	IEC62321-6 - GC/MS	3057.3		-0.14	
2355	IEC62321-6 - GC/MS	2903.0		-0.33	
2358	IEC62321-6 - GC/MS	3057.3		-0.14	
2363	IEC62321-6 - GC/MS	2918.1		-0.32	
2365	IEC62321-6 - GC/MS	2971.5		-0.25	
2366	IEC62321-6 - GC/MS	2990		-0.22	
2370	IEC62321-6 - GC/MS	2930		-0.30	
2375	IEC62321-6 - GC/MS	3330		0.21	
2379	IEC62321-6 - GC/MS	3277.388		0.14	
2382	IEC62321-6 - GC/MS	2917.9		-0.32	
2384	IEC62321-6 - GC/MS	3318.06		0.20	
2386	IEC62321-6 - GC/MS	3430		0.34	
2387	IEC62321-6 - GC/MS	3325.53		0.21	
2390		-----		-----	
2392	IEC62321-6 - GC/MS	3315.4189		0.19	
2415		-----		-----	
2481	In house	4020		1.10	
2482	IEC62321-6 - GC/MS	3092		-0.09	
2488	IEC62321-6 - GC/MS	2934.5		-0.29	
2532	IEC62321-6 - GC/MS	3383		0.28	
2568	IEC62321-6 - GC/MS	3423.72		0.33	
2590	IEC62321-6 - GC/MS	3858.5	C	0.89	first reported 9694.53
2620	IEC62321-6 - GC/MS	3803		0.82	
2621	IEC62321-6 - GC/MS	2525.637		-0.82	
2665	In house	4407.5		1.59	
2674	IEC62321-6 - GC/MS	3587		0.54	
2737	ISO17881-1	2750.9		-0.53	
2743	ISO17881-1	3964.088076		1.02	
2826	IEC62321-6 - GC/MS	3410		0.31	
2835	IEC62321-6 - GC/MS	1370.49	C	-2.30	first reported 14091.179
2864		1803.65		-1.74	
2910	IEC62321-6 - GC/MS	3620		0.58	
2916	IEC62321-6 - GC/MS	3250		0.11	
2930	IEC62321-6 - HPLC-PDA/UV	3450		0.37	
2970	IEC62321-6 - GC/MS	5490.66	R(0.05)	2.98	
2972	IEC62321-6 - GC/MS	2378.20		-1.01	
2981	IEC62321-6 - GC/MS	1034	R(0.05)	-2.73	
3100	GB/T26125	2656		-0.65	
3146	In house	4071.72		1.16	
3163		-----		-----	

lab	method	value	mark	z(targ)	remarks
3172	IEC62321-6 - GC/MS	3531.964		0.47	
3176	ISO17881-1	3475.5		0.40	
3182	IEC62321-6 - GC/MS	2650.90		-0.66	
3185	IEC62321-6 - GC/MS	2660.56		-0.64	
3210		-----		-----	
3214	IEC62321-6 - GC/MS	2763		-0.51	
3225	IEC62321-6 - GC/MS	3521		0.46	
3237	IEC62321-6 - GC/MS	2664.3		-0.64	
	normality	OK			
	n	69			
	outliers	4			
	mean (n)	3164.163			
	st.dev. (n)	578.3762	RSD = 18%		
	R(calc.)	1619.453			
	st.dev.(IEC62321-6:15)	781.1242			
	R(IEC62321-6:15)	2187.148			





APPENDIX 2 Other reported Brominated Flame retardants

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)

Sample #21705; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
110	-----	-----	-----	-----	-----	-----	-----
339	inférieur à 1	inférieur à 2	inférieur à 10	inférieur à 1	inférieur à 2	inférieur à 10	non analysé
523	not detected	not detected	not detected	not detected	not detected	9.85	not detected
551	-----	-----	-----	-----	-----	601.689	-----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected
826	-----	-----	-----	-----	-----	-----	-----
841	-----	-----	-----	-----	-----	-----	-----
1213	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2115	-----	-----	-----	-----	-----	-----	-----
2139	-----	-----	-----	-----	-----	-----	-----
2156	< 20	< 20	< 20	< 20	< 20	< 20	< 20
2159	N.D	N.D	N.D	N.D	9.91	27.31	N.D
2175	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2176	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not determined *)
2184	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2199	<50	<50	<50	<50	<50	<50	not applicable
2201	ND[<100]	ND[<100]	ND[<100]	ND[<100]	ND[<100]	ND[<100]	not applicable
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2230	<5	<5	<5	<5	<5	<5	<5
2232	-----	-----	-----	-----	-----	-----	-----
2236	-----	-----	-----	-----	-----	-----	-----
2250	-----	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----	-----
2271	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2284	<10	<10	<10	<10	<10	<10	NA
2293	not detected	not detected	not detected	-----	not detected	not detected	-----
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	<50	Not Detected
2316	not det. [<50]	not det. [<50]	not det. [<50]	not det.[<50]	not det. [<50]	not det. [<50]	not determined
2320	-----	-----	-----	-----	-----	-----	-----
2347	<5	<5	<5	<5	<5	<5	-----
2350	<5	<5	<5	<5	<5	<5	not analyzed
2352	-----	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	ND	ND	ND
2355	<5mg/kg	<5mg/kg	<5mg/kg	<5mg/kg	<5mg/kg	<5mg/kg	-----
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	<5
2366	<5	<5	<5	<5	<5	<5	-----
2370	<5	<5	<5	<5	<5	<5	<5
2375	-----	-----	-----	-----	-----	25	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2382	＜5	＜5	＜5	＜5	＜5	＜5	＜5
2384	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	-----
2386	<25	<25	<50	<25	<25	<50	-----
2387	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	-----
2390	-----	-----	-----	-----	-----	-----	-----
2392	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2415	-----	-----	-----	-----	-----	-----	-----
2481	not analysed	not analysed	not analysed	below DL	not analysed	below DL	below DL *)
2482	-----	-----	-----	-----	-----	-----	-----
2488	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2568	<10	<10	<10	<10	<10	<10	-----
2590	-----	-----	-----	-----	-----	11.17	-----
2620	-----	-----	-----	-----	-----	-----	-----

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
2621	-----	-----	-----	-----	-----	-----	-----
2665	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not detected	not analyzed
2674	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2737	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	0.987744121	-----
2826	<50	<50	<50	<50	<50	<50	-----
2835	Not Detected	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
2910	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2916	not detected	not detected	not detected	not detected	not detected	not detected	-----
2930	not det. <206	-----	not det. <178	not det. <239	-----	not det. <244	-----
2970	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2972	not analyzed	not analyzed	not detected	not detected	not detected	not detected	not analyzed
2981	not detected	not detected	not detected	not detected	not detected	not detected	-----
3100	not detected	not detected	not detected	not detected	not detected	not detected	not determined
3146	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3163	-----	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	-----	< 5	-----
3176	-----	-----	-----	-----	-----	-----	-----
3182	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3185	not detected	not detected	not detected	not detected	not detected	not detected	-----
3210	-----	-----	-----	-----	-----	-----	-----
3214	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3225	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]
3237	-----	-----	-----	-----	-----	-----	-----

*)
Lab 2176 reported Hepta-BDE: Less than LOQ
Lab 2481 reported Other Brominated Flame Retardant(s) analysed: tetrabromodiphenylether, pentabromodiphenylether, hexabromodiphenylether, heptabromodiphenylether

Sample #21706; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
110	-----	-----	-----	-----	-----	-----
339	inférieur à 1	inférieur à 2	inférieur à 10	4.7	80.1	Non analysé
523	not detected	not detected	not detected	10.618	Not analyzed	not detected
551	-----	-----	-----	42.242	-----	-----
623	not detected	not detected	not detected	not detected	not detected	not detected
826	-----	-----	-----	-----	-----	-----
841	-----	-----	-----	-----	-----	-----
1213	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2115	-----	-----	-----	2.36	-----	-----
2139	-----	-----	-----	-----	-----	-----
2156	< 20	< 20	< 20	< 20	< 20	< 20
2159	N.D	N.D	N.D	N.D	N.D	N.D
2175	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2176	Not detected	Not detected	Not detected	Not detected	-----	Not determined *)
2184	not detected	not detected	not detected	not detected	-----	not detected
2199	<50	<50	<50	<50	<20	not applicable
2201	ND[<100]	ND[<100]	ND[<100]	ND[<100]	not applicable	not applicable
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2230	<5	<5	<5	<5	<5	<5
2232	-----	-----	-----	-----	-----	-----
2236	-----	-----	-----	-----	-----	-----
2250	-----	-----	-----	-----	-----	-----
2256	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----
2271	<50	<50	<50	<50	not detected	not detected
2284	<10	<10	<10	<10	<10	NA
2293	not detected	not detected	not detected	-----	not detected	-----
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 det. [<50]	not det. [<50]	not det. [<50]	not det. [<50]	not det. [<50]	not determined
2320	-----	-----	-----	-----	-----	-----
2347	<5	<5	<5	<5	<10	-----
2350	<5	<5	<5	<5	not analyzed	not analyzed
2352	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	NA	ND
2355	<5mg/kg	<5mg/kg	<5mg/kg	<5mg/kg	<10mg/kg	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	＜5	＜5	＜5	＜5	＜5	＜5
2365	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<10	-----
2370	<5	<5	<5	<5	<5	<5
2375	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2382	＜5	＜5	＜5	＜5	＜5	＜5

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
2384	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	-----
2386	<25	<25	<50	<25	<50	-----
2387	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	Not Det. [<5]	-----	-----
2390	-----	-----	-----	-----	-----	-----
2392	Not detected	Not detected	Not detected	Not detected	Not analyzed	Not detected
2415	-----	-----	-----	-----	-----	-----
2481	not analysed	not analysed	not analysed	below DL	below DL	below DL
2482	-----	-----	-----	-----	-----	-----
2488	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2568	<10	<10	<10	67.89	-----	-----
2590	-----	-----	-----	-----	-----	-----
2620	-----	-----	-----	-----	-----	-----
2621	-----	-----	-----	-----	-----	-----
2665	not analyzed	not analyzed	not analyzed	not analyzed	not detected	not analyzed
2674	not detected	not detected	not detected	not detected	not applicable	not detected
2737	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----
2826	<50	<50	<50	<50	-----	-----
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
2910	not detected	not detected	not detected	not detected	not detected	not detected
2916	not detected	not detected	not detected	not detected	not detected	-----
2930	not det. <=206	-----	not det. <178	not det. <239	-----	-----
2970	not detected	not detected	not detected	1.53	-----	not detected
2972	not analyzed	not analyzed	not detected	not detected	2020.53	not analyzed
2981	not detected	not detected	not detected	not detected	-----	-----
3100	not detected	not detected	not detected	not detected	not determined	not determined
3146	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3163	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5	-----
3176	-----	-----	-----	-----	-----	-----
3182	<5.00	<5.00	<5.00	<5.00	<5.00	<5.00
3185	not detected	not detected	not detected	not detected	-----	-----
3210	-----	-----	-----	-----	-----	-----
3214	< 5	< 5	< 5	< 5	< 5	< 5
3225	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]	<5 [not det.]
3237	-----	-----	-----	-----	-----	-----

*)
Lab 2176 reported Tetra-BB: Less than LOQ
Lab 2481 reported Other Brominated Flame Retardant(s) analysed: tetrabromodiphenylether, pentabromodiphenylether, hexabromodiphenylether, heptabromodiphenylether

APPENDIX 3 Analytical details

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (min)	extraction temp (°C)
110	---	---		---			
339	---	---		---			
523	Yes	Further cut	2.0	Ultrasonic	Toluene	60	60
551	Yes	Further cut	0.1	Soxhlet	Toluene	120	
623	Yes	Further cut	2.2	Ultrasonic	Toluene	60	50, #21706: 60
826	Yes	Further grinded	#21705 1.0g/10 mL #21706 0.1g/10 mL	Ultrasonic	Toluene	90	60
841	---	---		---			
1213	Yes	Further cut	0.1	Ultrasonic	THF	30	40
2115	No	Used as received	0.5	Ultrasonic	Toluene	45 m	25
2139	Yes	Further cut	0.1g for 21705, 0.05g for 21706	Ultrasonic	Toluene	60	60
2156	Yes	Further cut	0.5	Ultrasonic	Toluene	120	60
2159	Yes	Further cut	0.3	Ultrasonic	toluene	120	70
2175	Yes	Further cut	Less than 0.5	Ultrasonic		*)	*)
2176	Yes	Further grinded	0.1 g/data	Soxhlet	Toluene	180 m	-
2184	Yes	Used as received	2	Ultrasonic	Toluene	180	60oC
2199	Yes	Further grinded	0.1	Soxhlet	Toluene	120m	not applicable
2201	Yes	Further grinded	0.1	Ultrasonic	Toluene	120	70℃
2202	Yes	Used as received	0.5	Stirrer	THF, toluene	over 12hrs	room temperature
2230	Yes	Used as received	0.5	Soxhlet *)	Toluene	120	NA
2232	Yes	Further cut	1	Soxhlet	toluene	120	
2236	---	---		---			
2250	Yes	Used as received	0,5	Soxhlet	Toluene	120	110
2256	Yes	Further cut	1.0	Soxhlet	Toluene	120	
2265	---	---		---			
2271	Yes	Further grinded	0.1	Soxhlet	Toluene	120	boiling Toluene
2284	Yes	Further cut	0.2	Ultrasonic	Toluene	60	55
2293	Yes	Further cut	0.1	Ultrasonic	Toluene	60	80
2310	Yes	Further cut	0.1	Ultrasonic	TOLUENE	60	60
2311	Yes	Further cut	0.1	Soxhlet	Toluene	60	60
2316	Yes	Further grinded	0.1 g flame retard., 0.3 g for HBCDD	Soxhlet	Toluene	2 Hours	70
2320	---	---		---			
2347	Yes	Further cut	0.1	Soxhlet	tolune	PBB:6h, HBCDD:60min	60
2350	Yes	Further cut	0.1	Ultrasonic	Toluene	120	50
2352	Yes	Further cut	0.1	Soxhlet	Toluene	4h	
2353	Yes	Further grinded	0.1	Soxhlet	toluene	120	soxhlet
2355	Yes	Further cut	0.1	Soxhlet	100ml	4h	280
2358	Yes	Further grinded	0.1	Soxhlet	Toluene	120	
2363	Yes	Further grinded	2.5	Soxhlet	toluene	300	
2365	Yes	Further cut	0.1	Soxhlet	toluene	4hours	270
2366	Yes	Further cut	0.1	Soxhlet	toluene		
2370	Yes	Further grinded	0.1	Soxhlet	toluene	1hr47mins	250
2375	Yes	Further cut	0,3	Ultrasonic	Toluene	60	60
2379	Yes	Further grinded	0.1	Soxhlet	Toluene	4 hr	-
2382	Yes	Further cut	#21705:0.5g, #21706:0.1g	#21705:Ultrasonic, #21706:Soxhlet	Toluene	#21705: 1h, #21706: 4h	#21705:60°C, #21706:110°C
2384	Yes	Further grinded	0.5	Soxhlet	toluene	960	reflux temperature
2386	Yes	Further grinded	1	Soxhlet	Toluene	240	Soxhlet conditions
2387	Yes	Further cut	1	Soxhlet	Toluene	960-1260	Reflux Temperature
2390	---	---		---			
2392	Yes	Further grinded	0.1	Soxhlet	Toluene	120	-
2415	---	---		---			
2481	Yes	Used as received	0.25	Ultrasonic	toluene	60	60
2482	Yes, HBCDD: No	Further cut	0,1	Ultrasonic	Toluene	60	60
2488	---	---		---			
2532	Yes	Further cut	0.5 gram, For HBCDD- 0.2 g	Ultrasonic	Toluene, For HBCDD - THF ,ACN, Water	45 min, HBCDD:1 hour	Room Temp., HBCDD-70 °C

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (min)	extraction temp (°C)
2568	Yes	Further grinded	0.1	Soxhlet	toluene	120	
2590	No	Further cut	0.5	Soxhlet	Tolune:DCM	360	N/A
2620	Yes, HBCDD: No	Further cut	4x 30mg each plus 3ml solvent	Ultrasonic	Toluene *)	60	60
2621	Yes	Further grinded	about 0.1	Soxhlet	Toluene	360	110
2665	Yes	sample was completely dissolved in THF	0,5	Mechanical Shaking	Tetrahydrofurane and Hexane	180	40
2674	Yes	Used as received	2.0	Ultrasonic	Toluene	3 hours	60
2737	Yes	#21705: Further Cut. #21706: Used as received	0.5	Ultrasonic	Toluene	60min	50°C
2743	Yes	Used as received	1	Ultrasonic	Toluene	60	Room temperature
2826	Yes	Further cut	0.1	Soxhlet	Toluene	2.5 hrs	
2835	Yes	Further cut	HBCDD - 0.2g PBB, PBDE - 0.5g	ASE	HBCDD - Methylene chloride, PBB/PBDE - Toulene	15	150
2864	Yes	Further cut	0.2	Ultrasonic	Toluene	180	60
2910	Yes	Further cut	2	Ultrasonic	Toluene	3h	60
2916	No	Further grinded	0,2	Ultrasonic	Toluene	30	60
2930	Yes	Further cut	0,1	Soxhlet	n-propanol	180	190
2970	Yes	Further grinded	0.6	Soxhlet	toluene	2 hours	-
2972	No	Further grinded	0.100 g aprox.	Soxhlet	Toluene	240	130 approx.
2981	Yes	Further grinded	0.5	Soxhlet	toluene	120	25
3100	Yes	Further cut	0.1	Soxhlet	Toluene	£¾120	
3146	Yes	Used as received	0,5	Ultrasonic	THF/ACN	30	70
3163	---	---		---			
3172	Yes	---		---			
3176	No	Used as received	1	Ultrasonic	Toluene	45	room temperature
3182	Yes	Further grinded	0.3	Soxhlet	Toluene	360	use soxhlet to extract at 2.5 minutes/cycle
3185	Yes	Further cut	0.2	Soxhlet	Toluene	240	not applicable
3210	---	---		---			
3214	Yes	Further grinded	1	Soxhlet	Toluene	480	--
3225	Yes	Further cut	0.5	Soxhlet	Toluene	960	over 120
3237	Yes	Further cut	0,25	Soxhlet	Toluen	120	-

*)
Lab 2175: Baked sample at 50C for 30mins before analyzing
Lab 2230: Soxhlet and each cycle being approximately 2 to 3 minutes
Lab 2620: measure of 1:10 diluted extracts

APPENDIX 4

Number of participants per country

1 lab in BRAZIL
1 lab in COLOMBIA
3 labs in FRANCE
9 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
4 labs in INDIA
1 lab in INDONESIA
4 labs in ITALY
3 labs in JAPAN
3 labs in MALAYSIA
1 lab in MEXICO
16 labs in P.R. of CHINA
1 lab in PAKISTAN
4 labs in SINGAPORE
4 labs in SOUTH KOREA
1 lab in SRI LANKA
5 labs in TAIWAN
4 labs in THAILAND
1 lab in THE NETHERLANDS
5 labs in TURKEY
2 labs in U.S.A.
3 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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
13 F. Cordeiro, I. Verbist, P. Robouch, T. Linsinger, M.B. de la Calle, IMEP-26: Determination of brominated flame retardants in plastic, EUR 24874 EN2011