Results of Proficiency Test
Total Brominated Flame retardants
August 2019

Organised 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 Pentabromodiphenyl ether (Penta-BDE) and Octabromodiphenyl ether (Octa-BDE). The European Union decided to ban the use of both PBB and PBDE in electrical and electronic devices. This ban was formalised 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 Authorisation. HBCDD is slowly banned worldwide.

Since 2009, the Institute for Interlaboratory Studies organizes a proficiency scheme for the determination of Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyls Ethers (PBDE) and the PT was extended with Hexabromocyclododecane (HBCDD) in 2015. During the annual proficiency testing program 2019/2020, it was decided to continue with the proficiency test for the analysis of total Brominated Flame Retardants in polymers. In this interlaboratory study 75 laboratories in 20 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2019 Brominated Flame Retardants 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 organiser of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different polymer samples. One sample contained approximately 3 grams of small blue squares, labelled #19605 and the other sample contained approximately 3 grams of beige granulate, labelled #19606. 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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

Sample #19605 was a blue colored PVC, artificially fortified with Decabromodiphenyl ether (Deca-BDE). After homogenisation, plastic bags were filled with 3 grams and labelled #19605. The homogeneity of subsamples #19605 was checked by the determination of HBCDD content on 8 stratified randomly selected subsamples.

	Deca-BDE in mg/kg
sample #19605-1	1967
sample #19605-2	1960
sample #19605-3	1929
sample #19605-4	1907
sample #19605-5	2041
sample #19605-6	1982
sample #19605-7	1988
sample #19605-8	1907

Table 1: homogeneity test results of subsamples #19605

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding target 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)	127
reference test method	IEC62321-6:15
0.3 x R (ref. test method)	408

Table 2: evaluation of the repeatability of subsamples #19605

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

Sample #19606 was a beige colored Acrylonitrile Butadiene Styrene (ABS), artificially fortified with Hexabromocyclododecane (HBCDD). After homogenisation, subsamples were filled with 3 grams and labelled #19606. The homogeneity of subsamples #19606 was checked by the determination of HBCDD content on 8 stratified randomly selected subsamples.

	HBCDD in mg/kg
sample #19606-1	912
sample #19606-2	981
sample #19606-3	973
sample #19606-4	998
sample #19606-5	931
sample #19606-6	1027
sample #19606-7	941
sample #19606-8	1002

Table 3: homogeneity test results of subsamples #19606

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

	HBCDD in mg/kg
r (observed)	111
reference test method	IMEP-26:11
0.3 x R (ref. test method)	204

Table 4: evaluation of the repeatabilities of subsamples #19606

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

To each of the participating laboratories one sample labelled #19605 and one sample labelled #19606 were sent on August 7, 2019.

2.5 ANALYSES

The participants were requested to determine on both samples: Octabromobiphenyl (Octa-BB), Nonabromobiphenyl (Nona-BB), Decabromobiphenyl (Deca-BB), Octabromodiphenyl ether (Octa-BDE), Nonabromodiphenyl ether (Nona-BDE), Decabromodiphenyl ether (Deca-BDE) and Hexabromocyclododecane (HBCDD). It was also requested to report if the laboratory was accredited for the requested components that were determined. Also, some method details were requested to report.

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 results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such test results can't 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 appropriate reference test methods 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 were 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 original test results are placed under 'Remarks' in the test 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 visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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. The Kernel Density Graph 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 in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.

The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Five participants reported test results after the final reporting date and eight participants did not report any test result at all. Not all laboratories were able to report all components requested.
Finally, the 67 reporting laboratories reported 168 numerical test results. Observed were 8 outlying test results, which is 4.8% 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. The abbreviations, used in these tables, are listed 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. From the IMEP-26 results (ref. 17) it was clear that target standard deviations of 3 – 12% earlier used in iis PTs were not realistic for non-expert laboratories and a more realistic PT target standard deviation is 25% of the assigned value. The reproducibility is calculated to be 2.8 times the standard deviation of 25% of the assigned value.

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 have been used for the evaluation of Nona-BDE and Deca-BDE.

Sample #19605

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

Deca-BDE: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of the target reproducibility from IEC62321-6:2015.

Sample #19606

HBCDD: Participants reported test results as low as 6 mg/kg and as high as 1020 mg/kg. Because of this large variability, it was decided not to calculate z-scores for this determination. Difficulties in releasing the analyte from the matrix of Acrylonitrile Butadiene Styrene may be a factor in this (see also the discussion in paragraph 5).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average results, 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	55	90.0	108.0	75.6
Deca-BDE	mg/kg	61	1429	968	995

Table 5: reproducibilities of components on sample #19605

Component	unit	n	average	2.8 * sd	R (lit)
HBCDD	mg/kg	44	(176)	(745)	(123)*

Table 6: reproducibility of conponents on sample #19606

*) when R(lit) in brackets, no z-scores were calculated due to the large variability of the test results.

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF AUGUST 2019 WITH PREVIOUS PTS

	August 2019	September 2018	September 2017	September 2016	August 2015
Number of reporting labs	67	77	66	60	58
Number of results reported	168	256	195	160	165
Number of statistical outliers	8	9	14	11	10
Percentage outliers	4.8%	3.5%	7.2%	6.9%	5.7%

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 below table.

Component	August 2019	Sept. 2018	Sept. 2017	Sept. 2016	August 2015	2014/ 2009	target **)
Nona-BDE	43%	n.e.	38%	36%	46%	15-51%	26-44% (<500mg/kg)
Deca-BDE	24%	28%	16%	18%	17%	10-37%	26-29% (<5000mg/kg)
Deca-BB	n.e.	22%	n.e.	n.e.	n.e.	n.e.	25%
HBCDD	(151%)*	17%	39%	24%	49%	n.e.	25%

Table 8: comparison with previous proficiency tests

*) no z-scores were calculated

**) For PTs of 2015 and earlier the value of 25% from IMEP-26 has been taken for the target uncertainty of the BDEs as well as for HBCDD. From 2016 onwards, a target uncertainty range from IEC62321-1 was used for the BDEs as the uncertainty is concentration dependent.

The uncertainty observed for Deca-BDE in this PT is in line with the uncertainties observed in previous PTs. For Nona-BDE it is slightly higher with the uncertainties observed in previous PTs. Both uncertainties are in line with the uncertainty requirements of the target method.

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 (see appendix 3). Based on the answers given by the participants the following can be summarized:
Sixty-two participants answered to be ISO/IEC17025 accredited for the determination of Brominated Flame Retardants in polymers (= 93% of the reported participants).
About 35% of the participants used the samples as received, about 30% of the participants grinded the samples further prior to analyses and about 35% cut the samples further prior to analyses.
The technique used to release the PBDE and HBCDD from the samples differs. About 45% of the participants used ultrasonic as technique and about 55% of the participants 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 24 min. to 16 hours. About 30% of the participants used an extraction time of 120 min.
The extraction temperature used differs from room temperature to 500°C. About 30% of the participants used an extraction temperature in the range of 40°C up to and including 60°C.

When the analytical details where investigated separately, it appeared that the effect on the determination of the analytes is negligible.

5 DISCUSSION

The material of the samples in this PT was PVC for sample #19605 and ABS for sample #19606. To extract the requested components (see chapter 2.5) from a polymer, the extraction solvent, the extraction conditions and the contact surface area are important. Sample #19605 of PVC was made positive on Deca-BDE. It appeared that none of the requested analytical details were dominant as the calculated reproducibility for the Deca-BDE determination, using all reported test results and thus including all different test details, were in agreement with the requirements of the target reproducibility limits of IEC62321-1.

The majority of the group identified all added PBDE correctly: sample #19605 contained Nona-BDE and Deca-DBE. HBCDD was detected in sample #19606 by most participants, although there were problems with the extraction of HBCDD in the ABS matrix is more difficult for some laboratories.
It was found by our expert that when a high volume of ABS will be dissolved in Toluene it could result in a sticky solution. Because the ABS sample was made positive with a high amount of HBCDD, a small volume of sample should have been enough to dissolve the sample in Toluene(mixture) and therefore it is expected that the effect of the matrix will be less profound as it is observed in this PT. For this sample only a few of the laboratories reported a high amount of HBCDD. Most laboratories did not manage to extract the HBCDD completely from the matrix. Therefore, it was decided to calculate no z-scores. One should be aware that ABS is a widely available polymer and when dealing with ABS the extraction should be done with care.

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 90% of the reporting laboratories would reject sample #19605 based on the test results of Deca-BDE. Only one laboratory would reject the sample based on the test results of Nona-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 #19606 for containing to much HBCDD.

6 CONCLUSION

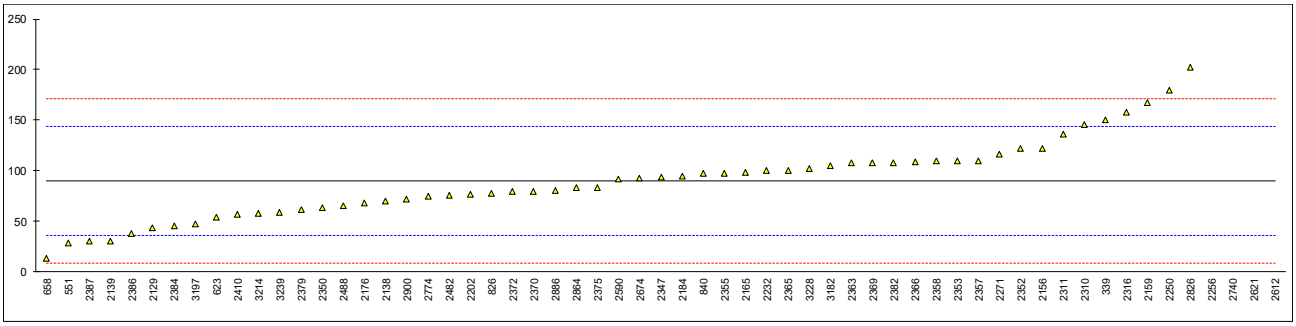
Although it can be concluded that most of the participants have no problem with the determination on PBDE in PVC, but most likely have a problem with Brominated Flame Retardants in ABS. Each participating laboratory has to evaluate its performance in this study and decide about any corrective actions 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.

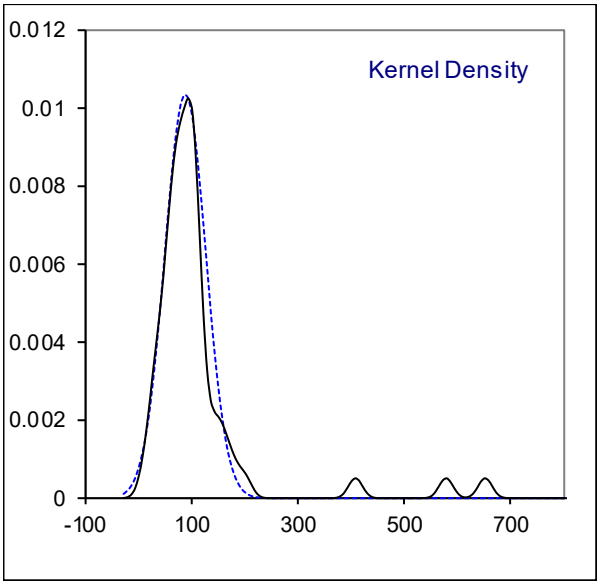
APPENDIX 1

Determination of Nonabromodiphenylether (Nona-BDE) on sample #19605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	150.35		2.23	
551	IEC62321-6 - GC/MS	28.93		-2.27	
623	IEC62321-6	54.46		-1.32	
658	IEC62321-6 - GC/MS	13.769		-2.83	
826	IEC62321-6 - GC/MS	78		-0.45	
840		97		0.26	
2129	IEC62321-6 - GC/MS	43.17		-1.74	
2138	IEC62321-6 - GC/MS	70.3		-0.73	
2139	IEC62321-6	30.81		-2.20	
2156	IEX62321-6- GC/ECD	122.08		1.19	
2159	IEC62321-6 - GC/MS	167.44		2.87	
2165	IEC62321-6 - GC/MS	98		0.29	
2176	In house	68.2	C	-0.81	first reported: 250.1
2184	IEC62321-6 - GC/MS	95		0.18	
2202	IEC62321-6 - GC/MS	77	C	-0.48	first reported: 377
2232	IEC62321-6 - GC/MS	100		0.37	
2250	ISO17881-1Mod.	180	C	3.33	first reported: 760
2256	IEC62321-6 - GC/MS	409.8	C,R(0.01)	11.85	first reported: 819.6
2271	IEC62321-6 - GC/MS	116.2		0.97	
2310	IEC62321-6 - GC/MS	145.17		2.04	
2311	IEC62321-6 - GC/MS	136.5		1.72	
2316	IEC62321-6 - GC/MS	158.3		2.53	
2347	IEC62321-6 - GC/MS	94		0.15	
2350	IEC62321-6 - GC/MS	63.0		-1.00	
2352	IEC62321-6 - GC/MS	122.04		1.18	
2353	IEC62321-6 - GC/MS	109.51		0.72	
2355	IEC62321-6 - GC/MS	97.6		0.28	
2357	IEC62321-6 - GC/MS	110		0.74	
2358	IEC62321-6 - GC/MS	109.51		0.72	
2363	IEC62321-6 - GC/MS	108		0.66	
2365	IEC62321-6 - GC/MS	100.1		0.37	
2366	IEC62321-6 - GC/MS	109		0.70	
2369	IEC62321-6 - GC/MS	108		0.66	
2370	IEC62321-6 - GC/MS	79.4		-0.40	
2372	IEC62321-6 - GC/MS	79.20		-0.40	
2375	IEC62321-6 - GC/MS	83.0		-0.26	
2379	IEC62321-6 - GC/MS	62		-1.04	
2382	IEC62321-6 - GC/MS	108.1		0.67	
2384	IEC62321-6 - GC/MS	45.83		-1.64	
2386	IEC62321-6 - GC/MS	38		-1.93	
2387	IEC62321-6 - GC/MS	30.033		-2.23	
2390		-----		-----	
2410	IEC62321-6 - GC/MS	57.2		-1.22	
2415		-----		-----	
2481		-----		-----	
2482	EN62321-6	76.07		-0.52	
2488	EN62321-6	65.4		-0.91	
2590	IEC62321-6 - GC/MS	92.13		0.08	
2612	IEC62321-6 - GC/MS	1755.72	C,R(0.01)	61.73	first reported: 2413.08
2621	IEC62321-6 - GC/MS	653.5	C,R(0.01)	20.88	first reported: 349
2672	IEC62321-6 - GC/MS	< 100		-----	
2674	IEC62321-6 - GC/MS	93		0.11	
2705		-----		-----	
2740	In house	580	R(0.01)	18.16	
2743		-----		-----	
2774	In house	75		-0.56	
2816		-----		-----	
2826	IEC62321-6 - GC/MS	202	C	4.15	first reported: 426
2857		-----	W	-----	first reported: ND
2864	IEC62321-6 - GC/MS	82.90		-0.27	
2886	In house	80		-0.37	
2890		-----		-----	
2900	IEC62321-6 - GC/MS	72.1		-0.67	
3118		-----		-----	
3146		-----		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	< 5		<-3.15	possible false negative test result?
3182	IEC62321-6 - GC/MS	105.38		0.57	
3197	IEC62321-6 - GC/MS	47.1		-1.59	
3210		-----		-----	
3213		-----		-----	

lab	method	value	mark	z(targ)	remarks
3214	IEC62321-6 - GC/MS	57.95		-1.19	
3228	IEC62321-6 - GC/MS	102		0.44	
3239	IEC62321-6 - GC/MS	58.87		-1.16	
3246		n.a.		-----	
	normality	OK			
	n	55			
	outliers	4			
	mean (n)	90.075			
	st.dev. (n)	38.5688	RSD = 43%		
	R(calc.)	107.993			
	st.dev.(IEC62321-6:15)	26.9844			
	R(IEC62321-6:15)	75.556			

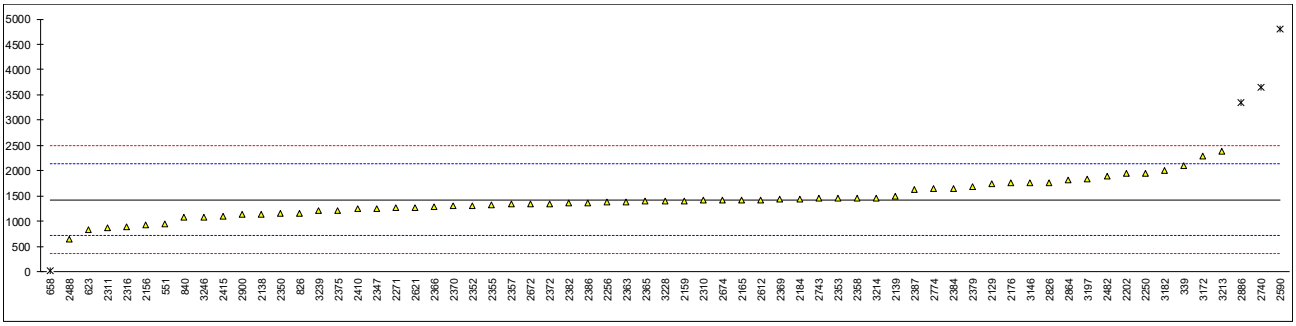


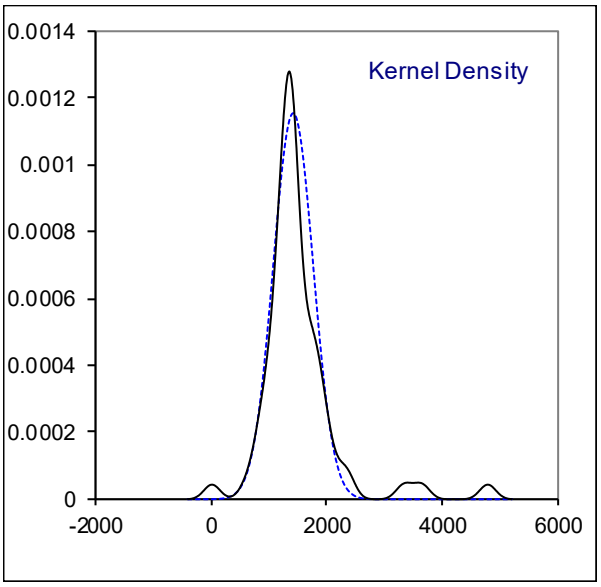


Determination of Decabromobiphenylether (Deca-BDE) on sample #19605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	2095.46		1.88	
551	IEC62321-6 - GC/MS	947.05		-1.36	
623	IEC62321-6	829.08		-1.69	
658	IEC62321-6 - GC/MS	23.279	R(0.01)	-3.95	
826	IEC62321-6 - GC/MS	1163		-0.75	
840		1075.6		-0.99	
2129	IEC62321-6 - GC/MS	1747		0.90	
2138	IEC62321-6 - GC/MS	1137.4		-0.82	
2139	IEC62321-6	1502.59		0.21	
2156	IEX62321-6- GC/ECD	935.93		-1.39	
2159	IEC62321-6 - GC/MS	1404.65		-0.07	
2165	IEC62321-6 - GC/MS	1418		-0.03	
2176	In house	1759.4		0.93	
2184	IEC62321-6 - GC/MS	1445		0.05	
2202	IEC62321-6 - GC/MS	1949		1.46	
2232		-----		-----	
2250	ISO17881-1Mod.	1950		1.47	
2256	IEC62321-6 - GC/MS	1376.1		-0.15	
2271	IEC62321-6 - GC/MS	1266.3		-0.46	
2310	IEC62321-6 - GC/MS	1412		-0.05	
2311	IEC62321-6 - GC/MS	872		-1.57	
2316	IEC62321-6 - GC/MS	890.2		-1.52	
2347	IEC62321-6 - GC/MS	1258		-0.48	
2350	IEC62321-6 - GC/MS	1149.6		-0.79	
2352	IEC62321-6 - GC/MS	1312.95		-0.33	
2353	IEC62321-6 - GC/MS	1460.13		0.09	
2355	IEC62321-6 - GC/MS	1333.4		-0.27	
2357	IEC62321-6 - GC/MS	1345		-0.24	
2358	IEC62321-6 - GC/MS	1460.13		0.09	
2363	IEC62321-6 - GC/MS	1381		-0.13	
2365	IEC62321-6 - GC/MS	1392.4		-0.10	
2366	IEC62321-6 - GC/MS	1285		-0.40	
2369	IEC62321-6 - GC/MS	1442		0.04	
2370	IEC62321-6 - GC/MS	1310		-0.33	
2372	IEC62321-6 - GC/MS	1352.4		-0.21	
2375	IEC62321-6 - GC/MS	1222.0		-0.58	
2379	IEC62321-6 - GC/MS	1688		0.73	
2382	IEC62321-6 - GC/MS	1369.2		-0.17	
2384	IEC62321-6 - GC/MS	1647.63		0.62	
2386	IEC62321-6 - GC/MS	1370		-0.16	
2387	IEC62321-6 - GC/MS	1629.502		0.57	
2390		-----		-----	
2410	IEC62321-6 - GC/MS	1250.0		-0.50	
2415	IEC62321-6 - GC/MS	1100	C	-0.92	first reported: 2800
2481		-----		-----	
2482	EN62321-6	1888		1.29	
2488	EN62321-6	637.2		-2.23	
2590	IEC62321-6 - GC/MS	4793.7	C, R(0.01)	9.47	first reported: 3732.29
2612	IEC62321-6 - GC/MS	1419.32		-0.03	
2621	IEC62321-6 - GC/MS	1276		-0.43	
2672	IEC62321-6 - GC/MS	1346		-0.23	
2674	IEC62321-6 - GC/MS	1412		-0.05	
2705		-----		-----	
2740	In house	3650	R(0.01)	6.25	
2743	ISO17881-1	1451.80		0.07	
2774	In house	1644		0.61	
2816		-----		-----	
2826	IEC62321-6 - GC/MS	1764	C	0.94	first reported: 3516
2857		-----	W	-----	first reported: ND
2864	IEC62321-6 - GC/MS	1814.40		1.09	
2886	In house	3348	R(0.01)	5.40	
2890		-----		-----	
2900	IEC62321-6 - GC/MS	1134.3		-0.83	
3118		-----		-----	
3146		1761	C	0.94	first reported as Decabromobiphenyl
3163		-----		-----	
3172	IEC62321-6 - GC/MS	2297		2.44	
3182	IEC62321-6 - GC/MS	1999.59		1.61	
3197	IEC62321-6 - GC/MS	1838.0		1.15	
3210		-----		-----	
3213	IEC62321-6 - GC/MS	2375.8		2.67	

lab	method	value	mark	z(targ)	remarks
3214	IEC62321-6 - GC/MS	1467.38		0.11	
3228	IEC62321-6 - GC/MS	1401		-0.08	
3239	IEC62321-6 - GC/MS	1209.32		-0.62	
3246		1075.91		-0.99	
	normality	OK			
	n	61			
	outliers	4			
	mean (n)	1428.625			
	st.dev. (n)	345.6467	RSD = 24%		
	R(calc.)	967.811			
	st.dev.(IEC62321-6:15)	355.3594			
	R(IEC62321-6:15)	995.006			

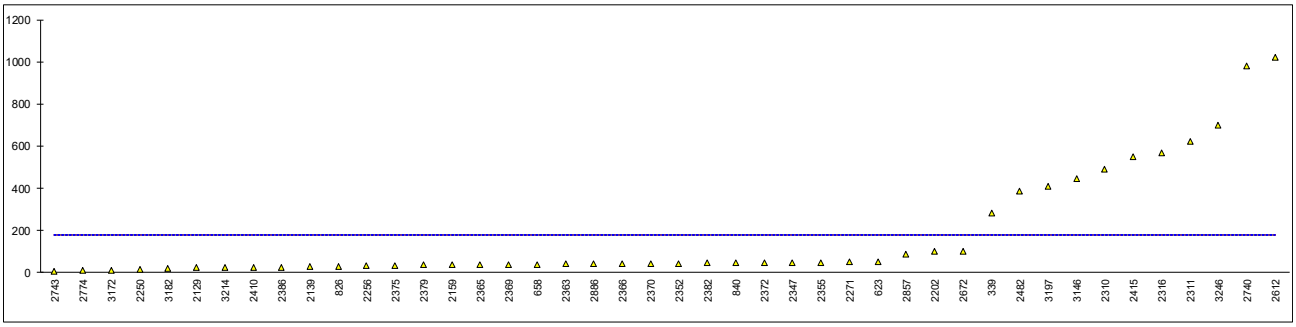


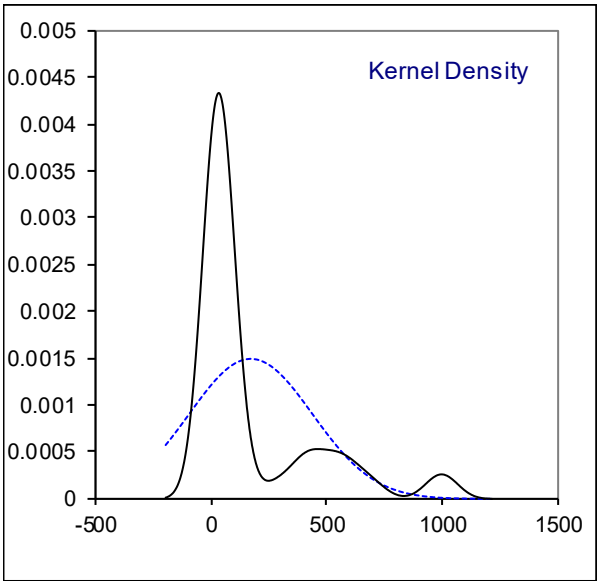


Determination of Hexabromocyclododecane (HBCDD) on sample #19606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	284		-----	
551		-----		-----	
623	INH-216	52.61		-----	
658	IEC62321-6 - GC/MS	39.422		-----	
826		28		-----	
840		44.8		-----	
2129	ISO17881-2	21.52		-----	
2138	IEC62321-6 - GC/MS	NA		-----	
2139	IEC62321-6	26.71		-----	
2156	IEC62321-6 - GC/ECD	< 20.00		-----	
2159	IEC62321-6 - GC/MS	37.66		-----	
2165	IEC62321-6 - GC/MS	n.a.		-----	
2176		-----		-----	
2184		-----		-----	
2202	IEC62321-6 - GC/MS	99		-----	
2232		-----		-----	
2250	ISO17881-2	15.86		-----	
2256	IEC62321-6 - HPLC-PDA/UV	30.5		-----	
2271	In house	49.3		-----	
2310	IEC62321-6 - GC/MS	491.15		-----	
2311	IEC62321-6 - GC/MS	623.5		-----	
2316	In house	567.7		-----	
2347	IEC62321-6 - GC/MS	46		-----	
2350	IEC62321-6 - GC/MS	NA		-----	
2352	IEC62321-6 - GC/MS	42.77		-----	
2353	IEC62321-6 - GC/MS	NA		-----	
2355	IEC62321-6 - GC/MS	46.0		-----	
2357		-----		-----	
2358	IEC62321-6 - GC/MS	N/A		-----	
2363	IEC62321-6 - GC/MS	40		-----	
2365	IEC62321-6 - GC/MS	38.1		-----	
2366	In house	42		-----	
2369	IEC62321-6 - GC/MS	39		-----	
2370	IEC62321-6 - GC/MS	42.2		-----	
2372	IEC62321-6 - GC/MS	45.75		-----	
2375	IEC62321-6 - GC/MS	34.0		-----	
2379	IEC62321-6 - GC/MS	36		-----	
2382	In house	44.3		-----	
2384		-----		-----	
2386	IEC62321-6 - GC/MS	25.6		-----	
2387		-----		-----	
2390		-----		-----	
2410	In house	24.8		-----	
2415	IEC62321-6 - GC/MS	550.0		-----	
2481		-----		-----	
2482	In house	384.2		-----	
2488	DIN EN 62321-6	Not Detected		-----	
2590		-----		-----	
2612		1019.83		-----	
2621	IEC62321-6 - GC/MS	N.D.		-----	
2672	IEC62321-6 - GC/MS	99.5		-----	
2674	IEC62321-6 - GC/MS	n.a		-----	
2705		-----		-----	
2740	In house	980		-----	
2743	ISO17881-1	6.22		-----	
2774	In house	9		-----	
2816		-----		-----	
2826	IEC62321-6 - GC/MS	NA		-----	
2857	IEC62321-6 - GC/MS	87.39		-----	
2864		-----		-----	
2886	In house	41.76		-----	
2890		-----		-----	
2900	IEC62321-6 - GC/MS	NA		-----	
3118		-----		-----	
3146		444		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	9.93		-----	
3182	IEC62321-6 - GC/MS	20.12		-----	
3197	IEC62321-6 - GC/MS	411.0		-----	
3210		-----		-----	
3213		-----		-----	

lab	method	value	mark	z(targ)	remarks
3214	IEC62321-6 - GC/MS	22.07		-----	
3228	IEC62321-6 - GC/MS	n.a.		-----	
3239		-----		-----	
3246		700.6		-----	
	normality	not OK			
	n	44			
	outliers	0			
	mean (n)	(175.997)			
	st.dev. (n)	(266.0193)	RSD = 151%		
	R(calc.)	744.854			
	st.dev.(IMEP-26:11)	(43.9993)			
	R(IMEP-26:11)	(123.198)			





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 #19605; results in mg/kg

Lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
339	<1	<2	<10	8.73	<10	<10
551	N.D.	N.D.	N.D.	N.D.	-----	-----
623	-----	-----	-----	-----	-----	-----
658	n.d.	-----	n.d.	n.d.	12.797	-----
826	-----	-----	-----	-----	-----	-----
840	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2129	-----	-----	-----	-----	-----	-----
2138	ND	ND	ND	ND	NA	NA
2139	-----	-----	-----	-----	-----	-----
2156	< 20.00	< 20.00	< 20.00	26.86	< 20.00	< 20.00
2159	<5,0	<5,0	<5,0	<5,0	<5,0	<5,0
2165	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.
2176	ND	ND	ND	ND	-----	-----
2184	ND	ND	ND	ND	-----	-----
2202	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2232	-----	-----	-----	2034.0587 C	-----	-----
2250	<5	<5	<5	<5	<5	<5
2256	-----	-----	-----	22.8	-----	-----
2271	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
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	ND	ND	ND	ND	ND	-----
2347	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	NA	NA
2352	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	ND	ND
2355	<5	<5	<5	<5	<10	<5
2357	<5	<5	<5	<5	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	NA
2365	＜5.0	＜5.0	＜5.0	＜5.0	＜10.0	-----
2366	<5	<5	<5	<5	<5	<5
2369	<5	<5	<5	<5	<5	-----
2370	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2375	-----	-----	-----	<5	-----	-----
2379	ND	ND	ND	ND	ND	Not tested
2382	<5	<5	<5	<5	<5	<5
2384	Not det. <5]	Not det. <5]	Not det. [<5]	Not det. [<5]	Not det. [<5]	-----
2386	<25	<25	<50	<25	<50	-----
2387	N.D.[<5]	N.D.[<5]	N.D.[<5]	N.D.[<5]	-----	-----
2390	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----
2415	-----	-----	-----	-----	-----	-----
2481	-----	-----	-----	-----	-----	-----
2482	-----	-----	-----	-----	-----	-----
2488	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	16.78	-----	-----
2612	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
2621	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2672	< 100	< 100	< 100	< 100	< 80	-----
2674	n.d.	n.d.	n.d.	n.d.	n.a.	-----
2705	-----	-----	-----	-----	-----	-----
2740	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----
2774	-----	-----	-----	-----	-----	-----

Lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
2816	-----	-----	-----	-----	-----	-----
2826	< 100	< 100	< 100	< 100	NA	< 100
2857	ND	ND	ND	ND	ND	-----
2864	N.D.	N.D.	N.D.	N.D.	-----	-----
2886	-----	-----	-----	4	-----	-----
2890	-----	-----	-----	-----	-----	-----
2900	ND	ND	ND	ND	NA	NA
3118	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5	< 5
3182	<5	<5	<5	10.25	<5	<5
3197	<20	<20	<20	<20	<20	-----
3210	-----	-----	-----	-----	-----	-----
3213	-----	-----	-----	-----	-----	-----
3214	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.
3239	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.a.	n.d.	n.a.

Lab 2232 first reported as Decabromobiphenyl

Other reported Brominated Flame retardants in sample #19606; 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	<10
551	N.D.	N.D.	N.D.	N.D.	N.D.	27.18	-----
623	-----	-----	-----	-----	-----	-----	-----
658	n.d.	-----	n.d.	n.d.	n.d.	17.148	-----
826	-----	-----	-----	-----	-----	-----	-----
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2129	-----	-----	-----	-----	-----	-----	-----
2138	ND	ND	ND	ND	ND	ND	NA
2139	-----	-----	-----	-----	-----	-----	-----
2156	< 20.00	< 20.00	< 20.00	< 20.00	< 20.00	< 20.00	< 20.00
2159	<5,0	<5,0	<5,0	<5,0	<5,0	21.80	<5,0
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
2176	1.9	ND	ND	ND	ND	ND	-----
2184	ND	ND	ND	ND	ND	ND	-----
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2232	-----	-----	-----	-----	-----	-----	-----
2250	<5	<5	<5	<5	<5	<5	<5
2256	-----	-----	-----	-----	-----	-----	-----
2271	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
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	Not Detected	Not Detected
2316	ND	ND	ND	ND	ND	ND	-----
2347	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	NA
2352	-----	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	ND	ND	ND
2355	<5	<5	<5	<5	<5	<5	<5
2357	<5	<5	<5	<5	<5	<5	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND	NA
2365	＜5.0	＜5.0	＜5.0	＜5.0	＜5.0	＜5.0	-----
2366	<5	<5	<5	<5	<5	<5	<5
2369	<5	<5	<5	<5	<5	<5	-----
2370	<5	<5	<5	<5	<5	<5	<5
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2375	-----	-----	-----	-----	<5	<5	-----
2379	ND	ND	ND	ND	ND	ND	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	N.D. [<5]	N.D. [<5]	N.D. [<5]	N.D. [<5]	N.D. [<5]	N.D. [<5]	-----
2390	-----	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----	-----
2415	-----	-----	-----	-----	-----	-----	-----
2481	-----	-----	-----	-----	-----	-----	-----
2482	-----	-----	-----	-----	-----	-----	-----
2488	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	10.22	-----
2612	< LOQ	< LOQ	< LOQ	< LOQ	478.29	< LOQ	< LOQ
2621	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2672	< 100	< 100	< 100	< 100	< 100	<100 C	-----
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----

Lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	Nona-BDE	Deca-BDE	Other
2705	-----	-----	-----	-----	-----	-----	-----
2740	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----
2774	-----	-----	-----	-----	-----	-----	-----
2816	-----	-----	-----	-----	-----	-----	-----
2826	< 100	< 100	< 100	< 100	< 100	< 100	< 100
2857	ND	ND	ND	ND	ND	ND	-----
2864	N.D.	N.D.	N.D.	N.D.	N.D.	7.20	-----
2886	-----	-----	-----	-----	-----	-----	-----
2890	-----	-----	-----	-----	-----	-----	-----
2900	ND	ND	ND	ND	ND	ND	NA
3118	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5	< 5	< 5
3182	<5	<5	<5	<5	<5	<5	<5
3197	<20	<20	<20	<20	<20	<20	-----
3210	-----	-----	-----	-----	-----	-----	-----
3213	-----	-----	-----	-----	-----	-----	-----
3214	<5	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
3239	-----	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	n.a.	n.a.	n.a.

Lab 2672 first reported 130

APPENDIX 3

Analytical details

Lab	Accred. for ISO/IEC17025 for the rep. comp.	Sample pretreatment	technique release/extract the analyte(s)	Solvent (mixture) used to release	Extraction time (min)	Extraction temp. (°C)
339	No	Used as received	Ultrasonic	Toluene	60	60
551	Yes	Further Cut	Soxhlet	Toluene	120 min	75°C
623	Yes	Further Cut	Ultrasonic	Toluene	30 minutes	Room temperature
658	Yes	Further Cut	Ultrasonic	Toluene	60 mins	60 degree Celsius
826	Yes	Further Cut	Ultrasonic	Toluene	60 mins	60
840	Yes	Further Cut	Ultrasonic	Toluene	60	50
2129	Yes	Further Grinded	Ultrasonic	DCM (LC-TOF-MS: Toluene)	30 min (LC-TOF-MS:60 min)	RT °C (LC-TOF-MS: 60 °C)
2138	Yes	Used as received	Ultrasonic	Toluene	About 2 hours	70 degree
2139	Yes	Used as received	Ultrasonic	Toluene	60	60
2156	Yes	Further Cut	Ultrasonic	Toluene	2 hours	60 degree celcius
2159	Yes	Used as received	Ultrasonic	Toluene	60 min	70 C
2165	Yes	Used as received	Ultrasonic	Toluene	180	60
2176	Yes	Further Cut	Soxhlet	Toluene for PVC Acetone for ABS	180 minutes	
2184	Yes	Used as received	Ultrasonic	Toluene	180 min	60°C
2202	Yes	Used as received	Stirrer	(THF or CH2Cl2/ Toluene/Hexane	24	Room temperature
2232	Yes	Used as received	Soxhlet	Toluene	120	500
2250	Yes	Used as received	Ultrasonic	PBB's+PBDE's: toluene HBCDD; Acetone	PBB's+PBDE's: 2 h; HBCDD: 1 h	PBB's+PBDE's: 70°C; HBCDD: 40 °C
2256	Yes	Further Cut	Soxhlet	TOLUENE	120 minutes	
2271	Yes	Further Grinded	Soxhlet	Toluene	120	2-3min/cycle
2310	Yes	Used as received	Ultrasonic	Toluene	1hr	50
2311	Yes	Further Grinded	Soxhlet	Toluene	120	120
2316	Yes	Further Grinded	Soxhlet	Toluene	150 min	70 to 80° C
2347	Yes	Further Cut	Soxhlet	toluene	360min	
2350	Yes	Used as received	Ultrasonic	Toluene	120min	50
2352	Yes	Further Cut	Soxhlet	Toluene	60min	60
2353	Yes	Further Grinded	Soxhlet	Toluene	120 minutes	Under reflux
2355	Yes	Further Cut	Soxhlet	Toluene	PBB/PBDE:16h HBCDD:60min	HBCDD:60
2357	Yes	Further Grinded	Soxhlet	toluene	420min	
2358	Yes	Further Grinded	Soxhlet	Toluene	120 min	
2363	Yes	Further Cut	Soxhlet	Toluene	240min	NA
2365	Yes	Further Cut	Soxhlet	Toluene	6h	290℃
2366	Yes	Further Cut	19605 Soxhlet 19606 Ultrason.	toluene	PBB/PBDE : 6h HBCDD 1h	PBB/PBDE : reflux HBCDD 60°C
2369	Yes	Further Cut	Soxhlet	Toluene	4h	
2370	Yes	Further Grinded	Soxhlet	Toluene	107min	250
2372	Yes	Further Grinded	Ultrasonic	Toluene	60 min	60 °C
2375	Yes	Further Cut	Ultrasonic	Toluene	60 min	60
2379	Yes	Further Grinded	Soxhlet	Toluene		
2382	Yes	Further Cut	Soxhlet	Toluene	240	
2384	Yes	Further Grinded	Soxhlet	Toluene	16 hours	reflux temperature
2386	Yes	Further Grinded	Soxhlet	Toluene	240	
2387	Yes	Further Grinded	Soxhlet	Toluene	960-1260	Reflux Temperature
2390	---	---	---			
2410	Yes	Further Grinded	Soxhlet	Toluene	120 min	60
2415	Yes	Further Cut	Ultrasonic	Toluene / THF	60	60
2481	---	---	---			
2482	Yes	Furter Cut	Ultrasonic	Toluene	120 min	60
2488	Yes	Used as received	Soxhlet			
2590	No	Used as received	Soxhlet	Toluene:DCM 1:1	360	not applicable
2612	Yes	Further Cut	Soxhlet	toluene	3 h	110.6 °C
2621	Yes	Used as received	Soxhlet	Toluene	240-300	Boiling Temp
2672	Yes	Milled (cryogenic)	Ultrasonic	toluene	60 min	70 °C
2674	Yes	Used as received	Ultrasonic	Toluene, Hexane	180mins	60
2705	---	---	---			
2740	No	Used as received	Ultrasonic	toluene	30 min + 15 min	room temperature
2743	Yes	Used as received	Ultrasonic	Toluene	30 min. + 15 min.	Room temperature

Lab	Accred. for ISO/IEC17025 for the rep. comp.	Sample pretreatment	technique release/extract the analyte(s)	Solvent (mixture) used to release	Extraction time (min)	Extraction temp. (°C)
2774	Yes	Used as received	19605 Soxhlet 19606 Ultrason.	Toluene with internal standard	19605: 120 Minutes 19606: 60 Minutes	16905: 200°C 19606: 70°C
2816	---	---	---			
2826	Yes	Further Grinded	Soxhlet	Toluene	180	
2857	Yes	Further Cut	Ultrasonic	19605 toluene 19606 toluene/methanol	60	60 degree Celsius
2864	Yes	Further Cut	Soxhlet	Toluene	2hr	110
2886	No	Used as received	Ultrasonic	cyclohexane/acetone 50:50	120 min	30-50
2890	---	---	---			
2900	Yes	Used as received	Ultrasonic	Toluene	2 hours	70 degree
3118	---	---	---			
3146	Yes	Used as received	Ultrasonic	Tertrahydrofurane	30	70
3163	---	---	---			
3172	Yes	Used as received	Ultrasonic	Toluene / Acetone	45	25
3182	Yes	Further Grinded	Soxhlet	Toluene	360 minutes	at 2.5 minutes/cycle
3197	Yes	Further Cut	Soxhlet	Toluene	120 minutes	Reflux Temperature
3210	---	---	---			
3213	Yes	Further Grinded	Soxhlet	Toluene	about 360 min.	about 90~100 °C
3214	Yes	Further Grinded	Soxhlet	Toluene	240	
3228	No	Used as received	Ultrasonic	Toluene	180mins	60
3239	Yes	Further Grinded	Soxhlet	Toluene	120 mins	115
3246	Yes	Further Cut	Soxhlet	Toluene	120 min	boiling p. of Toluene

APPENDIX 4

Number of participants per country

1 lab in	BRAZIL
1 lab in	COLOMBIA
2 labs in	DENMARK
3 labs in	FRANCE
9 labs in	GERMANY
4 labs in	HONG KONG
3 labs in	INDIA
2 labs in	INDONESIA
3 labs in	ITALY
1 lab in	LUXEMBOURG
3 labs in	MALAYSIA
14 labs in	P.R. of CHINA
1 lab in	PAKISTAN
3 labs in	SINGAPORE
7 labs in	SOUTH KOREA
6 labs in	TAIWAN R.O.C.
3 labs in	THAILAND
1 lab in	THE NETHERLANDS
4 labs in	TURKEY
4 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

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