Results of Proficiency Test
Brominated Flame retardants
September 2018

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 Diphenyls 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.

A proficiency scheme for the determination of Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyls Ethers (PBDE) was started by the Institute for Interlaboratory Studies in 2009 and the PT was extended with Hexabromocyclododecane (HBCDD) in 2015. It was decided to continue with the interlaboratory study for the determination of Brominated Flame Retardants in the annual proficiency testing program 2018/2019. In this interlaboratory study 85 laboratories in 24 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different plastic samples, both PVC. One sample contained approx. 3 grams of small green/blue squares, labelled #18605 and the other sample contained approx. 3 grams of small red squares, labelled #18606. 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/IEC 17043:2010 (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 #18605 was a green/blue coloured PVC, artificially fortified with Hexabromocyclododecane (HBCDD). After homogenisation, subsamples were filled with 3 grams and labelled #18605. The homogeneity of subsamples #18605 was checked by the determination of HBCDD content on 8 stratified randomly selected subsamples.

	HBCDD in mg/kg
sample #18605-1	962
sample #18605-2	978
sample #18605-3	942
sample #18605-4	914
sample #18605-5	948
sample #18605-6	933
sample #18605-7	916
sample #18605-8	947

Table 1: homogeneity test results of subsamples #18605

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 ISO 13528, Annex B2 in the next table;

	HBCDD in mg/kg
r (observed)	61
reference test method	IMEP-26:11
0.3 x R (ref. test method)	198

Table 2: evaluation of the repeatability of subsamples #18605

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 #18606 was a red coloured PVC, artificially fortified with Decabromodiphenylether (Deca-BDE) and Decabromobiphenyl (Deca-BB). After homogenisation, subsamples were filled with 3 grams and labelled #18606. The homogeneity of subsamples #18606 was checked by the determination of Deca-BDE and Deca-BB content on 8 stratified randomly selected subsamples.

	Deca-BDE in mg/kg	Deca-BB in mg/kg
sample #18606-1	125.5	82.5
sample #18606-2	127.5	88.7
sample #18606-3	131.2	90.2
sample #18606-4	123.4	86.2
sample #18606-5	119.8	81.7
sample #18606-6	124.5	83.0
sample #18606-7	127.5	83.1
sample #18606-8	121.8	80.4

Table 3: homogeneity test results of subsamples #18606

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

	Deca-BDE in mg/kg	Deca-BB in mg/kg
r (observed)	10.1	9.8
reference test method	IEC62321-6:15	IMEP-26:11
0.3 x R (ref. test method)	29.9	17.7

Table 4: evaluation of the repeatabilities of subsamples #18606

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories 1 sample labelled #18605 and 1 sample labelled #18606 was sent on August 8, 2018.

2.5 ANALYSES

The participants were requested to determine on both samples: Decabromodiphenyl ether (Deca-BDE), Octabromodiphenyl ether (Octa-BDE), Nonabromodiphenyl ether (Nona-BDE), Decabromobiphenyl (Deca-BB), Octabromobiphenyl (Octa-BB), Nonabromobiphenyl (Nona-BB) 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 calculations.

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 reanalyses). 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 ISO 5725 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, some problems were encountered with the dispatch of the samples. Three 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 77 reporting laboratories reported 256 numerical test results. Observed were 9 outlying test results, which is 3.5% of the statistically evaluated numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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 Deca-BDE. Regretfully, the IEC62321-6 method does not mention precision data for PBB. For the evaluation of Deca-BB the reproducibility is calculated as described for HBCDD, according to the IMEP-26 results using a target standard deviation of 25% of the assigned value.

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.

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 methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Sample #18605

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

Sample #18606

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

Deca-BB: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of the target reproducibility from IMEP-26:2011.

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 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)
HBCDD	mg/kg	49	756	363	529

Table 5: reproducibility of tests on sample #18605

Component	unit	n	average	2.8 * sd	R (lit)
Deca-BDE	mg/kg	73	129.3	101.5	102.5
Deca-BB	mg/kg	67	81.0	49.6	56.7

Table 6: reproducibilities of tests on sample #18606

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

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2018 AGAINST PREVIOUS PTS

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs, see below table.

	Sept. 2018	Sept. 2017	Sept. 2016	August 2015	Sept. 2014	target *)	
						IMEP-26	IEC62321-1
Nona-BDE	n.e.	38%	36%	46%	32-33%	25%	26 - 44% (<500mg/kg)
Deca-BDE	28%	16%	18%	17%	24%	25%	26 - 29% (<5000mg/kg)
Deca-BB	22%	n.e.	n.e.	n.e.	n.e.	25%	n.a.
HBCDD	17%	39%	24%	49%	n.e.	25%	n.a.

Table 7: comparison with previous proficiency tests

*) 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 slightly higher than the uncertainties observed in previous PTs. The uncertainty observed for HBCDD in this PT has improved in comparison with the previous PTs. All uncertainties in this PT (Deca-BDE, Deca-BB and HBCDD) 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 large majority participants used for the determination of PBDE, PBB and HBCDD IEC62321-6-GC/MS as test method. A few laboratories used a different test method for the determination of HBCDD than for the determination of PBDE and PBB.
Also a few laboratories were able to identify HBCDD but not able to quantify HBCDD (e.g. due to the lack of a suitable reference standard).

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-five of the participants answered to be ISO/IEC17025 accredited for the determination of brominated flame retardants in polymers (= 84%).
A few laboratories used a (slightly) different analytical pathway for the determination of HBCDD than for to the determination of PBDE and PBB.
About 25% of the participants used the samples as received, about 30% of the participants grinded the samples further prior to analyses and about 40% cut the samples further prior to analyses.
The technique used to release the PBDE, PBB and HBCDD from the samples differs. About 45% of the participants used ultrasonic as technique and about 45% of the participants used Soxhlet. Toluene or a Toluene mixture was used as solvent to release the analytes by about 85% of the participants.
The extraction time used differs from 15 min. to overnight. About 35% of the participants used an extraction time of 120 min.

The extraction temperature used differs from room temperature to 180 °C. About 35% of the participants used an extraction temperature in the range of 40 °C upto and including 60 °C.

5 DISCUSSION

The material of both samples in this PT (sample #18605 and sample #18606) was PVC. To extract the requested components (see chapter 2.5) from a solid like a polymer, the extraction solvent, the extraction conditions and the contact surface area are important variables.

In the PT of 2018 on Brominated Flame Retardants it was decided, to use PVC samples positive on Deca-BDE, Deca-BB and HBCDD. It appeared that none of the requested analytical details was dominant as the calculated reproducibilities for the Deca-BDE, Deca-BB and HBCDD 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 or IMEP-26.

The majority of the group identified all added PBDE, PBB and HBCDD correctly: sample #18605 contained HBCDD and sample #18606 contained Deca-BDE and Deca-BB.

According to the RoHS Directive 2011/65/EU, electrical and electronic equipment are not allowed to contain more than 1000 mg/kg PBDE or PBB (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 accept sample #18606.

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 #18605 for containing to much HBCDD.

6 CONCLUSION

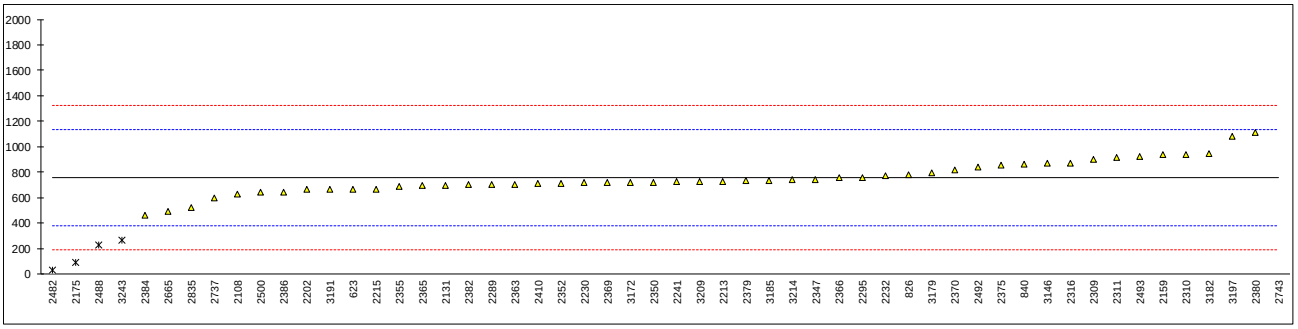
Although it can be concluded that most of the participants have no problem with the determination on Deca-PBDE, Deca-PBB and HBCDD in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation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

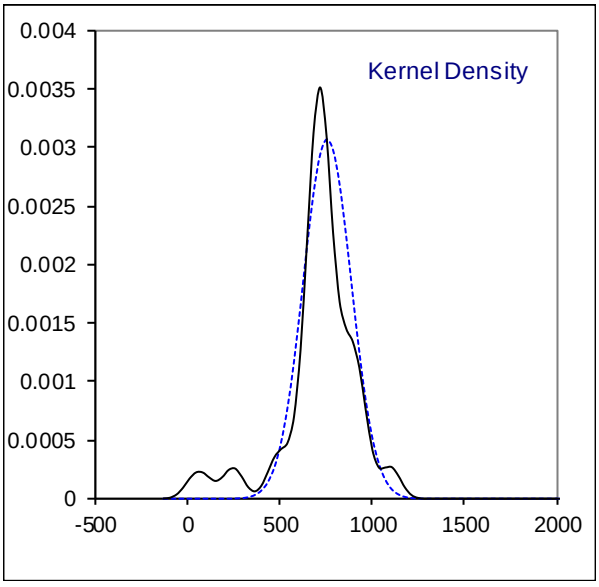
APPENDIX 1

Determination of Hexabromocyclododecane (HBCDD) on sample #18605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
324		-----		-----	
339	IEC62321 adapted	<10		<-3.95	possibly a false negative test result?
551		-----		-----	
623	IEC62321	669.55	C	-0.46	first reported 1483.293
826		780		0.13	
840	IEC62321-6 - GC/MS	861		0.55	
841		-----		-----	
2108	ISO 17881-1	628.9		-0.67	
2131	In house GC/MS	700.3		-0.30	
2159	In house	937.14	C	0.96	first reported <5 (fr. as other Br. Flame Ret.)
2165		-----		-----	
2175	IEC62321-6 - GC/MS	93.81	C,R(0.05)	-3.50	first reported 66.81
2184		-----		-----	
2202	IEC62321-6 - GC/MS	665		-0.48	
2213	IEC62321-6 - GC/MS	728.1		-0.15	
2215	In house	670.15		-0.46	
2230	In house	720	C	-0.19	first reported 0.747
2232	IEC62321-6 - HPLC-MS	769.6	C	0.07	first reported 0.7696240762
2236		-----		-----	
2241	IEC62321-6 - GC/MS	725.61		-0.16	
2247	IEC62321-6 - GC/MS	Detected		-----	identified but not quantified
2289	IEC62321-6 - HPLC-PDA/UV	706		-0.27	
2293		-----		-----	
2295	In house	760		0.02	
2309	IEC62321-6 - GC/MS	900.0		0.76	
2310	IEC62321-6 - GC/MS	940.4		0.97	
2311	IEC62321-6 - GC/MS	916.3		0.85	
2316	IEC62321-6 - GC/MS	870		0.60	
2347	IEC62321-6 - GC/MS	743		-0.07	
2350	IEC62321-6 - GC/MS	722.06		-0.18	
2352	IEC62321-6 - GC/MS	713.8		-0.22	
2353	IEC62321-6 - GC/MS	NA		-----	
2355	In house	691.1		-0.34	
2358	IEC62321-6 - GC/MS	N/A		-----	
2363	IEC62321-6 - GC/MS	707		-0.26	
2365	In house	697.404		-0.31	
2366	IEC62321-6 - GC/MS	759.1		0.01	
2369	IEC62321-6 - GC/MS	720		-0.19	
2370	IEC62321-6 - GC/MS	820		0.34	
2375	In house	854.7		0.52	
2379	IEC62321-6 - GC/MS	731.5664		-0.13	
2380	In house	1112.21		1.88	
2382	IEC62321-6 - GC/MS	702.4		-0.29	
2384	IEC62321-6 - GC/MS	463.93		-1.55	
2386	IEC62321-6 - GC/MS	647		-0.58	
2387		-----		-----	
2389		-----		-----	
2410	In house HPLC/MS	710		-0.24	
2424		-----		-----	
2482	IEC62321-6 - GC/MS	29.1755	C,R(0.05)	-3.85	first no test results reported (mix-up with #18606)
2488	ISO17881-1	228.1	R(0.05)	-2.79	
2492	In house	843.05		0.46	
2493	In house	926.0		0.90	
2500	IEC62321-6 - GC/MS	642.23		-0.60	
2612	IEC62321-6 - GC/MS	< LOQ		-----	
2665	IEC62321-6 - GC/MS	493.9		-1.39	
2674		-----		-----	
2705		-----		-----	
2737	In house	600	C	-0.83	first reported ND
2743	ISO17881-1	8727.69	R(0.01)	42.16	
2774	IEC62321-6 - GC/MS	not detected		-----	
2835	IEC62321-6 - GC/MS	524.78		-1.22	
2846	IEC62321-6 - GC/MS	Detected		-----	identified but not quantified
2854		-----		-----	
2855	IEC62321-6 - GC/MS	Detected		-----	identified but not quantified
2857		-----		-----	
2858		-----		-----	
3146	IEC62321-6 - LC/MS	868		0.59	
3153		-----		-----	
3154		-----		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	721.9		-0.18	
3179	IEC62321-6 - GC/MS	793.64		0.20	

lab	method	value	mark	z(targ)	remarks
3182	IEC62321-6 - GC/MS	946.92		1.01	
3185	IEC62321-6 - GC/MS	736.3		-0.11	
3191	IEC62321-6 - GC/MS	668.2		-0.47	
3197	IEC62321-6 - GC/MS	1084.5		1.74	
3209	In house	726.00		-0.16	
3210		-----		-----	
3213		-----		-----	
3214	IEC62321-6 - GC/MS	739.7		-0.09	
3228		-----		-----	
3239		-----		-----	
3243	IEC62321-6 - GC/MS	264.92	R(0.05)	-2.60	
	normality	OK			
	n	49			
	outliers	5			
	mean (n)	756.295			
	st.dev. (n)	129.7863			RSD = 17.2%
	R(calc.)	363.402			
	st.dev.(IMEP-26:11)	189.0737			
	R(IMEP-26:11)	529.406			

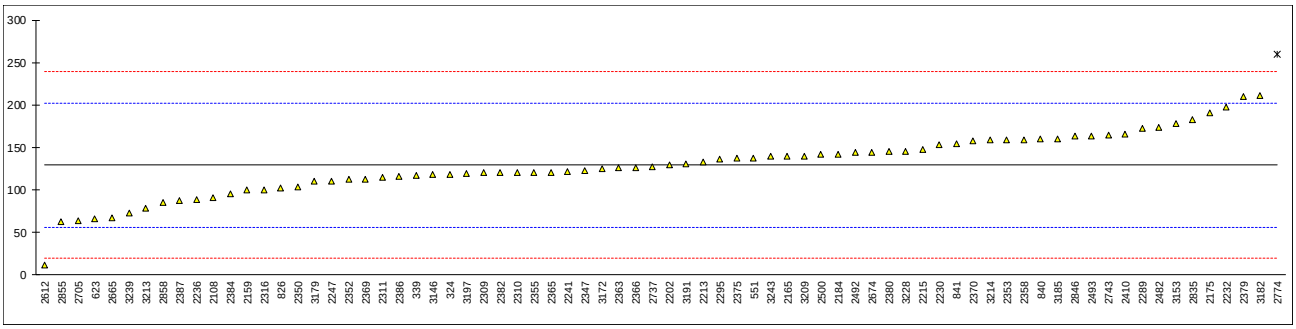


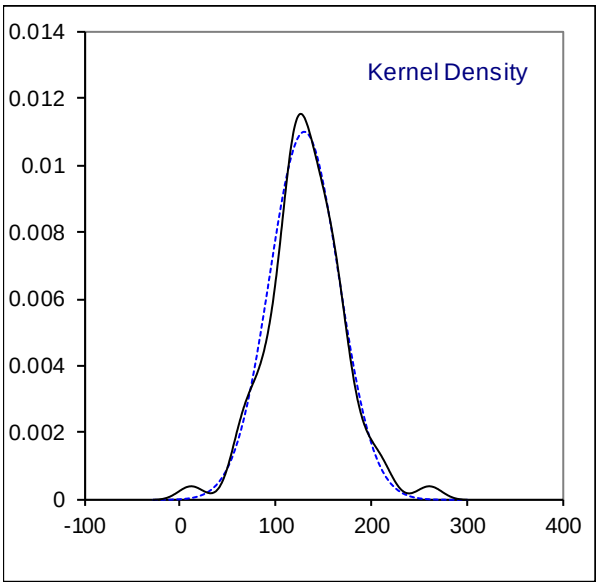


Determination of Decabromodiphenylether (Deca-BDE) on sample #18606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
324	IEC62321-6 - GC/MS	118.4		-0.30	
339	IEC62321 adapted	117		-0.33	
551	IEC62321-6 - GC/MS	137.5579		0.23	
623	IEC62321	66.42		-1.72	
826	IEC62321-6 - GC/MS	101.71		-0.75	
840	IEC62321-6 - GC/MS	160		0.84	
841	IEC62321-6 - GC/MS	154.7		0.70	
2108	ISO 17881-1	91.42		-1.03	
2131		-----		-----	
2159	In house	99.65		-0.81	
2165	IEC62321-6 - GC/MS	140		0.29	
2175	IEC62321-6 - GC/MS	190.73		1.68	
2184	IEC62321-6 - GC/MS	142		0.35	
2202	IEC62321-6 - GC/MS	129		-0.01	
2213	IEC62321-6 - GC/MS	132.38		0.09	
2215	In house	147.17		0.49	
2230	IEC62321-6 - GC/MS	153		0.65	
2232	IEC62321-6 - GC/MS	197.497768565		1.86	
2236	IEC62321-6 - GC/MS	88.28		-1.12	
2241	IEC62321-6 - GC/MS	121.56		-0.21	
2247	IEC62321-6 - GC/MS	110.34		-0.52	
2289	IEC62321-6 - GC/MS	173		1.20	
2293		-----		-----	
2295	In house	136		0.18	
2309		120.0		-0.25	
2310	IEC62321-6 - GC/MS	120.2		-0.25	
2311	IEC62321-6 - GC/MS	114.7		-0.40	
2316	IEC62321-6 - GC/MS	100		-0.80	
2347	IEC62321-6 - GC/MS	123		-0.17	
2350	IEC62321-6 - GC/MS	103.92		-0.69	
2352	IEC62321-6 - GC/MS	112.0		-0.47	
2353	IEC62321-6 - GC/MS	158.7		0.80	
2355	IEC62321-6 - GC/MS	120.4		-0.24	
2358	IEC62321-6 - GC/MS	158.7		0.80	
2363	IEC62321-6 - GC/MS	126		-0.09	
2365	QC/T 944	120.805		-0.23	
2366	IEC62321-6 - GC/MS	126.1		-0.09	
2369	IEC62321-6 - GC/MS	112		-0.47	
2370	IEC62321-6 - GC/MS	158		0.79	
2375	In house	136.8		0.21	
2379	IEC62321-6 - GC/MS	210		2.21	
2380	In house	144.93		0.43	
2382	IEC62321-6 - GC/MS	120.1		-0.25	
2384	IEC62321-6 - GC/MS	95.07		-0.93	
2386	IEC62321-6 - GC/MS	116		-0.36	
2387	IEC62321-6 - GC/MS	87.50		-1.14	
2389		-----		-----	
2410	IEC62321-6-LC-PDA/UV	166		1.00	
2424		-----		-----	
2482	In house	173.56	C	1.21	first no test results reported (mix-up with #18605)
2488		-----		-----	
2492	In house	143.70		0.39	
2493	In house	163.9		0.95	
2500	IEC62321-6 - GC/MS	141.98		0.35	
2612	IEC62321-6 - GC/MS	11.018	C	-3.23	first reported 19.283
2665	IEC62321-6 - GC/MS	67	C	-1.70	first reported 6.7
2674	IEC62321-6 - GC/MS	144.6		0.42	
2705		63.2		-1.80	
2737	In house	127.09		-0.06	
2743	ISO17881-1	164.52		0.96	
2774	IEC62321-6 - GC/MS	260	R(0.05)	3.57	
2835	IEC62321-6 - GC/MS	182.27		1.45	
2846	IEC62321-6 - GC/MS	163.29	C	0.93	first no test results reported (mix-up with #18605)
2854		-----		-----	
2855	IEC62321-6 - GC/MS	62.2		-1.83	
2857		-----		-----	
2858	In house	84.7		-1.22	
3146	IEC62321-6 - LC/MS	118		-0.31	
3153	IEC62321-6 - GC/MS	178.2		1.34	
3154		-----		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	124.5		-0.13	
3179	IEC62321-6 - GC/MS	110.23		-0.52	

lab	method	value	mark	z(targ)	remarks
3182	IEC62321-6 - GC/MS	210.6	C	2.22	first reported 244.5
3185	IEC62321-6 - GC/MS	160.2		0.85	
3191	IEC62321-6 - GC/MS	130.2		0.03	
3197	IEC62321-6 - GC/MS	118.7		-0.29	
3209	IEC62321-6 - GC/MS	140.00		0.29	
3210		-----		-----	
3213	IEC62321-6 - GC/MS	78		-1.40	
3214	IEC62321-6 - GC/MS	158.6		0.80	
3228	IEC62321-6 - GC/MS	145		0.43	
3239	IEC62321-6 - GC/MS	72.240	C	-1.56	first reported 68.943 (fr. as Deca-BB)
3243	IEC62321-6 - GC/MS	139.12		0.27	
	normality	OK			
	n	73			
	outliers	1			
	mean (n)	129.251			
	st.dev. (n)	36.2620			RSD = 28.1%
	R(calc.)	101.534			
	st.dev.(IEC62321-6:15)	36.5953			
	R(IEC62321-6:15)	102.467			

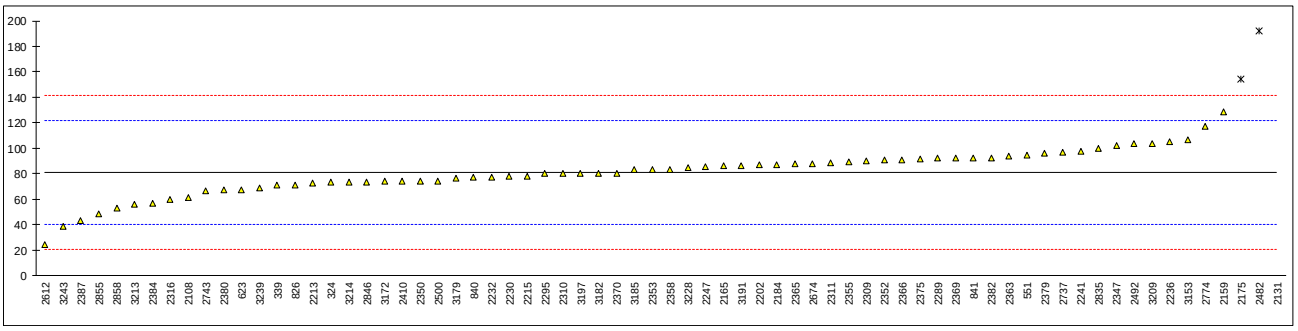


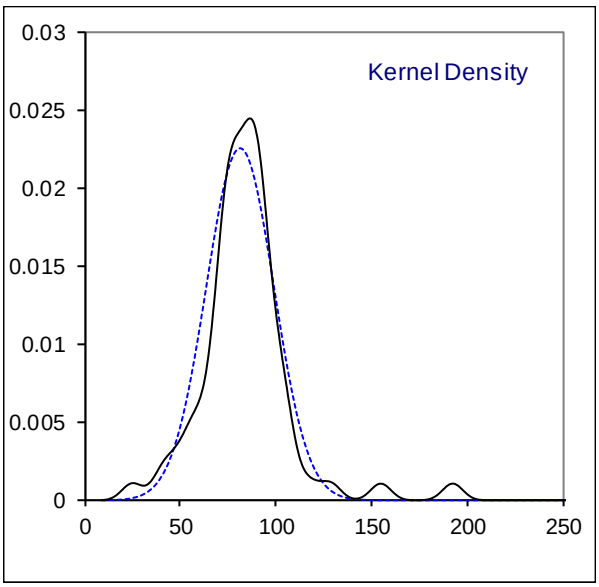


Determination of Decabromobiphenyl (Deca-BB) on sample #18606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
324	IEC62321-6 - GC/MS	73.6		-0.37	
339	IEC62321 adapted	71.1		-0.49	
551	IEC62321-6 - GC/MS	94.5082		0.67	
623	IEC62321:08	67.42		-0.67	
826	IEC62321-6 - GC/MS	71.30		-0.48	
840	IEC62321-6 - GC/MS	77		-0.20	
841	IEC62321-6 - GC/MS	92.21		0.55	
2108	ISO 17881-1	61.34		-0.97	
2131	In house GC/MS	295.9	R(0.01)	10.61	
2159	In house	128.33		2.34	
2165	IEC62321-6 - GC/MS	86		0.25	
2175	IEC62321-6 - GC/MS	154.625	R(0.01)	3.63	
2184	IEC62321-6 - GC/MS	87		0.29	
2202	IEC62321-6 - GC/MS	87		0.29	
2213	IEC62321-6 - GC/MS	72.64		-0.41	
2215	In house	78.22		-0.14	
2230	IEC62321-6 - GC/MS	77.7		-0.16	
2232	IEC62321-6 - GC/MS	77.3694390715		-0.18	
2236	IEC62321-6 - GC/MS	105.14		1.19	
2241	IEC62321-6 - GC/MS	97.96		0.84	
2247	IEC62321-6 - GC/MS	85.23		0.21	
2289	IEC62321-6 - GC/MS	92		0.54	
2293		-----		-----	
2295	In house	80.1		-0.05	
2309		90.0		0.44	
2310	IEC62321-6 - GC/MS	80.3		-0.04	
2311	IEC62321-6 - GC/MS	88.2		0.35	
2316	IEC62321-6 - GC/MS	60		-1.04	
2347	IEC62321-6 - GC/MS	102		1.04	
2350	IEC62321-6 - GC/MS	74.12		-0.34	
2352	IEC62321-6 - GC/MS	90.9		0.49	
2353	IEC62321-6 - GC/MS	83.4		0.12	
2355	IEC62321-6 - GC/MS	89.0		0.39	
2358	IEC62321-6 - GC/MS	83.4		0.12	
2363	IEC62321-6 - GC/MS	94		0.64	
2365	QC/T 944	87.509		0.32	
2366	IEC62321-6 - GC/MS	91.1		0.50	
2369	IEC62321-6 - GC/MS	92		0.54	
2370	IEC62321-6 - GC/MS	80.6		-0.02	
2375	In house	91.2		0.50	
2379	IEC62321-6 - GC/MS	96		0.74	
2380	In house	67.05		-0.69	
2382	IEC62321-6 - GC/MS	92.3		0.56	
2384	IEC62321-6 - GC/MS	56.57		-1.21	
2386	IEC62321-6 - GC/MS	<50		-----	
2387	IEC62321-6 - GC/MS	43.51		-1.85	
2389		-----		-----	
2410	IEC62321-6 - GC/MS	74		-0.35	
2424		-----		-----	
2482	In house	192.25	C,R(0.01)	5.49	first no test results reported (mix-up with #18605)
2488		-----		-----	
2492	In house	103.49		1.11	
2493		-----		-----	
2500	IEC62321-6 - GC/MS	74.32		-0.33	
2612	IEC62321-6 - GC/MS	24.541		-2.79	
2665		-----		-----	
2674	IEC62321-6 - GC/MS	87.6		0.32	
2705		-----		-----	
2737	In house	96.6	C	0.77	first reported 296.6
2743	ISO17881-1	66.54		-0.72	
2774	IEC62321-6 - GC/MS	117		1.78	
2835	IEC62321-6 - GC/MS	100.14		0.94	
2846	IEC62321-6 - GC/MS	73.77	C	-0.36	first no test results reported (mix-up with #18605)
2854		-----		-----	
2855	IEC62321-6 - GC/MS	48.2		-1.62	
2857		-----		-----	
2858	In house	52.8		-1.39	
3146	IEC62321-6 - LC/MS	<100		-----	
3153	IEC62321-6 - GC/MS	106.6		1.26	
3154		-----		-----	
3163		-----		-----	
3172	IEC62321-6 - GC/MS	73.9		-0.35	
3179	IEC62321-6 - GC/MS	76.39		-0.23	

lab	method	value	mark	z(targ)	remarks
3182	IEC62321-6 - GC/MS	80.39		-0.03	
3185	IEC62321-6 - GC/MS	83.2		0.11	
3191	IEC62321-6 - GC/MS	86.2		0.26	
3197	IEC62321-6 - GC/MS	80.3		-0.04	
3209	IEC62321-6 - GC/MS	104.00		1.13	
3210		-----		-----	
3213	IEC62321-6 - GC/MS	56		-1.24	
3214	IEC62321-6 - GC/MS	73.6		-0.37	
3228	IEC62321-6 - GC/MS	85		0.20	
3239	IEC62321-6 - GC/MS	68.943	C	-0.60	first reported 72.240 (fr. as Deca-BDE)
3243	IEC62321-6 - GC/MS	38.99		-2.08	
	normality	suspect			
	n	67			
	outliers	3			
	mean (n)	81.027			
	st.dev. (n)	17.7006			RSD = 21.8%
	R(calc.)	49.562			
	st.dev.(IMEP-26:11)	20.2569			
	R(IMEP-26:11)	56.719			





APPENDIX 2

Abbreviations of components:

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

Other reported Brominated Flame retardants in sample #18605; results in mg/kg

Lab	Deca-BDE	Octa-BDE	Nona-BDE	Deca-BB	Octa-BB	Nona-BB	Other
110	-----	-----	-----	-----	-----	-----	-----
324	< 10	< 10	< 10	< 10	< 10	< 10	-----
339	<10	<1	<2	<10	<1	<2	<loq
551	ND	ND	ND	ND	ND	ND	-----
623	ND	ND	ND	ND	ND	ND	ND
826	-----	-----	-----	-----	-----	-----	-----
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	ND	ND	ND	ND	ND	ND	-----
2108	-----	-----	-----	-----	-----	-----	-----
2131	-----	-----	-----	-----	-----	-----	-----
2159	<5	<5	<5	<5	<5	<5	<5 C
2165	ND	ND	ND	ND	ND	ND	-----
2175	0	0	0	0	0	0	0
2184	ND	ND	ND	ND	ND	ND	-----
2202	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2213	<1	<1	<1	<1	<1	<1	<1
2215	ND	ND	ND	ND	ND	ND	ND
2230	<5	<5	<5	<5	<5	<5	ND
2232	-----	-----	-----	-----	-----	-----	-----
2236	<25	<25	<25	<25	<25	<25	-----
2241	<10	<10	<10	<10	<10	<10	<10
2247	ND [DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]
2289	<5	<5	<5	<5	<5	<5	<5
2293	-----	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----	-----
2309	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]
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	-----
2316	ND	ND	ND	ND	ND	ND	--
2347	<5	<5	<5	<5	<5	<5	<5
2350	< 5	< 5	< 5	< 5	< 5	< 5	< 5
2352	-----	-----	-----	-----	-----	-----	-----
2353	ND	ND	ND	ND	ND	ND	ND
2355	<5	<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	ND
2365	<5	<5	<5	<5	<5	<5	<5
2366	<5	<5	<5	<5	<5	<5	<5
2369	<5	<5	<5	<5	<5	<5	-----
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not test
2380	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2384	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	-----
2386	<50	<25	<25	<50	<25	<25	-----
2387	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	-----
2389	-----	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----	-----
2424	-----	-----	-----	-----	-----	-----	-----
2482	----- C	-----	----- C	----- C	-----	-----	-----
2488	-----	-----	-----	-----	-----	-----	-----
2492	-----	-----	-----	-----	-----	-----	-----
2493	0	0	0	-----	-----	-----	-----
2500	<5	<5	<5	<5	<5	<5	<5
2612	12.341	7.401	7.812	< LOQ	5.431	5.428	5.183

Lab	Deca-BDE	Octa-BDE	Nona-BDE	Deca-BB	Octa-BB	Nona-BB	Other
2665	-----	-----	-----	-----	-----	-----	-----
2674	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----
2705	0.0	-----	-----	-----	-----	-----	-----
2737	ND	ND	ND	ND	ND	ND	ND
2743	-----	-----	-----	-----	-----	-----	-----
2774	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2835	-----	-----	-----	-----	-----	-----	-----
2846	----- C	-----	----- C	----- C	-----	-----	-----
2854	-----	-----	-----	-----	-----	-----	734.17
2855	-----	-----	-----	-----	-----	-----	-----
2857	-----	-----	-----	-----	-----	-----	-----
2858	-----	-----	-----	-----	-----	-----	-----
3146	<100	<100	<100	<100	<100	<100	-----
3153	<20	<20	<20	<20	<20	<20	-----
3154	-----	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----
3179	-----	-----	-----	-----	-----	-----	-----
3182	less than 5	less than 5	less than 5	less than 5	less than 5	less than 5	less than 5
3185	<5	<5	<5	<5	<5	<5	-----
3191	<10	<10	<10	<10	<10	<10	<10
3197	<10	<10	<10	<10	<10	<10	<10
3209	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----
3213	-----	-----	-----	-----	-----	-----	-----
3214	<5	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----
3239	-----	-----	-----	-----	-----	-----	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 2159: first reported 937.14 (fr. as HBCD)
Lab 2482: first reported 1735.58, 20.66, 1922.45 respectively (mix-up with #18506)
Lab 2846: first reported 163.29, <5, 73.77 respectively (mix-up with #18506)

Other reported Brominated Flame retardants in sample #18606; results in mg/kg

Lab	Octa-BDE	Nona-BDE	Octa-BB	Nona-BB	HBCDD	Other
110	-----	-----	-----	-----	-----	-----
324	< 10	< 10	< 10	< 10	-----	-----
339	<1	3.6	<1	<2	<10	<loq
551	ND	ND	ND	ND	-----	-----
623	ND	6.37	ND	ND	ND	ND
826	-----	7.55	-----	-----	-----	-----
840	n.d.	10.7	n.d.	n.d.	n.d.	n.d.
841	ND	10.3	ND	ND	-----	-----
2108	-----	3.37	-----	-----	-----	-----
2131	-----	5.25	-----	-----	-----	-----
2159	<5	<5	<5	<5	<5	<5
2165	ND	ND	ND	ND	-----	-----
2175	0	0	0	0	0	0
2184	ND	ND	ND	ND	-----	-----
2202	N.D.	17	N.D.	N.D.	N.D.	-----
2213	<1	3.82	<1	<1	<1	<1
2215	ND	ND	ND	ND	ND	ND
2230	<5	<5	<5	<5	<5	<5
2232	-----	-----	-----	-----	-----	-----
2236	<25	<25	<25	<25	-----	-----
2241	<10	<10	<10	<10	<10	<10
2247	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]	ND[DL-5mg/kg]
2289	<5	<5	<5	<5	<5	<5
2293	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----
2309	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-50mg/kg]	ND[DL-100mg/kg]	ND[DL-50mg/kg]
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2311	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	7.55	< 5	< 5	< 5	< 5
2352	-----	10.3	-----	-----	-----	-----
2353	ND	12.0	ND	ND	NA	ND
2355	<5	<5	<5	<5	<10	<5
2358	n.d.	12.0	n.d.	n.d.	N/A	n.d.
2363	ND	12	ND	ND	ND	ND
2365	<5	<5	<5	<5	<10	<5
2366	<5	<5	<5	<5	<10	<5
2369	<5	11	<5	<5	<10	-----
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab	Octa-BDE	Nona-BDE	Octa-BB	Nona-BB	HBCDD	Other
2375	<5	7.1	<5	<5	-----	-----
2379	Not detected	10	Not detected	Not detected	Not detected	Not test
2380	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2384	Not detected [<5]	Not detected [<5]	Not detected [<5]	Not detected [<5]	Not detected [<5]	-----
2386	<25	<25	<25	<25	<50	-----
2387	Not Detected<5	Not Detected<5	Not Detected<5	Not Detected<5	-----	-----
2389	-----	-----	-----	-----	-----	-----
2410	-----	-----	-----	-----	-----	-----
2424	-----	-----	-----	-----	-----	-----
2482	-----	2.07 C	-----	-----	----- C	-----
2488	-----	-----	-----	-----	<10	-----
2492	-----	-----	-----	-----	-----	-----
2493	104.9	172.6	-----	-----	0	-----
2500	<5	<5	<5	<5	<5	<5
2612	8.602	9.028	18.951	6.765	< LOQ	13.44
2665	-----	-----	-----	-----	-----	-----
2674	n.d.	n.d.	n.d.	n.d.	-----	-----
2705	-----	-----	-----	-----	-----	-----
2737	ND	ND	ND	ND	74.14	ND
2743	-----	-----	-----	-----	-----	-----
2774	not detected	<50	not detected	not detected	not detected	not detected
2835	-----	-----	-----	-----	-----	-----
2846	-----	<5 C	-----	-----	-----	-----
2854	-----	-----	-----	-----	-----	220.25
2855	-----	20.1	-----	-----	-----	-----
2857	-----	-----	-----	-----	-----	-----
2858	n.d	n.d	n.d	n.d	n.d	n.d
3146	<100	<100	<100	<100	<100	-----
3153	<20	<20	<20	<20	-----	-----
3154	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----
3179	-----	6.67	-----	-----	-----	-----
3182	less than 5	9.42	less than 5	less than 5	less than 5	less than 5
3185	<5	<5	<5	<5	<5	-----
3191	<10	<50	<10	<10	<10	<10
3197	<10	9.0	<10	<10	<10	<10
3209	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----
3213	-----	-----	-----	-----	-----	-----
3214	<5	<5	<5	<5	<5	<5
3228	n.d.	n.d.	n.d.	n.d.	-----	-----
3239	-----	-----	-----	-----	-----	-----
3243	n.d.	1.45	n.d.	n.d.	18.56	n.d.

Lab 2482: first not reported, first reported 29.1755 respectively (mix-up with #18505)
Lab 2846: first not reported (mix-up with #18505)

APPENDIX 3

Analytical details

Lab	Accred. for ISO/IEC17025 for the rep. component(s)	Sample pretreatment	technique release/extract the analyte(s)	Solvent (mixture) used to release	Extraction time (min)	Extraction temp. (°C)
110	Yes	Further Cut	Ultrasonic	Toluene	60	60
324	No	Further Cut	Soxhlet	Toluene	1200	---
339	Yes	Used as received	Ultrasonic AND Thermal desorption	Toluene / Hexane	60	60
551	Yes	Further Grinded	Soxhlet	Toluene	120	---
623	Yes	Further Cut	Ultrasonic	Toluene	60	50
826	Yes	Further Cut 1 x 1 x 1 mm	Ultrasonic	----	60	60
840	Yes	Further Cut	Soxhlet	Toluene	2 hour	---
841	Yes	Further Cut	Ultrasonic	Toluene	60	50
2108	Yes	Used as received	Ultrasonic	Toluene	30 + 15	RT
2131	No	Used as received	Ultrasonic	Toluene	45	Ambient
2159	No	Further Cut (no clean up)	Ultrasonic	Toluene	2 hour	70
2165	Yes	Used as received	Ultrasonic	Toluene	180	60
2175	Yes	Used as received	Soxhlet	Toluene	2 hours and half	---
2184	Yes	Used as received	Ultrasonic	Toluene	180	60
2202	Yes	Used as received	Mechanical Shaking	THF/Toluene/ Hexane	24hr	Room temperature
2213	Yes	Further Cut	Ultrasonic		---	---
2215	Yes	Further Cut	Ultrasonic	Toluene	120min	70
2230	Yes	Further Cut	Soxhlet	PBB/PBDE-Toluene HBCD-THF	PBB/PBDE-120 HBCD-60	HBCD-40
2232	Yes	Used as received	Soxhlet	Toluene	120	---
2236	Yes	Further Cut	Ultrasonic	Toluene	60	70
2241	Yes	Further Cut	Ultrasonic	Toluene	60	60
2247	Yes	Further Cut	Soxhlet	Toluene	120	110.0
2289	Yes	Further Cut	Soxhlet	Toluene	2h	---
2293	---	---	---		---	---
2295	Yes	Used as received	Ultrasonic	Toluene	150	200
2309	Yes	Further Grinded	Soxhlet	Toluene	2hrs	120
2310	Yes	Used as received	Soxhlet	Toluene	120	---
2311	Yes	Further Grinded	Soxhlet	Toluene	120	120
2316	Yes	Further Grinded	Soxhlet	Toluene	120	85
2347	Yes	Further Cut	Soxhlet	Toluene	360	---
2350	Yes	Further Grinded	Ultrasonic	Toluene	180	50
2352	Yes	Further Cut	Soxhlet	Toluene	240	120
2353	Yes	Further Grinded	Soxhlet	Toluene	120	Reflux only
2355	Yes	Further Cut	HBCDD: Ultrasonic PBB/PBDE: Soxhlet	Toluene	HBCDD:120 PBBPBDE:16h	HBCDD:60
2358	Yes	Further Grinded	Soxhlet	Toluene	120	Reflux only
2363	Yes	Further Grinded	Soxhlet	Methylbenzene	4h	---
2365	Yes	Further Cut	#18605: Ultrasonic; #18606: Soxhlet	Toluene	#18605:120 #18606: about 7hours	#18605:60; #18606:280
2366	Yes	Further Cut	Ultrasonic	Toluene	60	60
2369	---	---	---		---	---
2370	Yes	Further Grinded	Soxhlet	Toluene	107	300
2375	Yes	Further Cut	Ultrasonic	Toluene	60	60
2379	Yes	Further Grinded	Soxhlet	Toluene	2 hr	58
2380	Yes	Used as received	Ultrasonic	Toluene	60	60
2382	No	Further Cut	Soxhlet	Toluene	240	120
2384	Yes	Further Grinded	Other	Toluene	2 hours	under reflux temp.
2386	Yes	Further Grinded	Soxhlet	Toluene	240	---
2387	Yes	Further Cut	Soxhlet	Toluene	960-1260	Reflux Temperature
2389	---	---	---	---	---	---
2410	Yes	Further Grinded	Soxhlet	Toluene, THF	2 hr	60
2424	---	---	---	---	---	---
2482	Yes	Further Grinded	Ultrasonic	Toluene	60	60
2488	---	---	---	---	---	---
2492	Yes	Further Cut	Ultrasonic	Toluene	45	Room Temperature
2493	No	Used as received	Ultrasonic	Toluene/THF	overnight	room temp.
2500	Yes	Used as received	Ultrasonic	Toluene	120	70
2612	Yes	Further Cut	Ultrasonic	THF / n-Hexane	1 hour	60

Lab	Accred. for ISO/IEC17025 for the rep. component(s)	Sample pretreatment	technique release/extract the analyte(s)	Solvent (mixture) used to release	Extraction time (min)	Extraction temp. (°C)
2665	Yes	Used as received	Other	THF	90	20
2674	Yes	Used as received	Ultrasonic	Toluene Hexane	180	60
2705	No	Used as received	ASE	Hexane/Aceton 90/10	15	160
2737	Yes	Used as received	Ultrasonic	Toluene	60	50
2743	Yes	Further Cut	Ultrasonic	Toluene	30 + 15	60
2774	---	---	---	---	---	---
2835	Yes	Further Cut	ASE	Toluene	15	150
2846	Yes	Further Cut	Ultrasonic	THF and toluene	Over 60	40
2854	No	Further Grinded	Other	---	---	---
2855	No	Further Grinded	Ultrasonic	Toluene	60	50
2857	---	---	---	---	---	---
2858	Yes	Further Cut	Ultrasonic	Toluene	30	23
3146	Yes	Used as received	Ultrasonic	THF/ACN	60	60
3153	Yes	Further Grinded	Soxhlet	Toluene	4 hours reflux	---
3154	---	---	---	---	---	---
3163	---	---	---	---	---	---
3172	---	---	---	---	---	---
3179	Yes	Used as received	Thermal Desorption	Toluene	2h	100
3182	Yes	Further Grinded	Soxhlet	Toluene	About 360	soxhlet to extract: 2.5 minutes/cycle
3185	Yes	Further Cut	Soxhlet	Toluene	240	Extract by electric heating mantle
3191	Yes	Further Grinded	Soxhlet	Toluene	2h	230
3197	Yes	Further Cut	Soxhlet	Toluene	120	Reflux Temperature
3209	---	---	---	---	---	---
3210	---	---	---	---	---	---
3213	Yes	Further Grinded	Soxhlet	Toluene	4 hr	> 110
3214	Yes	Further Grinded	Soxhlet	Toluene	240	---
3228	Yes	Used as received	Ultrasonic	Toluene	180	60
3239	Yes	Further Grinded	Soxhlet	Toluene	2hrs	115
3243	Yes	Further Cut	Soxhlet	Toluene	120	115

APPENDIX 4

Number of participating laboratories per country

2 labs in BANGLADESH
1 lab in BELGIUM
2 labs in BRAZIL
2 labs in FRANCE
10 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
1 lab in HUNGARY
6 labs in INDIA
1 lab in INDONESIA
2 labs in ITALY
6 labs in KOREA
1 lab in LUXEMBOURG
2 labs in MALAYSIA
19 labs in P.R. of CHINA
1 lab in PAKISTAN
4 labs in SINGAPORE
1 lab in SWITZERLAND
4 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
5 labs in TURKEY
4 labs in U.S.A.
2 labs in VIETNAM

APPENDIX 5

Abbreviations:

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
f.r. = first reported
IMEP = International Measurement Evaluation Programme

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