



Institute for
Interlaboratory Studies

Results of Proficiency Test Total Brominated Flame Retardants in Polymers September 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis23P67

October 2023

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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. HBCDD is persistent, bio-accumulative and toxic to water-living organisms and slowly banned worldwide. The European Union decided to ban the use of both PBB and PBDE in electrical and electronic devices. In the RoHS Directive 2011/65/EU on the restriction of the use of certain hazardous substances in electrical and electronic equipment and an upper limit of 1000 mg/kg PBB or PBDE was set. Hexabromocyclododecane (HBCDD) was listed in the Annex XIV of REACH. In 2019 the EU issued a regulation on Persistent Organic Pollutants EU 2019/1021, which has been amended for the Brominated Flame Retardants in 2022. This amendment EU 2022/2400 of 23 November 2022 describes limits for Deca-BDE of 500 mg/kg till the end of 2025, reducing to 200 mg/kg from 2028. For HBCDD it describes a limit of 500 mg/kg till the end of 2027, further reduction still has to be adopted into legislative proposals.

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

In this interlaboratory study 71 laboratories in 21 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Total Brominated Flame Retardants in Polymers proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send two different PVC samples of approximately 3 grams each labelled #23705 and #23706 respectively.

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

For the first sample a batch of pink PVC blocks was selected which was artificially fortified with Nona- and Deca-BDE. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23705.

The batch for sample #23705 was used in a previous proficiency test on Total Brominated Flame Retardants in Polymers as sample #15152 in iis15P07. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of yellow PVC blocks was selected which was artificially fortified with Hexabromocyclododecane (HBCDD). After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23706.

The homogeneity of the subsamples was checked by the determination of HBCDD according to an in-house method on 8 stratified randomly selected subsamples.

	HBCDD in mg/kg
sample #23706-1	1136.6
sample #23706-2	1215.6
sample #23706-3	1166.0
sample #23706-4	1124.4
sample #23706-5	1184.8
sample #23706-6	1135.4
sample #23706-7	1206.0
sample #23706-8	1198.6

Table 1: homogeneity test results of subsamples #23706

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

	HBCDD in mg/kg
r (observed)	99
reference test method	IMEP-26:11
0.3 x R (reference test method)	246

Table 2: evaluation of the repeatability of subsamples #23706

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

To each of the participating laboratories two PVC samples labelled #23705 and #23706 respectively were sent on August 16, 2023.

2.5 ANALYZES

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

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

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or

corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and seven other participants did not report any test results. Not all participants were able to report all tests requested. In total 64 participants reported 159 numerical test results. Observed were 6 outlying test results, which is 3.8%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of PBB and PBDE method IEC62321-6 is considered to be the official IEC test method. The 2015 version of IEC62321 does mention precision data for PBDE and these have been used for the evaluation of Nona-BDE and Deca-BDE.

Unfortunately, no official test method exists for the determination of HBCDD. In its PTs when no (suitable) reproducibility requirement from a test method is available the target reproducibility is estimated from the Horwitz equation. Fortunately, an Interlaboratory Comparison report is available: IMEP-26, Determination of Brominated Flame Retardants in plastic (see lit. 13). Although HBCDD is not mentioned in IMEP-26 the relative target standard deviation for Brominated Flame Retardants mentioned in IMEP-26 is used for the evaluation of HBCDD in this report.

sample #23705

Nona-BDE: The group of participants had difficulty to meet the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC62321-6:15.

Deca-BDE: The group of participants met the target requirements. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IEC62321-6:15.

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

sample #23706

HBCDD: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of IMEP-26:11.

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

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(lit)
Nona-BDE	mg/kg	52	103.2	116.3	84.6
Deca-BDE	mg/kg	57	2032	1174	1409

Table 3: reproducibilities of tests on sample #23705

Component	unit	n	average	2.8 * sd	R(lit)
HBCDD	mg/kg	44	918	736	643

Table 4: reproducibility of test on sample #23706

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2023 WITH PREVIOUS PTS

	September 2023	September 2022	September 2021	September 2020	August 2019
Number of reporting laboratories	64	80	73	84	67
Number of test results	159	180	183	193	168
Number of statistical outliers	6	8	9	8	8
Percentage of statistical outliers	3.8%	4.4%	4.9%	4.1%	4.8%

Table 5: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	September 2023	September 2022	September 2021	September 2020	2019 -2009	Target*)
Nona-BDE	40%	44%	47%	45%	15-51%	25-34%
Deca-BDE	21%	21%	18%	24%	10-37%	25-34%
Deca-BB	--	--	--	--	22%	25%
HBCDD	29%	12%	25%	23%	17-49%	25%

Table 6: development of the uncertainties over the years

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

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

Sample #23705 was used in a previous PT as sample #15152 in iis15P07. The averages and the reproducibility found in both PTs for this sample are good comparable.

Component	unit	sample #23705			sample #15152		
		n	average	R(calc)	n	average	R(calc)
Nona-BDE	mg/kg	52	103.2	116.3	47	104.5	133.7
Deca-BDE	mg/kg	57	2032	1174	51	2054	1006

Table 7: comparison of sample #23705 with #15152

4.4 EVALUATION OF THE ANALYTICAL DETAILS

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

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

- A majority (94%) of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- 22% of the participants used the sample as received and 78% did further cut or further grind the samples prior to analysis.
- About 55% used less than 0.5 grams of sample intake and about 45% used a sample intake of 0.5 to 2 gram.
- To release the components from the sample about 49% of the participants used Ultrasonic and about 48% used Soxhlet.
- 88% of the participants used Toluene or a Toluene mixture as solvent to release the analytes.
- The extraction time used differs from 15 minutes to 16 hours. About 35% used an extraction time between 120 and 180 minutes, about 42% used an extraction time less than 120 minutes.
- The extraction temperature used differs from room temperature to 260 °C. About 45% used an extraction temperature between 60 °C and 70 °C, about 22% used an extraction time temperature below 60 °C.

For Deca-BDE and HBCDD the calculated reproducibility is below or close to the requirements of the target reproducibility, therefore no further analysis has been performed to investigate the effect of the analytical details.

5 DISCUSSION

The Regulation EU 2022/2400 describes limits for Deca-BDE and HBCDD of 500 mg/kg (see paragraph 1). When the results of this interlaboratory study were compared with respect to the above regulation it is noticed that all reporting laboratories, except two, would have rejected sample #23705 based on the test results of Deca-BDE.

Based on the test results of HBCDD all reporting laboratories, except three, would have rejected sample #23706.

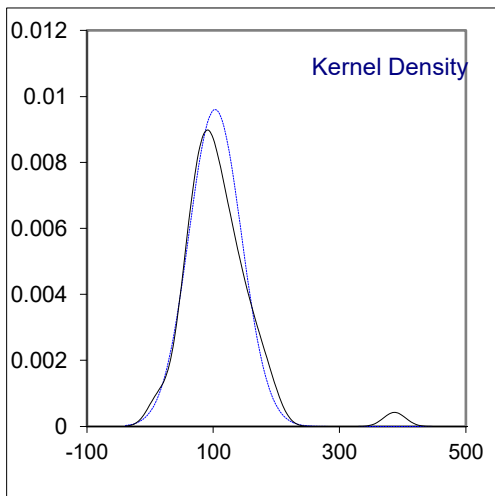
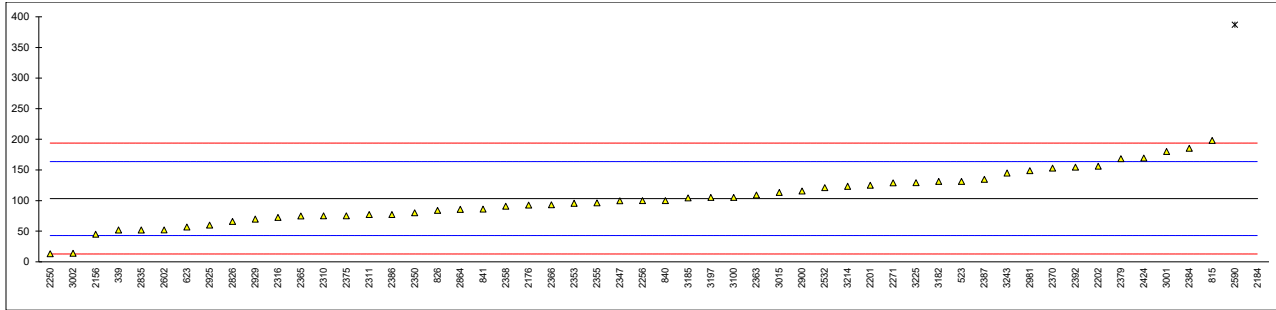
6 CONCLUSION

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

APPENDIX 1**Determination of Nonabromodiphenylether (Nona-BDE) on sample #23705; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
339	In house	51.8		-1.70	
523	IEC62321-6 - GC/MS	131.40		0.93	
623	IEC62321-6 - GC/MS	56.81		-1.54	
815	IEC62321-6 - GC/MS	198		3.14	
826	IEC62321-6 - GC/MS	84		-0.64	
840	IEC62321-6 - GC/MS	100		-0.11	
841	IEC62321-6 - GC/MS	86		-0.57	
1861		----		----	
2115		----		----	
2121		----		----	
2156	IEC62321-6 - GC/MS	45		-1.93	
2176	In house	92.58		-0.35	
2184	IEC62321-6 - GC/MS	1880	C,R(0.01)	58.81	First reported not detected
2201	IEC62321-6 - GC/MS	124.78		0.71	
2202	IEC62321-6 - GC/MS	156		1.75	
2250	IEC62321-6 - GC/MS	13.0		-2.99	
2256	In house	100		-0.11	
2265		----		----	
2271	IEC62321-6 - GC/MS	128.66		0.84	
2293		----		----	
2295		----		----	
2310	IEC62321-6 - GC/MS	75		-0.93	
2311	IEC62321-6 - GC/MS	76.92		-0.87	
2316	IEC62321-6 - GC/MS	72.40		-1.02	
2347	IEC62321-6 - GC/MS	99.77		-0.11	
2350	IEC62321-6 - GC/MS	80.1		-0.77	
2353	IEC62321-6 - GC/MS	95.54		-0.25	
2355	QCT944	96.1		-0.24	
2358	In house	90.54		-0.42	
2363	In house	109		0.19	
2365	IEC62321-6 - GC/MS	74.7		-0.94	
2366	IEC62321-6 - GC/MS	93		-0.34	
2370	IEC62321-6 - GC/MS	153		1.65	
2375	IEC62321-6 - GC/MS	75		-0.93	
2379	IEC62321-6 - GC/MS	168.3304		2.16	
2384	IEC62321-6 - GC/MS	185.23	C	2.71	First reported as test result for Nona-BB
2386	IEC62321-6 - GC/MS	77		-0.87	
2387	IEC62321-6 - GC/MS	134.52		1.04	
2392	IEC62321-6 - GC/MS	154.48		1.70	
2424	IEC62321-6 - GC/MS	169.4		2.19	
2426		----		----	
2469	IEC62321-6 - GC/MS	<500		----	
2532	IEC62321-6 - GC/MS	121		0.59	
2590		387.088	C,R(0.01)	9.40	First reported as test result for #23706 as Nona-BDE
2602	In house	52.15		-1.69	
2649		----		----	
2674		----		----	
2826	IEC62321-6 - GC/MS	66.0340		-1.23	
2835	IEC62321-6 - GC/MS	51.859	C	-1.70	First reported 298.4474
2846		----		----	
2864	IEC62321-6 - GC/MS	85.46		-0.59	
2900	IEC62321-6 - GC/MS	115.546		0.41	
2925	IEC62321-6 - GC/MS	59.6	C	-1.44	First reported as test result for Nona-BB
2929	In house	69.63		-1.11	
2981	IEC62321-6 - GC/MS	148.5		1.50	
3001	ISO17881-1	180		2.54	
3002	IEC62321-6 - GC/MS	13.85		-2.96	
3015	IEC62321-6 - GC/MS	113		0.32	
3027		----	W	----	Test result withdrawn, reported <0.005
3100	IEC62321-6 - GC/MS	105.06		0.06	
3163		----		----	
3172		----		----	
3182	IEC62321-6 - GC/MS	131.28		0.93	
3185	IEC62321-6 - GC/MS	104.30		0.04	
3197	In house	105		0.06	
3210		----		----	
3214	IEC62321-6 - GC/MS	123.1		0.66	
3225	IEC62321-6 - GC/MS	129.12		0.86	
3237		----		----	
3239	IEC62321-6 - GC/MS	<50		----	
3243	IEC62321-6 - GC/MS	145		1.38	

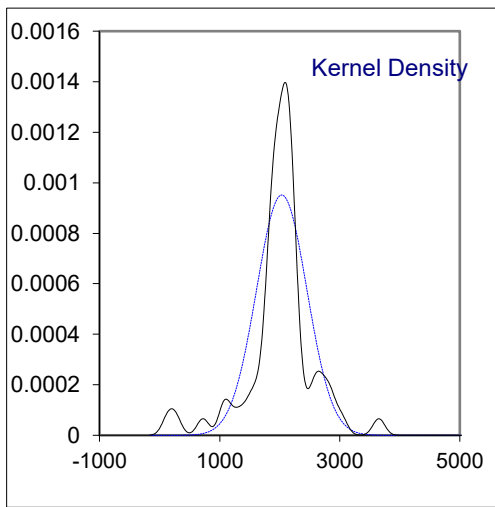
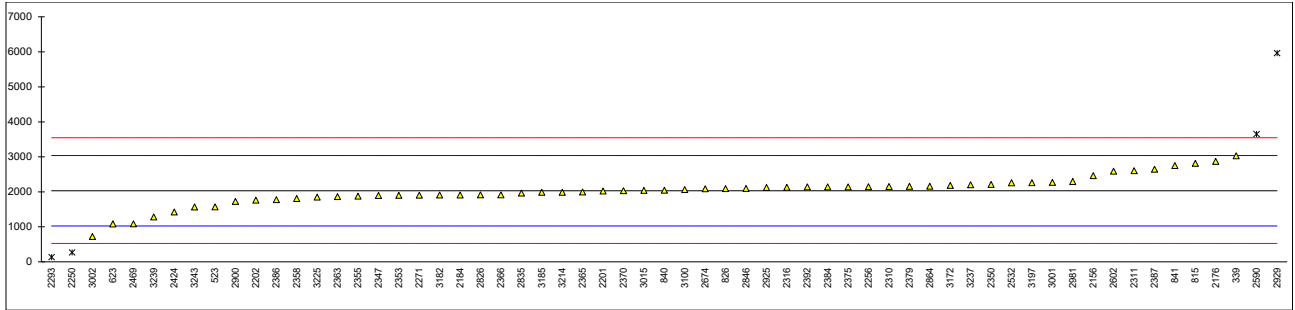
normality	OK	
n	52	
outliers	2	
mean (n)	103.222	
st.dev. (n)	41.5391	RSD = 40%
R(calc.)	116.309	
st.dev.(IEC62321-6:15)	30.2097	
R(IEC62321-6:15)	84.587	



Determination of Decabromodiphenylether (Deca-BDE) on sample #23705; results in mg/kg

lab	method	Value	mark	z(targ)	remarks
339	In house	3030		1.98	
523	IEC62321-6 - GC/MS	1568.35		-0.92	
623	IEC62321-6 - GC/MS	1080.37		-1.89	
815	IEC62321-6 - GC/MS	2810		1.55	
826	IEC62321-6 - GC/MS	2090		0.12	
840	IEC62321-6 - GC/MS	2044		0.02	
841	IEC62321-6 - GC/MS	2749		1.43	
1861		----		----	
2115		----		----	
2121		----		----	
2156	IEC62321-6 - GC/MS	2460		0.85	
2176	In house	2867.41		1.66	
2184	IEC62321-6 - GC/MS	1906		-0.25	
2201	IEC62321-6 - GC/MS	2022.54		-0.02	
2202	IEC62321-6 - GC/MS	1760		-0.54	
2250	IEC62321-6 - GC/MS	265	C,R(0.05)	-3.51	First reported 257.0
2256	In house	2146		0.23	
2265		----		----	
2271	IEC62321-6 - GC/MS	1902.17		-0.26	
2293	In house	131.85	R(0.05)	-3.77	
2295		----		----	
2310	IEC62321-6 - GC/MS	2150		0.24	
2311	IEC62321-6 - GC/MS	2601.15		1.13	
2316	IEC62321-6 - GC/MS	2126.70		0.19	
2347	IEC62321-6 - GC/MS	1894.15		-0.27	
2350	IEC62321-6 - GC/MS	2210		0.35	
2353	IEC62321-6 - GC/MS	1898.42		-0.26	
2355	QCT944	1872.6		-0.32	
2358	In house	1803.42		-0.45	
2363	In house	1862		-0.34	
2365	IEC62321-6 - GC/MS	1994.0		-0.07	
2366	IEC62321-6 - GC/MS	1910		-0.24	
2370	IEC62321-6 - GC/MS	2030		0.00	
2375	IEC62321-6 - GC/MS	2142		0.22	
2379	IEC62321-6 - GC/MS	2157.9457		0.25	
2384	IEC62321-6 - GC/MS	2141.68	C	0.22	First reported as test result for Deca-BB
2386	IEC62321-6 - GC/MS	1773		-0.51	
2387	IEC62321-6 - GC/MS	2646.03		1.22	
2392	IEC62321-6 - GC/MS	2141.18		0.22	
2424	IEC62321-6 - GC/MS	1419.6		-1.22	
2426		----		----	
2469	IEC62321-6 - GC/MS	1086		-1.88	
2532	IEC62321-6 - GC/MS	2256		0.45	
2590		3649.61	C,R(0.05)	3.21	First reported as test result for #23706 as Deca-BDE
2602	In house	2590		1.11	
2649		----		----	
2674	IEC62321-6 - GC/MS	2086		0.11	
2826	IEC62321-6 - GC/MS	1909.8107		-0.24	
2835	IEC62321-6 - GC/MS	1962.26	C	-0.14	First reported 4135.2206
2846	IEC62321-6 - GC/MS	2096.68		0.13	
2864	IEC62321-6 - GC/MS	2161.64		0.26	
2900	IEC62321-6 - GC/MS	1722.899		-0.61	
2925	IEC62321-6 - GC/MS	2125.7	C	0.19	First reported as test result for Deca-BB
2929	In house	5963	C,R(0.01)	7.81	First reported 13816.17
2981	IEC62321-6 - GC/MS	2292		0.52	
3001	ISO17881-1	2270		0.47	
3002	IEC62321-6 - GC/MS	719.90		-2.61	
3015	IEC62321-6 - GC/MS	2040		0.02	
3027		----	W	----	Test result withdrawn, reported <0.005
3100	IEC62321-6 - GC/MS	2063.59		0.06	
3163		----		----	
3172	IEC62321-6 - GC/MS	2182.4		0.30	
3182	IEC62321-6 - GC/MS	1905.71		-0.25	
3185	IEC62321-6 - GC/MS	1986.02		-0.09	
3197	In house	2263		0.46	
3210		----		----	
3214	IEC62321-6 - GC/MS	1987.2		-0.09	
3225	IEC62321-6 - GC/MS	1847.43		-0.37	
3237	IEC62321-6 - GC/MS	2201		0.34	
3239	IEC62321-6 - GC/MS	1277.38		-1.50	
3243	IEC62321-6 - GC/MS	1565		-0.93	

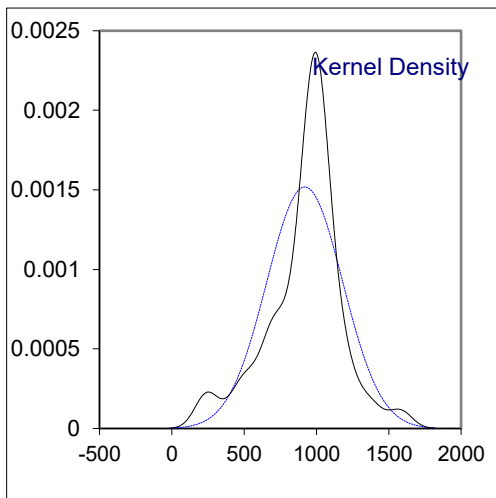
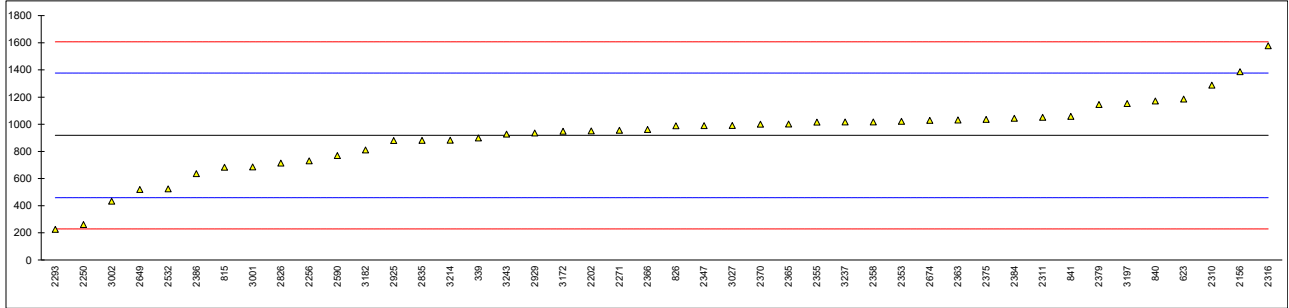
normality	suspect	
n	57	
outliers	4	
mean (n)	2031.708	
st.dev. (n)	419.3374	RSD=21%
R(calc.)	1174.145	
st.dev.(IEC62321-6:15)	503.3085	
R(IEC62321-6:15)	1409.264	



Determination of Hexabromocyclododecane (HBCDD) on sample #23706; results in mg/kg

lab	method	value	mark	z(tart)	Remarks
339	In house	898		-0.09	
523		----		----	
623	In house	1185.13		1.16	
815		683		-1.02	
826	IEC62321-9 - GC/MS	989		0.31	
840	IEC62321-6 - GC/MS	1172		1.11	
841	IEC62321-6 - GC/MS	1058		0.61	
1861		----		----	
2115		----		----	
2121		----		----	
2156	IEC62321-6 - GC/MS	1387		2.04	
2176		----		----	
2184		----		----	
2201		----		----	
2202	IEC62321-6 - GC/MS	950		0.14	
2250	EN62321-9	261	C	-2.86	First reported 240.2
2256	In house	730		-0.82	
2265		----		----	
2271	IEC62321-6 - GC/MS	955.58		0.16	
2293	In house	226.17		-3.01	
2295		----		----	
2310	IEC62321-6 - GC/MS	1288		1.61	
2311	IEC62321-6 - GC/MS	1051.09	C	0.58	First reported 1481.48
2316	IEC62321-6 - GC/MS	1577.93		2.87	
2347	IEC62321-6 - GC/MS	990.06		0.31	
2350		----		----	
2353	IEC62321-6 - GC/MS	1021.41		0.45	
2355	In house	1016.0		0.43	
2358	In house	1017.22		0.43	
2363	In house	1031		0.49	
2365		1000.8		0.36	
2366	In house	961		0.19	
2370	GB/T29493.1	1000		0.36	
2375	IEC62321-6 - GC/MS	1036		0.51	
2379	IEC62321-9	1145.2983		0.99	
2384	IEC62321-6 - GC/MS	1044.19		0.55	
2386		635.1		-1.23	
2387		----		----	
2392		----		----	
2424		----		----	
2426		----		----	
2469		----		----	
2532	IEC62321-6 - HPLC-PDA/UV	523		-1.72	
2590		768.586	C	-0.65	First reported as test result for #23705 as HBCDD
2602		----		----	
2649		519.2		-1.74	
2674	IEC62321-6 - GC/MS	1028		0.48	
2826	IEC62321-9 - GC/MS	713		-0.89	
2835	IEC62321-6 - GC/MS	880.259	C	-0.17	First reported 155.24
2846		----		----	
2864		----		----	
2900		----		----	
2925		880.0		-0.17	
2929	In house	934.6		0.07	
2981		----		----	
3001	ISO17881-1	685		-1.02	
3002	IEC62321-6 - GC/MS	432.53		-2.12	
3015		----		----	
3027	In house	990.45		0.31	
3100		----		----	
3163		----		----	
3172	IEC62321-6 - GC/MS	948.4		0.13	
3182	IEC62321-6 - GC/MS	809.75		-0.47	
3185		----		----	
3197	In house	1152		1.02	
3210		----		----	
3214	IEC62321-6 - GC/MS	883.0		-0.15	
3225		----		----	
3237	IEC62321-6 - GC/MS	1017		0.43	
3239		----		----	
3243	IEC62321-6 - GC/MS	927		0.04	

normality	suspect	
n	44	
outliers	0	
mean (n)	918.222	
st.dev. (n)	262.7112	RSD=29%
R(calc.)	735.591	
st.dev.(IMEP-26:11)	229.5554	
R(IMEP-26:11)	642.755	



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 #23705; results in mg/kg

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
339	<1	<2	<10	3.53	<100	<10
523	not detected	not detected	not detected	not detected	not analyzed	not detected
623	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
815	<10	<10	<10	<10	<20	----
826	----	----	----	----	----	----
840	not detected	not detected	not detected	not detected	not detected	----
841	<5	<5	<5	<5	<5	<5
1861	not detected	not detected	not detected	not determined	not analyzed	not analyzed
2115	----	----	----	----	----	----
2121	----	----	----	----	----	----
2156	<20	<20	<20	<20	<20	<20
2176	not detected	not detected	not detected	not detected	not analyzed	not analyzed
2184	not detected	not detected	not detected	not detected	----	----
2201	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2202	not detected	not detected	not detected	not detected	not detected	not detected
2250	< 5	< 5	< 5	< 5	< 5	----
2256	----	----	----	----	----	----
2265	----	----	----	----	----	----
2271	not detected	not detected	not detected	not detected	not detected	not detected
2293	----	----	----	----	----	----
2295	----	----	----	----	----	----
2310	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2316	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2347	<5	<5	<5	<5	<10	<5
2350	<5	<5	<5	<5	not analyzed	not analyzed
2353	not detected	not detected	not detected	not detected	not detected	not detected
2355	<5	<5	<5	<5	<10	----
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	<50	<50	<50	<50	<50	<50
2365	<5	<5	<5	<5	<20	<5
2366	----	----	----	----	----	----
2370	<5	<5	<5	<5	<5	<5
2375	----	----	----	----	----	----
2379	Not detected	Not detected	Not detected	Not detected	Not Analyzed	Not Analyzed
2384	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	< 25	< 25	< 50	< 25	< 50	not determined
2387	Not Detected	Not Detected	Not Detected	Not Detected	----	Not Detected
2392	not detected	not detected	not detected	not detected	not determined	not determined
2424	----	----	----	6.2	----	----
2426	----	----	----	----	----	----
2469	not detected	not detected	not detected	not detected	not analyzed	not detected
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	----
2590	----	----	----	----	----	C
2602	----	----	----	not detected	----	----
2649	----	----	----	----	----	----
2674	not detected	not detected	not detected	not detected	not detected	----
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2835	not detected	not detected	not detected	not detected	not detected	not detected
2846	----	----	----	----	----	----
2864	not detected	not detected	not detected	not detected	----	----
2900	ND	ND	ND	ND	NA	NA
2925	not detected	not detected	not detected	not detected	not detected	not applicable
2929	below det. limit	below det. limit	below det. limit	below det. limit	below det. limit	----
2981	not detected	not detected	not detected	not detected	----	----
3001	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

lab	Octa-BB	Nona-BB	Deca-BB	Octa-BDE	HBCDD	Other
3002	not determined	----	not determined	----	not determined	----
3015	<50	<50	<50	<50	----	----
3027	----	<0,005	<0,005	<0,005	<0,005	----
3100	< 100	< 100	< 100	< 100	not analyzed	not analyzed
3163	----	----	----	----	----	----
3172	< 1	< 1	< 1	< 1	< 1	----
3182	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3185	not detected[<5]	not detected[<5]	not detected[<5]	not detected[<5]	----	----
3197	<5	<5	<5	<5	<5	<5
3210	----	----	----	----	----	----
3214	< 5	< 5	< 5	< 5	< 5	----
3225	Not Detected	Not Detected	Not Detected	28.8	----	Not Detected
3237	----	----	----	----	----	----
3239	Not detected	Not detected	Not detected	Not detected	----	Not detected
3243	not detected	----	not detected	not detected	not detected	----

Lab 2384 first reported as test results for Nona-BDE and Deca-BDE respectively

Lab 2590 first reported as test result for HBCDD #23706

Lab 2925 first reported as test results for Nona-BDE and Deca-BDE respectively

Other reported Brominated Flame Retardants in sample #23706; 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
523	not detected	not detected	not detected	not detected	not detected	not detected	not detected
623	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
815	<10	<10	<10	<10	<10	<10	----
826	----	----	----	----	----	----	----
840	not detected	not detected	not detected	not detected	not detected	not detected	----
841	<5	<5	<5	<5	<5	<5	<5
1861	not detected	not detected	not detected	not detected	not detected	not detected	not analyzed
2115	----	----	----	----	----	----	----
2121	----	----	----	----	----	----	----
2156	<20	<20	<20	<20	<20	<20	<20
2176	not detected	not detected	not detected	not detected	not detected	not detected	not analyzed
2184	not detected	not detected	not detected	not detected	not detected	not detected	----
2201	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2202	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	< 5	< 5	< 5	< 5	< 5	< 5	----
2256	----	----	----	----	----	49.2	----
2265	----	----	----	----	----	----	----
2271	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2293	----	----	----	----	----	----	----
2295	----	----	----	----	----	----	----
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	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2347	<5	<5	<5	<5	<5	<5	<5
2350	<5	<5	<5	<5	<5	<5	not analyzed
2353	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2355	<5	<5	<5	<5	<5	<5	----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<50	<50	<50	<50	<50	<50	<50
2365	<5	<5	<5	<5	<5	<5	<5
2366	----	----	----	----	----	----	----
2370	<5	<5	<5	<5	<5	<5	<5
2375	----	----	----	----	----	----	----
2379	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed
2384	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	< 25	< 25	< 50	< 25	< 25	< 50	not determined
2387	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2392	not detected	not detected	not detected	not detected	not detected	not detected	not determined
2424	----	----	----	----	----	----	----
2426	----	----	----	----	----	----	----
2469	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	----
2590	----	----	----	----	----	----	----
2602	----	----	----	not detected	not detected	not detected	----
2649	----	----	----	----	----	----	----
2674	not detected	not detected	not detected	not detected	not detected	not detected	----
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2835	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2846	----	----	----	----	----	----	----
2864	not detected	not detected	not detected	not detected	not detected	41.89	----
2900	ND	ND	ND	ND	ND	ND	NA
2925	not detected	not detected	not detected	not detected	not detected	not detected	not applicable
2929	below det limit	below det limit	below det limit	below det limit	below det limit	below det limit	----
2981	not detected	not detected	not detected	not detected	not detected	not detected	----
3001	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3002	not determined	----	not determined	----	0.81	19.17	----
3015	<50	<50	<50	<50	<50	<50	----
3027	----	47.99	6.02	----	----	----	----
3100	<100	<100	<100	<100	<100	<100	not analyzed
3163	----	----	----	----	----	----	----
3172	< 1	< 1	< 1	< 1	----	< 1	----
3182	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3185	not	not	not	not	not	not	----
3197	<5	<5	<5	<5	<5	<5	<5
3210	----	----	----	----	----	----	----
3214	<5	<5	<5	<5	<5	<5	----
3225	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3237	----	----	----	----	----	----	----
3239	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3243	not detected	----	not detected	not detected	not detected	not detected	----

Lab 2590 first reported as test results for #23705

APPENDIX 3 Analytical details

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (minutes)	extraction temp (°C)
339	No	Used as received	1g	Ultrasonic	Toluene	60min	60°C
523	Yes	Further cut	0.5 g	Ultrasonic	Toluene	60 minutes	50 °C
623	Yes	Further cut	0.1 gr	Ultrasonic	Toluene	30	60
815	Yes	Further grinded	0.1g	Soxhlet	Toluene	30min	260°C
826	Yes	Further grinded	#23705 0.1 g/10 mL	Ultrasonic	#23705 Toluene	90 min	60 °C
840	Yes	Further cut	#23706 0.5 g/20 mL	Ultrasonic	#23706 THF	60	50
841	Yes	Further cut	0.5 gams	Ultrasonic	toluene	60 minutes	50 C
1861	Yes	Used as received	0.1 grams	Soxhlet	Toluene	More than 2 hours	Appr. 110.6°C (Toluene's boiling point)
2115	---	---	---	---	---	---	---
2121	---	---	---	---	---	---	---
2156	Yes	Further cut	1 g	Soxhlet	Toluene	120 minutes	-
2176	Yes	Further cut	0.1 gram per data	Soxhlet	Toluene	Total extraction time 180 min	65 degree C
2184	Yes	Used as received	0.5 gram	Ultrasonic	Toluene	180 minutes	60°C
2201	Yes	Further cut	0.1g	Soxhlet	Toluene	240min	70 degrees
2202	Yes	Used as received	0.5 g	Stirrer	THF/toluene/hexane	12hr	Room temp.
2250	Yes	Used as received	0,1 g	Ultrasonic	THF/MeOH	60 minutes	60 °C
2256	Yes	Further cut	1.0g	Ultrasonic	Toluene	120mins	70°C
2265	---	---	---	---	---	---	---
2271	Yes	Further grinded	0.1g	Soxhlet	Toluene	120 min	boiling
2293	Yes	Used as received	0.2 grams	Ultrasonic	THF	90 minutes	70°C
2295	---	---	---	---	---	---	---
2310	Yes	Further cut	0.5	Ultrasonic	Toluene	60	60
2311	Yes	Further cut	0.2	Ultrasonic	Toluene	60	60
2316	Yes	Further grinded	0.1 gram	Ultrasonic	Toluene	30 minutes	Not Applicable
2347	Yes	Further cut	0.1g	Soxhlet	/	6h	/
2350	Yes	Further cut	0.1g	Ultrasonic	Toluene	120min	50°C
2353	Yes	Further grinded	0.1	Soxhlet	Toluene	120	Soxhlet
2355	No	Further cut	PBBPBDE:0.1g, HBCDD:0.2g	Soxhlet	toluene	PBBPBDE:4h, HBCDD: ultrasonic 2h	PBBPBDE:280°C, HBCDD:60°C
2358	Yes	Further grinded	0.1	Soxhlet	toluene	120	Soxhlet
2363	Yes	Further cut	0.1g		toluene	705:4h 706:1h	706:60°C
2365	Yes	Further grinded	0.1g	Soxhlet	Toluene	4h	<260°C
2366	---	---	---	---	---	---	---
2370	Yes	Further grinded	IEC 62321-6:0.1g, GB/T29493.1:0.15g		Toluene	IEC 62321-6: 1hr56mins, GB/T29493.1: 30mins	IEC 62321-6: 250°C, GB/T29493.1: room temp.
2375	Yes	Further cut	0,5 gram	Ultrasonic	Toluene	60 min	60 °C
2379	Yes	Further grinded	0.1 g	Soxhlet	-	-	-
2384	Yes	Further grinded	0.1g	Soxhlet	Toluene	900 minutes	reflux temp. Soxhlet
2386	Yes	Further grinded	1 g	Soxhlet	Toluol	240 min	Condition(111°C)
2387	No	Further grinded	0.1g	Soxhlet	Toluene	120 minutes	Reflux temp.
2392	Yes	Further grinded	0.1 grams	Soxhlet	Toluene	120 minutes	-
2424	Yes	Further cut	0.5	Ultrasonic	Toluene	180	60
2426	---	---	---	---	---	---	---
2469	Yes	Further grinded	0.1 +/- 0.01 g	Soxhlet	toluene	2 h	about 110°C
2532	Yes	Further cut	0.5grams	Ultrasonic	Toluene /THF	30 min+15 min double extraction,	room temp.
2590	Yes	Further cut	1g	Ultrasonic	Toluene (ISO 17881-1) Extraction with THF, evaporated, resolved in Toluene	30 min + 15 min (ISO 17881-1)	room temperature
2602	Yes	Used as received	0,1 g	Ultrasonic		60 min	40 °C
2649	---	---	---	---	---	---	---
2674	Yes	Used as received	2.0g	Ultrasonic	Toluene	3 hours	60
2826	Yes	Used as received	0.1g		Toluene, THF	60 mins	NA
2835	Yes	Further cut	PBB/PBDE-0.5g	ASE	PBB/PBDE-Toluene	15 min	150 °C
2846	Yes	Further cut	1.008 g & 1.034 g	Stirrer	Tetrahydrofuran and Methanol	60 min	45 degrees temperature
2864	Yes	Further cut	0.2 g	Ultrasonic	toluene	60 min	60 °C
2900	Yes	Further cut	0.22217	Ultrasonic	Toluene	120 min	70
2925	Yes	Used as received	0.5 grams	Ultrasonic	Toluene	180 minutes	60°C
2929	Yes	Further cut	0,1	Ultrasonic	Dichloromethane	60 min ultrasonic	60
2981	Yes	Further grinded	0.5g	Soxhlet	toluene	+12 hrs without ultrasonic	
3001	Yes	Used as received	0.5	Ultrasonic	Toluene	180min	Room temp
						45	

lab	ISO17025 accredited	sample further grinded or cut	sample intake (g)	release/extract technique	release/extract solvent	extraction time (minutes)	extraction temp (°C)
3002	Yes	Further cut	0,5	Mechanical			
3015	Yes	Further cut	0.1g	Shaking Soxhlet	THF- Isooctane 1:2 toluene	60 120min	60
3027	No	Used as received	0,5 gr	Thermal Desorption	hexane,acetone	15dk	120°C
3100	Yes	Further cut	0.2250g	Soxhlet	toluene	6hour	
3163	---	---		---			
3172	Yes	---		---			
3182	Yes	Further grinded	0.1 g	Soxhlet	Toluene	360	60
3185	Yes	Further grinded	0.2g	Soxhlet	Toluene	240minutes	Not Applicable
3197	Yes	Further cut	1 g	Ultrasonic	toluene	45 min.	22±3°C
3210	---	---		---			
3214	Yes	Further grinded	1 gram	Soxhlet	Toluene	240 mins	N/A
3225	Yes	Further cut	0.5	Soxhlet	Toluene	16 hours	Reflux temp
3237	Yes	Used as received	0,5	Soxhlet	Toluene	120 min	300
3239	Yes	Further cut	0.1 g	Soxhlet	Toluene	120 minutes	115 °C
3243	Yes		cryomilling the samples, 0,5 g	Ultrasonic	toluene	1 h	70°c

APPENDIX 4

Number of participants per country

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

APPENDIX 5

Abbreviations

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

Literature

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