

**Results of Proficiency Test
Brominated Flame retardants
September 2016**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis16P08

October 2016

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1 INTRODUCTION

Worldwide, many consumer products with plastic parts are produced which contain brominated compounds as flame retardants. These brominated compounds are exceptionally effective for fire prevention.

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 electric 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 for the sum of PBB and 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 testing scheme (laboratory-evaluating interlaboratory study) for the determination of Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyls Ethers (PBDE) was started by the Institute for Interlaboratory Studies in 2009. It was decided to continue with the interlaboratory study for the determination of Brominated Flame Retardants in the annual proficiency testing program 2016/2017. In this interlaboratory study 69 laboratories from 21 different countries have registered for participation (see appendix 3). In this report, the results of the 2016 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 2 different plastic samples which are clearly positive on a number of Brominated Flame Retardants and labelled #16605 and #16606 respectively. Participants were requested to report rounded and unrounded test results and some details of the test methods used. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkensisse, 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 April 2014 (iis-protocol, version 3.3). 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

Two different samples were selected. The first material (#16605) was a Poly Vinyl Chloride (PVC) granulate, fortified with decabromodiphenylether (Deca-BDE). The second material (#16606) was an PVC granulate that was fortified with hexabromocyclo-dodecane (HBCDD). Sample #16605 and sample #16606 were both divided over 85 subsamples of approx. 3 grams. The homogeneity of subsamples #16605 and #16606 was checked by the determination of Deca-BDE and HBCD content respectively on 8 stratified randomly selected subsamples.

	Deca-BDE #16605 in mg/kg	HBCDD #16606 in mg/kg
Sample 1	2896	1137
Sample 2	2931	1216
Sample 3	2881	1166
Sample 4	2904	1124
Sample 5	2985	1185
Sample 6	2976	1135
Sample 7	2989	1206
Sample 8	2945	1199

Table 1: test results of the homogeneity study on the subsamples #16605 and #16606

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 #16605 in mg/kg	HBCDD #16606 in mg/kg
r (observed)	118	99
reference test method	IEC62321-6:15	IMEP-26 see ref. 17
0.3 x R (ref. test method)	476	246

Table 2: evaluation of the repeatabilities of the subsamples #16605 and #16606

Both observed repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples of #16605 and #16606 was assumed.

To each of the participating laboratories one set of samples; 1x sample #16605 and 1x sample #16606, both 3 grams each was sent on August 10, 2016.

2.5 ANALYSES

The participants were requested to determine on both samples: octa-Bromodiphenyl ether, nona-Bromodiphenyl ether, deca-Bromodiphenyl ether, hexa-Bromocyclo Dodecane, octa-Bromodiphenyl, nona-Bromodiphenyl and deca-Bromodiphenyl. 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 results can't be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.

The laboratories were also requested to confirm the sample receipt on the same data entry portal together with some details of the test methods used.

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 reanalysis). 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 April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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.

The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Two participants reported results after the final reporting date and nine participants did not report any test result at all. Not all laboratories were able to report all components requested. Finally, the 60 reporting laboratories reported 160 numerical test results. Observed were 11 outlying results, which is 6.9% of all reported numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

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 and have been used for the evaluation of the two PBDEs found in sample #16605.

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 that 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.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per component and per sample.

The participants were requested to report octa-, nona-, and deca-BDE as well as octa-, nona-, and deca-BB and HBCDD. The majority of participants did only detect nona-BDE and deca-BDE for sample #16605 and HBCDD for sample #16606. This means that for #16605 only Nona- and Deca-BDE were statistically evaluated and for #16606 only HBCDD.

The participants were also requested to report the analytical details of the methods. The analytical details are listed in Appendix 2. The participants were also requested to report whether or not they are accredited to perform these tests. Of all 60 reporting laboratories, 82% are ISO/IEC 17025 accredited.

Sample #16605:

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

Deca-BDE: This determination was problematic for a number of laboratories. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the target reproducibility from test method IEC62321-6:2015.

Sample #16606:

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

4.2 PERFORMANCE EVALUATION OF 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 calculated reproducibilities and the target reproducibilities derived from the literature reference test method are compared in the next tables.

Analytes	unit	n	Average	2.8 * sd	R (target)
Nona-BDE	mg/kg	52	115.0	114.5	160.8
Deca-BDE	mg/kg	52	2313	1161	1254

Table 3: performance overview for sample #16605

Analytes	unit	n	Average	2.8 * sd	R (target)
HBCDD	mg/kg	33	1258	857	881

Table 4: performance overview for sample #16606

4.3 COMPARISON OF CURRENT PT WITH PREVIOUS PROFICIENCY TESTS

The uncertainties in the test results of the determined Brominated Flame Retardants in the iis16P08 PT are listed in the next table and are comparable with previous proficiency tests.

	2016	2015	2014	2013	2012	2011	target *)
hexa-BDE	n.e.	n.e.	n.e.	n.e.	n.e.	28%	25%
hepta-BDE	n.e.	n.e.	n.e.	n.e.	n.e.	15%	25%
octa-BDE	n.e.	n.e.	n.e.	n.e.	41%	25%	25%
nona-BDE	36%	46%	32 - 33%	33 - 38%	40 - 51%	15 - 23%	30%-25%
deca-BDE	18%	17%	24%	14 - 21%	15 - 16%	20 - 25%	30%-25%
HBCDD	24%	49%	n.e.	n.e.	n.e.	n.e.	25%

Table 5: development of uncertainties over the last years

*) 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. For the PT of 2016 the target value of 30% from test method IEC62321-6:2015 has been taken for the BDEs. The observed uncertainties in 2016 PT are in line with the target values.

5 DISCUSSION

For the determination of PBBs and PBDEs, the IEC62321-6 test method is considered to be the official EC test method. In this proficiency test 75% of the participants used a version of IEC62321 for the determination of PBBs and PBDEs.

In this PT 59 of the 60 participants answered the additional questions about the analytical details. It appeared that 81% of the participants reduced the sample size before testing and almost all participants (95%) used Toluene or a Toluene mixture as extraction solvent. Surprisingly, only 66% of the participants used a Soxhlet extraction, as described in test method IEC62321-6 and 32% of the participants used the ultrasonic technique (also mentioned in IEC62321-6). The ultrasonic technique according to IEC62321-6 is only applicable to soluble polymers. The used polymer in this PT is PVC and it is not likely to be soluble in Toluene, but in THF. Most laboratories that used the ultrasonic technique also used Toluene as extraction solvent. However, when evaluated separately, the effect of using two different extraction methods for these particular samples appears to be small (see appendix 1).

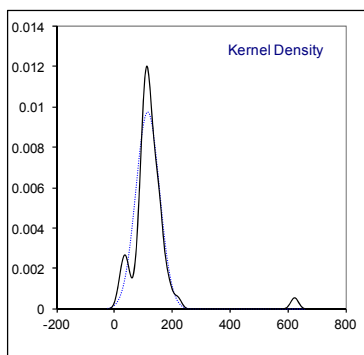
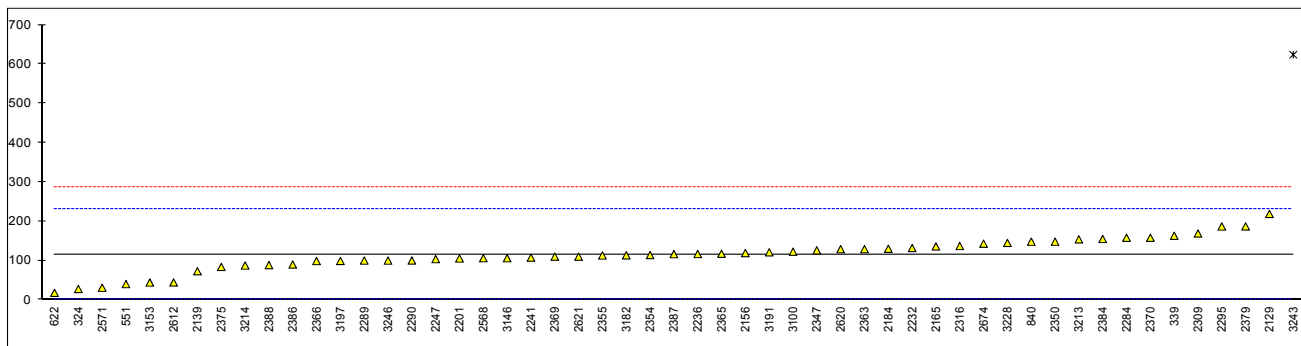
This was the second time that the performance of the HBCDD determination was evaluated in an iis PT. The variation in the current PT was in line with the variation observed in IMEP-26 study and much smaller than observed in the first iis PT. Remarkable is that most participants mentioned to use an in-house method and some participants described a (slightly) different analytical pathway compared to the determinations of PBBs and PBDEs (see appendix 2).

APPENDIX 1

Determination Nonabromodiphenyl ether CAS no. 63387-28-0 on sample #16605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
324	IEC62321	27.7		-1.52	
339	IEC62321Mod.	163		0.84	
551	IEC62321	40.065		-1.30	
622	IEC62321	17.864		-1.69	
840	IEC62321	148	C	0.58	first reported: n.d.
1099		----		----	
2115		----		----	
2129	IEC62321	218.67		1.81	
2132	In house	<200		----	
2139	In house	72.8		-0.73	
2156	IEC62321	119		0.07	
2165	IEC62321	136		0.37	
2184	IEC62321	130		0.26	
2201	IEC62321	105.3		-0.17	
2212		----		----	
2216		----		----	
2232	In house	131.87		0.29	
2236	In house	116.59	C	0.03	first reported: 302.64
2241	IEC62321	107.5		-0.13	
2247	IEC62321	103.67		-0.20	
2284	IEC62321	158.0		0.75	
2289	IEC62321	100		-0.26	
2290	IEC62321	100.2		-0.26	
2295	In house	186.8		1.25	
2309	IEC62321	169.0		0.94	
2316	IEC62321	137		0.38	
2347	IEC62321	126		0.19	
2350	IEC62321	148		0.58	
2354	IEC62321	114		-0.02	
2355	IEC62321	113.01		-0.03	
2363	IEC62321-6	129		0.24	
2365	IEC62321	117.2		0.04	
2366	IEC62321	98.7		-0.28	
2369	IEC62321	110		-0.09	
2370	IEC62321-6	158		0.75	
2375	In house	83.9		-0.54	
2379	IEC62321	186.95		1.25	
2384	IEC62321	155.01		0.70	
2386	IEC62321	90		-0.44	
2387	IEC62321	116.30		0.02	
2388	IEC62321	88.44		-0.46	
2497		----		----	
2508		----		----	
2557		----		----	
2568	IEC62321	106		-0.16	
2571	In house	30.7956		-1.47	
2612	IEC62321	44.36		-1.23	
2620	In house	128.8		0.24	
2621	IEC62321	110		-0.09	
2674	IEC62321	143		0.49	
2749		----		----	
2757		----		----	
3100	IEC62321	122.65		0.13	
3146	In house	106		-0.16	
3153	IEC62321-6	44.08		-1.23	
3163		----		----	
3172	IEC62321	n.d.		----	
3182	IEC62321	113.3		-0.03	
3191	IEC62321	121		0.10	
3197	IEC62321	99		-0.28	
3200		----		----	
3210		----		----	
3213	IEC62321	154		0.68	
3214	IEC62321	87.36		-0.48	
3228	IEC62321	145		0.52	
3237		----		----	
3243	IEC62321	624	C,R(0.01)	8.87	first reported: 379
3246	In house	100		-0.26	

normality	OK	
n	52	
outliers	1	
mean (n)	114.979	
st.dev. (n)	40.9104	RSD=35.6%
R(calc.)	114.549	
R(IEC62321-6:15)	160.757	Compare R(IMEP-26)=80.485



Effect of sample size reduction

	Overall	Sample size used as received	Sample size reduced
normality	OK	Suspect	OK
n	52	8	43
outliers	1	0	1
mean (n)	114.979	142.709	110.163
st.dev. (n)	40.9104	24.0019	41.9628
R(calc.)	114.549	67.205	117.496
R(IEC62321-6:15)	160.757	189.322	155.797

Effect of extraction techniques

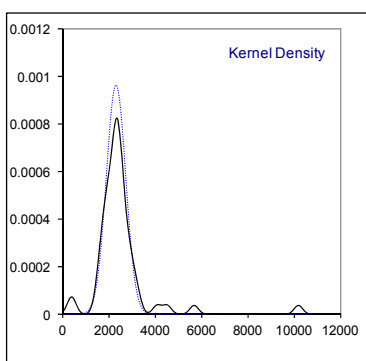
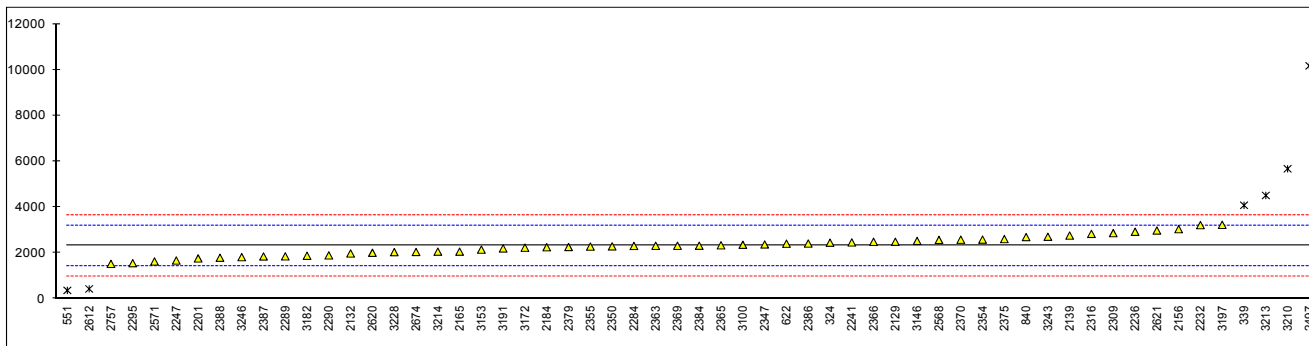
	Overall	Soxhlet	Ultrasonic
normality	OK	OK	Suspect
n	52	33	17
outliers	1	1	0
mean (n)	114.979	110.430	124.583
st.dev. (n)	40.9104	42.1351	40.3679
R(calc.)	114.549	117.978	113.030
R(IEC62321-6:15)	160.757	156.072	170.651

Determination of Decabromodiphenyl ether CAS no. 1163-19-5 on sample #16605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
324	IEC62321	2450		0.31	
339	IEC62321Mod.	4081	R(0.01)	3.95	
551	IEC62321	367.945	R(0.01)	-4.34	
622	IEC62321	2406.847		0.21	
840	IEC62321	2695	C	0.85	first reported: n.d.
1099		----		----	
2115		----		----	
2129	IEC62321	2491		0.40	
2132	In house	1980		-0.74	
2139	In house	2759.9		1.00	
2156	IEC62321	3040.3		1.62	
2165	IEC62321	2060		-0.56	
2184	IEC62321	2250		-0.14	
2201	IEC62321	1763.2		-1.23	
2212		----		----	
2216		----		----	
2232	In house	3216.06		2.02	
2236	In house	2927.17	C	1.37	first reported: 10517.96
2241	IEC62321	2460.5		0.33	
2247	IEC62321	1671.97		-1.43	
2284	IEC62321	2308.0		-0.01	
2289	IEC62321	1853		-1.03	
2290	IEC62321	1896.3		-0.93	
2295	In house	1555		-1.69	
2309	IEC62321	2868.0		1.24	
2316	IEC62321	2836		1.17	
2347	IEC62321	2374		0.14	
2350	IEC62321	2289		-0.05	
2354	IEC62321	2584		0.61	
2355	IEC62321	2280.46		-0.07	
2363	IEC62321-6	2316		0.01	
2365	IEC62321	2335.7		0.05	
2366	IEC62321	2490.5		0.40	
2369	IEC62321	2316		0.01	
2370	IEC62321-6	2580		0.60	
2375	In house	2607.4	C	0.66	first reported: 5214.8
2379	IEC62321	2260.75		-0.12	
2384	IEC62321	2317.17		0.01	
2386	IEC62321	2414		0.23	
2387	IEC62321	1848.91		-1.04	
2388	IEC62321	1792.45		-1.16	
2497		10168	C,R(0.01)	17.53	first reported: 20354.64
2508		----		----	
2557		----		----	
2568	IEC62321	2570		0.57	
2571	In house	1629.1087		-1.53	
2612	IEC62321	433	R(0.01)	-4.20	
2620	In house	2009.2		-0.68	
2621	IEC62321	2980		1.49	
2674	IEC62321	2045		-0.60	
2749		----		----	
2757	IEC62321	1526.37		-1.75	
3100	IEC62321	2360.27		0.11	
3146	In house	2526		0.48	
3153	IEC62321-6	2147.78		-0.37	
3163		----		----	
3172	IEC62321	2231		-0.18	
3182	IEC62321	1878.4		-0.97	
3191	IEC62321	2200		-0.25	
3197	IEC62321	3231		2.05	
3200		----		----	
3210	IEC62321	5673.46	R(0.01)	7.50	
3213	IEC62321	4511	R(0.01)	4.91	
3214	IEC62321	2058.86		-0.57	
3228	IEC62321	2040		-0.61	
3237		----		----	
3243	IEC62321	2708		0.88	
3246	In house	1820		-1.10	

normality OK
 n 52
 outliers 6
 mean (n) 2312.607
 st.dev. (n) 414.8144
 R(calc.) 1161.480
 R(IEC62321-6:15) 1254.426

RSD=17.9%
 Compare R(IMEP-26)=1618.825



Effect of sample size reduction

	Overall	Sample size used as received	Sample size reduced
normality	OK	Not OK	OK
n	52	7	44
outliers	6	3	3
mean (n)	2312.607	2241.723	2333.345
st.dev. (n)	414.8144	518.6872	401.5665
R(calc.)	1161.480	1452.324	1124.386
R(IEC62321-6:15)	1254.426	1216.701	1265.462

Effect of extraction techniques

	Overall	Soxhlet	Ultrasonic
normality	OK	OK	OK
n	52	35	15
outliers	6	3	3
mean (n)	2312.607	2308.196	2309.684
st.dev. (n)	414.8144	426.0180	382.9498
R(calc.)	1161.480	1192.850	1072.260
R(IEC62321-6:15)	1254.426	1252.078	1252.870

Determination of Octabromodiphenyl ether CAS no. 32536-52-0 and Hexabromocyclododecane CAS no. 3194-55-6 on sample #16605; results in mg/kg

lab	method	Octa-BDE	mark	method	HBCDD	mark
110		----			----	
324	IEC62321	<10			----	
339	IEC62321Mod.	2.80		IEC62321Mod.	<100	
551	IEC62321	ND			NA	C, fr n.d.
622	IEC62321	< 10			----	
840	IEC62321	ND		In house	ND	
1099		----			----	
2115		----			----	
2129		----			----	
2132	In house	<200		In house	<200	
2139		----			----	
2156	IEC62321	5			----	
2165	IEC62321	ND			NA	
2184	IEC62321	not detected			----	
2201	IEC62321	ND		In house	ND	
2212		----			----	
2216		----			----	
2232		----			----	
2236		----			----	
2241	IEC62321	<10		In house	<10	
2247	IEC62321	nd		In house	nd	
2284		----			----	
2289		----			----	
2290	IEC62321	<20		In house	<20	
2295	In house	35.4	C, fr. 95.2		----	
2309	IEC62321	ND[DL-50mg/kg]			----	
2316	IEC62321	ND			----	
2347	IEC62321	0		In house	0	
2350	IEC62321	<5		In house	<5	
2354	IEC62321	<5			----	
2355	IEC62321	<5			<10	
2363	IEC62321	<5		In house	<10	
2365	IEC62321	<5		IEC62321-6	<10	
2366	IEC62321	<5			<5	
2369	IEC62321	<5		In house	<10	
2370	IEC62321-6	n.d.		IEC62321-6	n.d.	
2375	In house	< 5			----	
2379	IEC62321	Not detected			----	
2384	IEC62321	<5		In house	<5	W, fr. n.d.
2386	IEC62321	< 25			< 50	
2387	IEC62321	<5			----	
2388	IEC62321	<5			----	
2497		326.66	false pos?		----	
2508		----			----	
2557		----		In house	<5	
2568	IEC62321	<10			----	
2571	In house	ND		In house	ND	
2612	IEC62321	15.83		In house	not detected	
2620	In house	<10 [per congener]			----	
2621	IEC62321	N.D.		In house	N.D.	
2674	IEC62321	n.d.		In house	N/A	
2749		----			----	
2757		----			----	
3100	IEC62321	<5		In house	<5	
3146		----			----	
3153	IEC62321-6	ND		IEC62321-6	ND	
3163		----			----	
3172	IEC62321	n.d.		In house	n.d.	
3182	IEC62321	N.D.			N.D.	
3191		----			----	
3197	IEC62321	ND			ND	
3200		----			----	
3210	IEC62321	9.714			----	
3213		----			----	
3214	IEC62321	n.d.			----	
3228	IEC62321	ND		In house	NA	
3237		----			----	
3243	IEC62321	n.d.		In house	n.d.	
3246	In house	n.d.		In house	n.d.	
n		47		n	32	
outliers		1 (false positive)		outliers	0	
mean (n)		<200		mean (n)	<200	

Determination of Octabromodiphenyl CAS no. 69278-61-1 and Nonabromodiphenyl CAS no. 69278-62-2 on sample #16605; results in mg/kg

lab	method	Octa-BDP	mark	method	Nona-BDP	mark
110		----			----	
324	IEC62321	<5.0		IEC62321	<5.0	
339	IEC62321Mod.	<1		IEC62321Mod.	<2	
551	IEC62321	ND		IEC62321	ND	
622	IEC62321	<10		IEC62321	<10	
840	IEC62321	ND		IEC62321	ND	C, fr. 148
1099		----			----	
2115		----			----	
2129		----			----	
2132	In house	<200		In house	<200	
2139		----			----	
2156	IEC62321	5		IEC62321	5	
2165	IEC62321	ND		IEC62321	ND	
2184	IEC62321	not detected		IEC62321	not detected	
2201	IEC62321	ND		IEC62321	ND	
2212		----			----	
2216		----			----	
2232		----			----	
2236		----			----	
2241	IEC62321	<10		IEC62321	<10	
2247	IEC62321	nd		IEC62321	nd	
2284		----			----	
2289		----			----	
2290	IEC62321	<20		IEC62321	<20	
2295		----			----	
2309	IEC62321	ND[DL-50mg/kg]		IEC62321	ND[DL-50mg/kg]	
2316	IEC62321	ND		IEC62321	ND	
2347	IEC62321	0		IEC62321	0	
2350		----			----	
2354	IEC62321	<5		IEC62321	<5	
2355	IEC62321	<5		IEC62321	<5	
2363	IEC62321-6	<5		IEC62321-6	<5	
2365	IEC62321	<5		IEC62321	<5	
2366	IEC62321	<5		IEC62321	<5	
2369	IEC62321	<5		IEC62321	<5	
2370	IEC62321-6	n.d.		IEC62321-6	n.d.	
2375		----			----	
2379	IEC62321	Not detected		IEC62321	Not detected	
2384	IEC62321	<5		IEC62321	<5	
2386	IEC62321	< 25		IEC62321	< 25	
2387	IEC62321	<5		IEC62321	<5	
2388	IEC62321	<5		IEC62321	<5	
2497		----			----	
2508		----			----	
2557		----			----	
2568	IEC62321	<10		IEC62321	<10	
2571	In house	ND		In house	ND	
2612	IEC62321	not detected		IEC62321	not detected	
2620	In house	<10 [per congener]		In house	<20 [per congener]	
2621	IEC62321	N.D.		IEC62321	N.D.	
2674	IEC62321	n.d.		IEC62321	n.d.	
2749		----			----	
2757		----			----	
3100	IEC62321	<5		IEC62321	<5	
3146		----			----	
3153	IEC62321-6	ND		IEC62321-6	ND	
3163		----			----	
3172	IEC62321	n.d.		IEC62321	n.d.	
3182	IEC62321	N.D.		IEC62321	N.D.	
3191		----			----	
3197	IEC62321	ND		IEC62321	ND	
3200		----			----	
3210		----			----	
3213		----			----	
3214	IEC62321	n.d.		IEC62321	n.d.	
3228	IEC62321	ND		IEC62321	ND	
3237		----			----	
3243	IEC62321	n.d.		IEC62321	n.d.	
3246	In house	n.d.		In house	n.d.	
n		43		n	43	
outliers		0		outliers	0	
mean (n)		<200		mean (n)	<200	

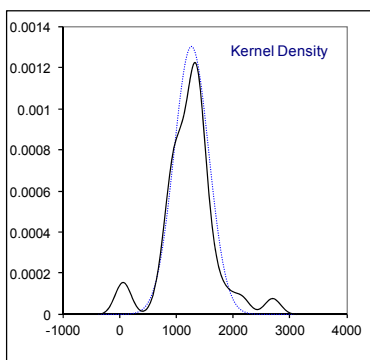
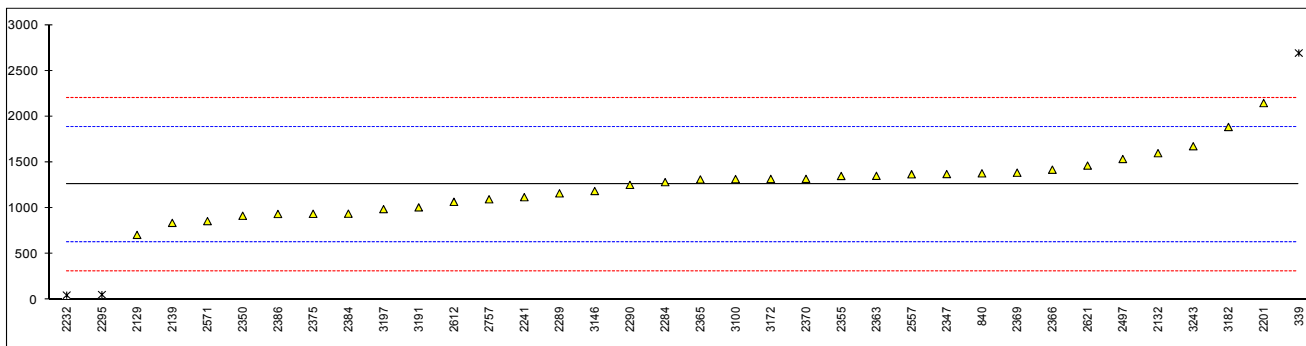
Determination of Decabromodiphenyl CAS no. 39282-95-6 and Other Brominated Flame Retardant(s) on sample #16605; results in mg/kg

lab	method	Deca-BDP	mark	method	Other BFRs	mark
110		----			----	
324	IEC62321	<5.0			----	
339	IEC62321Mod.	<10			----	
551	IEC62321	ND			ND	
622	IEC62321	<10			----	
840	IEC62321	ND	C, fr. 2695	In house	ND	
1099		----			23000	sum of PBBs/PBDEs
2115		----			----	
2129		----			----	
2132	In house	<200		In house	<200	
2139		----			----	
2156	IEC62321	5			----	
2165	IEC62321	ND			ND	
2184	IEC62321	not detected		IEC62321	not detected	
2201	IEC62321	ND		In house	ND	
2212		----			----	
2216		----			----	
2232		----			----	
2236		----			----	
2241	IEC62321	<10		In house	<10	
2247	IEC62321	nd			----	
2284		----			----	
2289		----			----	
2290	IEC62321	<20			<20	
2295		----			----	
2309	IEC62321	ND[DL-50mg/kg]			----	
2316	IEC62321	ND			----	
2347	IEC62321	0		In house	0	
2350		----			----	
2354	IEC62321	<5			----	
2355	IEC62321	<5			----	
2363	IEC62321-6	<5		IEC62321-6	<5	
2365	IEC62321	<5			----	
2366	IEC62321	<5			----	
2369	IEC62321	<5			----	
2370	IEC62321-6	n.d.		IEC62321-6	n.d.	
2375		----			----	
2379	IEC62321	Not detected			----	
2384	IEC62321	<5			----	
2386	IEC62321	< 50			----	
2387	IEC62321	<5			----	
2388	IEC62321	<5			----	
2497		----			----	
2508		----			----	
2557		----			----	
2568	IEC62321	<10			----	
2571	In house	ND		In house	ND	
2612	IEC62321	not detected		In house	not detected	
2620	In house	<25			----	
2621	IEC62321	N.D.		In house	N.D.	
2674	IEC62321	n.d.		In house	n.d.	
2749		----			----	
2757		----			----	
3100	IEC62321	<5		In house	<5	
3146		----			----	
3153	IEC62321-6	ND		IEC62321-6	ND	
3163		----			----	
3172	IEC62321	n.d.		In house	n.d.	
3182	IEC62321	N.D.			N.D.	
3191		----			----	
3197	IEC62321	ND			ND	
3200		----			----	
3210		----			----	
3213		----			----	
3214	IEC62321	n.d.			----	
3228	IEC62321	ND		In house	NA	
3237		----			----	
3243	IEC62321	n.d.		In house	n.d.	
3246	In house	n.d.		In house	n.d.	
n		43		n	23	
outliers		0		outliers	0	
mean (n)		<200		mean (n)	<200	

Determination of Hexabromocyclododecane CAS no. 3194-55-6 on sample #16606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
324		----		----	
339	IEC62321-6	2693	R(0.05)	4.56	
551		NA	C	----	first reported: n.d.
622		----		----	
840	In house	1380	C	0.39	first reported: n.d.
1099		----		----	
2115		----		----	
2129	ISO17881-2mod.	708.67		-1.75	
2132	In house	1600		1.09	
2139	In house	839.3		-1.33	
2156		----		----	
2165		NA		----	
2184		----		----	
2201	In house	2146.8		2.82	
2212		----		----	
2216		----		----	
2232	In house	50.72	C,R(0.05)	-3.84	first reported: 24.94
2236		----		----	
2241	In house	1120.4		-0.44	
2247	In house	1000-1500		----	
2284	In house	1284.0		0.08	
2289		1162		-0.31	
2290		1254.1		-0.01	
2295	In house	55.8	C,R(0.05)	-3.82	first reported: 26.5
2309		----		----	
2316		----		----	
2347	In house	1372		0.36	
2350	In house	917		-1.09	
2354		----		----	
2355		1351.12		0.29	
2363	In house	1353		0.30	
2365	IEC62321-6	1313.413		0.17	
2366	IEC62321	1419.8	C	0.51	first reported: <5
2369	In house	1387		0.41	
2370	IEC62321-6	1320		0.20	
2375	In house	938.7		-1.02	
2379		----	W	----	first reported: n.d.
2384	In house	939.90		-1.01	
2386		937		-1.02	
2387		----		----	
2388		----		----	
2497		1536.51		0.88	
2508		----		----	
2557	In house	1370		0.35	
2568		----		----	
2571	In house	859.1656		-1.27	
2612	In house	1069.51		-0.60	
2620		----		----	
2621	In house	1465		0.66	
2674	In house	N/A		----	
2749		----		----	
2757	In house	1096.89		-0.51	
3100	In house	1317.52		0.19	
3146	In house	1187		-0.23	
3153	IEC62321-6	ND		----	possibly a false negative test result?
3163		----		----	
3172	In house	1319		0.19	
3182	In house	1886.2		2.00	
3191		1009		-0.79	
3197		990		-0.85	
3200		----		----	
3210		----		----	
3213		----		----	
3214		----		----	
3228	In house	NA		----	
3237		----		----	
3243	In house	1676.3		1.33	
3246	In house	n.d.		----	possibly a false negative test result?

normality	suspect	
n	33	
outliers	3	
mean (n)	1258.373	
st.dev. (n)	306.1794	RSD=24.3%
R(calc.)	857.302	
R(IMEP-26:11)	880.861	



Effect of sample size reduction

Only 2 participants used sample as received, too few to able to draw conclusions

Effect of extraction techniques

	Overall	Soxhlet	Ultrasonic
normality	suspect	OK	not OK
n	33	20	12
outliers	3	1	2
mean (n)	1258.373	1275.985	1229.375
st.dev. (n)	306.1794	269.0779	382.3252
R(calc.)	857.302	753.418	1070.511
R(IMEP-26:11)	880.861	893.189	860.562

Determination of Octabromodiphenyl ether CAS no. 32536-52-0, Nonabromodiphenyl ether CAS no. 63387-28-0 and Decabromodiphenyl ether CAS no. 1163-19-5 on sample #16606; results in mg/kg

Lab	method	Octa-BDE	mark	Nona-BDE	mark	Deca-BDE	mark
110		----		----		----	
324	IEC62321	<10		<10		40.7	
339	IEC62321-6	<1		<2		<10	
551	IEC62321	ND		ND		ND	
622	IEC62321	<10		0.789		<10	
840	IEC62321	ND		ND		ND	
1099		----		----		----	
2115		----		----		----	
2129		----		----		----	
2132	In house	<200		<200		<200	
2139		----		----		----	
2156		----		----		----	
2165	IEC62321	ND		ND		ND	
2184	IEC62321	not detected		not detected		not detected	
2201	IEC62321	ND		ND		ND	
2212		----		----		----	
2216		----		----		----	
2232		----		----		----	
2236		----		<25		<25	
2241	IEC62321	<10		<10		<10	
2247	IEC62321	nd		nd		nd	
2284		----		----		33.0	
2289		----		----		----	
2290	IEC62321	<20		<20		<20	
2295		----		----		----	
2309	IEC62321	ND[DL-50mg/kg]		ND[DL-50mg/kg]		ND[DL-50mg/kg]	
2316	IEC62321	ND		ND		ND	
2347	IEC62321	0		0		0	
2350	IEC62321	<5		<5		<5	
2354	IEC62321	<5		<5		<5	
2355	IEC62321	<5		<5		<5	
2363	IEC62321-6	<5		<5		<5	
2365	IEC62321	<5		<5		<5	
2366	IEC62321	<5		<5		<5	
2369	IEC62321	<5		<5		<5	
2370	IEC62321-6	n.d.		n.d.		n.d.	
2375		----		----		----	
2379	IEC62321	Not detected		Not detected		Not detected	
2384	IEC62321	<5		<5		<5	
2386	IEC62321	< 25		< 25		< 50	
2387	IEC62321	<5		<5		39.29	
2388	IEC62321	<5		<5		15.76	
2497		----		----		----	
2508		----		----		----	
2557		----		----		----	
2568	IEC62321	<10		<10		<10	
2571	In house	ND		ND		ND	
2612	IEC62321	not detected		not detected		not detected	
2620	In house	<10 [per congener]		<25 [per congener]		<100	
2621	IEC62321	N.D.		N.D.		N.D.	
2674	IEC62321	n.d.		n.d.		n.d.	
2749		----		----		----	
2757		----		----		----	
3100	IEC62321	<5		<5		40.59	
3146		----		----		----	
3153	IEC62321-6	ND		ND		ND	
3163		----		----		----	
3172	IEC62321	n.d.		n.d.		n.d.	
3182	IEC62321	N.D.		N.D.		N.D.	
3191		----		----		----	
3197	IEC62321	ND		ND		ND	
3200		----		----		----	
3210		----		----		----	
3213		----		----		----	
3214	IEC62321	n.d.		n.d.		n.d.	
3228	IEC62321	ND		ND		ND	
3237		----		----		----	
3243	IEC62321	n.d.		n.d.		n.d.	
3246	In house	n.d.		n.d.		n.d.	
n		43		44		45	
outliers		0		0		0	
mean (n)		<200		<200		<200	

Determination of Octabromodiphenyl CAS no. 69278-61-1 and Nonabromodiphenyl CAS no. 69278-62-2 on sample #16606; results in mg/kg

lab	method	Octa-BDP	mark	method	Nona-BDP	mark
110		----			----	
324	IEC62321	<5.0		IEC62321	<5.0	
339	IEC62321-6	<1		IEC62321-6	<2	
551	IEC62321	ND		IEC62321	ND	
622	IEC62321	<10		IEC62321	<10	
840	IEC62321	ND		IEC62321	ND	
1099		----			----	
2115		----			----	
2129		----			----	
2132	In house	<200		In house	<200	
2139		----			----	
2156		----			----	
2165	IEC62321	ND		IEC62321	ND	
2184	IEC62321	not detected		IEC62321	not detected	
2201	IEC62321	ND		IEC62321	ND	
2212		----			----	
2216		----			----	
2232		----			----	
2236		----			----	
2241	IEC62321	<10		IEC62321	<10	
2247	In house	nd		IEC62321	nd	
2284		----			----	
2289		----			----	
2290	IEC62321	<20		IEC62321	<20	
2295		----			----	
2309	IEC62321	ND[DL-50mg/kg]		IEC62321	ND[DL-50mg/kg]	
2316	IEC62321	ND		IEC62321	ND	
2347	IEC62321	0		IEC62321	0	
2350		----			----	
2354	IEC62321	<5		IEC62321	<5	
2355	IEC62321	<5		IEC62321	<5	
2363	IEC62321-6	<5		IEC62321-6	<5	
2365	IEC62321	<5		IEC62321	<5	
2366	IEC62321	<5		IEC62321	<5	
2369	IEC62321	<5		IEC62321	<5	
2370	IEC62321-6	n.d.		IEC62321-6	n.d.	
2375		----			----	
2379	IEC62321	Not detected		IEC62321	Not detected	
2384	IEC62321	<5		IEC62321	<5	
2386	IEC62321	< 25		IEC62321	< 25	
2387	IEC62321	<5		IEC62321	<5	
2388	IEC62321	<5		IEC62321	<5	
2497		----			----	
2508		----			----	
2557		----			----	
2568	IEC62321	<10		IEC62321	<10	
2571	In house	ND		In house	ND	
2612	IEC62321	not detected		IEC62321	not detected	
2620	In house	<10 [per congener]		In house	<20 [per congener]	
2621	IEC62321	N.D.		IEC62321	N.D.	
2674	IEC62321	n.d.		IEC62321	n.d.	
2749		----			----	
2757		----			----	
3100	IEC62321	<5		IEC62321	<5	
3146		----			----	
3153	IEC62321-6	ND		IEC62321-6	ND	
3163		----			----	
3172	IEC62321	n.d.		IEC62321	n.d.	
3182	IEC62321	N.D.		IEC62321	N.D.	
3191		----			----	
3197	IEC62321	ND		IEC62321	ND	
3200		----			----	
3210		----			----	
3213		----			----	
3214	IEC62321	n.d.		IEC62321	n.d.	
3228	IEC62321	ND		IEC62321	ND	
3237		----			----	
3243	IEC62321	n.d.		IEC62321	n.d.	
3246	In house	n.d.		In house	n.d.	
n		42		n	42	
outliers		0		outliers	0	
mean (n)		<200		mean (n)	<200	

Determination of Decabromodiphenyl CAS no. 39282-95-6 and Other Brominated Flame Retardant(s) on sample #16606; results in mg/kg

lab	method	Deca-BDP	mark	method	Other BFRs	mark
110		----			----	
324	IEC62321	<5.0			----	
339	IEC62321-6	<10			----	
551	IEC62321	ND			ND	
622	IEC62321	<10			----	
840	IEC62321	ND		In house	ND	C, first reported: 1380
1099		----			1900	sum of PBBs/PBDEs
2115		----			----	
2129		----			----	
2132	In house	<200		In house	<200	
2139		----			----	
2156		----			----	
2165	IEC62321	ND			ND	
2184	IEC62321	not detected		IEC62321	not detected	
2201	IEC62321	ND		In house	ND	
2212		----			----	
2216		----			----	
2232		----			----	
2236		----			----	
2241	IEC62321	<10		In house	<10	
2247	IEC62321	nd		In house	nd	
2284		----			----	
2289		----			----	
2290	IEC62321	<20			<20	
2295		----			----	
2309	IEC62321	ND[DL-50mg/kg]			----	
2316	IEC62321	ND			----	
2347	IEC62321	0		In house	0	
2350		----			----	
2354	IEC62321	<5			----	
2355	IEC62321	<5			----	
2363	IEC62321-6	<5		IEC62321-6	<5	
2365	IEC62321	<5			----	
2366	IEC62321	<5			----	C, first reported. 1419.8
2369	IEC62321	<5			----	
2370	IEC62321-6	n.d.		IEC62321-6	n.d.	
2375		----			----	
2379	IEC62321	Not detected			----	
2384	IEC62321	<5			----	
2386	IEC62321	< 50			----	
2387	IEC62321	<5			----	
2388	IEC62321	<5			----	
2497		----			----	
2508		----			----	
2557		----			----	
2568	IEC62321	<10			----	
2571	In house	ND		In house	ND	
2612	IEC62321	not detected		In house	not detected	
2620	In house	<25			----	
2621	IEC62321	N.D.		In house	N.D.	
2674	IEC62321	n.d.		In house	n.d.	
2749		----			----	
2757		----			----	
3100	IEC62321	<5		In house	<5	
3146		----			----	
3153	IEC62321-6	ND		IEC62321-6	ND	
3163		----			----	
3172	IEC62321	n.d.		In house	n.d.	
3182	IEC62321	N.D.			N.D.	
3191		----			----	
3197	IEC62321	ND			ND	
3200		----			----	
3210		----			----	
3213		----			----	
3214	IEC62321	n.d.			----	
3228	IEC62321	ND		In house	NA	
3237		----			----	
3243	IEC62321	n.d.		In house	n.d.	
3246	In house	n.d.		In house	n.d.	
n		42		n	24	
outliers		0		outliers	0	
mean (n)		<200		mean (n)	<200	

APPENDIX 2: Analytical Details

lab	Analysis method	Sample pretreatment	Final estimated particle size	Technique release/extract the PBBs/PBDEs	Solvent (mixture) to release PBBs/PBDEs	Extraction temperature (°C) and time (minutes)	ISO/IEC17025 accredited for PBBs/PBDEs analyses
110	---	---	---	---	---	---	---
324	IEC62321	Grinded	<0.5 mm	Soxhlet	Toluene	110°C / 1200 min.	Yes
339	IEC62321-6-mod	Used as received	---	Ultrasonic	Toluene	60°C / 60 min.	No
551	IEC62321	Grinded	0.5 mm	Ultrasonic	Toluene	50°C / 30 min.	Yes
622	IEC62321	Cut	1 micro meter	Soxhlet	Toluene	120 min.	Yes
840	IEC62321	Cut	1 mm x 1 mm	Soxhlet	Toluene	120 min.	Yes
1099	In house method	Used as received	---	Soxhlet	Toluene	120 min.	No
2115	---	---	---	---	---	---	---
2129	ISO17881-1:14; HBCDD: ISO17881-2:14mod.	Grinded	not estimated, fine powder	Ultrasonic	DCM; HBCDD: Toluene	room T / 30 min.; HBCDD: 60°C / 60 min.	Yes
2132	In house method	Grinded	Powder	Soxhlet	Toluene	120 min. / 3 min cycle	No
2139	KATRI TM 2130	Grinded	< 1 mm	Ultrasonic	Toluene	60°C / 60 min.	Yes
2156	IEC62321	Cut	0.5 mm	Soxhlet	Toluene	reflux T / 300 min.(BP Toluene=110°C)	Yes
2165	IEC62321	Used as received	3 mm x 3 mm	Ultrasonic	Toluene	60°C / 180 min.	No
2184	IEC62321	Used as received	3 mm x 3 mm	Ultrasonic	Toluene	60°C / 180 min.	Yes
2201	IEC62321	Grinded	0.5 mm	Ultrasonic	Toluene	70°C / 60 min.	Yes
2212	---	---	---	---	---	---	---
2216	---	---	---	---	---	---	---
2232	Directive 2011/65/EU(RoHS)	Used as received	3 mm x 3 mm x 3 mm	Ultrasonic	Toluene; HBCDD: MeOH	70°C /120 min; HBCDD: 40°C /30 min	Yes
2236	IEC62321/C019.TP	Cut	1 mm x 1 mm	Stirrer	Chlorobenzene	refluxT/30 min(BP Chlorobenz.=131°C)	Yes
2241	IEC62321-6	Cut	1 mm x 1 mm	Ultrasonic	Toluene	60°C / 60 min.	Yes
2247	IEC62321	Grinded	< 1 mm	Soxhlet	Toluene	110°C	Yes
2284	IEC62321	Cut	1 mm x 1 mm	Ultrasonic	Toluene	55°C / 60 min.	Yes
2289	IEC62321	Grinded	< 0.5 mm	Soxhlet	Toluene	120 min.	Yes
2290	IEC62321	---	---	---	---	---	Yes
2295	In house method	Used as received	---	Soxhlet	Toluene	reflux T / 150 min.(BP Toluene=110°C)	No
2309	IEC62321	Grinded	---	Soxhlet	Toluene	70°C / 150 min.	Yes
2316	IEC62321-6:15	Cut	< 1 mm	Soxhlet	Toluene	70 - 80°C / 120 min.	Yes
2347	IEC62321	Cut	0.5 mm x 0.5 mm	Soxhlet	Toluene	360 min.	Yes
2350	IEC62321;HBCDD: in house	Grinded	< 0.5 mm	Ultrasonic	Toluene	50°C / 120 min.	Yes
2354	IEC62321-6:15	Grinded	< 0.5 mm	Soxhlet	Toluene	120 min.	Yes
2355	IEC62321;HBCDD:3550C	Cut	siever	Sox; HBCDD: Ultra	Toluene	960 min.; HBCDD: 60°C / 60 min.	Yes
2363	IEC62321-6:15	Cut	< 0.5 mm	Soxhlet (#16605) / Ultrasonic (#16606)	Toluene	240 min. (#16605) / 60 min. (#16606)	Yes
2365	IEC62321-6:15	Grinded	< 0.5 mm x 0.5 mm	Soxhlet	Toluene	480 min.	Yes

lab	Analysis method	Sample pretreatment	Final estimated particle size	Technique release/extract the PBBs/PBDEs	Solvent (mixture) to release PBBs/PBDEs	Extraction temperature (°C) and time (minutes)	ISO/IEC17025 accredited for PBBs/PBDEs analyses
2366	IEC62321	Grinded	< 0.5 mm x 0.5 mm	Soxhlet	Toluene	120 min.	Yes
2369	US EPA 3550C: 2007	Cut	1 mm x 1 mm	Soxhlet	Toluene	110°C / 960 min.	Yes
2370	IEC62321-6:15	Grinded	Sieved of 0.5 mm	Soxhlet	Toluene	120 min.	Yes
2375	In house method	Cut	2 mm x 2 mm	Ultrasonic	Toluene	50°C / 60 min.	Yes
2379	IEC62321-6:15	Grinded	---	Soxhlet	Toluene	58°C / 80-120 min. / 2-3 min cycle	Yes
2384	IEC62321-6-mod.	Cut	< 0.5 mm	Soxhlet	Toluene	reflux T / 960 - 1260 min. (BP =110°C)	Yes
2386	GC-MS	Grinded	< 1 mm	Soxhlet	Toluene	240 min.	Yes
2387	IEC62321-6-mod.	Cut	< 0.5 mm	Soxhlet	Toluene	reflux T / 960 - 1260 min. (BP =110°C)	Yes
2388	IEC62321-6-mod.	Cut	< 0.5 mm	Soxhlet	Toluene	reflux T / 960 - 1260 min. (BP =110°C)	Yes
2497	GB/T 24279-2009	Used as received	---	Ultrasonic	Hexane/Acetone	40°C / 15 min. two extractions	Yes
2508	---	---	---	---	---	---	---
2557	Solvent extraction-GCMS	Grinded	< 0.5 mm	Ultrasonic	Toluene	30°C / 90 min.	No
2568	IEC62321	Grinded	about 0.5 mm	Soxhlet	Toluene	120 min.	Yes
2571	GC/MS/MS	Grinded	50~300 um(data SEM)	Soxhlet	Toluene	110°C / 150 min.	Yes
2612	IEC62321 (GC-MS)	Cut	---	Soxhlet	Toluene	180 min.	Yes
2620	IEC62321-6-mod.	Grinded	---	Ultrasonic	Toluene	60°C / 30 min.	Yes
2621	IEC62321	Cut	< 0.5 mm	Soxhlet	Toluene / Propanol	BP mixture / 240 - 480 min.	Yes
2674	IEC62321	Used as received	3 mm x 3 mm	Ultrasonic	Toluene	60°C / 180 min.	No
2749	---	---	---	---	---	---	---
2757	EN 62321-1:2013	Cut	2 mm x 2 mm	Soxhlet	Toluene	120 min.	No
3100	IEC62321-6:15	Cut	< 0.5 mm	Soxhlet	Toluene	120 min. / 3 min cycle	Yes
3146	in house: SVHC	Used as received	---	Ultrasonic	THF/ACN 1:2	70°C / 2x30 min.	Yes
3153	IEC62321-6:15	Grinded	n.a.	Soxhlet	Toluene	reflux T / 240 min.(BP Toluene=110°C)	Yes
3163	---	---	---	---	---	---	---
3172	IEC62321	Cryomilled	Cryomilled (< 0.5 µm)	Soxhlet	Toluene	240 min.	Yes
3182	IEC62321	Grinded	0.5 mm	Soxhlet	Toluene	360 min.	Yes
3191	IEC62321-6:15	Grinded	0.5 mm	Soxhlet	Toluene	reflux T / 120 min.(BP Toluene=110°C)	Yes
3197	IEC62321	Cut	0.5 mm	Soxhlet	Toluene	80°C / 120 min.	Yes
3200	---	---	---	---	---	---	---
3210	IEC62321	Used as received	---	Soxhlet	Toluene	360 min.	No
3213	IEC62321-6:15	Grinded	0.5 mm	Soxhlet	Toluene	360 min.	Yes
3214	IEC62321	Grinded	0.5 mm	Soxhlet	Toluene	360 min.	Yes
3228	IEC62321	Used as received	3 mm x 3 mm	Ultrasonic	Toluene	60°C / 180 min.	Yes
3237	---	---	---	---	---	---	---
3243	GC-MS	Grinded	< 0.2 mm	Soxhlet	Toluene	120 min.	No
3246	In house method	Cut	> 1 mm	Ultrasonic	Toluene:Acetone (1:1)	70°C / 120 min.	No

APPENDIX 3

Number of participating laboratories per country

1 lab in BELGIUM
1 lab in BRAZIL
2 labs in FRANCE
8 labs in GERMANY
5 labs in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
3 labs in ITALY
2 labs in JAPAN
3 labs in KOREA
4 labs in MALAYSIA
16 labs in P.R. of CHINA
1 lab in POLAND
1 lab in SINGAPORE
1 lab in SWITZERLAND
4 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
4 labs in TURKEY
3 labs in U.S.A.
3 labs in VIETNAM

APPENDIX 4

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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= test result excluded from calculations
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
IMEP	= International Measurement Evaluation Programme

Literature:

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