Results of Proficiency Test Brominated Flame retardants September 2012

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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1 INTRODUCTION

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

Since the 1990s scientists have questioned the safety of the Poly Brominated Biphenyls (PBB) and Poly Brominated Diphenyls Ethers (PBDE), that bioaccumulate 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, and an upper limit of 1000 mg/kg for the sum of PBB and PBDE was set.

In February 2009, the Institute for Reference Materials and Measurements (IRMM) released the first certified reference materials (CRMs) to help analytical laboratories better detect these two classes of flame retardants. The certification study of 2007 was followed by an interlaboratory study in 2011. The test material used in this exercise was the earlier produced CRM. To avoid easy recognition by participants, the material was relabelled before use. Twenty-five laboratories from 15 countries participated in the exercise, from which 23 reported results. A proficiency testing scheme (laboratory-evaluating interlaboratory study) for the determination of PBB and PBDE was started by the Institute for Interlaboratory Studies in 2009. On request of several participants it was decided to continue the interlaboratory study for the determination of PBB and PBDE in the 2010 and 2011 iis' PT programs. In the interlaboratory study of September 2012, 79 laboratories from 21 different countries participated (See appendix 3). In this report, the results of the proficiency test are presented and discussed.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send 2 different plastic samples, of which one clearly positive on a number of brominated flame retardants and labelled #12082 and #12083 respectively. Participants were also requested to report some details of the methods used.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO/IEC 17043:2010. This ensures 100% confidentially of participant's data. Also, customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2). This protocol can be downloaded from the iis website http://www.iisnl.com.

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 (#12082) was a real world ivory coloured High Impact Poly Styrene containing 13-15% deca-BDE.

The second material (#12083) was a green coloured Poly Propylene that was artificially made positive on Deca-Brominated Diphenyl Ether (deca-BDE) by the addition of 0.28% deca-BDE. Sample #12082 was divided over 100 subsamples of approx. 3 grams and sample #12083 was divided over 100 subsamples of approx. 5 grams.

The homogeneity of subsample #12083 was checked by determination of Deca-BDE content on 8 stratified randomly selected subsamples.

	Deca-BDE #12083 in mg/kg
Sample 1	1962
Sample 2	2008
Sample 3	2142
Sample 4	2024
Sample 5	2112
Sample 6	1968
Sample 7	2234
Sample 8	1920

Table 1: test results of the homogeneity study on the subsamples #12083

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

	Deca-BDE #12083 in mg/kg
r (observed)	299
reference	see § 4 and ref. 17
0.3 x R (reference)	430

Table 2: evaluation of the observed repeatability against the requirement

The calculated repeatability was in agreement with 0.3 times the assigned target reproducibility. Therefore, homogeneity of the subsamples of #12083 was assumed.

To each of the participating laboratories one set of samples, (1* sample #12082 and 1* sample #12083) was sent on August 15, 2012.

2.5 ANALYSIS

The participants were requested to determine on both samples: octa-PBB, nona-PBB, deca-PBB, octa-BDE, nona-BDE and deca-BDE. It was explicitly requested to treat the samples as if it were routine samples and to report the analytical 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 not be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also, a letter of instructions was added to the package. The laboratories were requested to complete the report form with some details of the methods used.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report. The laboratories are represented by the code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2) For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>..." were not used in the statistical evaluation. Before further calculations, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. In the case of an anormal distribution, the statistical evaluation should be used with care. In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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 significant consequences for the evaluation of the test results.

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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 4; nos.14 and 15).

3.3 Z-SCORES

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

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.

The z-scores were calculated in accordance with:

 $z_{(target)} = (result - average of PT) / target standard deviation$

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

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 the fit-for-useness of the reported test result. See also appendix 4; ref. 16.

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 interlaboratory study, no problems were encountered with the dispatch of the samples. Eighteen participants reported results after the final reporting date and nine participants did not report any results at all. Not all laboratories were able to report all analytes requested. Finally, 70 of the 79 participants submitted analysis results. The 70 reporting laboratories reported 350 numerical test results. Observed were 33 outlying results, which is 8.6% of all reported numerial test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A not-Gaussian data distribution was found on sample #12082 for nona-BDE. Therefore, the statistical evaluation on this component should be used with due care.

For the determination of PBB and PBDE, the IEC62321 method is considered to be the official EC test method. Regretfully this method does not (yet) mention precision data.

Normally, when no (suitable) reproducibility requirements from a test method are available, target requirements are estimated from the Horwitz equation.

However, from the IMEP-26 results (ref. 17) it was clear that earlier target standard deviations of 3 – 12% were not realistic for non-expert laboratories and that a realistic PT target is 25% of the assigned value. This made the reproducibility requirements estimated by using the Horwitz equation to be unrealistically small.

Therefore, the target requirements were taken from the findings of interlaboratory study IMEP-26. In the IMEP-26 report the results of an interlaboratory study are presented on the determination of the sum of polybrominated biphenyls (PBB), the sum of polybrominated diphenylethers (PBDE) and several individual brominated diphenylethers (ref. 17).

4.1 EVALUATION PER COMPONENT AND PER SAMPLE

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

The participants were requested to report octa-, nona-, and deca-PBB and octa-, nona-, and deca-BDE. None of the participants detected octa-, nona-, and deca-PBB in any of the samples (#12082 and #12083). The participants were also requested to report the analytical details of the methods. The analytical details are listed in Appendix 2.

Two of the reported six test results (=33%) reported by laboratory 622 appeared to be statistical outliers and two other reported test results of this laboratory were very low. Because all test results of one laboratory are correlated, the remaining test results of laboratory 622 were excluded manually prior to the statistical analysis.

- <u>Octa-BDE</u>: Analytical problems were observed. For each of the samples (#12082 and #12083), the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the target reprocucibility. For sample #12082 four statistical outliers were observed and three other participants may have reported a false negative test result. For sample #12083 five statistical outliers were observed. The reported test results vary over a large range for each of the samples #12082 and #12083, respectively 1.6 1117 mg/kg and 0.2 320 mg/kg. However, the low concentration of Octa-BDE present in sample #12083 (12 mg/kg) may explain the above as this concentration will be near or below the limit of detection of most test procedures used. And therefore no z-scores were calculated.
- <u>Nona-BDE</u>: Analytical problems were observed. For sample #12082, the calculated reproducibility after rejection of four statistical outliers is not at all in agreement with the target reproducibility.
 The reported test results vary over a large range: 803 43952 mg/kg.
 For sample #12083, the calculated reproducibility after rejection of four statistical outliers is not in agreement with the target reproducibility.
 Here the reported range of the test results is 29 810 mg/kg.
- Deca-BDE:Analytical problems were observed for a number of individual laboratories.
For sample #12082, the calculated reproducibility after rejection of nine (!) statistical
outliers is in good agreement with the target reproducibility.
However, the reported test results vary over a large range: 5209 372440 mg/kg.
For sample #12083, the calculated reproducibility after rejection of seven statistical
outliers is also in good agreement with the target reproducibility.
However, the reported range of the test results is 127 12000 mg/kg.

4.2 **PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES**

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The calculated reproducibilities and the target reproducibilities derived from the literature standards are compared in the next tables.

Analytes	unit	n	Average	2.8 * sd	R (target)
Octa-BDE #12082	mg/kg	54	179	206	125
Nona-BDE #12082	mg/kg	61	5684	8192	3979
Deca-BDE #12082	mg/kg	59	117440	50316	82208

 Table 3: performance overview for sample #12082

Analytes	unit	n	Average	2.8 * sd	R (target)
Octa-BDE #12083	mg/kg	16	13	23	(8.9)
Nona-BDE #12083	mg/kg	61	214	242	150
Deca-BDE #12083	mg/kg	62	2461	1111	1723

Table 4: performance overview for sample #12083

*) For a reproducibility between brackets the evaluation should be used with care as the consensus value is near the detection limit

The observed reproducibilities for Octa-BDE and Nona-BDE are larger than the target reproducibility requirements and therefore it had to be concluded that the determination of octa and nona-BDE in the evaluated materials is rather problematic (see discussion in paragraph 6).

5 COMPARISON WITH PREVIOUS PROFICIENCY TESTS

The evolution of the relative reproducibilities for PBDE as observed in this proficiency scheme and the comparison with the findings in the previous iis' PTs is visualized in the next table.

	Hexa-BDE	Hepta-BDE	Octa-BDE	Nona-BDE	Deca-BDE
2009			60 – 102%	60 – 110%	36 – 103%
2010			97 – 108%	104 - 112%	29 – 39%
2011	78%	43%	70%	43 – 65%	57 – 69%
2012			115%	113 - 144%	43 - 45%

Table 5: comparison of the relative PBDE reproducibilities in the previous iis' PTs and in the present round

6 **DISCUSSION**

For the determination of PBB and PBDE, the IEC62321 method is considered to be the official EC test method. In this proficiency test the majority of the participants used a version of IEC62321 and almost all laboratories used GC/MS for separation, detection and quantification. Surprisingly not all laboratories that reported to have performed IEC62321 may have followed the guidelines for the sample preparation of this method. Several laboratories that reported to have performed IEC62321 answered the question "To what particle size was the sample reduced prior to analysis?" with "as received", "1mm", "2mm" or "<2mm", while the correct procedure should be grinding to <500 μ m. Cryogenic grinding is strongly recommended.

A number of participants used ultrasonification instead of Soxhlet extraction to release the components from the plastic matrix. These results were not significantly different from the other results, although several very low results are reported by these participants.

Other parts of the determination that may need attention are the possible congener degradation. IEC62321 mentions in Annex A.8 the necessary QC to check the congener degradation. Also the calibration and the subsequent calcualtions may need additional attention. One participating laboratory asked iis how the 3 nona-BDE congeners should be quantified using BDE-206 only. IEC62321 may be not clear on this point. This uncertainty may explain (partly) for the large spread (and the relatively low number of statistical outliers) in the determination of nona-BDE (with 3 congeners) and octa-BDE (with 6 congeners) in comparison with deca-BDE.

Determination of Octabromo diphenyl ether on sample #12082; results in mg/kg

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2173 IEC62321 143.5 0.79 2173 IEC62321 165.3 0.50 2174 IEC62321 164.1 0.33 2186 IEC62321 139 0.89 2201 IFA3540C 246 1.50 2202 inhouse 124.2 -1.22 2214 inhouse 186.7 0.18 2215 IEC62321 137.01 0.94 2246 IEC62321 137.01 0.94 2246 IEC62321 137.01 0.94 2246 IEC62321 1161 0.40 2280 2290 2303 2304 IEC62321 131.1 2.96 2305 IEC62321 142.0 -0.82 2306 IEC62321 144.5 -0.76 2304 IEC62321 145.6 -0.76 2370 IEC62321 145.6 -0.76 2370 IEC62321 146.6 <td>2169</td> <td>IEC62321</td> <td>335.8</td> <td></td> <td>3.51</td> <td></td>	2169	IEC62321	335.8		3.51	
2173 IEC62321 156.3 -0.50 2184 IEC62321 152.3 -0.59 2184 IEC62321 164.1 -0.33 2201 Inbuse 124.2 -1.22 2216 IEC6231 139 -0.89 2201 Inbuse 186.7 0.18 2216 IEC6231 151 -0.62 2233 IEC62321 137.01 -0.94 2246 IEC62321 137.01 -0.94 2246 IEC62321 137.01 -0.94 2246 IEC62321 1217 0.86 2271 IEC62321 1217 0.86 2284 IEC62321 1217 0.86 2284 IEC62321 141 -0.40 2286 IEC62321 311.8 2.96 239 IEC62321 314.85 -0.76 239 IEC62321 144.5 -0.76 239 IEC62321 144.5 -0.76 2370 IEC62321 174.6 -0.64 2371 IEC62321	2172	IEC62321	143.5		-0.79	
2119 IEC62321 152.3 -0.59 2146 IEC62321 154.1 -0.33 216 IEC62321 139 -0.89 2211 IPA540C 246 1.50 22221 inhouse 124.2 -1.22 2231 IEC62321 137.01 -0.62 2233 IEC62321 137.01 -0.64 2246 IEC62321 137.01 -0.40 2247 IEC62321 137.27 1.08 2240 IEC62321 132.85 3.00 2230 IEC62321 111 2.96 2303 IEC62321 143.6 -0.76 2304 IEC62321 143.6 -0.76 2354 IEC62321 143.6 -0.76 2354 IEC62321 145.5 -0.07 2375 IEC62321 145.6 -0.76 2376 IEC62321 145.6 -0.76 2376 IEC62321 145.6 -0.76 2376 IEC62321 175.6 -0.07 2376 IEC6232	2173	IEC62321	156.3		-0.50	
2144 IEC62321 194.1 -0.33 2106 IEC62321 139 -0.89 2201 Inhouse 124.2 -1.22 2216 IEC62321 150	2179	IEC62321	152.3		-0.59	
2196 IEC62321 139 -0.89 2201 IFA8540C 246 1.50 2212 in house 124.2 -1.22 2231 IEC62321 137.01 -0.62 2234 IEC62321 137.01 -0.94 2246 IEC62321 137.01 -0.62 2248 IEC62321 137.01 -0.62 2258 IEC62321 161 -0.40 2260 2303 2304 IEC62321 312.85 3.00 2305 IEC62321 131.2 2.96 2306 IEC62321 142.0 -0.62 2306 IEC62321 143.6 -0.79 2306 IEC62321 143.6 -0.79 2306 IEC62321 143.6 -0.70 2375 INH-216 105.14 -1.65 2379 IEC62321 175.6 -0.07 2376 IEC62321 276.4 -0.60 2416 IEC62321 166.1 -0.60	2184	IEC62321	164.1		-0.33	
2201 iPA3540C 246 1.50 2202 in house 124.2 1.22 2216 iEC6231 100 2237 IEC6231 151 -0.62 2243 IEC62321 137.01 -0.94 2244 IEC62321 137.01 -0.40 2246 IEC62321 137.01 -0.40 2247 IEC62321 127.7 1.08 2296 2309 IEC62321 311.8 2.96 2304 IEC62321 312.85 3.00 2305 IEC62321 142.0 -0.82 2366 IEC62321 142.0 -0.82 2367 IEC62321 143.6 -0.79 2370 IEC62321 143.6 -0.79 2370 IEC62321 134.6 -0.61 2370 IEC62321 135.4 -0.64 2381 IEC62321 136.6 -0.79 2382 IEC62321 136.4 -1.45 2370 IEC62321 136.4 -1.65 2371 IEC62321 136.4 -0.67 2486 IEC62321 128.4 -1.10 2410 <td< td=""><td>2196</td><td>IEC62321</td><td>139</td><td></td><td>-0.89</td><td></td></td<>	2196	IEC62321	139		-0.89	
2212 in house 124.2 -1.22 2214 in house 186.7 0.18 2237 ENG321 151 -0.62 2248 ECG2321 137.01 -0.94 2246 ECG2321 137.01 -0.94 2246 ECG2321 127 0.65 2271 ECG2321 127 0.64 2284 ECG2321 227 1.08 2290 2303 2304 ECG2321 312.85 3.00 2316 ECG2321 142.0 -0.62 2350 ECG2321 145. -0.79 2356 ECG2321 143.6 -0.79 2370 ECG2321 175.6 -0.07 2375 IN-216 105.14 -1.45 2370 ECG2321 175.6 -0.07 2386 ECG2321 175.6 -0.07 2376 IECG2321 155.4 -0.60 24415 ECG2321 155.4 -0.60	2201	EPA3540C	246		1.50	
2212 in house 186.7 0.18 2236 IEC62321 151 -0.62 2237 IEC62321 137.01 -0.94 2246 IEC62321 183.54 0.10 2256 IEC62321 183.54 0.10 2256 IEC62321 127 0.85 2271 IEC62321 127 1.08 2280 2303 2304 IEC62321 111 2.96 2305 IEC62321 142.0 -0.82 2304 IEC62321 145.5 -0.76 2305 IEC62321 145.6 -0.76 2306 IEC62321 145.6 -0.76 2370 IEC62321 145.6 -0.76 2370 IEC62321 133.1 0.09 2370 IEC62321 145.6 -0.76 2375 IEC62321 136.1 0.09 2370 IEC62321 120.7 -0.64 2431 IEC62321 120.6 -0.40 <td>2202</td> <td>in house</td> <td>124.2</td> <td></td> <td>-1.22</td> <td></td>	2202	in house	124.2		-1.22	
2216 EC62321 <100	2212	in house	186.7		0.18	
2237EV62321151-0.622243EC62321137.01-0.942244EC62321137.01-0.942256EC62321183.540.102254EC623212170.852274IEC623212271.0823032304IEC6232131.2.853.002316IEC62321142.0-0.822354IEC62321143.6-0.762354IEC62321143.6-0.762356IEC62321143.6-0.762356IEC62321143.6-0.762356IEC62321145.5-0.762356IEC62321145.6-0.702377IEC62321175.6-0.072378IEC62321175.6-0.072379IEC62321195.4-1.652410IEC62321195.4-1.652431IEC62321160.8-0.402443IEC62321160.8-0.402443IEC62321160.8-0.402443IEC62321116.80.882504IEC62321116.80.842504IEC62321195.567470.373100IEC62321198.90.453131IEC62321198.90.453144in house55-2.773150in house55-2.773161in house55-2.773162IEC62321342.583.66 <td>2216</td> <td>IEC62321</td> <td><100</td> <td></td> <td></td> <td></td>	2216	IEC62321	<100			
2243 IEC62321 137.01 -0.94 2246 IEC62321 137.54 0.10 2256 IEC62321 217 0.85 2271 IEC62321 227 1.08 2280 2303 IEC62321 31.285 3.00 2316 IEC62321 31.285 3.00 2316 IEC62321 31.45 -0.76 2350 IEC62321 142.0 -0.82 2354 IEC62321 143.6 -0.76 2359 IEC62321 183.1 0.09 2370 IEC62321 183.4 -1.45 2370 IEC62321 175.6 -0.07 2379 IEC62321 175.6 -0.07 2379 IEC62321 176.40 -1.45 2379 IEC62321 176.96 G(0.01) 2403 IEC62321 176.96 G(0.01) 2415 IEC62321 106.8 -0.40 2416 IEC62321 10.4 2433 </td <td>2237</td> <td>EN62321</td> <td>151</td> <td></td> <td>-0.62</td> <td></td>	2237	EN62321	151		-0.62	
2246 IEC62321 183.54 0.10 2256 IEC62321 161 -0.40 2284 IEC62321 227 1.08 2290 2290 2303 2304 IEC62321 312.85 3.00 2305 IEC62321 312.85 3.00 2306 IEC62321 142.0 -0.86 2354 IEC62321 143.6 -0.76 2359 IEC62321 143.6 -0.76 2370 IEC62321 175.6 -0.07 2375 INH-216 105.14 -1.45 2376 IEC62321 207.4 -0.60 2376 IEC62321 207.4 -0.60 2478 IEC62321 207.4 -0.60 2478 IEC62321 105.14 1.65 2360 IEC62321 106.4 2471 IEC62321 106.4 2488 IEC62321 106.4 2479 IEC62321 116.8 -0.40 2480 IEC62321 10.8 2481 IEC62321 10.8 -	2243	IEC62321	137.01		-0.94	
2256 IEC62321 217 0.85 2271 IEC62321 227 1.08 2280 2303 2304 IEC62321 312.85 3.00 2305 IEC62321 311. 2.96 2305 IEC62321 142.0 -0.82 2354 IEC62321 145. -0.76 2355 IEC62321 143.6 -0.79 2366 IEC62321 143.1 0.09 2375 INH-216 105.14 -1.45 2379 IEC62321 176.96 G(0.01) 2379 IEC62321 207.4 0.64 2403 IEC62321 152 -0.60 2415 IEC62321 152 -0.60 2415 IEC62321 160.8 -0.40 2416 IEC62321 160.8 -0.40 2418 IEC62321 100 2439 2439	2246	IEC62321	183.54		0.10	
2271 IEC62321 161 -0.40 2284 IEC62321 227 1.08 2303 2304 IEC62321 312.85 3.00 2316 IEC62321 312.85 3.00 2351 IEC62321 142.0 -0.82 2354 IEC62321 145.6 -0.76 2359 IEC62321 143.6 -0.79 2366 IEC62321 153.6 -0.07 2375 INH-216 105.14 -1.45 2376 IEC62321 175.6 -0.07 2376 IEC62321 207.4 0.64 2438 IEC62321 207.4 0.64 2438 IEC62321 152 -0.60 2415 IEC62321 160.8 -2.01 2443 INH-62321 160.8 -0.40 2448 INH-62321 116.94 -2.600 2448 INH-62321 116.94 -0.40 2448 2449	2256	IEC62321	217		0.85	
2284 IEC62321 227 1.08 2296 2303 IEC62321 312.85 3.00 2316 IEC62321 311 2.96 2350 IEC62321 142.0 -0.82 2354 IEC62321 143.6 -0.79 2366 IEC62321 143.6 -0.79 2371 IEC62321 143.1 0.09 2372 IEC62321 175.6 -0.07 2373 IEC62321 207.4 0.64 2403 IEC62321 207.4 0.64 2410 IEC62321 1.04 24110 IEC62321 1.6 2423 IEC62321 n.d. 2438 ENC2321 160.8 -0.40 2448 2475 2476 2478 IEC62321 160.8 -0.40 2489	2271	IEC62321	161		-0.40	
2290 2303 2304 IEC62321 312.85 3.00 2305 IEC62321 311 2.96 2306 IEC62321 142.0 -0.82 2305 IEC62321 143.6 -0.76 2306 IEC62321 143.6 -0.79 2306 IEC62321 143.6 -0.79 2307 IEC62321 175.6 -0.01 2375 INH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 152 -0.60 2415 IEC62321 160.8 -0.40 2415 IEC62321 160.8 -0.40 2428 INH-62321 <100	2284	IEC62321	227		1.08	
2296	2290					
2303 $$ $$ 2309IEC62321312.853.002316IEC62321142.0-0.822354IEC62321143.6-0.762359IEC62321143.6-0.792360IEC62321143.6-0.092370IEC62321175.6-0.072375INH-216105.14-1.652379IEC62321207.4-0.642403IEC62321207.4-0.642415IEC62321152-0.602416IEC62321152-0.602417IEC62321160.8-0.402418IEC62321160.8-0.4024282438ENC6321116.942500IEC62321116.54770.373100IEC62321195.567470.373100IEC62321194.043151ENC6321194.0343154in house55-2.77316631723182IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321342.583185IEC62321 <td< td=""><td>2296</td><td></td><td></td><td></td><td></td><td></td></td<>	2296					
2309IEC62321312.853.002316IEC623213112.962350IEC62321142.0-0.822354IEC62321143.6-0.792366IEC62321183.10.092370IEC62321114-1.452372IEC62321175.6-0.072375INH-216105.14-1.652376IEC62321207.40.642403IEC623212281.102410IEC623212281.102415IEC62321152-0.602415IEC62321160.8-2.0124752482INH-62321160.824932484INH-62321116.9249324942500IEC62321116.942513IEC62321116.57472504IEC62321198.5674731003151EN623211943151EN623211943151IEC623211943153IEC623211943154In house553154IEC623213163in house553163in house5531723182IEC62321342.583185IEC623213453185IEC62321342.583185IEC623213453185IEC62321345 </td <td>2303</td> <td></td> <td></td> <td></td> <td></td> <td></td>	2303					
2316 IEC62321 311 2.96 2350 IEC62321 142.0 -0.82 2354 IEC62321 143.6 -0.79 2366 IEC62321 183.1 0.09 2370 IEC62321 114 -1.45 2372 IEC62321 175.6 -0.07 2375 INH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 228 1.10 2410 IEC62321 208.4 1.0 2415 IEC62321 n.d. 2473 IEC62321 160.8 -0.40 2488 2493 2493 2494 2500 IEC62321 116.94 0.6(0.01) 2488 2500 IEC62321 116.94 0.37 2513 IEC62321 198.9 0.45 2513 IEC62321 198.9 <td>2309</td> <td>IEC62321</td> <td>312.85</td> <td></td> <td>3.00</td> <td></td>	2309	IEC62321	312.85		3.00	
2550 IEC62321 142.0 -0.82 2354 IEC62321 143.6 -0.76 2366 IEC62321 143.6 -0.79 2370 IEC62321 114 -1.45 2377 IEC62321 175.6 -0.07 2375 INH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 152 -0.60 2410 IEC62321 152 -0.60 2415 IEC62321 88.80 -2.01 2478 IEC62321 160.8 -0.40 2482 INH-62321 160.8 -0.40 2488 2493 2494 2493 2494 2493	2316	IEC62321	311		2.96	
2354 IEC62321 143.6 -0.76 2366 IEC62321 183.1 0.09 2370 IEC62321 114 -1.45 2372 IEC62321 175.6 -0.07 2375 INH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 228 1.10 2410 IEC62321 182. -0.60 2415 IEC62321 182. -0.60 2415 IEC62321 182. -0.60 2479 IEC62321 160.8 -0.40 2488 IEC62321 160.8 -0.40 2488 IEC62321 116.94 -0.60 2493 IEC62321 180.8 0.40 2488 IEC62321 180.8 0.40 2488 IEC62321 196.96 0.40 2493 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3153 IEC62321 198.9 0.45	2350	IEC62321	142.0		-0.82	
2359IEC62321143.6 -0.79 2366IEC62321183.10.092370IEC62321174.6 -0.07 2375INH-216105.14 -1.65 2379IEC62321207.40.642403IEC623212281.102410IEC62321152 -0.60 2415IEC6232188.80 -2.01 24752479IEC62321160.8 -0.40 2482INH-62321160.8 -0.40 2483EN62321160.8 -0.40 248424942500IEC62321195.56747 0.37 3100IEC62321198.9 0.45 3151IEC62321194. 0.34 3153IEC62321194. 0.34 3154in house55 -2.77 3163in house55 -2.77 31643185IEC62321342.58 3.66 3185IEC62321215 0.81	2354	IEC62321	145		-0.76	
2360IEC62321183.1 0.09 2370IEC62321114-1.452372IEC62321175.6-0.072373IEC62321796.96G(0.01)13.822386IEC62321207.40.642403IEC623212281.102410IEC62321152-0.602415IEC6232188.80-2.0124752479IEC62321160.8-0.4024882493249324942500IEC623211116.94C.G(0.01)200IEC62321195.567470.373100IEC62321198.90.453146in house<100	2359	IEC62321	143.6		-0.79	
2370 IEC62321 114 -1.45 2372 IEC62321 175.6 -0.07 2375 INH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 228 1.10 2410 IEC62321 152 -0.60 2415 IEC62321 88.80 -2.01 2475 2482 INH-62321 160.8 -0.40 2483 EN62321 160.8 -0.40 2484 2493 2483 INH-62321 <100	2366	IEC62321	183.1		0.09	
2372 IR-C62321 175.6 -0.07 2375 IRH-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 152 -0.60 2410 IEC62321 152 -0.60 2415 IEC62321 152 -0.60 2415 IEC62321 $n.d.$ $$ 2475 $$ $$ 2478 INH-62321 160.8 -0.40 2488 $$ $$ 2493 $$ $$ 2493 $$ $$ 2493 $$ $$ 2493 $$ $$ 2493 $$ $$ 2494 $$ $$ 2504 IEC62321 1116.94 $C,G(0.01)$ 20.98 100 IEC62321 198.9 0.45 3146 in house <100 $$ 3153 IEC62321 194 0.34	2370	IEC62321	114		-1.45	
2373 INT-216 105.14 -1.65 2379 IEC62321 207.4 0.64 2403 IEC62321 228 1.10 2410 IEC62321 152 -0.60 2415 IEC62321 n.d. 2438 EN62321 88.80 -2.01 2475 2482 INH-62321 160.8 -0.40 2483 2484 2493 2494 2493 2494 2493 2494 2500 IEC62321 218.5 0.88 2504 IEC62321 196.56747 0.37 3100 IEC62321 198.9 0.45 3145 IEC62321 194 0.34 3153 IEC62321 194 0.34 3154 <	2372	IEC62321	175.6		-0.07	
2379 IEC62321 796.96 G(0.01) 13.82 2386 IEC62321 207.4 0.64 2410 IEC62321 152 -0.60 2415 IEC62321 152 -0.60 2415 IEC62321 88.80 -2.01 2475 2479 IEC62321 160.8 -0.40 2488 2493 2493 2494 2493 2494 2504 IEC62321 116.94 C,G(0.01) 20.98 2513 IEC62321 198.9 0.45 3146 in house <100	2375	INH-216	105.14	0(0.04)	-1.65	
2360 IEC62321 207.4 0.64 2403 IEC62321 152 -0.60 2415 IEC62321 $n.d.$ $$ 2438 EN62321 88.80 -2.01 2475 $$ $$ 2476 100 $$ 2477 IEC62321 160.8 -0.40 2482 INH-62321 <100 $$ 2493 $$ $$ 2494 $$ $$ 2504 IEC62321 218 0.88 2504 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100 $$ 3151 IEC62321 198.9 0.45 3153 IEC62321 194 0.34 3154 $$ $$ $$ $$ $$ 3163 in house 55 -2.77 3164 $$ $$ $$ $$ </td <td>2379</td> <td>IEC62321</td> <td>796.96</td> <td>G(0.01)</td> <td>13.82</td> <td></td>	2379	IEC62321	796.96	G(0.01)	13.82	
2410 IEC62321 152 -0.60 2415 IEC62321 $n.d.$ $$ 2438 EN62321 88.80 -2.01 2475 $$ $$ 2479 IEC62321 160.8 -0.40 2482 INH-62321 <100 $$ 2488 $$ $$ 2493 $$ $$ 2494 $$ $$ 2500 IEC62321 218 0.88 2504 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100 $$ 3151 EN62321 90 -1.99 3153 IEC62321 194 0.34 3154 $$ $$ 3163 in house 55 -2.77 3166 $$ $$ 3172 $$ $$ 3185 IEC62321 342.58 3.66	2386	IEC62321	207.4		0.64	
2410 IEC62321 152 -0.60 2415 IEC62321 $n.d.$ $$ 2475 $$ $$ 2479 IEC62321 160.8 -0.40 2488 $$ $$ 2493 $$ $$ 2494 $$ $$ 2494 $$ $$ 2500 IEC62321 218 0.88 2504 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2403	IEC62321	228		1.10	
2413 IEU02321 11.0. 2413 EN62321 88.80 -2.01 2475 2479 IEC62321 160.8 -0.40 2482 INH-62321 <100	2410		102 nd		-0.60	
2435 EN02321 60.00 -2.01 2475 2479 IEC62321 160.8 -0.40 2482 INH-62321 <100 2488 2493 2494 2500 IEC62321 218 0.88 2504 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in bouse <100	2410	IEC02321	n.u.		2.01	
2473 IEC62321 160.8 -0.40 2482 INH-62321 <100	2430 2475	EIN02321	00.00		-2.01	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	2475	15060004	160.9		0.40	
2482 INN-62321 <100	2479		100.0		-0.40	
2493 2494 2500 IEC62321 218 0.88 2504 IEC62321 1116.94 C,G(0.01) 20.98 first reported: 1136.62 2513 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2482	INH-62321	<100			
2493 2494 2500 IEC62321 218 0.88 2504 IEC62321 1116.94 C,G(0.01) 20.98 first reported: 1136.62 2513 IEC62321 195.56747 0.37 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2400					
2494 $$ 2500 IEC62321 218 0.88 2504 IEC62321 1116.94 $C,G(0.01)$ 20.98 first reported: 1136.62 2513 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100 $$ 3151 EN62321 90 -1.99 3153 IEC62321 194 0.34 3154 $$ $$ 3163 in house 55 -2.77 3166 $$ $$ 3172 $$ $$ 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	2493					
2500 IEC62321 216 0.86 2504 IEC62321 1116.94 C,G(0.01) 20.98 first reported: 1136.62 2513 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2494	15060004			0.00	
2504 IEC62321 1110.94 C,G(0.01) 20.98 Instriptioned. 1136.62 2513 IEC62321 195.56747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2500		210	C C (0 01)	0.00	first reported, 1120 02
2513 IEC62321 195.36747 0.37 3100 IEC62321 198.9 0.45 3146 in house <100	2504		1110.94	C,G(0.01)	20.96	list reported: 1136.62
3100 IEC02021 190.5 0.45 3146 in house <100	2013	IEC62221	195.56/4/		0.37	
3150 Infloade C100 3151 EN62321 90 -1.99 3153 IEC62321 194 0.34 3154 3163 in house 55 -2.77 3166 3172 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3100		190.9 ~100		0.45	
3151 EC62321 194 0.34 3153 IEC62321 194 0.34 3154 3163 in house 55 -2.77 3166 3172 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3140	EN62221	< 100 00		.1.00	
3153 194 0.34 3154 3163 in house 55 -2.77 3166 3172 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3151		90 104		-1.99	
3163 in house 55 -2.77 3166 3172 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3153	1002321	194		0.34	
3166 3172 3182 IEC62321 342.58 3185 IEC62321 215	3104	in house	55			
3172 3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3166	mnouse			-2.11	
3182 IEC62321 342.58 3.66 3185 IEC62321 215 0.81	3172					
3185 IEC62321 215 0.81	3182	IEC62321	342 58		3 66	
	3185	IEC62321	215		0.00	

3190	IEC62321	<5		<-3.89	false negative?
3191	IEC62321	343		3.67	
3197	IEC62321	132.9		-1.03	
3213	IEC62321	69.0	С	-2.46	first reported: 43.3
3218	IEC62321	252		1.64	
3228	IEC62321	204		0.56	
3240	IEC62321	176		-0.06	
3242	in house	41.60		-3.07	
3243	in house	260		1.81	
	normality	ОК			
	n	54			
	outliers	4			
	mean (n)	178.85			
	st.dev. (n)	73.586			
	R(calc.)	206.04			
	R(target)	125.20			Compare Horwitz: 89.91





Determination of Nonabromo diphenyl ether on sample #12082; results in mg/kg

lah	method	value	mark	z(tara)	remarks
110		12209.25	IIIai K	2(laig)	Telliarks
324	INT-210	15506.25		-0.77	
324	in house	2300		-0.77	
551	IFC62321	2419.3		-2.30	
605	IEC62321	4212.88		-1.04	
607	IEC62321	4255.01		-1.01	
622	IEC62321	803.4	excl.	-3.43	see § 4.1
1132	IEC62321	2048.17		-2.56	
1213					
1370	IEC62321	8645.3		2.08	
2115					
2129	IEC62321Mod.	6013	0	0.23	first sea attack (200-40
2132	EIN62321	2517.99	C	-2.23	first reported: 629.49
2137	IEC62321	10036		3.06	
2156	IEC62321	1662		-2.83	
2165	IEC62321	5100		-0.41	
2169	IEC62321	16540.5	G(0.05)	7.64	
2172	IEC62321	7403		1.21	
2173	IEC62321	4239		-1.02	
2179	IEC62321	7053.6		0.96	
2184	IEC62321	4932.2		-0.53	
2196	IEC62321	3350		-1.64	
2201	EPA3540C	3609		-1.40	
2202	in house	2973.1		-1.91	
2212	IFC62321	5639		-0.03	
2237	EN62321	3770		-1.35	
2243	IEC62321	7733.47		1.44	
2246	IEC62321	5956.44		0.19	
2256	IEC62321	10471		3.37	
2271	IEC62321	10497		3.39	
2284	IEC62321	5743		0.04	
2290					
2303					
2309	IEC62321	3392.07		-1.61	
2316	IEC62321	4093		-1.12	
2350	IEC62321	7120.3		1.01	
2354	IEC62321	4430		-0.88	
2359	IEC62321	4514.9		-0.82	
2300	IEC02321	4477.5		-0.65	
2372	IEC62321	1729		-2.78	
2375	INH-216	12018.49	С	4.46	first reported: 15177.87
2379	IEC62321	43951.8	G(0.01)	26.93	•
2386	IEC62321	8581		2.04	
2403	IEC62321	10407		3.32	
2410	IEC62321	6360		0.48	
2415	EN62321	4960.06		-0.51	
2475	LINDEDEI				
2479	IEC62321	4139		-1.09	
2482	INH-62321	4520		-0.82	
2488					
2493					
2494	15000004				
2500	IEC02321	8206 17	C	3.14 1.78	first reported: 12/75 12
2513	IEC62321	12669 7086	0	4 92	
3100	IEC62321	6981.4		0.91	
3146	in house	4069		-1.14	
3151	EN62321	3548		-1.50	
3153	IEC62321	7370		1.19	
3154	in house				
3163	in nouse	1270		-3.11 2.27	
3172	III HOUSE			-3.21	
3182	IEC62321	7315.51		1.15	
3185	IEC62321	5305		-0.27	
3190	IEC62321	3360.7		-1.63	
3191	IEC62321	20034	G(0.01)	10.10	
3197	IEC62321	28851.2	C,G(0.01)	16.30	tirst reported: 18851.2

3213 3218 3228 3240 3242	IEC62321 IEC62321 IEC62321 IEC62321	6429 8119 5200 3520 	С	0.52 1.71 -0.34 -1.52	first reported: 17750.0, also reported: 21533.4
3243	in house normality	2584 not OK		-2.18	
	n outliers mean (n) st.dev. (n) R(calc.) R(target)	4 5683.71 2925.615 8191.72 3978.60	+1 excl.		Compare Horwitz: 1200.44





Determination of Decabromo diphenyl ether on sample #12082; results in mg/kg

lah	method	value	mark	z(targ)	remarks
110	INH-216	97268 /6	mark	-0 60	Iomana
324	IEC62321	139650		0.76	
339	in house	102000		-0.53	
551	IEC62321	5209.1	C,DG(0.01)	-3.82	first reported: 10929.4
605	IEC62321	129558.45		0.41	
607	IEC62321	128080.23	0(0.04)	0.36	
622	IEC62321	372439.7	G(0.01)	8.69	
1213	IEC02321	140087.8		-1.05	
1370	IEC62321	98149.6		-0.66	
2115					
2129	IEC62321Mod.	98020	С	-0.66	first reported: 13559
2132	EN62321	59952.04	C,DG(0.05)	-1.96	first reported: 14988.01
2137	IEC62321	120237.9		0.10	
2139	IEC62321	120584	C	0.31	first reported: 187004
2165	IEC62321	110000	0	-0.25	
2169	IEC62321	127547.6		0.34	
2172	IEC62321	123300		0.20	
2173	IEC62321	107600		-0.34	
2179	IEC62321	147151.7		1.01	
2104	IEC62321	90002.3		-0.64	
2201	EPA3540C	136512		0.65	
2202	in house	140237.0		0.78	
2212	in house	115839.0		-0.05	
2216	IEC62321	143141		0.88	
2237	EN62321	117000		-0.01	
2243	IEC62321	10073.20		-0.25	
2256	IEC62321	118916		0.05	
2271	IEC62321	102677		-0.50	
2284	IEC62321	119509		0.07	
2290					
2296					
2303	IEC62321	108452 88		-0.31	
2316	IEC62321	108621		-0.30	
2350	IEC62321	128599		0.38	
2354	IEC62321	115000		-0.08	
2359	IEC62321	108910.9		-0.29	
2366	IEC62321	110851.5		-0.22	
2370	IEC62321	129000		0.39	
2375	INH-216	78582.43		-1.32	
2379	IEC62321	90899.9		-0.90	
2386	IEC62321	115900		-0.05	
2403	IEC62321	120773		0.11	
2410	IEC62321	138000		0.70	
2438	EN62321	98852	С	-0.63	first reported: 234704
2475					•
2479	IEC62321	109600		-0.27	
2482	INH-62321	125156		0.26	
2488					
2493 2494					
2500	IEC62321	115752		-0.06	
2504	IEC62321	347653.64	C,G(0.01)	7.84	first reported: 915597.84
2513	IEC62321	55896.2988	DG(0.05)	-2.10	
3100	IEC62321	106455.3		-0.37	
3146	IN HOUSE	136800		0.66	
3153	IFC62321	107000		-0.12	
3154	002021				
3163	in house	35875	G(0.05)	-2.78	
3166	in house	12260	G(0.05)	-3.58	
3172	IEC62321	101480		-0.54	
3182	IEC62321	141933.15		0.83	
3190	IEC62321	106641 0		-0.31	
3191	IEC62321	81789		-1.21	
3197	IEC62321	>200000			false positive?

3213 3218 3228 3240 3242 3243	IEC62321 IEC62321 IEC62321 IEC62321 in house in house	153371.0 119000 120000 111350 7229.05 16600	DG(0.01) C,G(0.01)	1.22 0.05 0.09 -0.21 -3.75 -3.43	first reported: 8599
	normality n outliers mean (n) st.dev. (n) R(calc.) R(target)	OK 59 9 117439.60 17969.920 50315.78 82207.72			Compare Horwitz: 9078.40





Determination of Octabromo diphenyl ether on sample #12083; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	INH-216	11 74	mark	=(tai g/	Tomarko
324	IEC62321	<5			
339	in house	<25			
551	IEC62321	n.d.			
605	IEC62321	n.d.			
607	IEC62321	n.d.			•
622	IEC62321	3.2	excl.		see §4.1
1132	IEC62321	n.a.			
1213	IEC02321	<00 12.8			
2115	12002021	12.0			
2129					
2132	EN62321	n.d.			
2137	IEC62321	<1			
2139	IEC62321	<50			
2156	IEC62321	9			
2165	IEC62321	n.d.			
2169	IEC62321	6.67 -10			
2172	IEC02321	<10 n d			
2179	IEC62321	5.82			
2184	IEC62321	n.d.			
2196	IEC62321	n.d.			
2201	EPA3540C	<5			
2202	in house	n.d.			
2212	in house	16.0			
2216	In house	<100			
2237	EN62321	<50			
2243	IEC62321	20.4 I			
2240	IEC62321	< 100 n d			
2271	IEC62321	18.8			
2284	IEC62321	<5			
2290					
2296					
2303					
2309	IEC62321	<50			
2316	IEC62321	<50			
2350	IEC62321	n.a. n.d			
2354	IEC62321	n.u. n.d			
2366	IEC62321	n.d.			
2370	IEC62321	n.d.			
2372	IEC62321	7.509			
2375	INH-216	n.d.			
2379	IEC62321	n.d.			
2386	IEC62321	<50			
2403	IEC62321	0.20			
2410	IEC02321	<0 132 75	G(0.05)		
2438	E002321	<50	0(0.00)		
2475					
2479	IEC62321	n.d.			
2482	INH-62321	<100			
2488					
2493					
2494	15060004				
2500	IEC02321	107 16	C G(0.01)		first reported: 95.07
2513	IEC62321	9 62123	0,0(0.01)		
3100	IEC62321	n.d.			
3146	in house	<100			
3151	EN62321	n.d.			
3153	IEC62321	<20			
3154	in house		0(0.04)		
3163	in nouse	320	G(0.01)		
3172					
3182	IEC62321	70.99	G(0.01)		
3185	IEC62321	n.d.	2(0.01)		
3190	IEC62321	<5			
3191	IEC62321	10			
3197	IEC62321	16.8			







Determination of Nonabromo diphenyl ether on sample #12083; results in mg/kg

lah	method	value	mark	z(tara)	remarks
110	INH-216	326.44	mark	2 00	Temarka
324	IFC62321	164 0		-0.94	
339	in house	73.7		-2.62	
551	IEC62321	61.9	С	-2.85	first reported: 49.8
605	IEC62321	114.82		-1.86	
607	IEC62321	114.31		-1.87	
622	IEC62321	125.6	excl.	-1.66	see §4.1
1132	IEC62321	n.d.			
1213					
1370	IEC62321	267.5		0.99	
2115	IEC62221Mod			1 20	
2129	EU622211000.	203	C	1.20	first reported: 69.41
2132	IEC62321	273.05	C	-0.79	list reported. 00.41
2139	IEC62321	205		-0.18	
2156	IEC62321	29	С	-3.46	first reported: 39
2165	IEC62321	200		-0.27	
2169	IEC62321	265.9		0.96	
2172	IEC62321	223.3		0.17	
2173	IEC62321	151.2		-1.18	
2179	IEC62321	190.1		-0.45	
2184	IEC62321	210.4		-0.07	
2196	ED02321	150		-1.20	
2201	in house	123.5		-1.20	
2202	in house	347.3		2 48	
2216	in house	126.1		-1.65	
2237	EN62321	186		-0.53	
2243	IEC62321	350.32		2.54	
2246	IEC62321	174.49		-0.74	
2256	IEC62321	391		3.29	
2271	IEC62321	408		3.61	
2284	IEC62321	339		2.32	
2290					
2290					
2303	IEC62321	240.0		0.48	
2316	IEC62321	240.0		0.40	
2350	IEC62321	159.0		-1.03	
2354	IEC62321	161		-1.00	
2359	IEC62321	149.6		-1.21	
2366	IEC62321	177.5		-0.69	
2370	IEC62321	121		-1.74	
2372	INH-216	299.09		1.04	
2379	IEC62321	202.41		-0.22	
2386	IEC62321	295.9		1.52	
2403	IEC62321	306		1.71	
2410	IEC62321	160		-1.01	
2415	IEC62321	389.35	0	3.26	first see estad. C0. C0.
2430 2475		293.11	C	1.40	
2479	IEC62321	152.7		-1.15	
2482	INH-62321	104.2		-2.06	
2488					
2493					
2494	15000004				
2500	IEC62321	330	C	2.16	first reported: 246.08
2513	IEC62321	519 56445		-0.44 5.69	list reported. 240.00
3100	IEC62321	261.0	20(0.00)	0.87	
3146	in house	159		-1.03	
3151	EN62321	128		-1.61	
3153	IEC62321	296		1.52	
3154	in have a				
3163	in nouse	145		-1.29	
3172	III IIUUSE	240		0.59	
3182	IEC62321	810.31	G(0.01)	11.12	
3185	IEC62321	215	-(0.01	
3190	IEC62321	153.6		-1.13	
3191	IEC62321	685	G(0.01)	8.78	
3197	IEC62321	268.2		1.00	

3213 3218 3228 3240 3242	IEC62321 IEC62321 IEC62321 IEC62321	175 292 204 157 	С	-0.73 1.45 -0.19 -1.07	first reported: 500.0, also reported: 411.8
3243	in house	506	C,DG(0.05)	5.44	first reported:783
	normality n	OK 61			
	outliers	4	+1 excl.		
	mean (n) st.dev. (n)	214.39 86.366			
	R(calc.)	241.82			
	R(target)	150.08			Compare Horwitz: 74.16





Determination of Decabromo diphenyl ether on sample #12083; results in mg/kg

lah	mothed	volue	mark	T(tora)	romarka
	method		mark	Z(targ)	remarks
110	INH-216	1709.44		-1.22	
324	IEC02321	3215		1.22	
551		126.0		-3.70	first reported: 70.7
605	IEC62321	2345 94	00(0.00)	-0.19	
607	IEC62321	2219.18		-0.39	
622	IEC62321	2199.9	excl.	-0.42	see §4.1
1132	IEC62321	1640.17		-1.33	
1213	IEC62321	2363.8		-0.16	
1370	IEC62321	1820	С	-1.04	first reported: 6397.1
2115					
2129	IEC62321Mod.	2365		-0.16	
2132	EN62321	4878.05	CG(0.05)	3.93	first reported: 1219.51
2137	IEC62321	2370.9		-0.15	
2139	IEC62321	2602		0.23	
2100	IEC02321	2202		-0.29	
2169	IEC62321	2044 3		-0.20	
2172	IEC62321	2891		0.70	
2173	IEC62321	2676		0.35	
2179	IEC62321	2956		0.80	
2184	IEC62321	2530.3		0.11	
2196	IEC62321	2313		-0.24	
2201	EPA3540C	2582		0.20	
2202	in house	2735.0		0.44	
2212	in house	2356.7		-0.17	
2210	IN NOUSE	2954		0.80	
2237	EIN02321	3040 4227 14		0.94	
2245	IEC62321	2834 35	DO(0.01)	0.61	
2256	IEC62321	4192	DG(0.01)	2.81	
2271	IEC62321	2322	(,	-0.23	
2284	IEC62321	2683		0.36	
2290					
2296					
2303					
2309	IEC62321	2048.0		-0.67	
2316	IEC62321	2024		-0.71	
2350	IEC62321	2367.5		-0.15	
2304	IEC62321	2240		-0.30	
2366	IEC62321	2367 5		-0.45	
2370	IEC62321	2240		-0.36	
2372	IEC62321	2113		-0.57	
2375	INH-216	2904.37		0.72	
2379	IEC62321	1752.64		-1.15	
2386	IEC62321	2776		0.51	
2403	IEC62321	2709		0.40	
2410	IEC62321	2715		0.41	
2415	IEC62321 EN62221	2729.45		0.44	
2430	LINUZJZI				
2479	IEC62321	2578		0.19	
2482	INH-62321	1810		-1.06	
2488					
2493					
2494					
2500	IEC62321	3453		1.61	
2504	IEC62321	3101.33	С	1.04	first reported: 6238.09
2513	IEC62321	2154.16108		-0.50	
3146	in house	2392.0		-0.11	
3140	EN62321	2535		0.31	
3153	IEC62321	2210		-0.41	
3154					
3163	in house	175	G(0.01)	-3.72	
3166	in house	3160	•	1.14	
3172	IEC62321	2129		-0.54	
3182	IEC62321	2494.74		0.05	
3185	IEC62321	2346		-0.19	
3190	IEC02321	27 13.U 4041	G(0.05)	0.41	
3197	IEC62321	2616.1	0(0.00)	0.25	

3213 3218 3228 3240 3242 3243	IEC62321 IEC62321 IEC62321 IEC62321 in house in house	2882.0 2460 2204 2170 2679.52 12000	G(0.01)	0.68 0.00 -0.42 -0.47 0.35 15.50	first reported: 19542
	normality n outliers mean (n) st.dev. (n) R(calc.) R(target)	OK 62 7 2461.33 396.891 1111.29 1722.93	+1 excl.		Compare Horwitz: 340.43





Analytical details for samples #12082 & 12083

Lab	Max. particle size	Extraction	Detection	Other details
110	1mmx1mmx1mm	ultrasonic bath	GC-MS	
324	< 1mm	soxhlet extraction, toluene	GC-MS	
339	No Further grinding	soxhlet extraction, 6 hr toluene	GC-MS	
551	500µm	ultrasonic	GC-MS	
605	<0.5mm	soxhlet extraction	GC-MS	
607	<0.5mm	soxhlet extraction	GC-MS	
622	small size	extraction, 6 hr	GC-ECD	
1132	<500µm	soxhlet extraction, toluene	GC-MS	
1010	500		00,110,110	can not quantify nona-
1213	<500µm		GC-MS-MS	BDE and octa-BDE
1370	0.5-1 mm	soxniet extraction, toluene	GC-MS	
2129	grinding under liquefied nitrogen		GC-MS	
2132	1mmx1mmx1mm	ultrasonic	GC-MS	
2137	500µm	soxhlet extraction, toluene	GC-MSD	
2139	Cryomilling liquefied nitrogen	ultrasonic extraction	GC-MS and HPL-DAD	
2156	<1mm	<0.5mm	GC-MS	
2165	1mmx1mmx1mm	sonication	GC-MS	
2169	<0.5mm	soxhlet	GC-MS	
2172	0.5 mmx 0.5mmx0.5mm		GC-MS	
2173	<500µm	soxhlet extraction	GC-MS	
2179	powder	utrasonication	GC/MS	
2184	3mmx3mmx3mm	sonication	GC-MS	ref. to IEC62321
2196	500µm	utrasonic/50°C 1 hr/Toluene	GC-MS, Sim	
2201	500µm	soxhlet extraction, toluene	GC-MS, HPLC DAD	
2202	<250µm	soxhlet	GC-MS	
2212	n.a.	extraction, toluene	GC-MS	
2216	<1000 um	ultrasonication	GC-uECD, GCMS	
2237	<0.05	utrasonic	GC-MS	
2243	<0.1mm	soxhlet, toluene	GC-MS	
2246	2mmx2mm	soxhlet extraction	GC-QQQ	
2256	0.1mmx0.1mmx0.1mm	soxhlet extraction	GC-MS	
2271	<1mm	soxhlet extraction	GC-MS	
2284	1mmx1mmx1mm	soxhlet extraction	GC/MS	
2309	500um	soxhlet extraction		
2316	<500um	soxhlet extraction	GC-MSD	
2350		ultrasonic	GC-MSD	
2354	1mmx1mmx1mm	soxhlet extraction	GC-MSD	
2359	powder	soxhlet extraction	GC-MS	
2366	<500µm	soxhlet	GC-MS	
2370	powder	soxhlet extraction	GC-MS	
2372	<0.5mm	soxhlet extraction	GC-MS	
2375	grinded sample	ultrasonic extraction, toluene	GC-MS	
2379		auto soxhlet extraction	GC-MS	
2386	<1mm	soxhlet extraction, 4hr	GC-MS	
2403	1mmx1mmx1mm	soxtec	GC-MS	
2410	Cryo-milling with nitrogen<500µm	soxhlet extraction, toluene	GS-MS	

2415	almost powder	soxhlet extraction	GS-MS	
2438	around 2 mm	soxhlet extraction, toluene	GC-MS	
2479	<500µm	soxhlet extraction	GC-MS	
2500	500µm	IEC62321:2008	IEC62321:2008	
2504	1-2mm	soxhlet	GC-MSD	
2513	left as pellets	soxhlet extraction, toluene	GC-MS	
3100	grind to powder, 500µm	soxhlet extraction, toluene	SIM	SIM
3146	no further grinding	soxhlet extraction, toluene	GC-MS	
3151	no further grinding	soxhlet extraction,toluene for 2h	GC-MS	ca.05g/50ml
3153	<500µm	soxhlet extraction	GC-MS	
3163	0.5g in THF, not completly solved		GC-MS	
3166		sonication	GC-MS	
3172	<500um	soxhlet extraction, toluene	GC-MS	
3182	<500um	soxhlet extraction	GC-MS	
3185	<500µm	soxhlet extraction	GC-MS	
3190	<500µm	soxhlet extraction	HPLC	
3191	500µm	soxhlet extraction	GC-MS	
3197	0.5mm	soxhlet extraction	GC-MS	
3213	freeze mill	soxhlet extraction	GC-MS	quantification problems with nona-DBE
3218	<500µm	soxhlet extraction	GC-MSD	
3228	1mmx1mm	sonication	GC/MS Int.Std.	
3240	<500µm	ultrasonic extraction	GC-MS	
3242	as received	soxhlet extraction	GC-MS	
3243	<2mm	ASE (Dionex) in IPA	GC-MS	

Note that one laboratory reported deviating conditions for sample #12083:

Lab	Max. particle size	Extraction	Detection	Other details
2213	<500µm	extract with n-Hexane-Acetone 3:1 V/V	GC-MS-MS	

Number of participating laboratories per country

1 lab in BELGIUM 2 labs in BRAZIL 2 labs in FRANCE 8 labs in GERMANY 6 labs in HONG KONG 1 lab in HUNGARY 3 labs in INDIA 2 labs in INDONESIA 2 labs in ITALY 1 lab in JAPAN 6 labs in KOREA 3 labs in MALAYSIA 22 labs in P.R. of CHINA 1 lab in SLOVAKIA 2 labs in TAIWAN R.O.C. 4 labs in THAILAND 1 lab in THE NETHERLANDS 3 labs in TURKEY 4 labs in U.S.A. 2 labs in UNITED KINGDOM 3 labs in VIETNAM

Abbreviations:

С	= final result after checking of first reported suspect result	t
•	initial recall alter checking of mot reported caepeet recal	

- 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
- n.a. = not applicable
- n.d. = not detected
- IMEP = International Measurement Evaluation Programme

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