# Results of Proficiency Test Brominated Flame retardants September 2012


Organised by: Institute for Interlaboratory Studies
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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:

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

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.

Octa-BDE: 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.

Nona-BDE: 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 µm. 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.

## APPENDIX 1

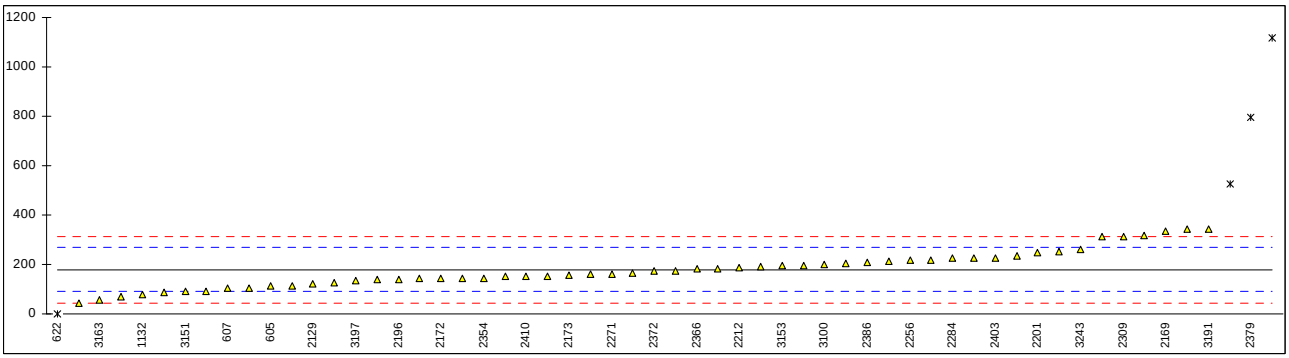
Determination of Octabromo diphenyl ether on sample #12082; results in mg/kg

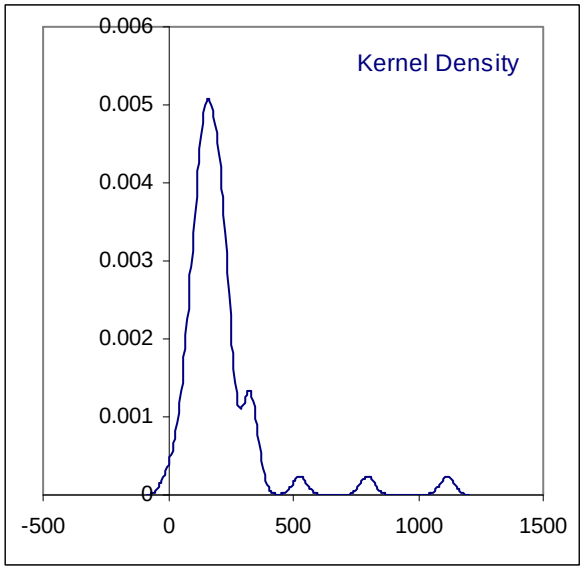
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 319.44 | | 3.14 | |
| 324 | IEC62321 | 93.18 | | -1.92 | |
| 339 | in house | <25 | | <-3.44 | false negative? |
| 551 | IEC62321 | n.d. | | ----- | |
| 605 | IEC62321 | 111.45 | | -1.51 | |
| 607 | IEC62321 | 102.67 | | -1.70 | |
| 622 | iEC62321 | 1.6 | G(0.05) | -3.96 | |
| 1132 | IEC62321 | 79.14 | | -2.23 | |
| 1213 | | ----- | | ----- | |
| 1370 | IEC62321 | 227.0 | | 1.08 | |
| 2115 | | ----- | | ----- | |
| 2129 | IEC62321Mod. | 120 | | -1.32 | |
| 2132 | EN62321 | n.d. | | ----- | |
| 2137 | IEC62321 | <1 | | <-3.98 | false negative? |
| 2139 | IEC62321 | 234 | | 1.23 | |
| 2156 | IEC62321 | 526 | C,G(0.01) | 7.76 | first reported: 426 |
| 2165 | IEC62321 | 190 | | 0.25 | |
| 2169 | IEC62321 | 335.8 | | 3.51 | |
| 2172 | IEC62321 | 143.5 | | -0.79 | |
| 2173 | IEC62321 | 156.3 | | -0.50 | |
| 2179 | IEC62321 | 152.3 | | -0.59 | |
| 2184 | IEC62321 | 164.1 | | -0.33 | |
| 2196 | IEC62321 | 139 | | -0.89 | |
| 2201 | EPA3540C | 246 | | 1.50 | |
| 2202 | in house | 124.2 | | -1.22 | |
| 2212 | in house | 186.7 | | 0.18 | |
| 2216 | IEC62321 | <100 | | ----- | |
| 2237 | EN62321 | 151 | | -0.62 | |
| 2243 | IEC62321 | 137.01 | | -0.94 | |
| 2246 | IEC62321 | 183.54 | | 0.10 | |
| 2256 | IEC62321 | 217 | | 0.85 | |
| 2271 | IEC62321 | 161 | | -0.40 | |
| 2284 | IEC62321 | 227 | | 1.08 | |
| 2290 | | ----- | | ----- | |
| 2296 | | ----- | | ----- | |
| 2303 | | ----- | | ----- | |
| 2309 | IEC62321 | 312.85 | | 3.00 | |
| 2316 | IEC62321 | 311 | | 2.96 | |
| 2350 | IEC62321 | 142.0 | | -0.82 | |
| 2354 | IEC62321 | 145 | | -0.76 | |
| 2359 | 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 | 796.96 | G(0.01) | 13.82 | |
| 2386 | 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 | | ----- | | ----- | |
| 2479 | IEC62321 | 160.8 | | -0.40 | |
| 2482 | INH-62321 | <100 | | ----- | |
| 2488 | | ----- | | ----- | |
| 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 | | ----- | |
| 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 | |

| | | | | | |
|---|---|---|---|---|---|
| 3190 | IEC62321 | <5 | | <-3.89 | false negative? |
| 3191 | IEC62321 | 343 | | 3.67 | |
| 3197 | IEC62321 | 132.9 | | -1.03 | |
| 3213 | IEC62321 | 69.0 | C | -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 | OK |
| | 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



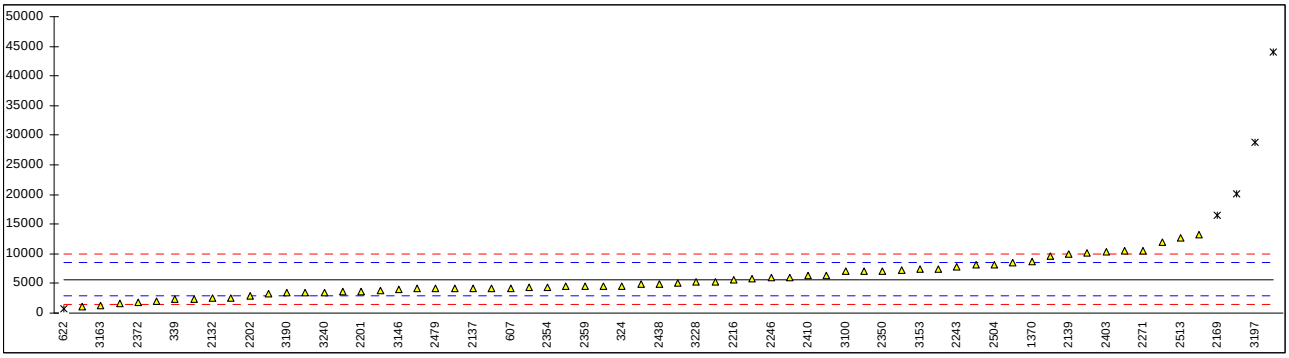



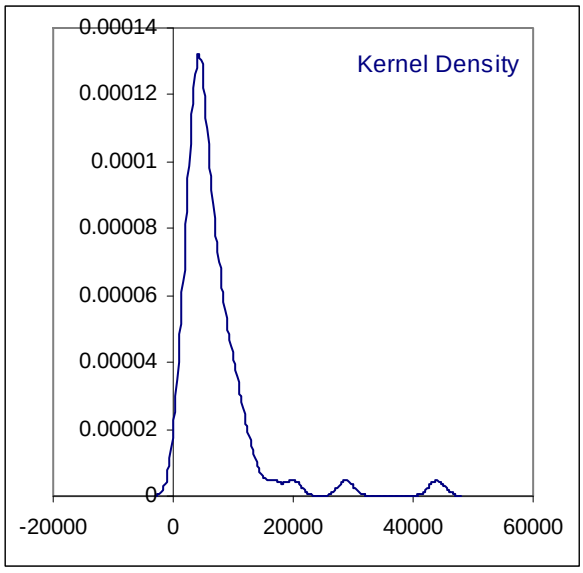
Determination of Nonabromo diphenyl ether on sample #12082; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 13308.25 | | 5.37 | |
| 324 | IEC62321 | 4589 | | -0.77 | |
| 339 | in house | 2390 | | -2.32 | |
| 551 | IEC62321 | 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 | | 0.23 | |
| 2132 | EN62321 | 2517.99 | C | -2.23 | first reported: 629.49 |
| 2137 | IEC62321 | 4232.8 | | -1.02 | |
| 2139 | 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 | | -1.46 | |
| 2202 | in house | 2973.1 | | -1.91 | |
| 2212 | in house | 9685.0 | | 2.82 | |
| 2216 | IEC62321 | 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 | | ----- | | ----- | |
| 2296 | | ----- | | ----- | |
| 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 | |
| 2366 | IEC62321 | 4477.5 | | -0.85 | |
| 2370 | IEC62321 | 4350 | | -0.94 | |
| 2372 | IEC62321 | 1729 | | -2.78 | |
| 2375 | INH-216 | 12018.49 | C | 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 | | ----- | | ----- | |
| 2438 | EN62321 | 4960.06 | | -0.51 | |
| 2475 | | ----- | | ----- | |
| 2479 | IEC62321 | 4139 | | -1.09 | |
| 2482 | INH-62321 | 4520 | | -0.82 | |
| 2488 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 2494 | | ----- | | ----- | |
| 2500 | IEC62321 | 10142 | | 3.14 | |
| 2504 | IEC62321 | 8206.17 | C | 1.78 | first reported: 12475.12 |
| 2513 | IEC62321 | 12669.7086 | | 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 | | ----- | | ----- | |
| 3163 | in house | 1270 | | -3.11 | |
| 3166 | in house | 1040 | | -3.27 | |
| 3172 | | ----- | | ----- | |
| 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 | first reported: 18851.2 |

| | | | | | |
|---|---|---|---|---|---|
| 3213 | IEC62321 | 6429 | C | 0.52 | first reported: 17750.0, also reported: 21533.4 |
| 3218 | IEC62321 | 8119 | | 1.71 | |
| 3228 | IEC62321 | 5200 | | -0.34 | |
| 3240 | IEC62321 | 3520 | | -1.52 | |
| 3242 | | ----- | | ----- | |
| 3243 | in house | 2584 | | -2.18 | |

| | | | |
|---|---|---|---|
| | normality | not OK | |
| | n | 61 | |
| | outliers | 4 | +1 excl. |
| | mean (n) | 5683.71 | |
| | st.dev. (n) | 2925.615 | |
| | R(calc.) | 8191.72 | |
| | R(target) | 3978.60 | Compare Horwitz: 1200.44 |



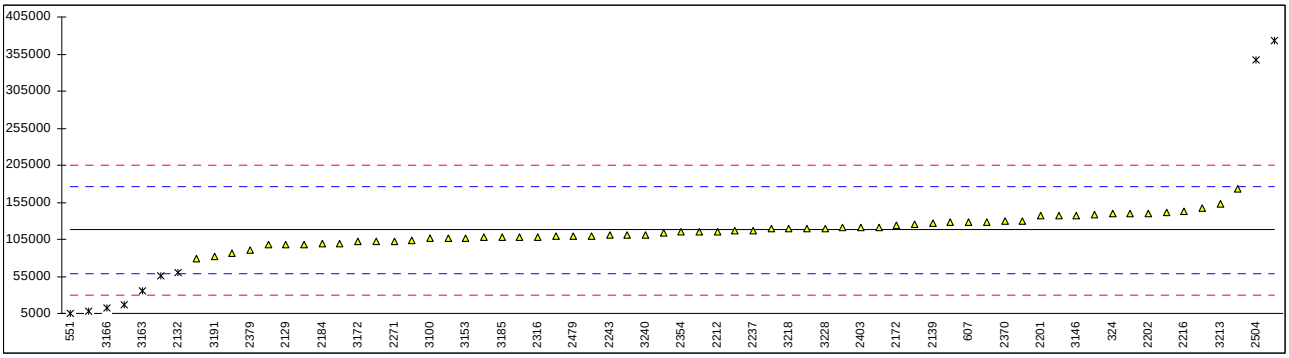
Determination of Decabromo diphenyl ether on sample #12082; results in mg/kg

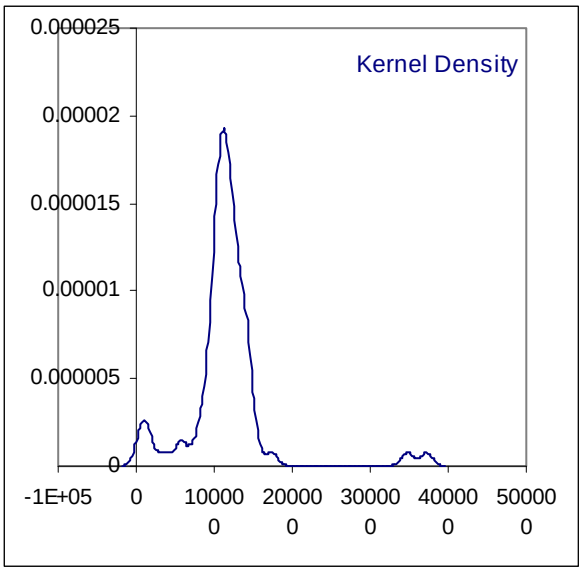
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 97268.46 | | -0.69 | |
| 324 | IEC62321 | 139650 | | 0.76 | |
| 339 | in house | 102000 | | -0.53 | |
| 551 | IEC62321 | 5209.1 | C,DG(0.01) | -3.82 | first reported: 10929.4 |
| 605 | IEC62321 | 129558.45 | | 0.41 | |
| 607 | IEC62321 | 128080.23 | | 0.36 | |
| 622 | IEC62321 | 372439.7 | G(0.01) | 8.69 | |
| 1132 | IEC62321 | 86667.17 | | -1.05 | |
| 1213 | IEC62321 | 140087.8 | | 0.77 | |
| 1370 | IEC62321 | 98149.6 | | -0.66 | |
| 2115 | | ----- | | ----- | |
| 2129 | IEC62321Mod. | 98020 | C | -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 | 126584 | | 0.31 | |
| 2156 | IEC62321 | 173582 | C | 1.91 | first reported: 187994 |
| 2165 | IEC62321 | 110000 | | -0.25 | |
| 2169 | IEC62321 | 127547.6 | | 0.34 | |
| 2172 | IEC62321 | 123300 | | 0.20 | |
| 2173 | IEC62321 | 107600 | | -0.34 | |
| 2179 | IEC62321 | 147151.7 | | 1.01 | |
| 2184 | IEC62321 | 98562.3 | | -0.64 | |
| 2196 | IEC62321 | 113000 | | -0.15 | |
| 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 | 110073.28 | | -0.25 | |
| 2246 | IEC62321 | 103218.25 | | -0.48 | |
| 2256 | IEC62321 | 118916 | | 0.05 | |
| 2271 | IEC62321 | 102677 | | -0.50 | |
| 2284 | IEC62321 | 119509 | | 0.07 | |
| 2290 | | ----- | | ----- | |
| 2296 | | ----- | | ----- | |
| 2303 | | ----- | | ----- | |
| 2309 | 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 | |
| 2372 | IEC62321 | 136565 | | 0.65 | |
| 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 | |
| 2415 | | ----- | | ----- | |
| 2438 | EN62321 | 98852 | C | -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 | |
| 3151 | EN62321 | 121032 | | 0.12 | |
| 3153 | IEC62321 | 107000 | | -0.36 | |
| 3154 | | ----- | | ----- | |
| 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 | |
| 3185 | IEC62321 | 108400 | | -0.31 | |
| 3190 | IEC62321 | 106641.0 | | -0.37 | |
| 3191 | IEC62321 | 81789 | | -1.21 | |
| 3197 | IEC62321 | >200000 | | ----- | false positive? |

| | | | | | |
|---|---|---|---|---|---|
| 3213 | IEC62321 | 153371.0 | | 1.22 | |
| 3218 | IEC62321 | 119000 | | 0.05 | |
| 3228 | IEC62321 | 120000 | | 0.09 | |
| 3240 | IEC62321 | 111350 | | -0.21 | |
| 3242 | in house | 7229.05 | DG(0.01) | -3.75 | |
| 3243 | in house | 16600 | C,G(0.01) | -3.43 | first reported: 8599 |

| | | |
|---|---|---|
| | normality | OK |
| | n | 59 |
| | outliers | 9 |
| | mean (n) | 117439.60 |
| | st.dev. (n) | 17969.920 |
| | R(calc.) | 50315.78 |
| | R(target) | 82207.72 |

Compare Horwitz: 9078.40

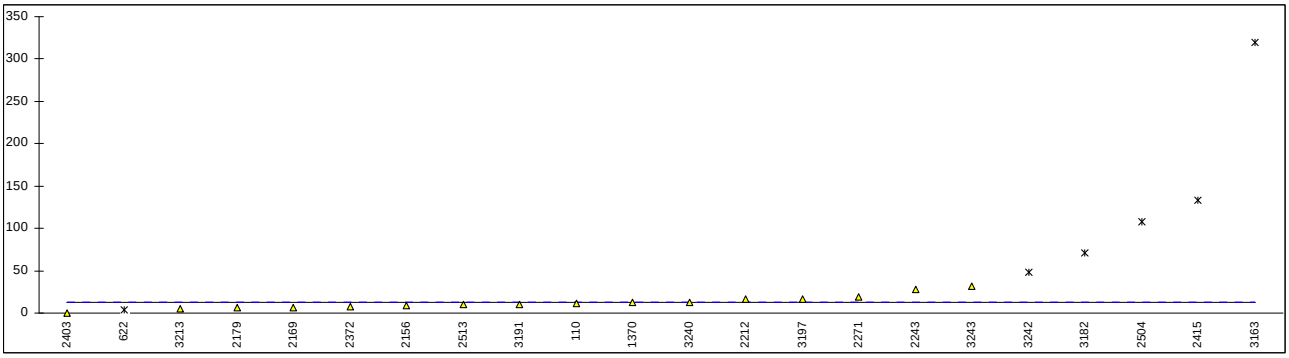


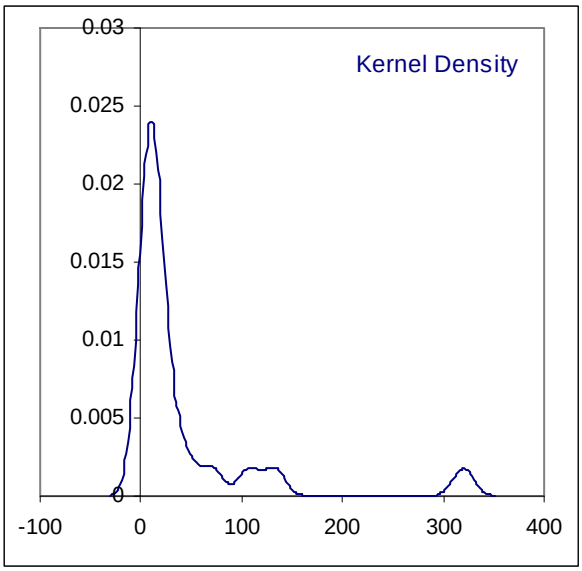



Determination of Octabromo diphenyl ether on sample #12083; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 11.74 | | ----- | |
| 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.d. | | ----- | |
| 1213 | IEC62321 | <50 | | ----- | |
| 1370 | IEC62321 | 12.8 | | ----- | |
| 2115 | | ----- | | ----- | |
| 2129 | | ----- | | ----- | |
| 2132 | EN62321 | n.d. | | ----- | |
| 2137 | IEC62321 | <1 | | ----- | |
| 2139 | IEC62321 | <50 | | ----- | |
| 2156 | IEC62321 | 9 | | ----- | |
| 2165 | IEC62321 | n.d. | | ----- | |
| 2169 | IEC62321 | 6.67 | | ----- | |
| 2172 | IEC62321 | <10 | | ----- | |
| 2173 | IEC62321 | 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 | 28.41 | | ----- | |
| 2246 | IEC62321 | <100 | | ----- | |
| 2256 | IEC62321 | n.d. | | ----- | |
| 2271 | IEC62321 | 18.8 | | ----- | |
| 2284 | IEC62321 | <5 | | ----- | |
| 2290 | | ----- | | ----- | |
| 2296 | | ----- | | ----- | |
| 2303 | | ----- | | ----- | |
| 2309 | IEC62321 | <50 | | ----- | |
| 2316 | IEC62321 | <50 | | ----- | |
| 2350 | IEC62321 | n.d. | | ----- | |
| 2354 | IEC62321 | n.d. | | ----- | |
| 2359 | IEC62321 | 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 | IEC62321 | <5 | | ----- | |
| 2415 | IEC62321 | 132.75 | G(0.05) | ----- | |
| 2438 | EN62321 | <50 | | ----- | |
| 2475 | | ----- | | ----- | |
| 2479 | IEC62321 | n.d. | | ----- | |
| 2482 | INH-62321 | <100 | | ----- | |
| 2488 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 2494 | | ----- | | ----- | |
| 2500 | IEC62321 | n.d. | | ----- | |
| 2504 | IEC62321 | 107.16 | C,G(0.01) | ----- | first reported: 95.07 |
| 2513 | IEC62321 | 9.62123 | | ----- | |
| 3100 | IEC62321 | n.d. | | ----- | |
| 3146 | in house | <100 | | ----- | |
| 3151 | EN62321 | n.d. | | ----- | |
| 3153 | IEC62321 | <20 | | ----- | |
| 3154 | | ----- | | ----- | |
| 3163 | in house | 320 | G(0.01) | ----- | |
| 3166 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3182 | IEC62321 | 70.99 | G(0.01) | ----- | |
| 3185 | IEC62321 | n.d. | | ----- | |
| 3190 | IEC62321 | <5 | | ----- | |
| 3191 | IEC62321 | 10 | | ----- | |
| 3197 | IEC62321 | 16.8 | | ----- | |

| | | | | | |
|---|---|---|---|---|---|
| 3213 | IEC62321 | 4.8 | | ----- | |
| 3218 | IEC62321 | n.d. | | ----- | |
| 3228 | | ----- | | ----- | |
| 3240 | IEC62321 | 13 | | ----- | |
| 3242 | in house | 47.76 | G(0.05) | ----- | |
| 3243 | in house | 32 | C | ----- | first reported:407 |
| | | | | | |
| | normality | OK | | | |
| | n | 16 | | | |
| | outliers | 5 | +1 excl. | | |
| | mean (n) | 12.70 | | | |
| | st.dev. (n) | 8.347 | | | |
| | R(calc.) | 23.37 | | | |
| | R(target) | (8.89) | | | Compare Horwitz: 9.51 |



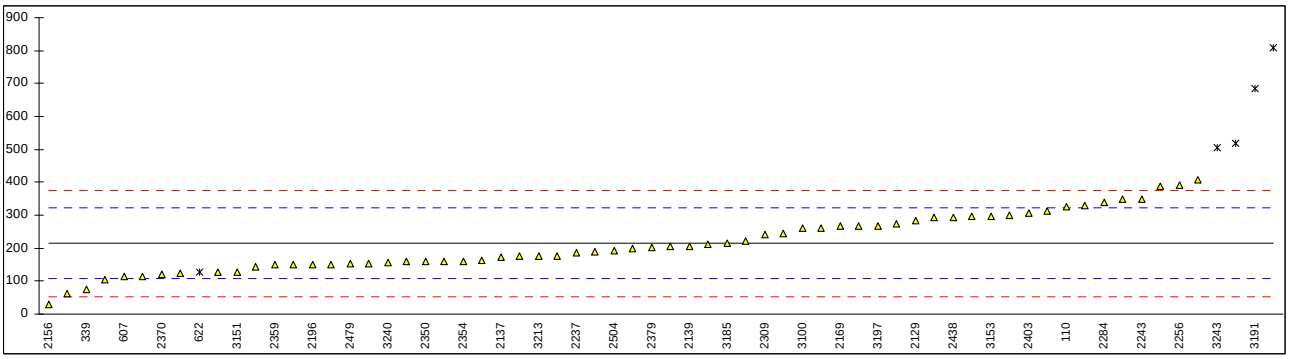



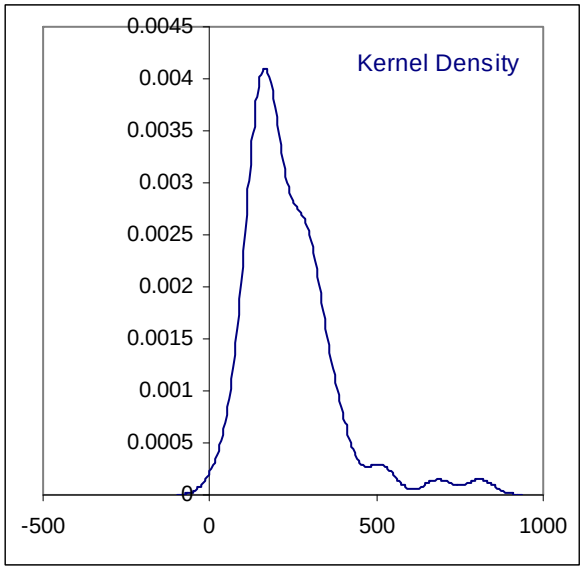
Determination of Nonabromo diphenyl ether on sample #12083; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 326.44 | | 2.09 | |
| 324 | IEC62321 | 164.0 | | -0.94 | |
| 339 | in house | 73.7 | | -2.62 | |
| 551 | IEC62321 | 61.9 | C | -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 | | ----- | | ----- | |
| 2129 | IEC62321Mod. | 283 | | 1.28 | |
| 2132 | EN62321 | 273.65 | C | 1.11 | first reported: 68.41 |
| 2137 | IEC62321 | 171.9 | | -0.79 | |
| 2139 | IEC62321 | 205 | | -0.18 | |
| 2156 | IEC62321 | 29 | C | -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 | IEC62321 | 150 | | -1.20 | |
| 2201 | EPA3540C | 150 | | -1.20 | |
| 2202 | in house | 123.5 | | -1.70 | |
| 2212 | 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 | | ----- | | ----- | |
| 2296 | | ----- | | ----- | |
| 2303 | | ----- | | ----- | |
| 2309 | IEC62321 | 240.0 | | 0.48 | |
| 2316 | IEC62321 | 261 | | 0.87 | |
| 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 | IEC62321 | 312.8 | | 1.84 | |
| 2375 | INH-216 | 299.09 | | 1.58 | |
| 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 | | 3.26 | |
| 2438 | EN62321 | 293.77 | C | 1.48 | first reported: 68.62 |
| 2475 | | ----- | | ----- | |
| 2479 | IEC62321 | 152.7 | | -1.15 | |
| 2482 | INH-62321 | 104.2 | | -2.06 | |
| 2488 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 2494 | | ----- | | ----- | |
| 2500 | IEC62321 | 330 | | 2.16 | |
| 2504 | IEC62321 | 191.07 | C | -0.44 | first reported: 246.08 |
| 2513 | IEC62321 | 519.56445 | DG(0.05) | 5.69 | |
| 3100 | IEC62321 | 261.0 | | 0.87 | |
| 3146 | in house | 159 | | -1.03 | |
| 3151 | EN62321 | 128 | | -1.61 | |
| 3153 | IEC62321 | 296 | | 1.52 | |
| 3154 | | ----- | | ----- | |
| 3163 | in house | 145 | | -1.29 | |
| 3166 | in house | 246 | | 0.59 | |
| 3172 | | ----- | | ----- | |
| 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 | IEC62321 | 175 | C | -0.73 | first reported: 500.0, also reported: 411.8 |
| 3218 | IEC62321 | 292 | | 1.45 | |
| 3228 | IEC62321 | 204 | | -0.19 | |
| 3240 | IEC62321 | 157 | | -1.07 | |
| 3242 | | ----- | | ----- | |
| 3243 | in house | 506 | C,DG(0.05) | 5.44 | first reported:783 |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 61 | |
| | outliers | 4 | +1 excl. |
| | mean (n) | 214.39 | |
| | st.dev. (n) | 86.366 | |
| | R(calc.) | 241.82 | |
| | R(target) | 150.08 | Compare Horwitz: 74.16 |



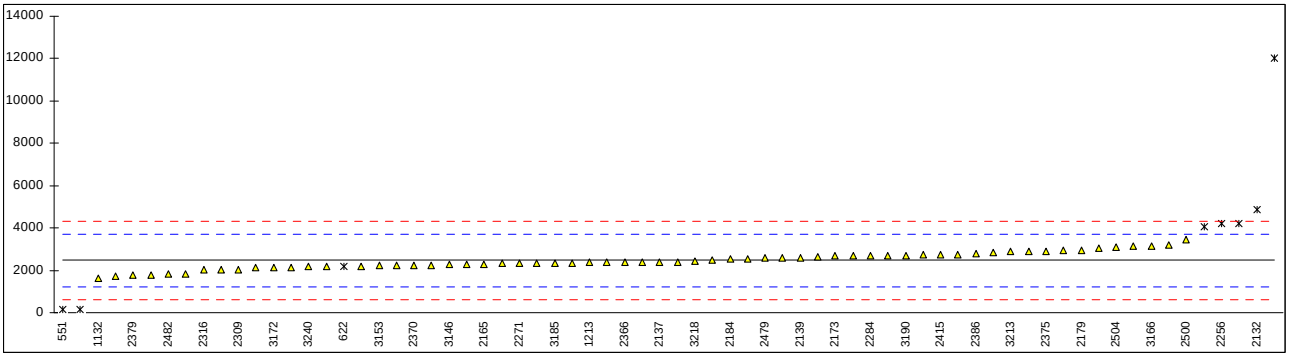



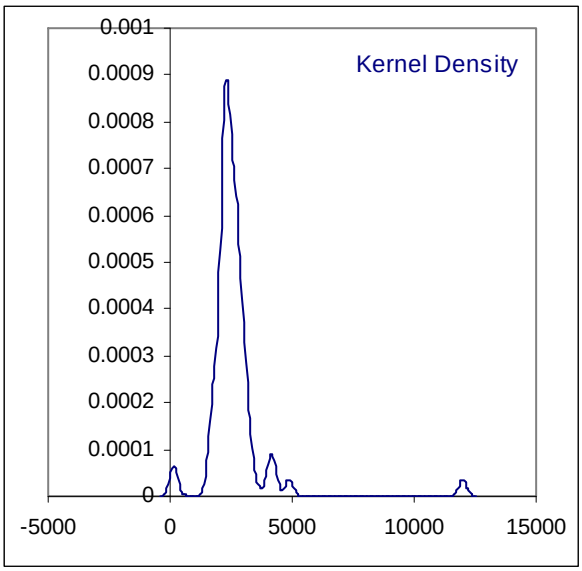
Determination of Decabromo diphenyl ether on sample #12083; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | INH-216 | 1709.44 | | -1.22 | |
| 324 | IEC62321 | 3215 | | 1.22 | |
| 339 | in house | 1800 | | -1.07 | |
| 551 | IEC62321 | 126.9 | CG(0.05) | -3.79 | first reported: 79.7 |
| 605 | IEC62321 | 2345.94 | | -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 | C | -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 | |
| 2156 | IEC62321 | 2282 | | -0.29 | |
| 2165 | IEC62321 | 2300 | | -0.26 | |
| 2169 | IEC62321 | 2044.3 | | -0.68 | |
| 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 | |
| 2216 | in house | 2954 | | 0.80 | |
| 2237 | EN62321 | 3040 | | 0.94 | |
| 2243 | IEC62321 | 4227.14 | DG(0.01) | 2.87 | |
| 2246 | IEC62321 | 2834.35 | | 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 | |
| 2354 | IEC62321 | 2240 | | -0.36 | |
| 2359 | IEC62321 | 2186.9 | | -0.45 | |
| 2366 | IEC62321 | 2367.5 | | -0.15 | |
| 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 | 2729.45 | | 0.44 | |
| 2438 | EN62321 | 3119.64 | | 1.07 | |
| 2475 | | ----- | | ----- | |
| 2479 | IEC62321 | 2578 | | 0.19 | |
| 2482 | INH-62321 | 1810 | | -1.06 | |
| 2488 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 2494 | | ----- | | ----- | |
| 2500 | IEC62321 | 3453 | | 1.61 | |
| 2504 | IEC62321 | 3101.33 | C | 1.04 | first reported: 6238.09 |
| 2513 | IEC62321 | 2154.16108 | | -0.50 | |
| 3100 | IEC62321 | 2392.8 | | -0.11 | |
| 3146 | in house | 2270 | | -0.31 | |
| 3151 | EN62321 | 2535 | | 0.12 | |
| 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 | IEC62321 | 2713.0 | | 0.41 | |
| 3191 | IEC62321 | 4041 | G(0.05) | 2.57 | |
| 3197 | IEC62321 | 2616.1 | | 0.25 | |

| | | | | | |
|---|---|---|---|---|---|
| 3213 | IEC62321 | 2882.0 | | 0.68 | |
| 3218 | IEC62321 | 2460 | | 0.00 | |
| 3228 | IEC62321 | 2204 | | -0.42 | |
| 3240 | IEC62321 | 2170 | | -0.47 | |
| 3242 | in house | 2679.52 | | 0.35 | |
| 3243 | in house | 12000 | G(0.01) | 15.50 | first reported: 19542 |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 62 | |
| | outliers | 7 | +1 excl. |
| | mean (n) | 2461.33 | |
| | st.dev. (n) | 396.891 | |
| | R(calc.) | 1111.29 | |
| | R(target) | 1722.93 | Compare Horwitz: 340.43 |



## APPENDIX 2

### 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 | |
| 1213 | <500µm | ultrasonic, toluene | GC-MS-MS | can not quantify nona-BDE and octa-BDE |
| 1370 | 0.5-1 mm | soxhlet extraction, toluene | GC-MS | |
| 2129 | grinding under liquefied nitrogen | ultrasonic, dichloormethane | 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 | |

## APPENDIX 3

### 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

**APPENDIX 4**

**Abbreviations**:

C = final result after checking of first reported suspect 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
n.a. = not applicable
n.d. = not detected
IMEP = International Measurement Evaluation Programme

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