Results of Proficiency Test Organotin Compounds in Textile December 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile & leather products on a voluntary basis. Well known organizations are for instance: Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL) and Oeko-Tex Standard 100 (Switzerland).

Since 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Organotin Compounds in textile every year. During the annual proficiency testing program of 2020/2021 it was decided to continue the proficiency test for the analysis of Organotin Compounds in textile.

In this interlaboratory study 92 laboratories in 27 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Organotin Compounds in textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, both positive on some Organotin compounds, labelled #20755 and #20756.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

For the first sample a batch of blue cotton textile was enriched with Monobutyltin (MBT). The batch was cut into small pieces and after homogenization divided over 120 subsamples of 3 grams each and labelled #20755.
The homogeneity of the subsamples was checked by determination of Monobutyltin (MBT) in accordance with an in house test method on 8 stratified randomly selected subsamples.

	Monobutyltin (MBT) in mg/kg
sample #20755-1	6.65
sample #20755-2	6.68
sample #20755-3	6.71
sample #20755-4	7.34
sample #20755-5	6.21
sample #20755-6	6.52
sample #20755-7	6.53
sample #20755-8	6.53

Table 1: homogeneity test results of subsamples #20755

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

	Monobutyltin (MBT) in mg/kg
r (observed)	0.90
reference test method	ISO/TS16179:12
0.3 x R (reference test method)	1.28

Table 2: evaluation of the repeatability of subsamples #20755

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

For the second sample a batch of light purple cotton textile was enriched with a number of Organotin components. The batch was cut into small pieces and after homogenization divided over 120 subsamples of 3 grams each and labelled #20756.
The homogeneity of the subsamples was checked by determination of Dibutyltin (DBT) in accordance with ISO17353 on 9 stratified randomly selected subsamples.

	Dibutyltin (DBT) in mg/kg
sample #20756-1	5.14
sample #20756-2	5.19
sample #20756-3	4.84
sample #20756-4	4.29
sample #20756-5	5.37
sample #20756-6	4.97
sample #20756-7	5.31
sample #20756-8	4.24
sample #20756-9	4.83

Table 3: homogeneity test results of subsamples #20756

From the above test results the relative standard deviation RSD_r was calculated and compared with 0.3 times the RSD's form previous iis PTs in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Dibutyltin (DBT)
RSD_r (observed)	8%
reference test method	ISO/TS16179:12
0.3 x RSD_R (reference test method)	7%
0.3 x RSD_R (previous iis PTs)	11%

Table 4: evaluation of the relative standard deviations of subsamples #20756

The calculated relative standard deviation RSD_r is almost in agreement with 0.3 times the RSD_R of the reference test method and did meet 0.3 times the RSD_R from previous proficiency tests (see chapter 4.3, table 7). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #20755 and one sample labelled #20756 were sent on November 18, 2020.

2.5 ANALYZES

The participants were requested to determine on both samples #20755 and #20756: Monomethyltin (MMT), Dimethyltin (DMT), Trimethyltin (TMT), Tripropyltin (TPT), Monobutyltin (MBT), Dibutyltin (DBT), Tributyltin (TBT), Tetrabutyltin (TeBT), Monooctyltin (MOT), Dioctyltin (DOT), Trioctyltin (TOT), Diphenyltin (DPhT), Triphenyltin (TPhT) and Tricyclohexyltin (TCyHT).

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

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers,

this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

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

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

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

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

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

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

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Five participants reported test results after the final reporting date and seven other participants did not report any test results. Not all participants were able to report all tests requested.
In total 85 participants reported 247 numerical test results. Observed were 12 outlying test results, which is 4.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of Organotin compounds in textile test method ISO/TS16179 is recommended to be the common test method. Unfortunately, test method ISO/TS16179 mentions for only three Organotin components precision data that varies greatly from one another (see table B.1 of ISO/TS16179:12), with MBT having an RSD of 23%. Therefore, for the evaluation of the test results we decided in iis PT of 2018 to compare all Organotin

Compounds with a target value of 23%. This means that the target reproducibility for each Organotin component will be 2.8 * 23 * mean PT /100.

Sample #20755

Monobutyltin (MBT): This determination was problematic for a number of laboratories. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO/TS16179:12.

Other Organotin components: The concentrations reported for all other Organotin components were near or below the detection limit. Therefore, no z-scores are calculated. See appendix 2 for the reported test results.

Sample #20756

Monobutyltin (MBT): This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO/TS16179:12.

Dibutyltin (DBT): This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO/TS16179:12.

Other Organotin components: The concentrations reported for all other Organotin components were near or below the detection limit. Therefore, no z-scores are calculated. See appendix 2 for the reported test results.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(target)
Monobutyltin (MBT)	mg/kg	77	6.06	3.45	3.91

Table 5: reproducibilities of tests on sample #20755

Parameter	unit	n	average	2.8 * sd	R(target)
Monobutyltin (MBT)	mg/kg	81	1.29	1.43	0.83
Dibutyltin (DBT)	mg/kg	77	3.90	1.99	2.51

Table 6: reproducibilities of tests on sample #20756

Without further statistical calculations, it can be concluded that for two of the three detected components there is a good compliance of the group of participants with the reference test method. The problematic test has been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

The development of the uncertainties of the proficiency tests over the years was compared, expressed as the relative standard deviations (RSD) of the PT, in the next table.

Component	December 2020	December 2019	December 2018	December 2017	December 2016	Reference ISO16179
Monomethyltin (MMT)	n.e.	37%	--	--	--	23%
Dimethyltin (DMT)	n.e.	22%	25-40%	38%	--	23%
Trimethyltin (TMT)	n.e.	26%				
Monobutyltin (MBT)	20-39%	33%	--	--	37%	23%
Dibutyltin (DBT)	18%	22%	21%	35%	--	23%
Tributyltin (TBT)	n.e.	--	29-31%	29%	--	23%

Table 7: development of uncertainties of the proficiency tests over the years

The RSDs observed in this PT are better or in line with RSDs observed in previous iis PTs. It is observed that when the Organotin Compound level is low the variability in the PT becomes higher.

Sample #20756 was used before in Proficiency Test iis19A17 as sample #19660. It is observed that the current PT findings show similar average concentration levels and calculated reproducibilities, see next table.

Component	unit	#20756			#19660		
		n	average	R(calc)	n	average	R(calc)
Monobutyltin (MBT)	mg/kg	81	1.29	1.43	76	0.78	0.72
Dibutyltin (DBT)	mg/kg	77	3.90	1.99	81	4.92	3.00

Table 8: comparison of sample #20756 with sample #19660

4.4 EVALUATION OF ANALYTICAL DETAILS

The participants were asked to provide some analytical details which are listed in appendix 3. Based on the reported answers the following can be summarized:

- 82% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- 57% used the samples as received prior to analysis and 43% further cut the samples
- Almost all participants used between 0.5 - 1 grams of sample intake; 36% around 0.5 grams and 55% around 1 gram.
- 93% used Ultrasonic technique to extract/release the components from the samples.
- 68% used a mixture of Methanol and Ethanol as extraction solvent, 20% used other mixtures and 12% used one solvent e.g. Hexane, Acetone or iso-Octane.
- Almost all participants used an extraction/release time of 60 minutes and 77% used an extraction/release temperature of 60°C, 14% used a lower temperature.
- 80% reported to observe a pH of 4.5 - 4.6. About 46% have adjusted the pH.

The effect of sample preparation prior to analysis and amount of sample intake on the determinations of Monobutyltin (MBT) in sample #20755 was further investigated in the next table.

Analytical Details	unit	n	average	RSD
Used as received	mg/kg	37	6.15	22%
Further cut	mg/kg	32	6.08	19%
around 1 g sample intake	mg/kg	38	5.96	21%
around 0.5 g sample intake	mg/kg	27	6.40	20%

Table 9: effect of sample preparation and amount of sample intake on MBT in textile sample #20755

It appeared that the effect of the analytical details on the determination of MBT is not statistically significant.

5 DISCUSSION

In this proficiency test for the determination of Organotin in textile, it was noticed that the majority of the participants was able to detect and quantify the Organotin components Monobutyltin (MBT) in sample #20755 and Dibutyltin in sample #20756. However, the quantification of Monobutyltin (MBT) in sample #20756 was problematic.

When the test results of this interlaboratory study were compared to the Oeko-Tex Standard 100 (see table 10), it could be noted that some laboratories would make a different decision about the acceptability of the textile.
Four reporting laboratories would accept sample #20755 based on MBT for all classes (less than 1 mg/kg). All other reporting laboratories would have rejected sample #20755.
Nineteen reporting laboratories would accept sample #20756 based on MBT+DBT for class 1 (less than 1 mg/kg) while all other reporting laboratories would have rejected sample #20756 for class 1. And 76 reporting laboratories would accept sample #20756 based on MBT+DBT for classes 2 to 4 (less than 2 mg/kg) while all other reporting laboratories would have rejected sample #20756 for classes 2 to 4.

Oeko-Tex Standard 100	Class 1 Baby clothes (mg/kg)	Class 2 Clothes direct skin contact (mg/kg)	Class 3 Clothes, no direct contact with skin (mg/kg)	Class 4 Decoration material (mg/kg)
TBT, TPhT	0.5	1.0	1.0	1.0
Other Organotin compounds	1.0	2.0	2.0	2.0

Table 10: Ecolabelling Standard and Requirements for Textiles in EU

6 CONCLUSION

Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

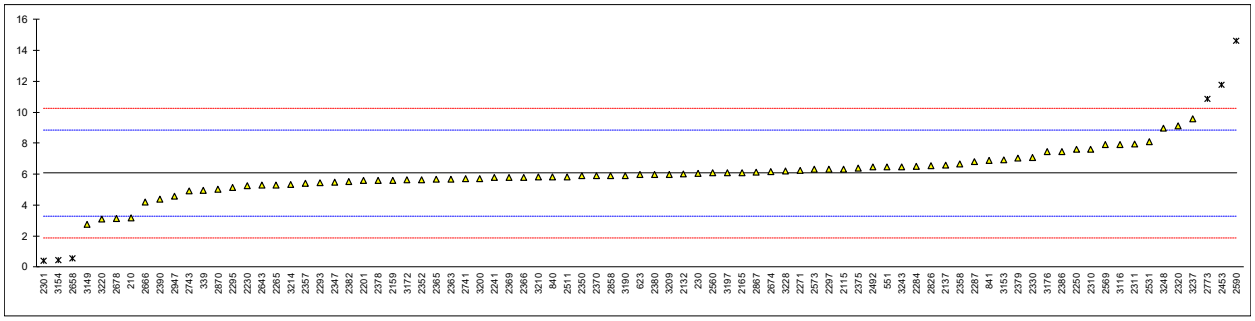
Determination of Monobutyltin (MBT) on sample #20755; results in mg/kg

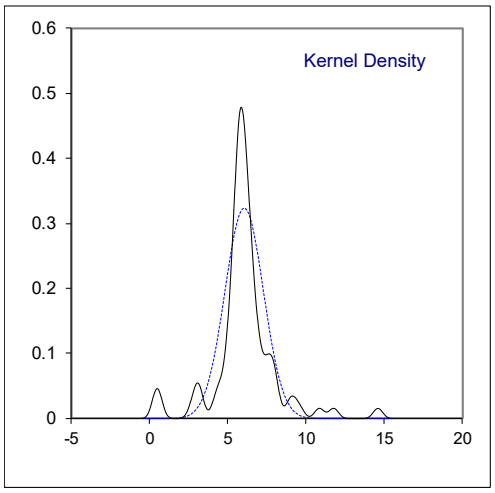
lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17353	3.18		-2.07	
230	ISO/TS 16179	6.052017	C	-0.01	first reported 12.10403
339	In house	4.97		-0.78	
551		6.49		0.31	
623	ISO/TS 16179	5.970		-0.07	
840	ISO/TS 16179	5.84		-0.16	
841	ISO/TS 16179	6.914		0.61	
2115	In house	6.32		0.18	
2129		-----		-----	
2132	ISO/TS 16179	6.0101		-0.04	
2137	KS K0737	6.605	C	0.39	first reported 10.602
2159	In house	5.616	C	-0.32	first reported 1.248
2165	ISO/TS 16179	6.11		0.03	
2201	ISO/TS 16179	5.599		-0.33	
2213		-----		-----	
2230		5.25		-0.58	
2241	ISO/TS 16179	5.780		-0.20	
2250	ISO/TS 16179	7.61		1.11	
2265	ISO/TS 16179	5.310		-0.54	
2271	ISO/TS 16179	6.27		0.15	
2284	ISO/TS 16179	6.525		0.33	
2287	ISO/TS 16179	6.838		0.55	
2293	ISO/TS 16179	5.470		-0.43	
2295	ISO/TS 16179	5.137		-0.66	
2297	ISO/TS 16179	6.32		0.18	
2301	ISO/TS 16179	0.431	R(0.01)	-4.04	
2310	ISO17353	7.62		1.12	
2311	ISO/TS 16179	7.9686		1.37	
2320	ISO/TS 16179	9.151		2.21	
2330	ISO/TS 16179	7.080		0.73	
2347		5.50		-0.40	
2350	ISO/TS 16179	5.899		-0.12	
2352	ISO/TS 16179	5.660		-0.29	
2357	ISO/TS 16179	5.41		-0.47	
2358	ISO17353	6.6794		0.44	
2363	ISO/TS 16179	5.7		-0.26	
2365	ISO/TS 16179	5.70		-0.26	
2366	ISO/TS 16179	5.80		-0.19	
2369	ISO/TS 16179	5.80		-0.19	
2370	ISO/TS 16179	5.90		-0.12	
2375	In house	6.4		0.24	
2378	ISO/TS 16179	5.601		-0.33	
2379	ISO/TS 16179	7.0409		0.70	
2380	ISO/TS 16179	5.982		-0.06	
2382	ISO/TS 16179	5.54		-0.38	
2386	ISO/TS 16179	7.468		1.01	
2390	ISO/TS 16179	4.4100		-1.19	
2453	In house	11.80	R(0.01)	4.11	
2492	In house	6.471		0.29	
2511	ISO/TS 16179	5.85		-0.15	
2531	ISO/TS 16179	8.12	C	1.47	first reported 0.74
2560	ISO17353	6.1		0.03	
2561		-----		-----	
2569	ISO/TS 16179	7.92		1.33	
2573	ISO/TS 16179	6.31		0.18	
2590	ISO/TS 16179	14.634	C,R(0.01)	6.14	first reported 15.905
2591		-----		-----	
2614		-----		-----	
2643	KS K0737	5.3	C	-0.55	first reported 11.2
2658		0.57	R(0.01)	-3.94	
2666	ISO/TS 16179	4.21796		-1.32	
2674	ISO/TS 16179	6.192		0.09	
2678	ISO/TS 16179	3.130		-2.10	
2741	ISO/TS 16179	5.707		-0.26	
2743	ISO/TS 16179	4.9189		-0.82	
2773	ISO16179	10.88	R(0.05)	3.45	
2804		-----		-----	
2826	ISO/TS 16179	6.5391		0.34	
2858	ISO/TS 16179	5.9		-0.12	
2864	ISO/TS 16179	not detected		-----	
2867	ISO/TS 16179	6.151		0.06	
2870	ISO23161Mod.	5.05		-0.73	

lab	method	value	mark	z(targ)	remarks
2947	In house	4.57		-1.07	
3116	ISO/TS 16179	7.934		1.34	
3118		-----		-----	
3149	ISO/TS 16179	2.75	C	-2.38	first reported 1.11
3153	ISO/TS 16179	6.93		0.62	
3154	ISO/TS 16179	0.46	C,R(0.01)	-4.02	first reported 1.523
3172	ISO/TS 16179	5.635		-0.31	
3176	ISO/TS 16179	7.46		1.00	
3190	ISO/TS 16179	5.908		-0.11	
3197	ISO/TS 16179	6.10		0.03	
3200	In house	5.73		-0.24	
3209	ISO/TS 16179	5.987		-0.06	
3210	In house	5.82		-0.18	
3214	ISO/TS 16179	5.345		-0.52	
3220	ISO/TS 16179	3.109		-2.12	
3228	ISO/TS 16179	6.22		0.11	
3237	ISO/TS 16179	9.6	C	2.53	first reported 14.5
3243	In house	6.49		0.31	
3248	In house	8.99		2.10	

			Only ISO16179:12 *)	
normality	suspect		suspect	
n	77		43	
outliers	6		2	
mean (n)	6.0643		6.0693	
st.dev. (n)	1.23183	RSD=20%	1.38745	RSD=23%
R(calc.)	3.4491		3.8849	
st.dev.(ISO/TS16179:12)	1.39479		1.39595	
R(ISO/TS16179:12)	3.9054		3.9087	

*) Followed ISO16179 with Methanol/Ethanol mixture and extraction temperature 60°C for 60 minutes



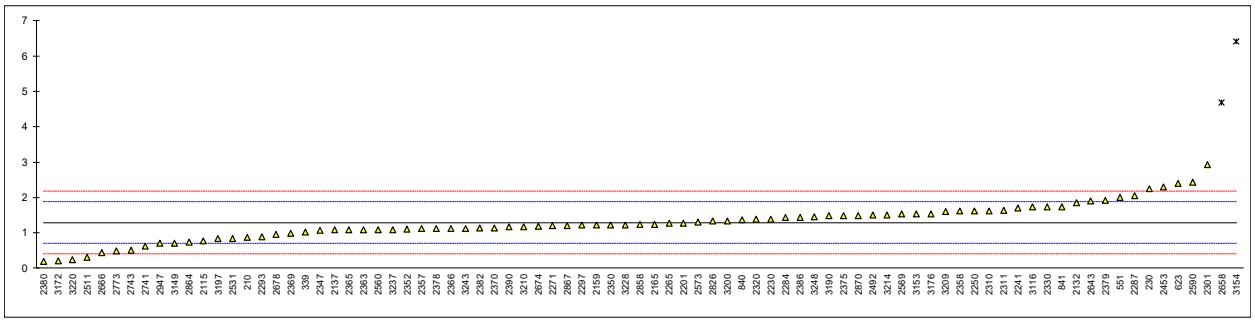


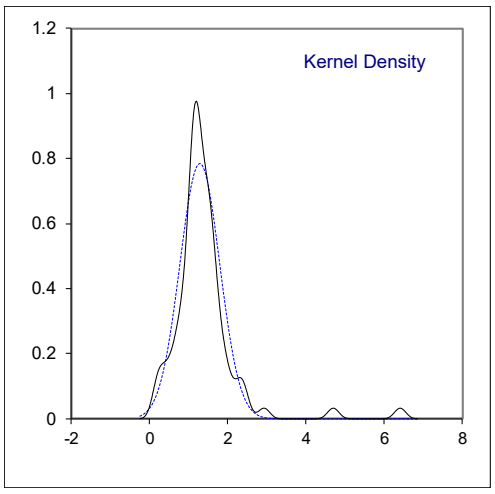
Determination of Monobutyltin (MBT) on sample #20756; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17353	0.88		-1.38	
230	ISO/TS 16179	2.26114	C	3.27	first reported 3.916069
339	In house	1.03		-0.88	
551		2.01		2.42	
623		2.405		3.75	
840	ISO/TS 16179	1.38		0.30	
841	ISO/TS 16179	1.743		1.52	
2115	In house	0.779		-1.72	
2129		-----		-----	
2132	ISO/TS 16179	1.8555		1.90	
2137	KS K0737	1.087	C	-0.69	first reported 1.745
2159		1.22	C	-0.24	first reported 0.246
2165	ISO/TS 16179	1.25		-0.14	
2201	ISO/TS 16179	1.282		-0.03	
2213		-----		-----	
2230		1.40		0.37	
2241	ISO/TS 16179	1.703		1.39	
2250	ISO/TS 16179	1.62		1.11	
2265	ISO/TS 16179	1.275		-0.05	
2271		1.21		-0.27	
2284	ISO/TS 16179	1.435		0.48	
2287	ISO/TS 16179	2.063		2.60	
2293	ISO/TS 16179	0.889		-1.35	
2295		-----		-----	
2297	ISO/TS 16179	1.22		-0.24	
2301	ISO/TS 16179	2.932		5.52	
2310		1.63		1.14	
2311	ISO/TS 16179	1.6383		1.17	
2320	ISO17353	1.388		0.33	
2330	ISO/TS 16179	1.734		1.49	
2347	ISO/TS 16179	1.07		-0.75	
2350	ISO/TS 16179	1.22488		-0.22	
2352	ISO/TS 16179	1.110		-0.61	
2357	ISO/TS 16179	1.12		-0.58	
2358	ISO17353	1.6182		1.10	
2363	ISO/TS 16179	1.1		-0.64	
2365	ISO/TS 16179	1.09		-0.68	
2366	ISO/TS 16179	1.13		-0.54	
2369	ISO/TS 16179	1.00		-0.98	
2370	ISO/TS 16179	1.15		-0.48	
2375	In house	1.5		0.70	
2378	ISO/TS 16179	1.122		-0.57	
2379	ISO/TS 16179	1.9187		2.11	
2380	ISO/TS 16179	0.198	C	-3.68	first reported 0.099
2382	ISO/TS 16179	1.14		-0.51	
2386	ISO/TS 16179	1.443		0.51	
2390	ISO/TS 16179	1.1800		-0.37	
2453	In house	2.31		3.43	
2492	In house	1.506		0.72	
2511	ISO/TS 16179	0.32		-3.27	
2531	ISO/TS 16179	0.851	C	-1.48	first reported 0.08
2560		1.1		-0.64	
2561		-----		-----	
2569		1.54		0.84	
2573	ISO/TS 16179	1.31		0.06	
2590	ISO/TS 16179	2.435		3.85	
2591		-----		-----	
2614		-----		-----	
2643	KS K0737	1.9		2.05	
2658		4.70	R(0.01)	11.48	
2666	ISO/TS 16179	0.4435	C	-2.85	first reported 0.10464
2674	ISO/TS 16179	1.193		-0.33	
2678	ISO/TS 16179	0.955		-1.13	
2741	ISO/TS 16179	0.630		-2.23	
2743	ISO/TS 16179	0.5217		-2.59	
2773	ISO16179	0.497		-2.67	
2804		-----		-----	
2826	ISO/TS 16179	1.3397		0.16	
2858	ISO/TS 16179	1.239		-0.18	
2864	ISO/TS 16179	0.75		-1.82	
2867	ISO/TS 16179	1.213		-0.26	
2870	ISO23161Mod.	1.50		0.70	

lab	method	value	mark	z(targ)	remarks
2947	In house	0.7066666		-1.97	
3116	ISO/TS 16179	1.733		1.49	
3118		-----		-----	
3149		0.71	C	-1.96	first reported 0.09
3153	ISO/TS 16179	1.55		0.87	
3154	ISO/TS 16179	6.41	C,R(0.01)	17.23	first reported 4.643
3172	ISO/TS 16179	0.2095		-3.64	
3176	ISO/TS 16179	1.55		0.87	
3190	ISO/TS 16179	1.484		0.65	
3197	ISO/TS 16179	0.85		-1.49	
3200	In house	1.34		0.16	
3209	ISO/TS 16179	1.613		1.08	
3210		1.18		-0.37	
3214	ISO/TS 16179	1.511		0.74	
3220	ISO/TS 16179	0.251		-3.50	
3228	ISO/TS 16179	1.23		-0.21	
3237	ISO/TS 16179	1.1		-0.64	
3243	In house	1.13		-0.54	
3248	In house	1.46		0.57	
					Only ISO16179:12 *)
	normality	suspect			not OK
	n	81			40
	outliers	2			0
	mean (n)	1.2913			1.2995
	st.dev. (n)	0.50917	RSD=39%		0.50987 RSD=39%
	R(calc.)	1.4257			1.4276
	st.dev.(ISO/TS16179:12)	0.29700			0.29889
	R(ISO/TS16179:12)	0.8316			0.8369

*) Followed ISO16179 with Methanol/Ethanol mixture and extraction temperature 60°C for 60 minutes





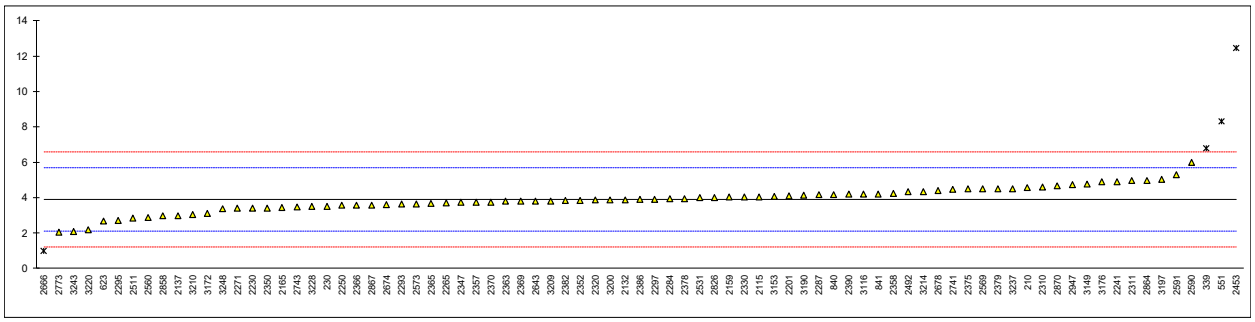
Determination of Dibutyltin (DBT) on sample #20756; results in mg/kg

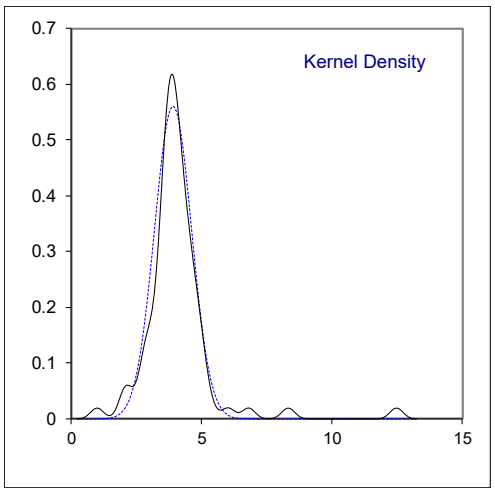
lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO17353	4.57		0.74	
230	ISO/TS 16179	3.510569	C	-0.44	first reported 7.021139
339	In house	6.8	R(0.01)	3.23	
551		8.32	R(0.01)	4.92	
623		2.678		-1.36	
840	ISO/TS 16179	4.18		0.31	
841	ISO/TS 16179	4.224		0.36	
2115	In house	4.057		0.17	
2129		-----		-----	
2132	ISO/TS 16179	3.8918		-0.01	
2137	KS K0737	2.989	C	-1.02	first reported 3.897
2159		4.036	C	0.15	first reported 1.009
2165	ISO/TS 16179	3.44		-0.52	
2201	ISO/TS 16179	4.109		0.23	
2213		-----		-----	
2230		3.42		-0.54	
2241	ISO/TS 16179	4.920		1.13	
2250	ISO/TS 16179	3.57		-0.37	
2265	ISO/TS 16179	3.713		-0.21	
2271		3.40		-0.56	
2284	ISO/TS 16179	3.940		0.04	
2287	ISO/TS 16179	4.178		0.31	
2293	ISO/TS 16179	3.657		-0.27	
2295	ISO/TS 16179	2.72		-1.32	
2297	ISO/TS 16179	3.91		0.01	
2301	ISO/TS 16179	not detected		-----	
2310		4.62		0.80	
2311	ISO/TS 16179	4.9586		1.18	
2320	ISO17353	3.871		-0.04	
2330	ISO/TS 16179	4.052		0.17	
2347	ISO/TS 16179	3.74		-0.18	
2350	ISO/TS 16179	3.42478		-0.53	
2352	ISO/TS 16179	3.850		-0.06	
2357	ISO/TS 16179	3.74		-0.18	
2358	ISO17353	4.2485		0.39	
2363	ISO/TS 16179	3.8		-0.11	
2365	ISO/TS 16179	3.67		-0.26	
2366	ISO/TS 16179	3.59		-0.35	
2369	ISO/TS 16179	3.80		-0.11	
2370	ISO/TS 16179	3.76		-0.16	
2375	In house	4.5		0.67	
2378	ISO/TS 16179	3.961		0.06	
2379	ISO/TS 16179	4.5039		0.67	
2380		-----		-----	
2382	ISO/TS 16179	3.83		-0.08	
2386	ISO/TS 16179	3.900		0.00	
2390	ISO/TS 16179	4.2100		0.34	
2453	In house	12.47	R(0.01)	9.54	
2492	In house	4.336		0.48	
2511	ISO/TS 16179	2.84		-1.18	
2531	ISO/TS 16179	4.00	C	0.11	first reported 0.37
2560		2.9		-1.12	
2561		-----		-----	
2569		4.5		0.67	
2573	ISO/TS 16179	3.66		-0.27	
2590	ISO/TS 16179	6.014		2.35	
2591		5.3		1.56	
2614		-----		-----	
2643	KS K0737	3.8		-0.11	
2658		-----		-----	
2666	ISO/TS 16179	0.9906	C,R(0.01)	-3.24	first reported 0.88922
2674	ISO/TS 16179	3.603		-0.33	
2678	ISO/TS 16179	4.40		0.55	
2741	ISO/TS 16179	4.493		0.66	
2743	ISO/TS 16179	3.4837		-0.47	
2773	ISO16179	2.04		-2.08	
2804		-----		-----	
2826	ISO/TS 16179	4.0111		0.12	
2858	ISO/TS 16179	2.975		-1.03	
2864	ISO/TS 16179	4.96		1.18	
2867	ISO/TS 16179	3.597		-0.34	
2870	ISO23161Mod.	4.67		0.85	

lab	method	value	mark	z(targ)	remarks
2947	In house	4.7433333		0.94	
3116	ISO/TS 16179	4.220		0.35	
3118		-----		-----	
3149		4.77	C	0.97	first reported 2.94
3153	ISO/TS 16179	4.09		0.21	
3154		-----		-----	
3172	ISO/TS 16179	3.1206		-0.87	
3176	ISO/TS 16179	4.91		1.12	
3190	ISO/TS 16179	4.132		0.26	
3197	ISO/TS 16179	5.03		1.26	
3200	In house	3.89		-0.01	
3209	ISO/TS 16179	3.828		-0.08	
3210		3.04		-0.96	
3214	ISO/TS 16179	4.351		0.50	
3220	ISO/TS 16179	2.183		-1.92	
3228	ISO/TS 16179	3.51		-0.44	
3237	ISO/TS 16179	4.51		0.68	
3243	In house	2.084		-2.03	
3248	In house	3.37		-0.59	

				Only ISO16179:12 *)	
	normality	OK		not OK	
	n	77		38	
	outliers	4		1	
	mean (n)	3.9027		4.0285	
	st.dev. (n)	0.71106	RSD=18%	0.60504	RSD=15%
	R(calc.)	1.9910		1.6941	
	st.dev.(ISO/TS16179:12)	0.89762		0.92655	
	R(ISO/TS16179:12)	2.5133		2.5943	

*) Followed ISO16179 with Methanol/Ethanol mixture and extraction temperature 60°C for 60 minutes





APPENDIX 2

Determination of other Organotin components on sample #20755; results in mg/kg

MMT = Monomethyltin / DMT = Dimethyltin / TMT = Trimethyltin / TPT = Tripropyltin / DBT = Dibutyltin / TBT = Tributyltin
TeBT = Tetrabutyltin / MOT = Monooctyltin / DOT = Dioctyltin / TOT = Trioctyltin / DPhT = Diphenyltin / TPhT = Triphenyltin
TCyHT = Tricyclohexyltin

lab	MMT	DMT	TMT	TPT	DBT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
230	n.d.	n.d.	n.d.	n.d.	n.d.	0.188114	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
339	<0.1	<0.1	-----	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	-----	<0.1	<0.1	<0.1
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2115	n.d.	0.023	n.d.	n.d.	n.d.	0.034	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2132	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2159	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2165	-----	-----	-----	-----	n.d.	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2201	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2230	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2241	<0.05	0.05	-----	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-----	<0.05	<0.05	<0.05
2250	-----	<0,025	-----	-----	-----	<0,025	-----	-----	-----	-----	-----	-----	-----
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2271	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2293	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2297	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2301	n.d.	n.d.	n.d.	n.d.	1.831	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2310	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2330	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2347	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2365	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2369	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2370	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2382	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
2386	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2531	-----	-----	-----	-----	-----	nd	-----	-----	-----	-----	-----	-----	-----
2560	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2569	-----	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2573	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2591	-----	-----	n.d.	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.	-----	n.d.	n.d.
2658	-----	-----	-----	-----	1.89	-----	-----	-----	-----	-----	-----	-----	-----
2666	-----	-----	-----	-----	-----	0.02115	-----	-----	-----	-----	-----	-----	-----
2674	-----	-----	-----	-----	n.d.	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2678	-----	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.
2741	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2743	-----	-----	-----	-----	-----	-----	-----	0.1030	-----	-----	-----	-----	-----
2773	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2826	-----	-----	-----	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	-----	<0.1	<0.1
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2864	-----	-----	-----	-----	n.d.	n.d.	n.d.	n.d.	-----	-----	-----	n.d.	n.d.
2867	-----	-----	-----	-----	n.d.	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2947	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3116	-----	-----	-----	-----	-----	0.0229	-----	-----	-----	-----	-----	-----	-----
3153	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3154	-----	-----	-----	-----	3.338	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3200	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3209	-----	<0.1	-----	-----	-----	<0.1	-----	-----	-----	-----	-----	-----	-----
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3214	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
3220	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3228	-----	-----	-----	-----	n.d.	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
3243	n.d.	-----	-----	-----	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	n.d.

Determination of other Organotin components on sample #20756; results in mg/kg

MMT = Monomethyltin / DMT = Dimethyltin / TMT = Trimethyltin / TPT = Tripropyltin / TBT = Tributyltin / TeBT = Tetrabutyltin
MOT= Monooctyltin / DOT = Dioctyltin / TOT = Trioctyltin / DPhT = Diphenyltin / TPhT = Triphenyltin / TCyHT = Tricyclohexyltin

lab	MMT	DMT	TMT	TPT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
230	n.d.	n.d.	n.d.	n.d.	0.264431	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
339	<0.1	<0.1	-----	<0.1	<0.1	<0.1	<0.1	<0.1	-----	<0.1	<0.1	<0.1
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2115	n.d.	0.044	0.032	n.d.	0.038	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2132	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2159	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2165	-----	-----	-----	-----	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2201	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2230	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2241	<0.05	0.06	-----	<0.05	<0.05	<0.05	<0.05	<0.05	-----	<0.05	<0.05	<0.05
2250	-----	0.03	0.03	-----	0.03	-----	-----	-----	-----	-----	-----	-----
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2271	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2293	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2297	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2301	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2310	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2330	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2347	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2365	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2369	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2370	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2379	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2380	-----	0.998	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
2386	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2390	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2531	-----	-----	-----	-----	nd	-----	-----	-----	-----	-----	-----	-----
2560	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2569	-----	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2573	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2591	-----	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.	-----	n.d.	n.d.
2666	-----	-----	-----	-----	1.47861	-----	-----	-----	-----	-----	-----	-----
2674	-----	-----	-----	-----	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2678	-----	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.
2741	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2743	-----	-----	-----	-----	-----	-----	0.8069	-----	-----	-----	-----	-----
2773	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2826	-----	-----	-----	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	-----	<0.1	<0.1
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2864	-----	-----	-----	-----	n.d.	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2867	-----	-----	-----	-----	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
2947	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3116	-----	0.0514	-----	-----	0.3030	-----	-----	-----	-----	-----	-----	-----
3153	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3200	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3209	-----	<0.1	-----	-----	<0.1	-----	-----	-----	-----	-----	-----	-----
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3214	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
3220	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3228	-----	-----	-----	-----	-----	n.d.	n.d.	n.d.	-----	-----	n.d.	n.d.
3243	n.d.	-----	-----	-----	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	n.d.

APPENDIX 3 Analytical details

lab	ISO/IEC 17025 accre-dited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding the buffer	Acidified to pH 4.5
110	---	---	---	---	---	---	---	---	---
210	No	---	---	---	---	---	---	---	---
230	Yes	Further cut	0.5	Ultrasonic	2,2,4 Trimethylpentane	60	60	4.5	Yes
339	No	Used as received	1	Ultrasonic	Acetone	60	40	4.5	Yes
551	---	---	---	---	---	---	---	---	---
623	Yes	Further cut	1	Ultrasonic	methanol-ethanol	60	60	---	---
840	Yes	Further cut	1	Ultrasonic	methanol: ethanol (8:2)	60	60	4.5	No
841	Yes	Used as received	0.5024	Ultrasonic	methanol + ethanol	60	60	4.5	Yes
2115	No	Used as received	1	Ultrasonic	Ethanol/acetic acid 95:5	60	40	5	No
2129	---	---	---	---	---	---	---	---	---
2132	No	Used as received	1	Ultrasonic	0.5% Tropolone in methanol/ethanol (4:1)	60	60	NA	No
2137	Yes	Used as received	3	Ultrasonic	HEXANE	60	60	4.5	Yes
2159	Yes	Used as received	1,0	Ultrasonic	Hexane	60	70	4.5	Yes
2165	Yes	Used as received	1	Ultrasonic	methanol and ethanol	60	60	+/- 4.5	No
2201	Yes	Used as received	0.5	Ultrasonic	MeOH/EtOH (4:1 V/V)	60	60	4.5	No
2213	---	---	---	---	---	---	---	---	---
2230	Yes	Used as received	1	Ultrasonic	1g sodium diethyldithiocarbamate trihydrate in 500mL methane	60	70	4.5	No
2241	Yes	Further cut	+/- 0.5	Ultrasonic	methanol/ethanol mix (80/20 in volume)	60	60	+/- 4.5	No
2250	Yes	Used as received	0,5	Ultrasonic	Methanol/Ethanol (4:1)	60	60	5,2	No
2265	No	Further cut	0,5	Ultrasonic	MeOH / EtOH 80/20	60	60	---	No
2271	Yes	Further cut	0.5	Ultrasonic	Methanol/Ethanol mix (80/20 in volume)	60	60	Medium-strength acidic	No
2284	Yes	Further cut	1	Mechanical Shaking	Methanol: Ethanol=4:1	60	60	---	No
2287	No	Further cut	0.5	Ultrasonic	methanol/ethanol mix 80/20 in volume	60	60	---	---
2293	Yes	Used as received	0.25	Mechanical Shaking & Ultrasonic	Methanol:Ethanol=4:1 V/V	60	60	---	No
2295	Yes	Further cut	1	Ultrasonic	methanol-ethanol mix	60	60	4.5	No
2297	Yes	Used as received	1	Ultrasonic	methanol	60	60	4.5	Yes
2301	No	Used as received	1	Ultrasonic	methanol/ethanol (80/20 v/v)	60	60	+/- 5	Yes
2310	Yes	Further cut	1	Ultrasonic	Acetone	60	40	4.5-5.0	Yes
2311	Yes	Further cut	0.5	Ultrasonic	Acetone	60	40	5.4	Yes
2320	Yes	Further cut	trial1: 0.5 both samples trial2: 0.25 for #20755 and 0.5 #20756	Ultrasonic	MeOH/EtOH(80:20 v/v) and Liquid-liquid extraction with iso-octane for ISO/TS 16179. Acetone for ISO 17353 and subsequently Liquid-liquid extraction with Hexane	60	40°C ISO17353 / 60°C ISO/TS 16179	4.5	No
2330	No	Further cut	0.5	Ultrasonic	EtOH:MeOH ratio 20:80 v/v	60	60±2	N/A	No
2347	Yes	Further cut	0.5	---	---	---	---	---	---
2350	---	---	---	---	---	---	---	---	---
2352	Yes	Further cut	>0.5	Ultrasonic	Methanol Ethanol	60	60	4.50	Yes
2357	---	---	---	---	---	---	---	---	---
2358	Yes	---	---	---	---	---	---	---	---
2363	Yes	Further cut	0.5	Ultrasonic	methanol:ethanol mix=4:1	60	60	4.5	Yes
2365	Yes	Further cut	1	Ultrasonic	methanol / ethanol=1:1	60	60	4.5-4.8	Yes
2366	No	Further cut	1	Ultrasonic	methanol-ethanol (80/20 v/v)	60	60	---	---
2369	---	---	---	---	---	---	---	---	---
2370	Yes	Further cut	1	Ultrasonic	MeOH:EtOH=4:1 mixture	60	60	4.5	Yes
2375	Yes	Further cut	0,5	Ultrasonic	Acetone	60	40	4,5	Yes
2378	Yes	Further cut	0.5	Mechanical Shaking	Isooctane	60	60	4.5	Yes
2379	Yes	Further cut	0.5	Ultrasonic	MeOH : EtOH (80 : 20)	60	60	4.5	Yes
2380	Yes	Used as received	1	Ultrasonic	Methanol	60	60	4.5	No

lab	ISO/IEC 17025 accre-dited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding the buffer	Aci-dified to pH 4.5
2382	Yes	Used as received	0.5	Ultrasonic	16ml methanol+4ml ethanol	60	60	4.5	No
2386	Yes	Used as received	0.5	Ultrasonic	Ethanol/Methanol (80:20, V/V)	60	60	5	No
2390	Yes	Further cut	1	Ultrasonic	methanol/ethanol(1:1)	60	60	4.5	Yes
2453	No	Further cut	2	Ultrasonic	---	---	---	---	---
2492	Yes	Used as received	1	Ultrasonic	Ethanol/glacial acetic acid	60	40	4.5	No
2511	---	---	---	---	---	---	---	---	---
2531	Yes	Used as received	1	Ultrasonic	Methanol/Ethanol	60	60	---	No
2560	Yes	Used as received	0.6	ISO17353	0.03% Sodium diethyl dithiocarbamate	60	30	5.5	Yes
2561	---	---	---	---	---	---	---	---	---
2569	Yes	Used as received	1	Ultrasonic	MEOH:ETOH mix	60	60	5	Yes
2573	Yes	Used as received	0.5	Ultrasonic	methanol/ethanol (4:1)	60	60	4.5	Yes
2590	Yes	Used as received	1	Ultrasonic	meoh/etoh, 80:20 v/v	60	60	4.5	No
2591	No	Used as received	---	Ultrasonic	Acetic Acid/Methanol	10	Room	---	---
2614	---	---	---	---	---	---	---	---	---
2643	Yes	Used as received	0.5	Soxhlet	Methanol, Hexane	60	60	4.5	Yes
2658	Yes	Used as received	---	---	---	---	---	---	---
2666	Yes	Further cut	1,0	Ultrasonic	Methanol(80)/ethanol (20)	60	60	---	Yes
2674	Yes	Used as received	1	Ultrasonic	methanol and ethanol	60	60	4.5	No
2678	No	Further cut	0.5	Ultrasonic	Methanol-Ethanol	60	60	---	---
2741	Yes	Further cut	0.5	Ultrasonic	methanol /ethanol	60	60	---	No
2743	Yes	Used as received	0.75	Ultrasonic	Mix Methanol/Ethanol 80:20 + 1ml Tropolone	60	60	4.5	No
2773	---	---	---	---	---	---	---	---	---
2804	---	---	---	---	---	---	---	---	---
2826	Yes	Used as received	1	Ultrasonic	Methanol/Ethanol, 80:20 in v/v	60	60	4.5	No
2858	Yes	Used as received	0.8035	Ultrasonic	Ethanol+Acetic acid+Tropolone	60	40	4.5	Yes
2864	Yes	Used as received	0.5	Ultrasonic	Methanol-Ethanol(8:2)	60	60	4.5	Yes
2867	Yes	Used as received	1	Ultrasonic	methanol and ethanol	60	60	4.5	Yes
2870	Yes	Used as received	1	Ultrasonic	Ethanol/glacial acetic acid solution 95/5 (v/v): + 0,25 g Tropolone (250 mg/L)	60	40	4.5	No
2947	No	Used as received	1	Mechanical Shaking	Ethanol/acetic acid	60	40	---	No
3116	Yes	Used as received	1	Ultrasonic	methanol/ethanol (80/20 v/v)	60	60	4.5	Yes
3118	---	---	---	---	---	---	---	---	---
3149	Yes	Used as received	1	Ultrasonic	Methanol/Ethanol (v/v/80/20)	60	60	---	Yes
3153	Yes	Further cut	0.5	Ultrasonic	n-hexane	60	60	4.5	No
3154	Yes	Used as received	1	Ultrasonic	MeOH/EtOH	60	70	---	---
3172	---	---	---	---	---	---	---	---	---
3176	Yes	Further cut	1	Ultrasonic	Hexane	60	60	4,5	Yes
3190	Yes	Used as received	1	Ultrasonic	Methanol/Ethanol 4:1 V/V	60	60	5.95	No
3197	Yes	Used as received	1	Ultrasonic	methanol/ethanol	60	60	4,5	No
3200	Yes	Used as received	0.5	Ultrasonic	methanol/ethanol	60	room temp.	4.5	No
3209	Yes	Used as received	1.0025	Ultrasonic	methanol/ethanol mix (80/20 in volume)	60	60	---	Yes
3210	Yes	Further cut	1	Ultrasonic	MeOH/EtOH (80/20)	60	60	4.50	No
3214	Yes	Further cut	1	Ultrasonic	Methanol ethanol mix (80:20 in volume)	60	60	4.5	Yes
3220	Yes	Used as received	1	Ultrasonic	Methanol / Ethanol mix (80/20) AND Isooctane	60	60	5.75	No
3228	Yes	Further cut	0.5	Ultrasonic	methanol and ethanol	60	60	+/- 4.5	No
3237	Yes	Used as received	0,5	Ultrasonic	Methanol%80+Ethanol %20	60	60	4,5	No
3243	No	Further cut	1	Ultrasonic	Methanol / Ethanol	60	60	---	Yes
3248	Yes	Used as received	1	Ultrasonic	METHANOL	60	70	---	No

APPENDIX 4

Number of participants per country

1 lab in	AUSTRIA
3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA
2 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
8 labs in	HONG KONG
8 labs in	INDIA
3 labs in	INDONESIA
6 labs in	ITALY
1 lab in	JAPAN
1 lab in	MAURITIUS
1 lab in	MOROCCO
22 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	PORTUGAL
3 labs in	SOUTH KOREA
1 lab in	SPAIN
1 lab in	SRI LANKA
4 labs in	TAIWAN
1 lab in	THAILAND
2 labs in	TUNISIA
7 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM
3 labs in	VIETNAM

APPENDIX 5

Abbreviations

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

Literature

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