

Results of Proficiency Test
Organotin components in textile
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Organised 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 organisations are for instance: Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL) and Öko-Tex Standard 100 (Switzerland).

Since 2016, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Organotin components in textile every year. During the annual proficiency testing program of 2018/2019, it was decided to continue the proficiency test for the analysis of Organotin components in textile.

In this interlaboratory study 106 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 2018 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 Spijkensisse, the Netherlands was the organiser of the proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, both positive on Organotin and resp. labelled #18660 and #18661. 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 Spijkensisse, the Netherlands, has implemented a quality system based on IEC/ISO17043:2010. 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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

Two different batches of textile were enriched with a number of Organotin components. Each bulk material was cut into pieces and mixed well per batch. For both samples #18660 and #18661, 162 subsamples of 3 grams each were packed.

The homogeneity of sample #18660 was checked by the determination of Dimethyltin (DMT) and the homogeneity of sample #18661 was checked by determination of Tributyltin (TBT) in accordance with test method ISO17353 on 8 stratified randomly selected subsamples for both samples.

	DMT in mg/kg #18660	TBT in mg/kg #18661
sample - 1	2.67	5.43
sample - 2	2.96	5.65
sample - 3	2.75	5.92
sample - 4	2.80	5.83
sample - 5	2.93	6.40
sample - 6	2.42	6.60
sample - 7	3.02	5.34
sample - 8	2.46	5.96

Table 1: homogeneity test results of subsamples #18660 and #18661

From the above test results the relative standard deviations were calculated and compared with 0.3 times the corresponding relative standard deviation of the reference test method, in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	DMT in mg/kg #18660	TBT in mg/kg #18661
RSD% (observed)	8	7
reference test method	ISO16179:12	ISO16179:12
0.3 x RSD% (ref. test method)	7	7

Table 2: evaluation of the relative standard deviation of subsamples #18660 and subsamples #18661

The RSD% for DMT (#18660) and TBT (#18661) are (almost) in agreement with 0.3 times the RSD% of the reference test method. Therefore, homogeneity of the subsamples #18660 and #18661 was assumed.

To each of the participating laboratories 1 sample labelled #18660 with 3 grams of textile and 1 sample labelled #18661 with 3 grams of textile were sent on November 14, 2018.

2.5 ANALYSES

The participants were requested to determine on samples #18660 and #18661 the concentrations of the following Organotin components: Monomethyltin (MMT), Dimethyltin (DMT), Trimethyltin (TMT), Tripropyltin (TPT), Monobutyltin (MBT), Dibutyltin (DBT), Tributyltin (TBT), Tetrabutyltin (TeBT), Monoctyltin (MOT), Dioctyltin (DOT), Trioctyltin (TOT), Diphenyltin (DPhT), Triphenyltin (TPhT) and Tricyclohexyltin (TCyHT) applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited to determine Organotin and to report some analytical details of the test method used.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but to 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 test results cannot be used for meaningful statistical evaluation.

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 appropriate 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 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 the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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. Additional or corrected test results are used for the data analysis and the original 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of 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.

According to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and/or Rosner's 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 and by R(0.01) for the Rosner test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner 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 visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 should be done in order to evaluate whether the reported test results are fit-for-use.

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

During the execution of this proficiency test, no problems occurred with the dispatch of the samples. Six laboratories decided not to report any test results. All other laboratories reported the test results in time. Not all laboratories were able to report all analyses requested. In total 100 participants reported 415 numerical test results. Observed were 7 outlying test results, which is 1.7% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER TEST PER SAMPLE AND PER COMPONENT

In this section, the reported 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 in appendix 1 together with the original data. The abbreviations used in these tables are listed in appendix 5.

For the determination of Organotin in textile ISO/TS16179:12 is recommended to be the test method (see Bluesign v6.0, July 2016 and Oekotex standard, January 2017). In ISO/TS16179 not for all listed Organotin components precision data are available (see table B.1 of ISO/TS16179:12), but only for three Organotin components. Regretfully, the given RSD% for all three components deviate much from each other. Therefore, the RSD% in ISO/TS16179 were compared with the RSD% of the Organotin components in this PT for laboratories that followed ISO/TS16179 by using Methanol/Ethanol as extraction solvent and 60°C as extraction temperature for 60 minutes (see also appendix 1 and table 6). The RSD% of the detected Organotin components are close to the RSD% of 23% for MBT. Therefore, it was decided to use the RSD of 23% for MBT for all Organotin components from ISO/TS16179:12 as reference.

The target reproducibility for each Organotin component will be $2.8 * 23 * \text{mean PT} / 100$.

The test results of laboratory 3150 showed a significant number of statistical outliers. Because the test results are not independent, it was decided to exclude the other test results of this laboratory from statistical evaluation.

Sample #18660

Dimethyltin (DMT): This determination was problematic at a concentration level of 2.84 mg/kg. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the reproducibility requirements of ISO/TS16179:12.

Tributyltin (TBT): This determination was problematic at a concentration level of 0.50 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility requirements of ISO/TS16179:12.

Sample #18661

Dimethyltin (DMT): This determination was not problematic at a concentration level of 0.15 mg/kg. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility requirements of ISO/TS16179:12.

Dibutyltin (DBT): This determination was not problematic at a concentration level of 0.12 mg/kg. No statistical outliers were observed. The calculated reproducibility is in full agreement with the reproducibility requirements of ISO/TS16179:12.

Tributyltin (TBT): This determination was problematic at a concentration level of 2.43 mg/kg. Two statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the reproducibility requirements of ISO/TS16179:12.

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 results, the average test result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility are compared in the next table:

Component	unit	n	average	2.8 * sd	R (target)
Dimethyltin (DMT)	mg/kg	73	2.84	3.15	1.83
Tributyltin (TBT)	mg/kg	92	0.50	0.41	0.32

Table 3: reproducibilities of Organotin components in sample #18660

Component	unit	n	average	2.8 * sd	R (target)
Dimethyltin (DMT)	mg/kg	69	0.15	0.10	0.09
Dibutyltin (DBT)	mg/kg	75	0.12	0.07	0.08
Tributyltin (TBT)	mg/kg	97	2.43	2.08	1.57

Table 4: reproducibilities of Organotin components in sample #18661

Without further statistical calculations, it can be concluded that for the observed Organotin components the group of participating laboratories may have difficulties with the analysis. See also the discussion in paragraph 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2018 WITH THE PREVIOUS PTS

The performance of the determinations of the proficiency test was compared, expressed as the relative standard deviation (RSD) of the PT, see below table.

Component	December 2018	December 2017	December 2016	Reference ISO16179
Dimethyltin (DMT)	25 – 40%	38%	--	23%
Monobutyltin (MBT)	--	--	37%	23%
Dibutyltin (DBT)	21%	35%	--	23%
Tributyltin (TBT)	29 – 31%	29%	--	23%

Table 5: development of uncertainties over the years

The uncertainty observed in this PT showed some improvement in comparison with the uncertainties observed in previous PTs.

4.4 EVALUATION OF ANALYTICAL DETAILS

For this PT, some analysis details were requested (see appendix 3). From the answers given the following can be summarized:

The majority of the participants (77%) is accredited according to ISO/IEC 17025 for the determination of Organotin components in textile.

About 54% of the participants used for intake 1 gram and 33% mentioned to have used 0.5 gram or less.

43% of the participants used a mixture of Methanol and Ethanol and 13% used Acetone as extraction solvent. Eighteen others used a different solvent (e.g. Hexane, iso-Octane).

The majority of the participants (87%) used ultrasonic bath for the extraction. Almost all participants (80%) used an extraction time of 60 minutes.

About 53% of the participants reported to extract at 60°C and 16% to extract at 40°C.

Furthermore, 56% of the group reported to observe a pH of 4.5 / 4.6 and 9 participants reported to observed a pH 5 / 5.6. Only 34% adjust the pH. Some participants mentioned that measuring (and adjusting) the pH is not a part of the test method used.

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 correctly the Organotin components Dimethyltin and Tributyltin in sample #18660 and Dimethyltin, Dibutyltin and Tributyltin in sample #18661.

In this proficiency tests on Organotin components it appeared that the choice of the extraction solvent used and the extraction conditions were important variables. These depend on the test method used. When the test results reported by using Methanol/Ethanol as extraction solvent, 60°C and 60 minutes (which is according ISO/TS16179) were evaluated separately, the following RSD% were calculated.

Component	Sample #18660	Sample #18661
Dimethyltin (DMT)	31%	23%
Dibutyltin (DBT)	--	20%
Tributyltin (TBT)	24%	27%

Table 6: uncertainties of the selected group based on MeOH/EtOH/60°C and 60 minutes test conditions of ISO/TS16179

The above uncertainties are close to the RSD of 23% for Monobutyltin (MBT) mentioned in ISO/TS16179:12 annex B table 1.

When the test results of this interlaboratory study were compared to the Öko-Tex Standard 100 (see table 7), it could be noted that some laboratories would make a different decision about the acceptability of the textile. Four reporting laboratories would accept sample #18660 based on DMT+TBT for all classes (less than 0.5 mg/kg). All other of the reporting laboratories would have rejected sample #18660. Sample #18661 was rejected by all reporting laboratories for too high level of Organotin present.

Öko-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	0.5	1.0	1.0	1.0
DMT, MBT, DBT	1.0	2.0	2.0	2.0

Table 7: Ecolabelling Standard and Requirements for Textiles in EU

For sample #18661 the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to proof the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.

Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias. Also, each test result reported by the laboratories that participate in the PT will

have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time, the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

6 CONCLUSION

Although it is clear that not all laboratories followed the reported test method completely, it can be concluded that the observed variation in this interlaboratory study may not be caused by just one critical point in the analysis. However, it is observed that choice of extraction solvent, extraction time and temperature has a significant effect on the variation.

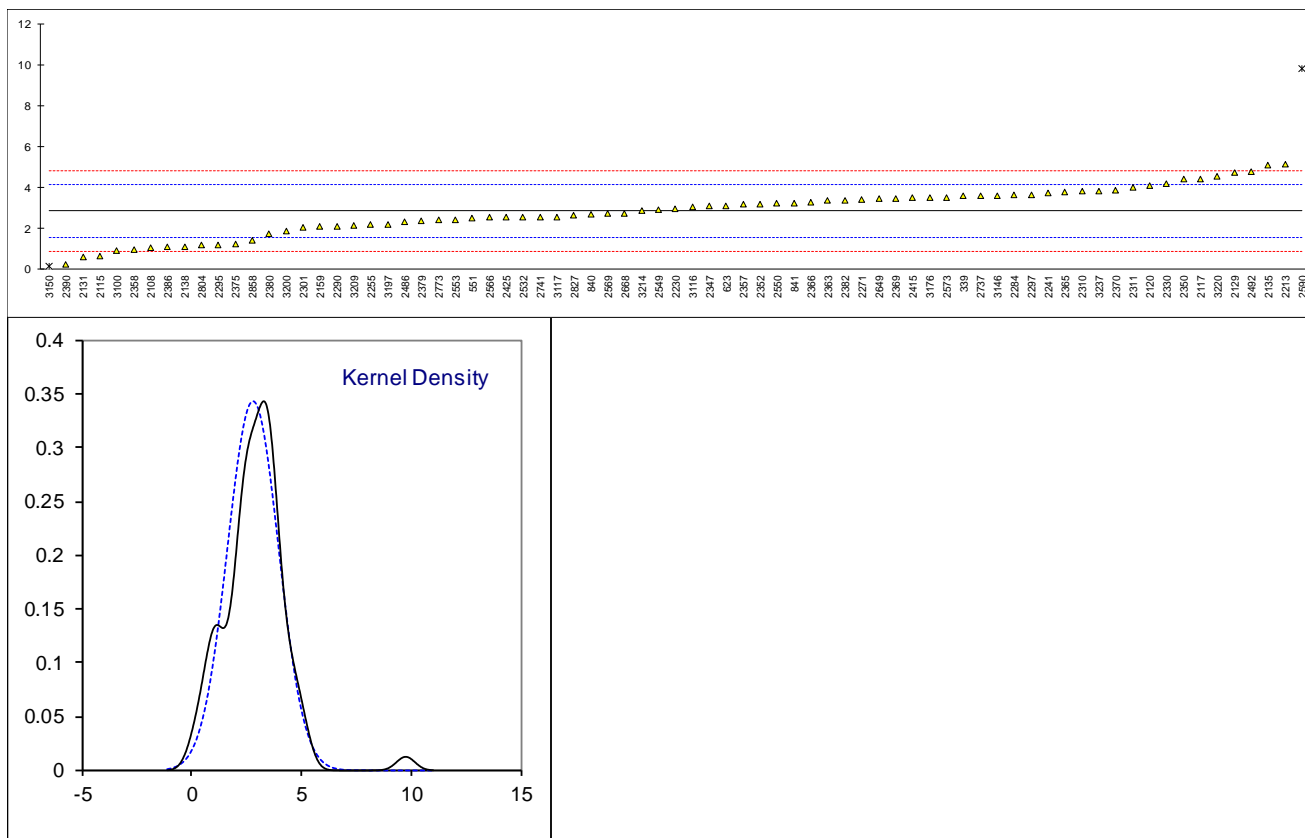
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1**Determination of Dimethyltin (DMT) on sample #18660; results in mg/kg**

lab	method	value	mark	z(targ)	Remarks
110		----		----	
213		----		----	
230		----		----	
339	In house	3.58		1.12	
551	In house	2.5199		-0.50	
623	ISO17353	3.11		0.41	
840	ISO TS 16179	2.67		-0.27	
841	ISO17353	3.25		0.62	
1099	EN71-3	n.a.		----	
2108	In house	1.068		-2.72	
2115	In house	0.654		-3.35	
2117	ISO TS 16179	4.4276		2.42	
2120	ISO TS 16179	4.07		1.87	
2129	ISO TS 16179	4.74		2.90	
2131	In house	0.595		-3.44	
2135		5.10		3.45	
2138	ISO17353	1.081		-2.70	
2159	In house	2.10		-1.14	
2184		----		----	
2213	ISO17353	5.11	C	3.46	First reported 1.26
2217		----		----	
2229		----		----	
2230	ISO17353	2.94		0.15	
2241	ISO TS 16179	3.7236		1.34	
2255	ISO TS 16179	2.19		-1.00	
2265		----		----	
2271	ISO17353	3.410		0.86	
2284	ISO TS 16179	3.6228		1.19	
2290	ISO TS 16179	2.103		-1.13	
2293		----		----	
2295	ISO17353	1.2		-2.51	
2297	ISO TS 16179	3.63		1.20	
2300	ISO TS 16179	ND		----	
2301	ISO17353	2.07		-1.18	
2310	ISO17353	3.80		1.46	
2311	ISO17353	3.999		1.76	
2330	ISO17353	4.1676	C	2.02	First reported 7.6895
2347	ISO TS 16179	3.10		0.39	
2350	ISO TS 16179	4.419		2.41	
2352	ISO TS 16179	3.20		0.54	
2357	ISO TS 16179	3.184		0.52	
2358	ISO17353	0.94		-2.91	
2363	ISO TS 16179	3.35		0.77	
2365	ISO TS 16179	3.752		1.39	
2366	ISO TS 16179	3.290		0.68	
2369	ISO TS 16179	3.47		0.96	
2370	ISO TS 16179	3.86		1.55	
2375	ISO17353	1.24		-2.45	
2379	ISO TS 16179	2.387		-0.70	
2380	ISO17353	1.719		-1.72	
2382	ISO TS 16179	3.362		0.79	
2386	ISO17353	1.079		-2.70	
2390	ISO17353	0.256		-3.96	
2415	ISO TS 16179	3.5		1.00	
2425	ISO TS 16179	2.551		-0.45	
2453		----		----	
2486	ISO TS 16179	2.3435		-0.77	
2492	In house	4.7663		2.94	
2495		----		----	
2497		----		----	
2525		----		----	
2532	ISO TS 16179	2.56		-0.43	
2549	In house	2.902		0.09	
2550	ISO TS 16179	3.23		0.59	
2553	In house	2.41		-0.66	
2561		----		----	
2566	ISO17353	2.54		-0.47	
2569	ISO TS 16179	2.74		-0.16	
2573	ISO TS 16179	3.502		1.00	
2590	ISO TS 16179	9.786	C,R(0.01)	10.61	First reported 11.132
2591		----		----	
2602		----		----	
2629	ISO TS 16179	< 0.2		<-4.04	Possibly a false negative test result?
2649	In house	3.44		0.91	

lab	method	value	mark	z(targ)	Remarks
2668	ISO TS 16179	2.74		-0.16	
2713		----		----	
2719		----		----	
2727		----		----	
2737	ISO TS 16179	3.581		1.13	
2741	ISO TS 16179	2.56		-0.43	
2743		----		----	
2773	ISO TS 16179	2.4		-0.68	
2804	In house	1.19		-2.53	
2826		----		----	
2827	In house	2.65		-0.30	
2845		----		----	
2858	In house	1.399		-2.21	
2864		----		----	
3100	ISO TS 16179	0.9211		-2.94	
3116	ISO TS 16179	3.042		0.30	
3117		2.5672		-0.42	
3118		----		----	
3146	ISO TS 16179	3.61		1.17	
3150	ISO TS 16179	0.1494	ex	-4.12	Result excluded see §4.1
3154		----		----	
3172		----		----	
3176	ISO17353	3.50		1.00	
3197	ISO17353	2.20		-0.99	
3200	In house	1.884		-1.47	
3209	ISO17353	2.121		-1.11	
3210		----		----	
3214	ISO TS 16179	2.870		0.04	
3220	ISO TS 16179	4.562		2.63	
3228		----		----	
3237	ISO TS 16179	3.83		1.51	
3243		----		----	
					<u>Only ISO16179:12 *)</u>
	normality	OK		OK	
	n	73		32	
	outliers	1 (+1excl)		1 (+1excl)	
	mean (n)	2.8445		3.0790	
	st.dev. (n)	1.12637	RSD = 40%	0.94381	RSD = 31%
	R(calc.)	3.1538		2.6427	
	st.dev.(ISO/TS16179:12)	0.65425		0.70817	
	R(ISO/TS16179:12)	1.8319		1.9829	

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes

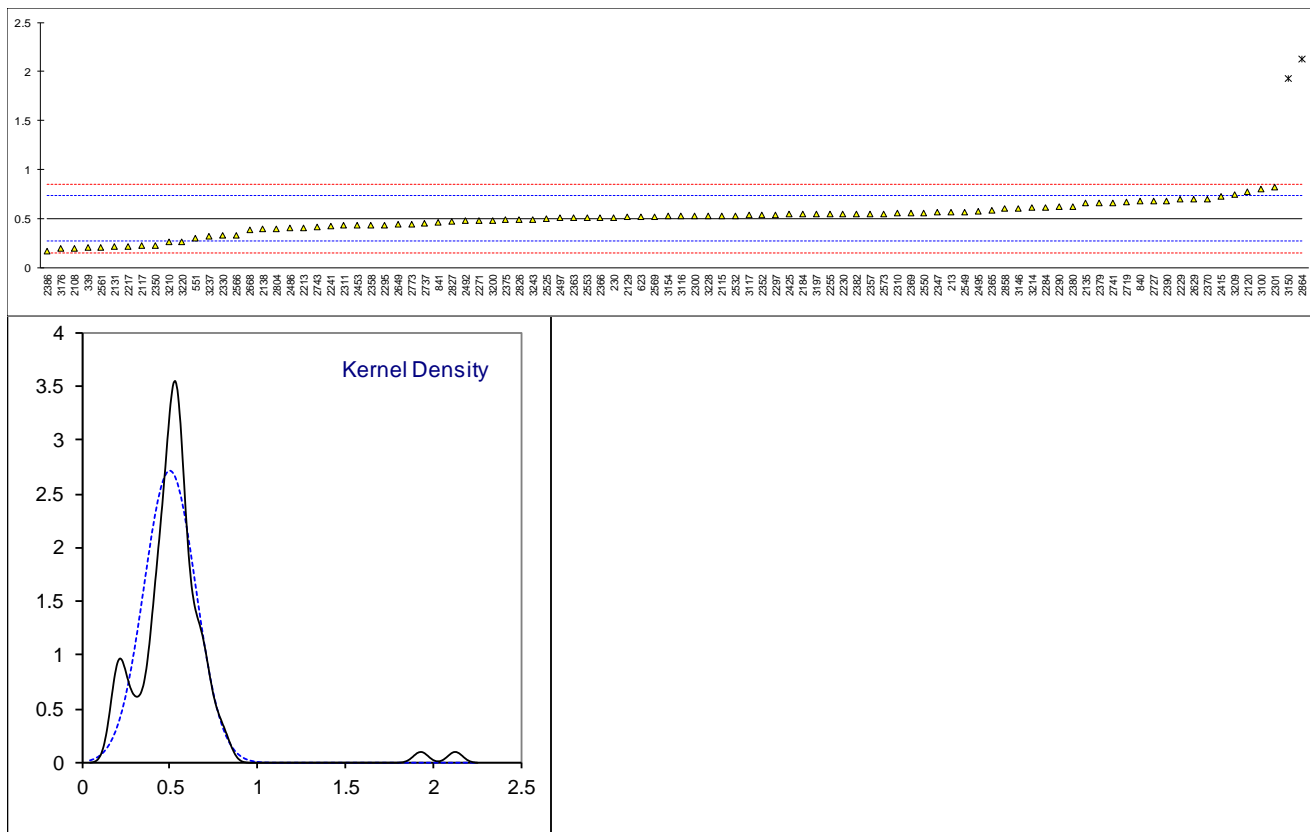


Determination of Tributyltin (TBT) on sample #18660; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
213	ISO17353	0.57		0.58	
230	ISO TS 16179	0.516		0.11	
339	In house	0.21		-2.53	
551	In house	0.3001		-1.75	
623	ISO17353	0.52		0.15	
840	ISO TS 16179	0.68		1.53	
841	ISO17353	0.46		-0.37	
1099	EN71-3	<0.2		----	
2108	In house	0.201		-2.61	
2115	In house	0.531		0.24	
2117	ISO TS 16179	0.2246		-2.41	
2120	ISO TS 16179	0.772		2.33	
2129	ISO TS 16179	0.519		0.14	
2131	In house	0.215		-2.49	
2135		0.66		1.36	
2138	ISO17353	0.396		-0.92	
2159	In house	<0.05		<-3.92	Possibly a false negative test result?
2184	ISO TS 16179	0.55		0.41	
2213	ISO17353	0.41		-0.80	
2217	ISO TS 16179	0.216		-2.48	
2229	ISO TS 16179	0.703		1.73	
2230	ISO17353	0.551		0.42	
2241	ISO TS 16179	0.4269		-0.66	
2255	ISO TS 16179	0.551		0.42	
2265		----		----	
2271	ISO17353	0.481		-0.19	
2284	ISO TS 16179	0.6151		0.97	
2290	ISO TS 16179	0.622		1.03	
2293		----		----	
2295	ISO17353	0.44		-0.54	
2297	ISO TS 16179	0.543		0.35	
2300	ISO TS 16179	0.53		0.23	
2301	ISO17353	0.82		2.74	
2310	ISO17353	0.56		0.49	
2311	ISO17353	0.4373		-0.57	
2330	ISO17353	0.3287		-1.51	
2347	ISO TS 16179	0.57		0.58	
2350	ISO TS 16179	0.229		-2.37	
2352	ISO TS 16179	0.54		0.32	
2357	ISO TS 16179	0.554		0.44	
2358	ISO17353	0.44		-0.54	
2363	ISO TS 16179	0.51		0.06	
2365	ISO TS 16179	0.590		0.75	
2366	ISO TS 16179	0.512		0.08	
2369	ISO TS 16179	0.56		0.49	
2370	ISO TS 16179	0.705		1.75	
2375	ISO17353	0.49		-0.11	
2379	ISO TS 16179	0.660	C	1.36	First reported 0.125
2380	ISO17353	0.624		1.05	
2382	ISO TS 16179	0.551		0.42	
2386	ISO17353	0.175		-2.83	
2390	ISO17353	0.685		1.57	
2415	ISO TS 16179	0.73		1.96	
2425	ISO TS 16179	0.546		0.37	
2453	ISO17353	0.438		-0.56	
2486	ISO TS 16179	0.4064		-0.83	
2492	In house	0.4798		-0.20	
2495	ISO TS 16179	0.5779		0.65	
2497	ISO TS 16179	0.508		0.04	
2525	ISO TS 16179	0.5038		0.01	
2532	ISO TS 16179	0.535		0.28	
2549	In house	0.573		0.61	
2550	ISO TS 16179	0.56		0.49	
2553	In house	0.51		0.06	
2561	ISO TS 16179	0.21		-2.53	
2566	ISO17353	0.337		-1.43	
2569	ISO TS 16179	0.52		0.15	
2573	ISO TS 16179	0.554		0.44	
2590		----		----	
2591	In house	<0.5		----	
2602		----		----	
2629	ISO TS 16179	0.704		1.74	
2649	In house	0.45		-0.46	

lab	method	value	mark	z(targ)	remarks
2668	ISO TS 16179	0.39		-0.98	
2713	In house	<0.5		-----	
2719	ISO TS 16179	0.67	C	1.44	First reported 1.15
2727	ISO TS 16179	0.6801		1.53	
2737	ISO TS 16179	0.455		-0.41	
2741	ISO TS 16179	0.66		1.36	
2743	ISO TS 16179	0.420		-0.72	
2773	ISO TS 16179	0.45		-0.46	
2804	In house	0.40		-0.89	
2826	In house	0.49		-0.11	
2827	In house	0.47		-0.28	
2845		-----		-----	
2858	In house	0.605		0.88	
2864	NIEA T504.30B	2.129	C,R(0.01)	14.06	First reported 3.129
3100	ISO TS 16179	0.8078		2.64	
3116	ISO TS 16179	0.5276		0.21	
3117		0.53731		0.30	
3118		-----		-----	
3146	ISO TS 16179	0.61		0.93	
3150	ISO TS 16179	1.9327	R(0.01)	12.36	
3154	ISO TS 16179	0.527		0.21	
3172		-----		-----	
3176	ISO17353	0.20		-2.62	
3197	ISO17353	0.55		0.41	
3200	In house	0.481		-0.19	
3209	ISO17353	0.751		2.14	
3210	ISO TS 16179	0.266		-2.05	
3214	ISO TS 16179	0.615		0.97	
3220	ISO TS 16179	0.267		-2.04	
3228	ISO TS 16179	0.53		0.23	
3237	ISO TS 16179	0.32		-1.58	
3243	In house	0.492		-0.09	
					Only ISO16179:12 *)
normality	OK			OK	
n	92			42	
outliers	2			1	
mean (n)	0.5029			0.5489	
st.dev. (n)	0.14668	RSD = 29%		0.13117	RSD = 24%
R(calc.)	0.4107			0.3673	
st.dev.(ISO/TS16179:12)	0.11567			0.12624	
R(ISO/TS16179:12)	0.3239			0.3535	

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes

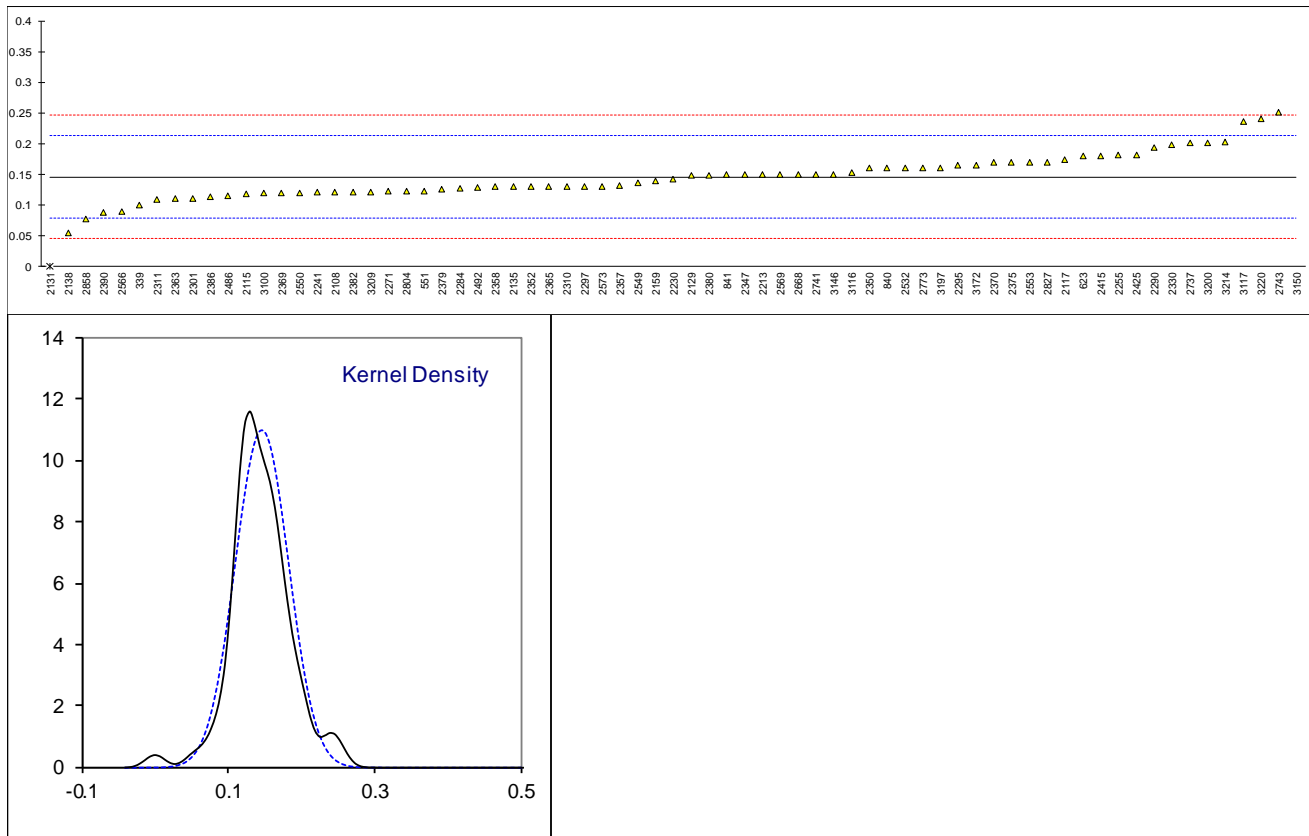


Determination of Dimethyltin (DMT) on sample #18661; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
213		----		----	
230		----		----	
339	In house	0.1		-1.37	
551	In house	0.1231	C	-0.68	First reported 0.5081
623	ISO17353	0.18		1.01	
840	ISO TS 16179	0.16		0.42	
841	ISO17353	0.15		0.12	
1099	EN71-3	n.a.		----	
2108	In house	0.121		-0.74	
2115	In house	0.118		-0.83	
2117	ISO TS 16179	0.1734		0.82	
2120	ISO TS 16179	< 0,30		----	
2129	ISO TS 16179	0.148		0.06	
2131	In house	0	R(0.05)	-4.35	
2135		0.13		-0.48	
2138	ISO17353	0.054		-2.74	
2159	In house	0.14		-0.18	
2184		----		----	
2213	ISO17353	0.15	C	0.12	First reported 0.41
2217		----		----	
2229		----		----	
2230	ISO17353	0.142		-0.12	
2241	ISO TS 16179	0.1208		-0.75	
2255	ISO TS 16179	0.181		1.04	
2265		----		----	
2271	ISO17353	0.123		-0.68	
2284	ISO TS 16179	0.1272		-0.56	
2290	ISO TS 16179	0.193		1.40	
2293		----		----	
2295	ISO17353	0.165		0.57	
2297	ISO TS 16179	0.131		-0.45	
2300	ISO TS 16179	ND		----	
2301	ISO17353	0.11		-1.07	
2310	ISO17353	0.13		-0.48	
2311	ISO17353	0.1088		-1.11	
2330	ISO17353	0.1982	C	1.56	First reported 0.4228
2347	ISO TS 16179	0.15		0.12	
2350	ISO TS 16179	0.160		0.42	
2352	ISO TS 16179	0.13		-0.48	
2357	ISO TS 16179	0.132		-0.42	
2358	ISO17353	0.13		-0.48	
2363	ISO TS 16179	0.11		-1.07	
2365	ISO TS 16179	0.130		-0.48	
2366	ISO TS 16179	< 0.2		----	
2369	ISO TS 16179	0.12		-0.77	
2370	ISO TS 16179	0.169		0.69	
2375	ISO17353	0.17		0.72	
2379	ISO TS 16179	0.125	C	-0.62	First reported 0.660
2380	ISO17353	0.149		0.09	
2382	ISO TS 16179	0.121		-0.74	
2386	ISO17353	0.113		-0.98	
2390	ISO17353	0.088		-1.73	
2415	ISO TS 16179	0.18		1.01	
2425	ISO TS 16179	0.182		1.07	
2453		----		----	
2486	ISO TS 16179	0.1156		-0.90	
2492	In house	0.1292		-0.50	
2495		----		----	
2497		----		----	
2525		----		----	
2532	ISO TS 16179	0.16		0.42	
2549	In house	0.137		-0.27	
2550	ISO TS 16179	0.12		-0.77	
2553	In house	0.17		0.72	
2561		----		----	
2566	ISO17353	0.09		-1.67	
2569	ISO TS 16179	0.15		0.12	
2573	ISO TS 16179	0.131		-0.45	
2590		----		----	
2591		----		----	
2602		----		----	
2629	ISO TS 16179	< 0.2		----	
2649		----		----	

lab	method	value	mark	z(targ)	remarks
2668	ISO TS 16179	0.15		0.12	
2713		-----		-----	
2719		-----		-----	
2727		-----		-----	
2737	ISO TS 16179	0.202		1.67	
2741	ISO TS 16179	0.15		0.12	
2743	ISO TS 16179	0.251		3.13	
2773	ISO TS 16179	0.16		0.42	
2804	In house	0.123		-0.68	
2826		-----		-----	
2827	In house	0.17		0.72	
2845		-----		-----	
2858	In house	0.077		-2.05	
2864		-----		-----	
3100	ISO TS 16179	0.1198		-0.78	
3116	ISO TS 16179	0.1536		0.23	
3117		0.2360	C	2.68	First reported 0
3118		-----		-----	
3146	ISO TS 16179	0.15		0.12	
3150	ISO TS 16179	1.6588	R(0.01)	45.06	
3154		-----		-----	
3172	ISO TS 16179	0.165		0.57	
3176		-----		-----	
3197	ISO17353	0.16		0.42	
3200	In house	0.202		1.67	
3209	ISO17353	0.1211		-0.74	
3210		-----		-----	
3214	ISO TS 16179	0.203		1.70	
3220	ISO TS 16179	0.240		2.80	
3228		-----		-----	
3237		-----		-----	
3243		-----		-----	
					<u>Only ISO16179:12 *)</u>
normality		OK		not OK	
n		69		31	
outliers		2		1	
mean (n)		0.1460	RSD= 25%	0.1538	RSD= 23%
st.dev. (n)		0.03638		0.03490	
R(calc.)		0.1019		0.0977	
st.dev.(ISO/TS16179:12)		0.03358		0.03537	
R(ISO/TS16179:12)		0.0940		0.0990	

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes

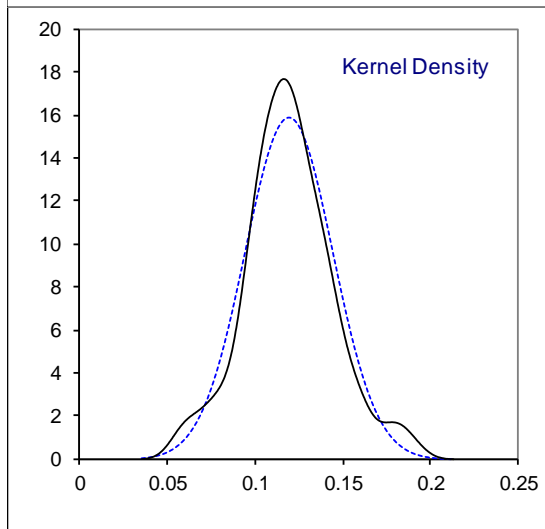
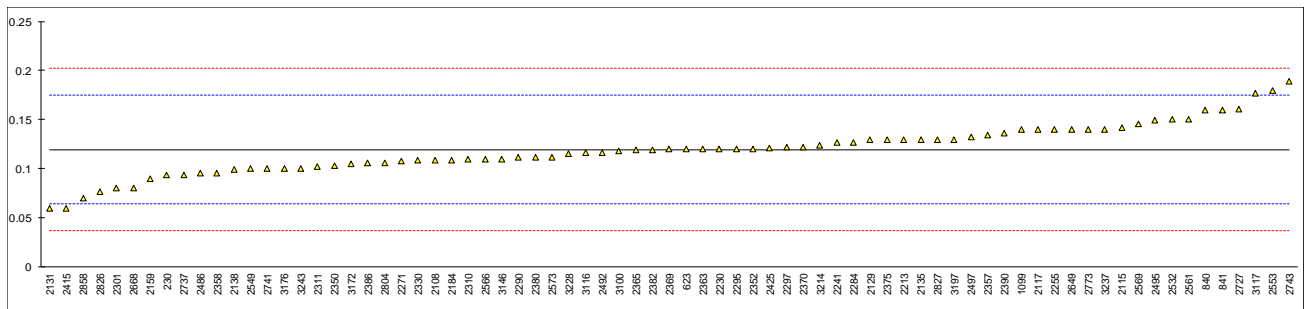


Determination of Dibutyltin (DBT) on sample #18661; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
213		----		----	
230	ISO TS 16179	0.0933		-0.96	
339	In house	<0.1		----	
551	In house	N.D.		----	
623	ISO17353	0.12		0.01	
840	ISO TS 16179	0.16		1.47	
841	ISO17353	0.16		1.47	
1099	EN71-3	0.140		0.74	
2108	In house	0.109		-0.39	
2115	In house	0.142		0.81	
2117	ISO TS 16179	0.1400		0.74	
2120	ISO TS 16179	< 0,30		----	
2129	ISO TS 16179	0.13		0.38	
2131	In house	0.06		-2.17	
2135		0.13		0.38	
2138	ISO17353	0.099		-0.75	
2159	In house	0.09		-1.08	
2184	ISO TS 16179	0.109		-0.39	
2213	ISO17353	0.13		0.38	
2217	ISO TS 16179	nd		----	
2229		----		----	
2230	ISO17353	0.120		0.01	
2241	ISO TS 16179	0.1264		0.25	
2255	ISO TS 16179	0.140		0.74	
2265		----		----	
2271	ISO17353	0.108		-0.42	
2284	ISO TS 16179	0.1265		0.25	
2290	ISO TS 16179	0.112		-0.28	
2293		----		----	
2295	ISO17353	0.12		0.01	
2297	ISO TS 16179	0.122		0.09	
2300	ISO TS 16179	ND		----	
2301	ISO17353	0.08		-1.44	
2310	ISO17353	0.11		-0.35	
2311	ISO17353	0.1025		-0.62	
2330	ISO17353	0.1084		-0.41	
2347	ISO TS 16179	<0.2		----	
2350	ISO TS 16179	0.103		-0.60	
2352	ISO TS 16179	0.12		0.01	
2357	ISO TS 16179	0.134		0.52	
2358	ISO17353	0.096		-0.86	
2363	ISO TS 16179	0.12		0.01	
2365	ISO TS 16179	0.119		-0.02	
2366	ISO TS 16179	< 0.2		----	
2369	ISO TS 16179	0.12		0.01	
2370	ISO TS 16179	0.122		0.09	
2375	ISO17353	0.13		0.38	
2379	ISO TS 16179	Not detected		----	
2380	ISO17353	0.112		-0.28	
2382	ISO TS 16179	0.119		-0.02	
2386	ISO17353	0.106		-0.49	
2390	ISO17353	0.136		0.60	
2415	ISO TS 16179	0.06		-2.17	
2425	ISO TS 16179	0.121		0.05	
2453		----		----	
2486	ISO TS 16179	0.0958		-0.87	
2492	In house	0.1168		-0.10	
2495	ISO TS 16179	0.1492		1.08	
2497	ISO TS 16179	0.132		0.45	
2525	ISO TS 16179	<0,05		----	
2532	ISO TS 16179	0.15		1.11	
2549	In house	0.1		-0.71	
2550		----		----	
2553	In house	0.18		2.20	
2561	ISO TS 16179	0.15		1.11	
2566	ISO17353	0.11		-0.35	
2569	ISO TS 16179	0.146		0.96	
2573	ISO TS 16179	0.112		-0.28	
2590		----		----	
2591	In house	<0.5		----	
2602		----		----	
2629	ISO TS 16179	< 0.2		----	
2649	In house	0.14		0.74	

lab	method	value	mark	z(targ)	remarks
2668	ISO TS 16179	0.08		-1.44	
2713	In house	<0.5		----	
2719		----		----	
2727	ISO TS 16179	0.1604		1.48	
2737	ISO TS 16179	0.094		-0.93	
2741	ISO TS 16179	0.10		-0.71	
2743	ISO TS 16179	0.189	C	2.52	First reported 0.266
2773	ISO TS 16179	0.14		0.74	
2804	In house	0.106		-0.49	
2826	In house	0.077		-1.55	
2827	In house	0.13		0.38	
2845		----		----	
2858	In house	0.070		-1.80	
2864		----		----	
3100	ISO TS 16179	0.1182		-0.05	
3116	ISO TS 16179	0.1162		-0.12	
3117		0.1770	C	2.09	First reported 0
3118		----		----	
3146	ISO TS 16179	0.11		-0.35	
3150	ISO TS 16179	<0,10		----	
3154		----		----	
3172	ISO TS 16179	0.105		-0.53	
3176	ISO17353	0.10		-0.71	
3197	ISO17353	0.13		0.38	
3200		----		----	
3209		----		----	
3210	ISO TS 16179	<0.5	C	----	First reported 0.258
3214	ISO TS 16179	0.124		0.16	
3220	ISO TS 16179	Not Detected		----	
3228	ISO TS 16179	0.115		-0.17	
3237	ISO TS 16179	0.14		0.74	
3243	In house	0.10		-0.71	
					<u>Only ISO16179:12 *)</u>
	normality	OK			suspect
	n	75			32
	outliers	0			0
	mean (n)	0.1196	RSD= 21%	0.1233	RSD= 20%
	st.dev. (n)	0.02506		0.02416	
	R(calc.)	0.0702		0.0677	
	st.dev.(ISO/TS16179:12)	0.02751		0.02836	
	R(ISO/TS16179:12)	0.0770		0.0794	

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes

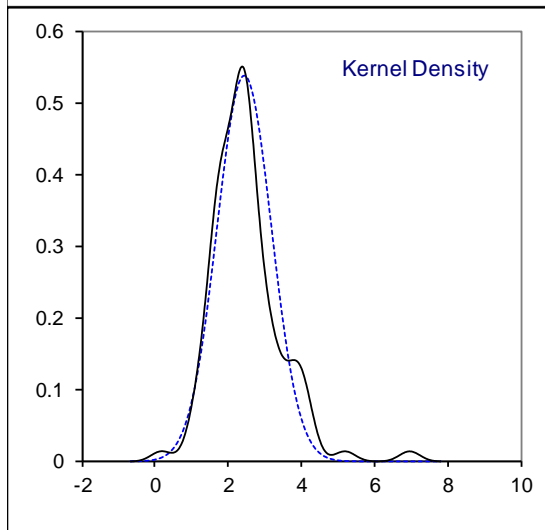
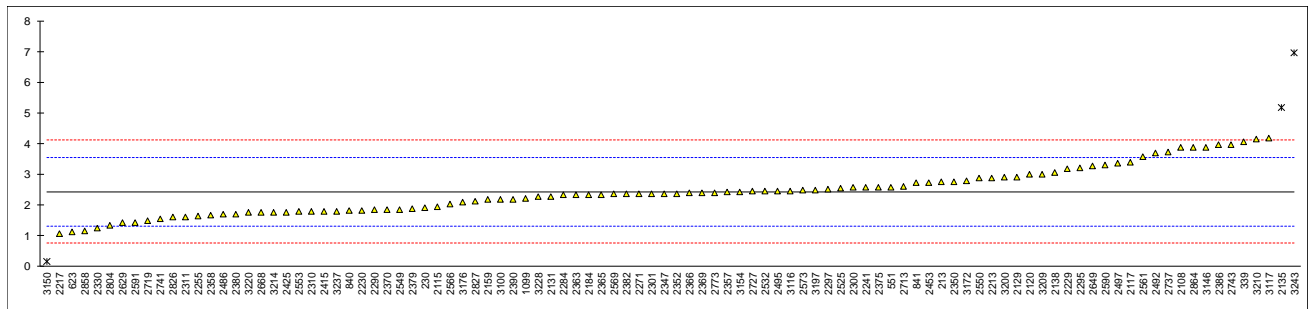


Determination of Tributyltin (TBT) on sample #18661; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
213	ISO17353	2.75		0.57	
230	ISO TS 16179	1.914		-0.92	
339	In house	4.07		2.93	
551	In house	2.5707		0.25	
623	ISO17353	1.11		-2.36	
840	ISO TS 16179	1.83		-1.07	
841	ISO17353	2.72		0.52	
1099	EN71-3	2.19895		-0.41	
2108	In house	3.865		2.57	
2115	In house	1.948		-0.86	
2117	ISO TS 16179	3.3975		1.73	
2120	ISO TS 16179	3.01		1.04	
2129	ISO TS 16179	2.917		0.87	
2131	In house	2.285		-0.26	
2135		5.18	R(0.05)	4.92	
2138	ISO17353	3.066		1.14	
2159	In house	2.18		-0.45	
2184	ISO TS 16179	2.341		-0.16	
2213	ISO17353	2.89		0.82	
2217	ISO TS 16179	1.06		-2.45	
2229	ISO TS 16179	3.183		1.35	
2230	ISO17353	1.83		-1.07	
2241	ISO TS 16179	2.5641		0.24	
2255	ISO TS 16179	1.652		-1.39	
2265		-----		-----	
2271	ISO17353	2.362		-0.12	
2284	ISO TS 16179	2.3292		-0.18	
2290	ISO TS 16179	1.841		-1.05	
2293		-----		-----	
2295	ISO17353	3.2		1.38	
2297	ISO TS 16179	2.51		0.14	
2300	ISO TS 16179	2.56		0.23	
2301	ISO17353	2.37		-0.11	
2310	ISO17353	1.80		-1.13	
2311	ISO17353	1.6147		-1.46	
2330	ISO17353	1.2543		-2.10	
2347	ISO TS 16179	2.37		-0.11	
2350	ISO TS 16179	2.754		0.58	
2352	ISO TS 16179	2.37		-0.11	
2357	ISO TS 16179	2.423		-0.01	
2358	ISO17353	1.66		-1.38	
2363	ISO TS 16179	2.34		-0.16	
2365	ISO TS 16179	2.343		-0.16	
2366	ISO TS 16179	2.39		-0.07	
2369	ISO TS 16179	2.39		-0.07	
2370	ISO TS 16179	1.86		-1.02	
2375	ISO17353	2.57		0.25	
2379	ISO TS 16179	1.876		-0.99	
2380	ISO17353	1.696		-1.31	
2382	ISO TS 16179	2.361		-0.12	
2386	ISO17353	3.965		2.74	
2390	ISO17353	2.194		-0.42	
2415	ISO TS 16179	1.8		-1.13	
2425	ISO TS 16179	1.771		-1.18	
2453	ISO17353	2.721		0.52	
2486	ISO TS 16179	1.6942		-1.32	
2492	In house	3.6906		2.25	
2495	ISO TS 16179	2.4533		0.04	
2497	ISO TS 16179	3.348		1.64	
2525	ISO TS 16179	2.5593		0.23	
2532	ISO TS 16179	2.45		0.03	
2549	In house	1.864		-1.01	
2550	ISO TS 16179	2.88		0.80	
2553	In house	1.78		-1.16	
2561	ISO TS 16179	3.57		2.04	
2566	ISO17353	2.04		-0.70	
2569	ISO TS 16179	2.35		-0.14	
2573	ISO TS 16179	2.473		0.08	
2590	ISO TS 16179	3.305		1.56	
2591	In house	1.42		-1.81	
2602		-----		-----	
2629	ISO TS 16179	1.414		-1.82	
2649	In house	3.26		1.48	

lab	method	value	mark	z(targ)	remarks
2668	ISO TS 16179	1.75		-1.22	
2713	In house	2.61		0.32	
2719	ISO TS 16179	1.50		-1.66	
2727	ISO TS 16179	2.441		0.02	
2737	ISO TS 16179	3.734		2.33	
2741	ISO TS 16179	1.55		-1.58	
2743	ISO TS 16179	3.971	C	2.76	First reported 6.434
2773	ISO TS 16179	2.4		-0.05	
2804	In house	1.35		-1.93	
2826	In house	1.6		-1.49	
2827	In house	2.11		-0.57	
2845		-----		-----	
2858	In house	1.146		-2.30	
2864	NIEA T504.30B	3.8694		2.57	
3100	ISO TS 16179	2.1807		-0.45	
3116	ISO TS 16179	2.456		0.05	
3117		4.18879		3.15	
3118		-----		-----	
3146	ISO TS 16179	3.87		2.57	
3150	ISO TS 16179	0.1708	ex	-4.04	Result excluded see §4.1
3154	ISO TS 16179	2.43		0.00	
3172	ISO TS 16179	2.781		0.63	
3176	ISO17353	2.10		-0.59	
3197	ISO17353	2.49		0.11	
3200	In house	2.896		0.83	
3209	ISO17353	3.012		1.04	
3210	ISO TS 16179	4.153		3.08	
3214	ISO TS 16179	1.766		-1.19	
3220	ISO TS 16179	1.747		-1.22	
3228	ISO TS 16179	2.26		-0.31	
3237	ISO TS 16179	1.80		-1.13	
3243	In house	6.947	R(0.01)	8.08	
					<u>Only ISO16179:12 *)</u>
normality		OK		OK	
n		97		42	
outliers		2 (+1excl)		2	
mean (n)		2.4305	RSD= 31%	2.3541	RSD= 27%
st.dev. (n)		0.74331		0.63829	
R(calc.)		2.0813		1.7872	
st.dev.(ISO/TS16179:12)		0.55902		0.54144	
R(ISO/TS16179:12)		1.5653		1.5160	

*) Followed ISO16179 with Methanol/Ethanol mix and extraction temp 60°C for 60 minutes



APPENDIX 2**Determination of other Organotin components on sample #18660; results in mg/kg**

lab	MMT	TMT	TPT	MBT	DBT	TeBT
110	----	----	----	----	----	----
213	----	----	----	----	----	----
230	----	----	----	----	----	----
339	----	----	----	<0.1	<0.1	<0.1
551	----	----	----	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND	ND
1099	n.a.	n.a.	n.a.	<0.2	<0.2	<0.2
2108	----	----	----	----	----	----
2115	----	----	----	----	----	----
2117	----	----	----	0.0100	0.0074	----
2120	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2131	0	0	0	0	0	0
2135	----	----	----	----	----	----
2138	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2159	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2184	----	----	----	not detected	not detected	not detected
2213	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2217	----	----	----	nd	nd	nd
2229	----	----	----	----	----	----
2230	<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
2255	nd	nd	nd	nd	nd	nd
2265	----	----	----	----	----	----
2271	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2284	----	----	----	----	----	----
2290	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2293	----	----	----	----	----	----
2295	----	----	----	----	----	----
2297	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2300	ND	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND	ND
2310	0.09	0.09	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2311	0.0738	0.0738	Not Detected	Not Detected	Not Detected	Not Detected
2330	0.084	0.084	ND	ND	ND	ND
2347	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2350	----	----	----	----	----	0.382
2352	----	----	----	----	----	----
2357	ND	ND	ND	ND	ND	ND
2358	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2363	ND	ND	ND	ND	ND	ND
2365	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2366	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2369	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	0.0641	0.0641	n.d.	n.d.	n.d.	n.d.
2375	----	----	----	----	----	----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	0.099	0.099	----	----	----	----
2382	----	----	----	----	----	----
2386	0.010	0.010	<0,01	<0,01	<0,01	<0,01
2390	----	----	----	----	----	----
2415	----	----	----	----	----	----
2425	ND	ND	ND	ND	ND	ND
2453	----	----	----	----	----	----
2486	ND	ND	ND	ND	ND	ND
2492	0.0400	0.0400	----	----	----	----
2495	----	----	----	0.0282	<0.02	<0.02
2497	2.39	2.39	----	----	----	----
2525	----	----	----	<0,05	<0,05	<0,05
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	ND	ND	ND	ND	ND	ND
2550	----	----	----	----	----	----
2553	ND	ND	ND	ND	ND	ND
2561	----	----	----	----	----	----
2566	ND	ND	ND	ND	ND	ND
2569	ND	ND	----	ND	ND	ND
2573	----	----	----	----	----	----
2590	----	----	----	----	----	----
2591	<0.5	<0.5	<0.5	----	<0.5	<0.5
2602	----	----	----	----	----	----
2629	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2649	----	----	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected

lab	MMT	TMT	TPT	MBT	DBT	TeBT
2713	----	----	----	----	<0.5	----
2719	----	----	----	----	----	----
2727	----	----	----	0.0160	0.0764	----
2737	0.084	0.084	----	ND	ND	ND
2741	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2743	----	----	----	----	----	----
2773	ND	ND	ND	ND	ND	ND
2804	----	----	----	----	----	----
2826	----	----	----	----	----	----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2845	----	----	----	----	----	----
2858	n.d	n.d	n.d	n.d	n.d	n.d
2864	----	----	----	----	----	----
3100	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3116	----	----	----	----	0.02515	----
3117	ND	ND	ND	ND	ND	ND
3118	----	----	----	----	----	----
3146	----	----	----	----	----	----
3150	<0,10	<0,10	<0,10	<0,10	0.1304	<0,10
3154	----	----	----	----	----	----
3172	----	----	----	----	----	----
3176	----	----	----	----	----	----
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3200	----	----	----	----	----	----
3209	----	----	----	----	----	----
3210	----	----	----	----	----	----
3214	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3228	----	----	----	N.D.	N.D.	N.D.
3237	----	----	----	----	----	----
3243	----	----	----	n.d.	n.d.	n.d.

MMT = Monomethyltin
TMT = Trimethyltin
TPT = Tripropyltin
MBT = Monobutyltin
DBT = Dibutyltin
TeBT = Tetrabutyltin

Determination of other Organotin components on sample #18660; results in mg/kg == continued

lab	MOT	DOT	TOT	DPhT	TPhT	TCyHT
110	----	----	----	----	----	----
213	----	----	----	----	----	----
230	----	----	----	----	----	----
339	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND	ND
1099	<0.2	<0.2	n.a.	<0.2	<0.2	n.a.
2108	----	----	----	----	----	----
2115	----	----	----	----	----	----
2117	----	----	----	----	----	----
2120	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2131	0	0	0	0	0	----
2135	----	----	----	----	----	----
2138	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2159	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2184	not detected	not detected	----	----	not detected	not detected
2213	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2217	nd	nd	----	----	nd	nd
2229	----	----	----	----	----	----
2230	<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
2255	nd	nd	nd	nd	nd	nd
2265	----	----	----	----	----	----
2271	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2284	----	----	----	----	----	----
2290	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2293	----	----	----	----	----	----
2295	----	----	----	----	----	----
2297	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2300	ND	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND	ND
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ND	ND	ND	ND	ND	ND
2347	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2350	----	----	----	----	----	----
2352	----	----	----	----	----	----
2357	ND	ND	ND	ND	ND	ND
2358	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2363	ND	ND	ND	ND	ND	ND
2365	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2366	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2369	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	----	----	----	----	----	----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	----	----	----	----	----	----
2382	----	----	----	----	----	----
2386	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01
2390	----	----	----	----	----	----
2415	----	----	----	----	----	----
2425	ND	ND	ND	ND	ND	ND
2453	----	----	----	----	----	----
2486	ND	ND	ND	ND	ND	ND
2492	----	----	----	----	----	----
2495	<0.02	<0.02	----	----	<0.02	<0.02
2497	----	----	----	----	----	----
2525	<0,05	<0,05	----	----	<0,05	<0,05
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	ND	ND	ND	ND	ND	ND
2550	----	----	----	----	----	----
2553	ND	ND	ND	ND	ND	ND
2561	----	----	----	----	----	----
2566	ND	ND	ND	ND	ND	ND
2569	ND	ND	ND	ND	ND	ND
2573	----	----	----	----	----	----
2590	----	----	----	----	----	----
2591	----	<0.5	<0.5	----	<0.5	<0.5
2602	----	----	----	----	----	----
2629	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2649	----	----	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected

lab	MOT	DOT	TOT	DPhT	TPhT	TCyHT
2713	----	<0.5	----	----	<0.5	----
2719	----	----	----	----	----	----
2727	----	----	----	----	----	----
2737	ND	ND	ND	ND	ND	ND
2741	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2743	----	----	----	----	----	----
2773	ND	ND	ND	ND	ND	ND
2804	----	----	----	----	----	----
2826	----	----	----	----	----	----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2845	----	----	----	----	----	----
2858	n.d	n.d	n.d	n.d	n.d	n.d
2864	----	----	----	----	----	----
3100	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3116	----	----	----	----	----	----
3117	ND	ND	ND	ND	ND	ND
3118	----	----	----	----	----	----
3146	----	----	----	----	----	----
3150	<0,10	<0,10	<0,10	<0,10	<0,10	<0,10
3154	----	----	----	----	----	----
3172	----	----	----	----	----	----
3176	----	----	----	----	----	----
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3200	----	----	----	----	----	----
3209	----	----	----	----	----	----
3210	----	----	----	----	----	----
3214	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3228	N.D.	N.D.	----	----	N.D.	N.D.
3237	----	----	----	----	----	----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

MOT = Monooctyltin
DOT = Dioctyltin
TOT = Trioctyltin
DPhT = Diphenyltin
TPhT = Triphenyltin
TCyHT = Tricyclohexyltin

Determination of other Organotin components on sample #18661; results in mg/kg

lab	MMT	TMT	TPT	MBT	TeBT	MOT
110	----	----	----	----	----	----
213	----	----	----	----	----	----
230	----	----	----	----	----	----
339	<0.1	----	----	<0.1	<0.1	<0.1
551	N.D.	----	----	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND	ND
1099	<0.2	n.a.	n.a.	<0.2	<0.2	<0.2
2108	----	----	----	----	----	----
2115	----	----	----	----	----	----
2117	----	----	----	0.0445	----	----
2120	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2131	0	0	0	0	0	0
2135	----	----	----	----	----	----
2138	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2159	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2184	----	----	----	not detected	not detected	not detected
2213	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2217	----	----	----	nd	nd	nd
2229	----	----	----	----	----	----
2230	<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
2255	nd	nd	nd	nd	nd	nd
2265	----	----	----	----	----	----
2271	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2284	----	----	----	----	----	----
2290	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2293	----	----	----	----	----	----
2295	----	----	----	----	----	----
2297	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2300	ND	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND	ND
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ND	ND	ND	ND	ND	ND
2347	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2350	----	----	----	----	0.344	----
2352	----	----	----	----	----	----
2357	ND	ND	ND	ND	ND	ND
2358	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2363	ND	ND	ND	ND	ND	ND
2365	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2366	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2369	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	----	----	----	----	----	----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	----	----	----	----	----	----
2382	----	----	----	----	----	----
2386	<0,01	0.007	<0,01	0.010	<0,01	<0,01
2390	----	----	----	----	----	----
2415	----	----	----	----	----	----
2425	ND	ND	ND	ND	ND	ND
2453	----	----	----	----	----	----
2486	ND	ND	ND	ND	ND	ND
2492	----	----	----	----	----	----
2495	----	----	----	0.0255	<0.02	<0.02
2497	----	0.111	----	----	----	----
2525	----	----	----	<0,05	<0,05	<0,05
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	ND	ND	ND	ND	ND	ND
2550	----	----	----	----	----	----
2553	ND	ND	ND	ND	ND	ND
2561	----	----	----	----	----	----
2566	ND	ND	ND	ND	ND	ND
2569	----	ND	----	ND	ND	ND
2573	----	----	----	----	----	----
2590	----	----	----	----	----	----
2591	----	<0.5	<0.5	----	<0.5	----
2602	----	----	----	----	----	----
2629	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2649	----	----	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected

lab	MMT	TMT	TPT	MBT	TeBT	MOT
2713	----	----	----	----	----	----
2719	----	----	----	----	----	----
2727	----	----	----	0.0349	----	----
2737	ND	ND	----	ND	ND	ND
2741	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2743	----	----	----	----	----	----
2773	ND	ND	ND	ND	ND	ND
2804	----	----	----	----	----	----
2826	----	----	----	----	----	----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2845	----	----	----	----	----	----
2858	n.d	n.d	n.d	n.d	n.d	n.d
2864	----	----	----	----	----	----
3100	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3116	----	----	----	0.0311	----	----
3117	ND	ND	ND	ND	ND	ND
3118	----	----	----	----	----	----
3146	----	----	----	----	----	----
3150	<0,10	<0,10	<0,10	<0,10	<0,10	<0,10
3154	----	----	----	0.258	----	----
3172	----	----	----	----	----	----
3176	----	----	----	----	----	----
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3200	----	----	----	----	----	----
3209	----	----	----	----	----	----
3210	----	----	----	----	----	----
3214	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3228	----	----	----	N.D.	N.D.	N.D.
3237	----	----	----	----	----	----
3243	n.d.	----	----	n.d.	n.d.	n.d.

MMT = Monomethyltin
 TMT = Trimethyltin
 TPT = Tripropyltin
 MBT = Monobutyltin
 TeBT = Tetrabutyltin
 MOT = Monoocetyl tin

Determination of other Organotin components on sample #18661; results in mg/kg == continued

lab	DOT	TOT	DPhT	TPhT	TCyHT
110	----	----	----	----	----
213	----	----	----	----	----
230	----	----	----	----	----
339	<0.1	<0.1	<0.1	<0.1	<0.1
551	N.D.	N.D.	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND
1099	<0.2	n.a.	<0.2	<0.2	n.a.
2108	----	----	----	----	----
2115	----	----	----	----	----
2117	----	----	----	----	----
2120	< 0,30	< 0,30	< 0,30	< 0,30	< 0,30
2129	<0,1	<0,1	<0,1	<0,1	<0,1
2131	0	0	0	0	0
2135	----	----	----	----	----
2138	n.d.	n.d.	n.d.	n.d.	n.d.
2159	<0.05	<0.05	<0.05	<0.05	<0.05
2184	not detected	----	----	not detected	not detected
2213	<0.1	<0.1	<0.1	<0.1	<0.1
2217	nd	----	----	nd	nd
2229	----	----	----	----	----
2230	<0.025	<0.025	<0.025	<0.025	<0.025
2241	<0.05	<0.05	<0.05	<0.05	<0.05
2255	nd	nd	nd	nd	nd
2265	----	----	----	----	----
2271	<0.025	<0.025	<0.025	<0.025	<0.025
2284	----	----	----	----	----
2290	<0.05	<0.05	<0.05	<0.05	<0.05
2293	----	----	----	----	----
2295	----	----	----	----	----
2297	<0.1	<0.1	<0.1	<0.1	<0.1
2300	ND	ND	ND	ND	ND
2301	ND	ND	ND	ND	ND
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ND	ND	ND	ND	ND
2347	<0.2	<0.2	<0.2	<0.2	<0.2
2350	----	----	----	----	----
2352	----	----	----	----	----
2357	ND	ND	ND	ND	ND
2358	<0.02	<0.02	<0.02	<0.02	<0.02
2363	ND	ND	ND	ND	ND
2365	<0.02	<0.02	<0.02	<0.02	<0.02
2366	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2369	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.
2375	----	----	----	----	----
2379	Not detected	Not detected	Not detected	Not detected	Not detected
2380	----	----	----	----	----
2382	----	----	----	----	----
2386	<0,01	<0,01	<0,01	<0,01	<0,01
2390	----	----	----	----	----
2415	----	----	----	----	----
2425	ND	ND	ND	ND	ND
2453	----	----	----	----	----
2486	ND	ND	ND	ND	ND
2492	----	----	----	----	----
2495	<0.02	----	----	<0.02	<0.02
2497	----	----	----	----	----
2525	<0,05	----	----	<0,05	<0,05
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	ND	ND	ND	ND	ND
2550	----	----	----	----	----
2553	ND	ND	ND	ND	ND
2561	----	----	----	----	----
2566	ND	ND	ND	ND	ND
2569	ND	ND	ND	ND	ND
2573	----	----	----	----	----
2590	----	----	----	----	----
2591	<0.5	<0.5	----	<0.5	<0.5
2602	----	----	----	----	----
2629	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2649	----	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected

lab	DOT	TOT	DPhT	TPhT	TCyHT
2713	<0.5	----	----	<0.5	----
2719	----	----	----	----	----
2727	----	----	----	----	----
2737	ND	ND	ND	ND	ND
2741	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2743	----	----	----	----	----
2773	ND	ND	ND	ND	ND
2804	----	----	----	----	----
2826	----	----	----	----	----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2845	----	----	----	----	----
2858	n.d	n.d	n.d	n.d	n.d
2864	----	----	----	----	----
3100	<0.05	<0.05	<0.05	<0.05	<0.05
3116	----	----	----	----	----
3117	ND	ND	ND	ND	ND
3118	----	----	----	----	----
3146	----	----	----	----	----
3150	<0,10	<0,10	<0,10	<0,10	<0,10
3154	----	----	----	----	----
3172	----	----	----	----	----
3176	----	----	----	----	----
3197	<0,05	<0,05	<0,05	<0,05	<0,05
3200	----	----	----	----	----
3209	----	----	----	----	----
3210	----	----	----	----	----
3214	<0.025	<0.025	<0.025	<0.025	<0.025
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3228	N.D.	----	----	N.D.	N.D.
3237	----	----	----	----	----
3243	n.d.	n.d.	n.d.	n.d.	n.d.

DOT = Dioctyltin
 TOT = Trioctyltin
 DPhT = Diphenyltin
 TPhT = Triphenyltin
 TCyHT = Tricyclohexyltin

APPENDIX 3

Analytical details

lab	ISO/IEC 17025 accredited	Sample intake (in g)	Type of extraction	Solvent used to extract/release	Extraction time/temp (min/°C)	pH after adding the buffer	Extraction solution acidified until pH 4.5
110	---	---	---	---	---	---	---
213	No	---	---	---	---	---	---
230	No	0.5	Ultrasonic ext.	Acetone	60/40	6.2	Yes
339	No	1	Ultrasonic ext.	Acetone	60/40	4.5	Yes
551	Yes	1.0	Ultrasonic ext.	Isocane	60/40	4.5	---
623	Yes	1	Ultrasonic ext.	Acetone	60/40	4.5	No
840	Yes	0.5	Ultrasonic ext.	methanol:ethanol(8:2)	60/60	4.5	No
841	Yes	1.25	Ultrasonic ext.	Acetone, Hexane	60/40	4.5	Yes
1099	Yes	0.5	Heat bath ext.	HCl solvent	120/37	circa 1,2 pH	No
2108	Yes	0,5	Ultrasonic ext.	---	---	---	---
2115	Yes	1	Ultrasonic ext.	Ethanol, acetic acid	60/40	5.0	No
2117	Yes	0.2-0.3	Ultrasonic ext.	Methanol/Ethanol (80:2)	60/60	4,5	No
2120	No	0,5	Ultrasonic ext.	Methanol/Ethanol + tropolone	60/60	not noted	No
2129	Yes	0,6	Ultrasonic ext.	Methanol, acidified with HCl	60/60	not checked	No
2131	---	---	---	---	---	---	---
2135	Yes	1	Ultrasonic ext.	Methanol/Ethanol	60/60	4,5	No
2138	Yes	0.5	Ultrasonic ext.	methanol	60/70	5.29	Yes
2159	Yes	1	Ultrasonic ext.	n-hexane	60/70	4.5	No
2184	Yes	0.5	Ultrasonic ext.	Methanol/Ethanol	60/60	pH 4.5	Yes
2213	Yes	---	Ultrasonic ext.	---	---	---	---
2217	Yes	0.5 – 1	Ultrasonic ext.	methanol-ethanol= 80:20	60/60	not measured-	No
2229	Yes	0.5	Ultrasonic ext.	methanol/ethanol= 80/20	60/60	---	No
2230	Yes	1	Ultrasonic ext.	MeOH	60/70	4.5	Yes
2241	Yes	0.5	Ultrasonic ext.	methanol:ethanol=4:1	60/60	4.5	No
2255	Yes	0.5	Ultrasonic ext.	n Hexane	60/70	4.5	Yes
2265	---	---	---	---	---	---	---
2271	Yes	1	Ultrasonic ext.	Methanol:Ethanol=8:2	60/60	4.5	No
2284	Yes	1.000	Ultrasonic ext.	methanol:ethanol=4:1	60/60	4.5	Yes
2290	---	---	---	---	---	---	---
2293	---	---	---	---	---	---	---
2295	Yes	1	Ultrasonic ext.	methanolðanol	60/room	4.5	Yes
2297	Yes	1	Ultrasonic ext.	methanol/ethanol	60/60	4.5	Yes
2300	Yes	1	Ultrasonic ext.	Methanol	60/60	4.5	No
2301	Yes	1	Ultrasonic ext.	Methanol	60/70	4.5	Yes
2310	Yes	1	Ultrasonic ext.	Acetone	60/40	4.5-5.0	Yes
2311	Yes	1	Ultrasonic ext.	Acetone	60/40	5.4	Yes
2330	No	1	Ultrasonic ext.	Acetone	60/60	5	Yes
2347	Yes	1	Ultrasonic ext.	---	---	---	---
2350	Yes	0.5	Ultrasonic ext.	Isocane	60/60	---	---
2352	Yes	0.5	Ultrasonic ext.	Acetone	60/60	4.5	Yes
2357	Yes	0.5	Ultrasonic ext.	isocane	60/60	---	Yes

lab	ISO/IEC 17025 accredited	Sample intake (in g)	Type of extraction	Solvent used to extract/release	Extraction time/temp (min/°C)	pH after adding the buffer	Extraction solution acidified until pH 4.5
2358	Yes	1 gram	Ultrasonic ext.	Acetone	60/40	4.5	No
2363	Yes	1	Ultrasonic ext.	Methanol/Ethanol	60/60	5.0	Yes
2365	Yes	1	Ultrasonic ext.	Methanol:Ethanol=4:1(v/v)	60/60	4.5	No
2366	No	0.5	Ultrasonic ext.	methanol : ethanol = 4:1	60/60	---	Yes
2369	---	---	---	---	---	---	---
2370	Yes	0.9946	Ultrasonic ext.	Methanol/Ethanol	60/60	4.5	Yes
2375	Yes	0.5	Ultrasonic ext.	Acetone	60/40	6.3	Yes
2379	No	1	Ultrasonic ext.	Methanol - Ethanol mixture (80/20 , v/v)	60/60	---	Yes
2380	Yes	0.80	Ultrasonic ext.	Acetone	60/40	4.5+/-0.3	Yes
2382	No	1	Ultrasonic ext.	methanol & ethanol	60/60	4.5	Yes
2386	Yes	1	Ultrasonic ext.	Acetone	60/40	4,5	Yes
2390	Yes	1.0	Ultrasonic ext.	Acetone	60/40	4.5	Yes
2415	Yes	1	Ultrasonic ext.	Methanol/Ethanol	60/60	4.5	No
2425	Yes	0.5	Ultrasonic ext.	methanol /ethanol= 80/20	60/60	--	No
2453	No	2	Ultrasonic ext.	Ethanol	60/60	4.5	Yes
2486	Yes	1	Ultrasonic ext.	N-Hexane	240/70	4.5	No
2492	Yes	0.2	Ultrasonic ext.	Ethanol/acetic acid solution 95/5 (v/v)	60/40	5	No
2495	No	1	Ultrasonic ext.	methanol:ethanol 4:1	60/60	4.7-4.8	Yes
2497	Yes	1	Ultrasonic ext.	Methanol	60/60	---	---
2525	No	1,00	Ultrasonic ext.	Methanol/Ethanol	60/60	5,9	No
2532	Yes	0.5	Ultrasonic ext.	80:20 Methanol:Ethanol	60/60	5.6	Yes
2549	Yes	0.5	Ultrasonic ext.	Methanol:Ethanol (80:20)	60/60	4.5	No
2550	Yes	1.0	Ultrasonic ext.	methanol/ethanol mixture and 1mL tropotone solution	60/60	4.5	No
2553	Yes	1	Ultrasonic ext.	Sodium diethyldithiocarbamate trihydrate in Methanol	60/70	4.5	Yes
2561	---	---	---	---	---	---	---
2566	Yes	1	Ultrasonic ext.	sodium diethyldithiocarbamate trihydrate in Methanol	60/70	4.5	No
2569	Yes	1	Ultrasonic ext.	methanol+Ethanol	60/60	4.45	Yes
2573	Yes	0.5	Ultrasonic ext.	methanol/ethanol mixture (4:1)	60/60	4.75	Yes
2590	Yes	1	Ultrasonic ext.	methanol/ethanol mixture 80/20	60/60	4.5	No
2591	No	0.2	Ultrasonic ext.	Acetic Acid/Methanol	10/room	---	---
2602	---	---	---	---	---	---	---
2629	Yes	1.0	Ultrasonic ext.	Methanol/Ethanol + tropolone	60/60	---	Yes
2649	Yes	1	Ultrasonic ext.	Methanol with sodium diethyldithiocarbamate trihydrate	60/70	4.5	Yes
2668	Yes	0.5	Ultrasonic ext.	Isooctane	60/60	4.5	Yes
2713	Yes	1	Ultrasonic ext.	Methanol with sodium diethyldithro carbamate trihydrate	60/60	---	No
2719	---	---	---	---	---	---	---
2727	Yes	1	Ultrasonic ext.	mixture methanol/ethanol (80:20)	60/60	4,5	No
2737	Yes	1	Ultrasonic ext.	methanol	60/70	4.5	Yes
2741	No	0.5	Ultrasonic ext.	Methanol:Ethanol Solution (8:2) with tropolone	60/60	---	No

lab	ISO/IEC 17025 accredited	Sample intake (in g)	Type of extraction	Solvent used to extract/release	Extraction time/temp (min/°C)	pH after adding the buffer	Extraction solution acidified until pH 4.5
2743	Yes	1,0	Ultrasonic ext.	mixture methanol/ethanol (80:20)	60/60	4.5	No
2773	Yes	0.50	Ultrasonic ext.	Methanol/Ethanol	---	6.12	Yes
2804	No	1	Ultrasonic ext.	Methanol	60/70	4.5	No
2826	Yes	1	---	methanol / ethanol (80:20)	60/60	4.5	No
2827	Yes	1	Ultrasonic ext.	4:1 methanol : ethanol mixture	60/60	4.5	No
2845	---	---	---	---	---	---	---
2858	Yes	0.5001	Ultrasonic ext.	n-hexane	60/40	5	Yes
2864	Yes	0.5	Soxhlet / AES extraction	0.05% HCl in methanol	---	2.0	No
3100	Yes	1.0	Ultrasonic ext.	Methanol/Ethanol	60/60	4.5	Yes
3116	Yes	1	Ultrasonic ext.	Mixture of methanol/ethanol	60/60	---	No
3117	Yes	1.005 1.017	Ultrasonic ext.	---	60/room	4.4	Yes
3118	---	---	---	---	---	---	---
3146	Yes	0,5	Ultrasonic ext.	MeOH/EtOH 80:20 v/v	60/60	5	No
3150	Yes	1	Ultrasonic ext.	Methanol/Ethanol 80:20	60/60	---	---
3154	---	---	---	---	---	---	---
3172	---	---	---	---	---	---	---
3176	Yes	1	Ultrasonic ext.	Methanol/ Hexane	30/room	4,5	Yes
3197	Yes	2	Ultrasonic ext.	Ethanol	120/room	4,5	No
3200	Yes	0.5	Ultrasonic ext.	Methanol/Ethanol	60/60	4.5	No
3209	Yes	1	Ultrasonic ext.	Ethanol	60/room	4.5	Yes
3210	---	---	---	---	---	---	---
3214	Yes	0.5	Ultrasonic ext.	MeOH/ EtOH	60/60	4.5	Yes
3220	Yes	1	Ultrasonic ext.	Methanol:Ethanol (8:2)	60/60	4.5	Yes
3228	Yes	0.5	Ultrasonic ext.	methanol /ethanol mixture (80/20 V/V)	60/60	4.5	Yes
3237	Yes	2	Other (mention in remarks)	methanol	120/room	---	No
3243	No	1	Ultrasonic ext.	Ethanol	60/60	4.5	No

APPENDIX 4

Number of participants per country

6 labs in BANGLADESH
1 lab in BRAZIL
1 lab in CAMBODIA
2 labs in FRANCE
12 labs in GERMANY
1 lab in GUATEMALA
6 labs in HONG KONG
1 lab in HUNGARY
12 labs in INDIA
3 labs in INDONESIA
6 labs in ITALY
2 labs in KOREA
1 lab in MAURITIUS
1 lab in MOROCCO
21 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in POLAND
3 labs in PORTUGAL
1 lab in SPAIN
1 lab in SRI LANKA
1 lab in SWITZERLAND
4 labs in TAIWAN R.O.C.
1 lab in THAILAND
8 labs in TURKEY
1 lab in U.S.A.
1 lab in UNITED KINGDOM
7 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	= possibly an error in calculations
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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- 18 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)