

# Results of Proficiency Test Organotin Compounds in Textile December 2023

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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Report: iis23T48

March 2024

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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 textile there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known organizations are for instance: Bluesign® (Germany), which has created a Bluesign® restricted substances list (RSL) and OEKO-TEX® Standard 100 (Switzerland).

Since 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Organotin Compounds in Textile every year. During the annual proficiency testing program of 2023, it was decided to continue the proficiency test for the determination of Organotin Compounds in Textile.

In this interlaboratory study 72 laboratories in 26 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send two different textile samples of approximately 3 grams each, both positive on some Organotin compounds, labelled #23805 and #23806 respectively. 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 positive on some Organotin compounds was selected. The batch was cut into small pieces. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23805.

The homogeneity of the subsamples was checked by determination of Monobutyltin (MBT) in accordance with ISO17353 on 8 stratified randomly selected subsamples.

	Monobutyltin (MBT) in mg/kg
sample #23805-1	2.44
sample #23805-2	2.40
sample #23805-3	2.52
sample #23805-4	2.63
sample #23805-5	3.02
sample #23805-6	2.87
sample #23805-7	2.56
sample #23805-8	2.65

Table 1: homogeneity test results of subsamples #23805

From the above test results the relative standard deviation (RSD) was calculated and compared with 0.3 times the average relative standard deviation obtained from different compounds in four its PTs test data from 2019 – 2022 (see table 8) in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Monobutyltin (MBT) in mg/kg
RSD (observed)	8%
reference method	iis PTs
0.3 x RSD (reference method)	7%

Table 2: evaluation of the relative standard deviation of subsamples #23805

The calculated relative standard deviation is in agreement with 0.3 times average relative standard deviation obtained from the previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of grey cotton textile positive on some Organotin compounds was selected. The batch was cut into small pieces. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23806.

The homogeneity of the subsamples was checked by determination of Dimethyltin (DMT) in accordance with ISO17353 on 8 stratified randomly selected subsamples.

	Dimethyltin (DMT) in mg/kg
sample #23806-1	1.42
sample #23806-2	1.46
sample #23806-3	1.51
sample #23806-4	1.51
sample #23806-5	1.39
sample #23806-6	1.43
sample #23806-7	1.51
sample #23806-8	1.42

Table 3: homogeneity test results of subsamples #23806

From the above test results the relative standard deviation (RSD) were calculated and compared with 0.3 times the average relative standard deviation obtained from different compounds in four iis PTs test data from 2019 - 2022 (see tabel 8) in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Dimethyltin (DMT) in mg/kg
RSD (observed)	3%
reference method	iis PTs
0.3 x RSD (reference method)	7%

Table 4: evaluation of the relative standard deviation of subsamples #23806

The calculated relative standard deviation is in agreement with 0.3 times the average relative standard deviation obtained from the previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two textile samples labelled #23805 and #23806 were sent on November 15, 2023.

#### 2.5 ANALYZES

The participants were requested to determine on both samples: 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 appendices 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 appendices 1 and 2. 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. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

#### 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 (derived from e.g. ISO or ASTM test methods), 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_{\text{(target)}} = \text{(test result - average of PT)} / \text{target standard deviation}
```

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

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

```
|z| < 1 good
1 < |z| < 2 satisfactory
2 < |z| < 3 questionable
3 < |z| unsatisfactory
```

#### 4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and three participants did not report any test results. Not all participants were able to report all components requested. In total 69 participants reported 283 numerical test results. Observed were 12 outlying test results, which is 4.2%. 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of Organotin compounds, test method ISO/TS16179 has been used in previous reports. This is a test method for Organotin Compounds in Footwear materials. However, in 2020 a test method has been published for Organotin Compounds in Textile, ISO22744-1. A number of participants have reported this method. It has been decided to use the precision of this method in this and future reports, due to the matrix used in this proficiency test. The precision of method ISO22744-1 is based on two samples with a known concentration of MBT, DBT and TBT and shows a good linear correlation. The reproducibility of this test method as found for these three components is 43.4% of the concentration and therefore the RSD is 43.4%/2.8=15.5%. iis decided to use this precision for all components in this PT. The calculated precision for ISO/TS16179 has been added as comparison under the tables in appendix 1.

#### sample #23805

- Monobutyltin (MBT): The group of participants had difficulty to meet the target requirements. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO22744-1:20.
- <u>Dibutyltin (DBT)</u>: The group of participants had difficulty to meet the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO22744-1:20.

Almost all participants agreed on a concentration near or below the limit of detection for all other components mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

#### sample #23806

- Monomethyltin (MMT): The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ISO22744-1:20.
- <u>Dimethyltin (DMT)</u>: The group of participants had difficulty to meet the target requirements.

  One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO22744-1:20.
- <u>Tributyltin (TMT)</u>: The group of participants had difficulty to meet the target requirements.

  Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO22744-1:20.

Almost all participants agreed on a concentration near or below the limit of detection for all other components mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

#### 4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the reproducibility as declared by the reference test method 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 derived from reference method are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Monobutyltin (MBT)	mg/kg	62	15.2	8.2	6.6
Dibutyltin (DBT)	mg/kg	34	0.082	0.046	0.035

Table 5: reproducibilities of tests on sample #23805

Component	unit	n	average	2.8 * sd	R(lit)
Monomethyltin (MMT)	mg/kg	61	1.23	1.28	0.53
Dimethyltin (DMT)	mg/kg	62	53.9	30.8	23.4
Trimethyltin (TMT)	mg/kg	52	0.267	0.153	0.116

Table 6: reproducibilities of tests on sample #23806

Without further statistical calculations it can be concluded that for the reported components there is a not good compliance of the group of participants with the reference test method. The problematic tests have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2023 WITH PREVIOUS PTS

	December 2023	December 2022	December 2021	December 2020	December 2019
Number of reporting laboratories	69	80	72	85	85
Number of test results	283	334	185	247	317
Number of statistical outliers	12	15	11	12	12
Percentage of statistical outliers	4.2%	4.5%	5.9%	4.9%	3.8%

Table 7: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	December 2023	December 2022	December 2021	December 2020	December 2019	2018 – 2016
Monomethyltin (MMT)	37%		34%		37%	
Dimethyltin (DMT)	20%		23%		22%	25-40%
Trimethyltin (TMT)	20%				26%	
Monobutyltin (MBT)	19%	22%	22%	20-39%	33%	37%
Dibutyltin (DBT)	20%	26%		18%	22%	21-35%
Tributyltin (TBT)		23%				29-31%
Monooctyltin (MOT)		23%				
Dioctyltin (DOT)		16%				

Table 8: development of uncertainties of the proficiency tests over the years

The RSDs observed in this PT are in line with RSDs observed in previous its PTs. Remarkably, the determination of MMT has more variation than that of the other Organotin components.

#### 4.4 EVALUATION OF THE 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:

- Eighty-nine percent of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- Thirty-three percent of the participants indicate to have used the samples as received and sixty-seven percent of the participants further cut the samples prior to analysis.
- Fifty-six percent of the participants used 1 gram as sample intake. About thirty-seven percent around 0.5 grams and six percent around 2 or more grams.
- All participants used an Ultrasonic technique to extract/release the components from the samples.
- Seventy-eight participants used Methanol or a mixture of Methanol and Ethanol as extraction solvent.
- Almost all participants used an extraction/release time of 60 minutes and an extraction/release temperature of between 60 and 70 °C.
- Almost all participants reported to observe a pH of 4.5 4.6. Fifty-nine percent have adjusted the pH.

As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

#### 5 DISCUSSION

In this PT the average of the homogeneity test results are 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 prove 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.

When the test results of this interlaboratory study were compared to the limits of well known ecolabelling standards as OEKO-TEX® Standard 100 and bluesign® RSL (table 9) it should be noted that some laboratories would have made a different decision about the acceptability or rejection of the textile.

For sample #23805 all laboratories, that reported a test result for Monobutyltin, would have rejected the sample based on the presence of this component. One laboratory only reported for Dibutyltin and based on that test result it would approve the sample.

For sample #23806 all laboratories, that reported a test result for Dimethyltin, would have rejected the sample based on the presence of this component. One laboratory only reported for Trimethyltin and based on that test result it would approve the sample.

	OEKO-TEX® Standard 100 direct skin contact in mg/kg	bluesign® RSL v14.0 next to skin use in mg/kg
TBT, TPhT	1.0	0.5
DMT, TMT, TPT, TeBT, TCyHT, TOT	2.0	0.5
MBT, DBT, DOT	2.0	1.0
MMT, MOT, DPhT,	2.0	2.0

Table 9: OEKO-TEX® and Bluesign® criteria for Organotin in textiles

#### 6 CONCLUSION

In this proficiency test for the determination of Organotin Compounds in Textile it was noticed that the majority of the participants was able to detect and quantify the Organotin components present in both samples.

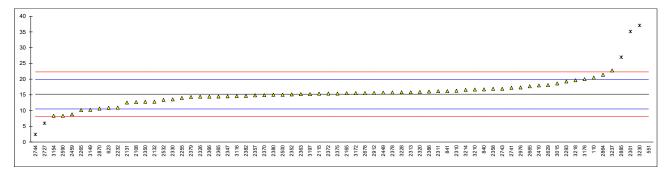
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

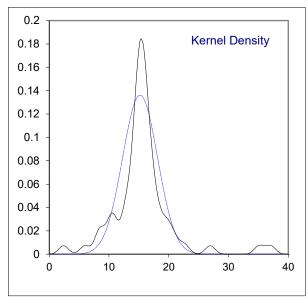
**APPENDIX 1** 

Determination of Monobutyltin (MBT) on sample #23805; results in mg/kg

					3805; results in mg/kg
lab	method	value	mark	z(targ)	remarks
110	In house	20.467		2.23	
210	100/70 40470		0.0(0.04)		<i>"</i>
551	ISO/TS 16179	109.126	C,R(0.01)	39.84	first reported: 27.62
623	ISO/TS 16179	10.848		-1.85	
840	ISO/TS 16179	16.73		0.65	
841	ISO/TS 16179	16.203		0.42	
2108	ISO22744-1	12.71		-1.06	
2115	In house	15.37		0.07	
2131	In house	12.55		-1.13	
2132	ISO/TS 16179	12.8		-1.02	
2165	ISO/TS 16179	15.51		0.13	
2232		10.914		-1.82	
2255	ISO/TS 16179	14.0		-0.51	
2265	ISO/TS 16179	10.16		-2.14	
2293	ISO/TS 16179	19.24		1.71	
2301	ISO/TS 16179	35.14	C,R(0.01)	8.46	first reported: 0.12, reported as #23806
2310	ISO22744-1	16.3		0.46	
2311	ISO22744-1	16.171		0.41	
2313	ISO/TS 16179	15.82		0.26	
2320	ISO22744-1	15.902		0.29	
2326	ISO/TS 16179	14.44	С	-0.33	first reported: 5.77
2330	ISO22744-1	13.537		-0.71	
2347	ISO17353	14.50		-0.30	
2350	ISO/TS 16179	12.789		-1.03	
2352	ISO22744-1	15.140		-0.03	
2357	ISO/TS 16179	14.831		-0.16	
2358		16.9		0.72	
2363	ISO22744	15.2		0.00	
2365	ISO/TS 16179	14.464		-0.32	
2366	ISO/TS 16179	14.44		-0.33	
2370	ISO22744-1	14.9		-0.13	
2372	ISO17353	15.4		0.08	
2375	ISO22744-1	15.4		0.08	
2378	ISO22744-1	15.74		0.23	
2379	ISO/TS 16179	14.3166		-0.38	
2380	ISO/TS 16179	15.0		-0.09	
2382	ISO22744-1	14.613		-0.25	
2386	ISO/TS 16179	16.04		0.35	
2410	ISO/TS 16179	17.99		1.18	
2449		15.702		0.21	
2453					
2459	ISO/TS 16179	8.78		-2.73	
2500	ISO/TS 16179	15.02		-0.08	
2532	ISO/TS 16179	13.4		-0.77	
2561					
2590	ISO/TS 16179	8.34		-2.91	
2602					
2629	ISO/TS 16179	18.11		1.23	
2665	ISO/TS 16179	17.717		1.06	
2678	ISO22744-1	15.56		0.15	
2727	ISO/TS 16179	5.961	R(0.05)	-3.92	
2741	ISO/TS 16179	17.243	,	0.86	
2743	ISO/TS 16179	16.90		0.72	
2744	ISO/TS 16179	2.4	C,R(0.05)	-5.43	first reported: not detected, reported as #23806
2864	ISO/TS 16179	21.35		2.61	
2870	ISO22744-1	10.62		-1.95	
2885	KS K0737	26.95	R(0.05)	4.98	
2912	ISO22744-1	15.614	C	0.17	first reported: 25.579
2976	ISO/TS 16179	17.326		0.90	
3015	ISO/TS 16179	18.62		1.45	
3116	ISO/TS 16179	14.6		-0.26	
3149	ISO22744-1	10.2		-2.12	
3154	ISO/TS 16179	8.31		-2.93	
3172	ISO/TS 16179	15.553		0.15	
3176	In house	20.0		2.03	
3197	ISO23161	15.2		0.00	
3210		16.68		0.62	
3214	ISO/TS 16179	16.572		0.58	
3218	ISO/TS 16179	19.618		1.87	
3228	ISO/TS 16179	15.8		0.25	
3230	In house	37.067	C,R(0.01)	9.27	first reported: 25.84
3237	ISO/TS 16179	22.75		3.20	

```
normality
                                 OK
                                 62
         outliers
                                 6
         mean (n)
                                 15.2084
                                             RSD = 19%
         st.dev. (n)
                                 2.92961
        R(calc.)
st.dev.(ISO22744-1)
R(ISO22744-1)
                                 8.2029
                                 2.35730
                                 6.6004
Compare
         R(ISO/TS16179:12)
                                 9.7942
```

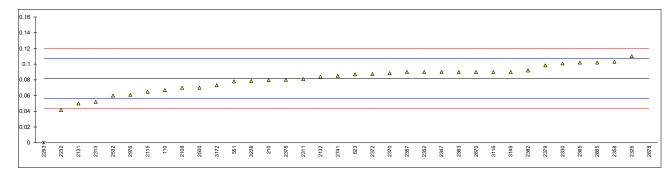


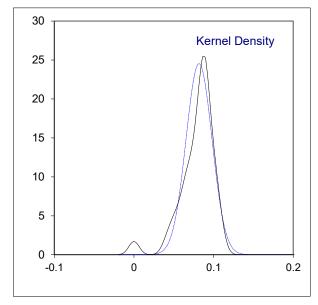


# Determination of Dibutyltin (DBT) on sample #23805; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	0.067	IIIdik	-1.16	Telliarks
210	III IIOUSE	0.0796		-0.16	
551	ISO/TS 16179	0.078		-0.29	
623	ISO/TS 16179	0.087		0.42	
840	ISO/TS 16179	not detected			
841	ISO/TS 16179	<0.1			
2108	ISO22744-1	0.07		-0.92	
2115	In house	0.065		-1.32	
2131	In house	0.05		-2.50	
2132	ISO/TS 16179	0.0841		0.19	
2165	ISO/TS 16179	Not Detected		2.47	
2232 2255	ISO/TS 16179	0.041492 Not Detected		-3.17	
2265	ISO/TS 16179	< 0,05			
2293	ISO/TS 16179	0	R(0.01)	-6.45	
2301	100/10 101/0		11(0.01)		
2310	ISO22744-1	<0.1			
2311	ISO22744-1	0.08089		-0.06	
2313	ISO/TS 16179	0.052		-2.34	
2320	ISO22744-1	<0.1			
2326	ISO/TS 16179	0.11		2.24	
2330	ISO22744-1	0.101		1.53	
2347 2350	ISO17353 ISO/TS 16179	0.09		0.66	
2350	ISO22744-1	< 0.5 0.090		0.66	
2357	ISO/TS 16179	0.09		0.66	
2358	100/10 101/0	0.103		1.68	
2363	ISO22744	0.09		0.66	
2365	ISO/TS 16179	0.102		1.61	
2366	ISO/TS 16179	<0.2			
2370	ISO22744-1	0.0886		0.55	
2372	ISO17353	0.0874		0.45	
2375 2378	ISO22744-1	0.08		-0.13	
2379	ISO/TS 16179	0.0984		1.32	
2380	ISO/TS 16179	<0.1			
2382	ISO22744-1	0.092		0.82	
2386	ISO/TS 16179	< 0.2			
2410					
2449					
2453	ICO/TC 16170	ND.			
2459 2500	ISO/TS 16179 ISO/TS 16179	ND 0.07		-0.92	
2532	ISO/TS 16179	0.06		-0.92 -1.71	
2561	100/10 101/0				
2590					
2602					
2629	ISO/TS 16179	Not Detected			
2665	ISO/TS 16179	0.102	D(0.04)	1.61	
2678 2727	ISO22744-1 ISO/TS 16179	0.78 < 0.2	R(0.01)	55.16 	
2721 2741	ISO/TS 16179	0.085		0.26	
2743	.55,15 10178	0.005			
2744	ISO/TS 16179	not detected			
2864	ISO/TS 16179	not detected			
2870	ISO22744-1	0.09		0.66	
2885	10000777				
2912	ISO22744-1	< 0.2		4.00	
2976 3015	ISO/TS 16179 ISO/TS 16179	0.061 <0.05		-1.63	
3116	ISO/TS 16179	0.0900		0.66	
3149	ISO22744-1	0.09		0.66	
3154					
3172	ISO/TS 16179	0.0731		-0.68	
3176					
3197	ISO23161	<0,01		<-5.66	possibly a false negative test result?
3210	ICO/TC 40470	<0.1			nessibly a false negative test v====10
3214 3218	ISO/TS 16179 ISO/TS 16179	<0.025 <0.050		<-4.48 	possibly a false negative test result?
3218	ISO/TS 16179 ISO/TS 16179	<0.050 0.0785		-0.25	
3230	In house	not detected			
3237					

```
normality
                                 OK
                                 34
         outliers
                                 2
         mean (n)
                                 0.08168
                                 0.016256
                                                  RSD = 20%
         st.dev. (n)
        R(calc.)
st.dev.(ISO22744-1)
R(ISO22744-1)
                                 0.04552
                                 0.012660
                                 0.03545
Compare
         R(ISO/TS16179:12)
                                 0.05260
```

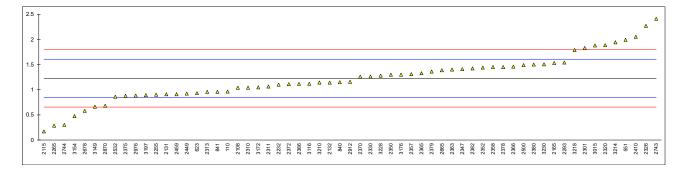


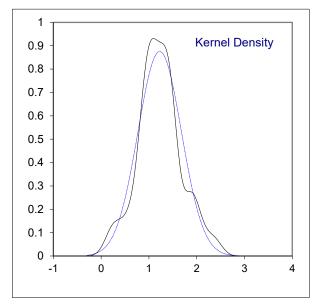


# Determination of Monomethyltin (MMT) on sample #23806; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	0.962		-1.39	
210					
551	ISO/TS 16179	1.99		4.02	
623	ISO/TS 16179 ISO/TS 16179	0.930		-1.56	
840 841	150/15 101/9	1.15 0.958		-0.40 -1.41	
2108	ISO22744-1	1.04		-0.98	
2115	In house	0.17	С	-5.56	first reported: 2.56
2131		0.91		-1.66	
2132	ISO/TS 16179	1.1409		-0.45	
2165	ISO/TS 16179	1.531		1.61	
2232	ISO/TS 16179	1.1000		-0.66	
2255	ISO/TS 16179	0.9		-1.71	
2265 2293	ISO/TS 16179 ISO/TS 16179	0.285 1.54		-4.95 1.66	
2301	ISO/TS 16179	1.83	С	3.18	first reported: not reported, reported as #23805
2310	ISO22744-1	1.04	Ü	-0.98	mot reported. Het reported, reported de #20000
2311	ISO22744-1	1.0638		-0.85	
2313	ISO/TS 16179	0.956		-1.42	
2320	ISO/TS 16179	1.885		3.47	
2326	ISO/TS 16179	2.27		5.50	
2330 2347	ISO22744-1 ISO/TS 16179	1.262 1.41		0.19 0.97	
2350	ISO/TS 16179	1.41		0.97	
2352	ISO22744-1	1.440		1.13	
2357	ISO/TS 16179	1.31		0.44	
2358		1.45		1.18	
2363	ISO22744-1	1.4		0.92	
2365	ISO/TS 16179	1.327		0.53	
2366	ISO/TS 16179	1.46		1.23	
2370 2372	ISO17353 ISO17353	1.26 1.11		0.18 -0.61	
2375	ISO22744-1	0.88		-1.82	
2378	ISO22744-1	1.45		1.18	
2379	ISO22744-1	1.3616		0.72	
2380	ISO/TS 16179	1.50		1.44	
2382	ISO22744-1	1.423		1.04	
2386	ISO/TS 16179	1.115		-0.58	
2410 2449	ISO/TS 16179	2.05 0.9231		4.34 -1.59	
2449		0.9231		-1.59	
2459	ISO17353	0.91		-1.66	
2500	ISO/TS 16179	1.49		1.39	
2532	ISO/TS 16179	0.86		-1.92	
2561					
2590					
2602 2629					
2665	ISO/TS 16179	1.385		0.84	
2678	ISO22744-1	0.58		-3.40	
2727					
2741					
2743	ISO/TS 16179	2.41	C	6.23	first reported: 4.96
2744	ISO/TS 16179	0.3	С	-4.87	first reported: not detected
2864 2870	ISO22744-1	0.68		 -2.87	
2885	13022744-1			-2.01	
2912	ISO22744-1	1.157		-0.36	
2976	ISO/TS 16179	0.884		-1.80	
3015	ISO/TS 16179	1.88		3.44	
3116	ISO/TS 16179	1.12		-0.56	
3149 3154	ISO22744-1	0.66		-2.98	
3154 3172	ISO/TS 16179	0.475 1.0448		-3.95 -0.95	
3172	In house	1.30		0.39	
3197	ISO23161	0.89		-1.77	
3210		1.14		-0.45	
3214	ISO/TS 16179	1.942		3.77	
3218	ISO/TS 16179	1.789		2.97	
3228	ISO/TS 16179	1.28		0.29	
3230 3237	In house	1.503 		1.46	
3231					

			ISO22744-1 only:	ISO/TS16179 only:
normality	OK		OK	OK
n	61		14	33
outliers	0		0	28
mean (n)	1.2256		1.1027	1.3573
st.dev. (n)	0.45556	RSD = 37%	0.30729 RSD = 28%	0.51824 RSD = 38%
R(calc.)	1.2756		0.8604	1.4511
st.dev.(ISO22744-1)	0.18996		0.17091	
R(ISO22744-1)	0.5319		0.4786	
Compare				
R(ISO/TS16179:12)	0.7893			0.8741

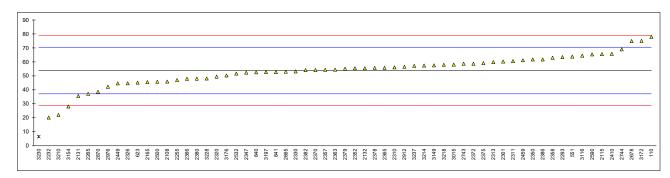


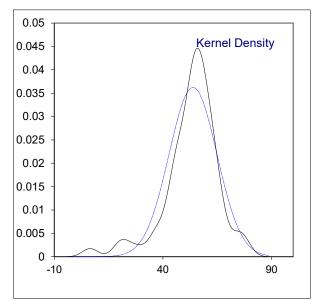


# Determination of Dimethyltin (DMT) on sample #23806; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	77.912	С	2.88	first reported: 26.183
210			•	2.00	
551	ISO/TS 16179	63.815		1.19	
623	ISO/TS 16179	45.189		-1.04	
840	ISO/TS 16179	52.5		-0.16	
841		52.718		-0.14	
2108	ISO22744-1	45.92		-0.95	
2115	In house	65.6		1.41	
2131 2132	ISO/TS 16179	35.61 55.3980		-2.19 0.18	
2165	ISO/TS 16179	45.58		-0.99	
2232	ISO/TS 16179	20.176	С	-4.03	first reported: 17.5650
2255	ISO/TS 16179	47.0		-0.82	
2265	ISO/TS 16179	37.14		-2.00	
2293	ISO/TS 16179	63.44		1.15	
2301	ISO/TS 16179	60.27	С	0.77	first reported: not reported, reported as #23805
2310	ISO22744-1	56.1		0.27	
2311 2313	ISO22744-1 ISO/TS 16179	60.557		0.80	
2320	ISO/TS 16179	59.861 49.430		0.72 -0.53	
2326	ISO/TS 16179	44.67	С	-1.10	first reported: ND
2330	ISO22744-1	53.119	Ü	-0.09	mot reported. NB
2347	ISO/TS 16179	52.34		-0.18	
2350	ISO/TS 16179	61.714		0.94	
2352	ISO22744-1	55.350		0.18	
2357	ISO/TS 16179	54.26		0.05	
2358	100007444	63		1.10	
2363	ISO22744-1 ISO/TS 16179	54.47		0.07	
2365 2366	ISO/TS 16179	55.716 47.88		0.22 -0.72	
2370	ISO17353	54.2		0.04	
2372	ISO17353	58.7		0.58	
2375	ISO22744-1	59.2		0.64	
2378	ISO22744-1	55.70		0.22	
2379	ISO22744-1	55.1704		0.16	
2380	ISO/TS 16179	48.0		-0.70	
2382 2386	ISO22744-1 ISO/TS 16179	54.102		0.03 0.94	
2410	ISO/TS 16179	61.72 65.77		1.43	
2449	100/10 101/3	44.596		-1.11	
2453					
2459	ISO17353	61.13		0.87	
2500	ISO/TS 16179	45.78		-0.97	
2532	ISO/TS 16179	51.65		-0.26	
2561	100/70 10170				
2590	ISO/TS 16179	65.31		1.37	
2602 2629					
2665	ISO/TS 16179	52.965		-0.11	
2678	ISO22744-1	74.88		2.52	
2727					
2741					
2743	ISO/TS 16179	58.67	_	0.58	
2744	ISO/TS 16179	69	С	1.81	first reported: not detected, reported as #23805
2864	10000744 4	 20 E4		-1.83	
2870 2885	ISO22744-1	38.54 		-1.03	
2912	ISO22744-1	56.534		0.32	
2976	ISO/TS 16179	42.219		-1.39	
3015	ISO/TS 16179	58.08		0.51	
3116	ISO/TS 16179	64.5		1.27	
3149	ISO22744-1	57.5		0.44	
3154	ISO/TS 16179	28.04		-3.09	
3172	la hacea	75.03		2.54	
3176	In house	50.27		-0.43	
3197 3210	ISO23161	52.7 22.03	С	-0.14 -3.81	first reported: 79.77
3214	ISO/TS 16179	57.366	J	0.42	mot opolica. 10.11
3218	ISO/TS 16179	57.852		0.48	
3228	ISO/TS 16179	48.1		-0.69	
3230	In house	6.527	C,R(0.01)	-5.67	first reported, 5.984
3237	ISO/TS 16179	57.15		0.39	

```
normality
                                 suspect
                                 62
         outliers
         mean (n)
                                 53.8579
         st.dev. (n)
                                 11.01498
                                               RSD = 20%
        R(calc.)
st.dev.(ISO22744-1)
R(ISO22744-1)
                                 30.8419
                                 8.34797
                                 23.3743
Compare
         R(ISO/TS16179:12)
                                 34.6845
```

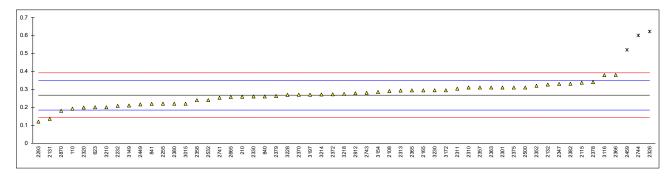


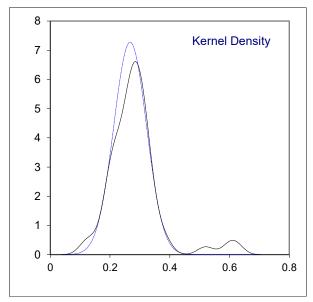


# Determination of Trimethyltin (TMT) on sample #23806; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	0.192	С	-1.82	first reported: 1.007
210		0.2589	-	-0.21	
551	ISO/TS 16179	not detected			
623	ISO/TS 16179	0.200		-1.63	
840	ISO/TS 16179	0.26		-0.18	
841	ISO22744 4	0.219		-1.17 0.54	
2108 2115	ISO22744-1 In house	0.29 0.337		0.54 1.68	
2113	110430	0.1353	С	-3.19	first reported: 0.47
2132	ISO/TS 16179	0.3264	=	1.42	F
2165	ISO/TS 16179	0.294		0.64	
2232	ISO/TS 16179	0.2080		-1.43	
2255	ISO/TS 16179	0.22	0	-1.14	fortunated 0.50 months follows:
2265 2293	ISO/TS 16179	<0.05	С	,-5.25	first reported: 0.52, possibly a false negative test result?
2301	ISO/TS 16179 ISO/TS 16179	0.12 0.31	С	-3.56 1.03	first reported: not reported, reported as #23805
2310	ISO22744-1	0.31	O	1.03	mat reported. Not reported, reported as #20000
2311	ISO22744-1	0.3038		0.88	
2313	ISO/TS 16179	0.293		0.62	
2320	ISO/TS 16179	0.199		-1.65	
2326	ISO/TS 16179	0.622	C,R(0.01)	8.55	first reported: not detected
2330 2347	ISO22744-1 ISO/TS 16179	0.260 0.33		-0.18 1.51	
2347	ISO/TS 16179	0.33 < 0.5		1.51	
2352	ISO22744-1	0.320		1.27	
2357	ISO/TS 16179	0.31		1.03	
2358		0.24		-0.66	
2363	ISO22744-1	0.31		1.03	
2365	ISO/TS 16179	0.294		0.64	
2366 2370	ISO/TS 16179 ISO17353	0.38 0.270		2.72 0.06	
2372	ISO17353	0.270		0.00	
2375	ISO22744-1	0.31		1.03	
2378	ISO22744-1	0.34		1.75	
2379	ISO22744-1	0.2625		-0.12	
2380	ISO/TS 16179	0.22		-1.14	
2382 2386	ISO22744-1 ISO/TS 16179	0.331 < 0.2		1.53	
2410	100/10 101/10				
2449		0.21606		-1.24	
2453					
2459	ISO17353	0.52	R(0.01)	6.09	
2500	ISO/TS 16179	0.31		1.03	
2532 2561	ISO/TS 16179	0.24		-0.66 	
2590					
2602					
2629					
2665	ISO/TS 16179	0.257		-0.25	
2678 2727	ISO22744-1	Not detected			
2727 2741	ISO/TS 16179	0.253		-0.35	
2743	ISO/TS 16179	0.28		0.30	
2744	ISO/TS 16179	0.6	C,R(0.01)	8.02	first reported: not detected
2864	10000=:::				
2870	ISO22744-1	0.18		-2.11	
2885 2912	ISO22744-1	0.278		0.25	
2976	100221 77-1	0.276		0.25	
3015	ISO/TS 16179	0.22		-1.14	
3116	ISO/TS 16179	0.379		2.69	
3149	ISO22744-1	0.21		-1.39	
3154	ISO/TS 16179	0.285		0.42	
3172 3176		0.29455		0.65 	
3176	ISO23161	0.27		0.06	
3210		0.20		-1.63	
3214	ISO/TS 16179	0.271		0.09	
3218	ISO/TS 16179	0.274		0.16	
3228	ISO/TS 16179	0.269	0	0.04	first new autodu wat data stad
3230 3237	In house	0.2945	С	0.65	first reported: not detected
3231					

```
normality
                                 OK
                                 52
        outliers
                                 3
        mean (n)
                                 0.26744
                                                  RSD = 20%
        st.dev. (n)
                                 0.054813
        R(calc.)
st.dev.(ISO22744-1)
R(ISO22744-1)
                                 0.15348
                                 0.041454
                                 0.11607
Compare
        R(ISO/TS16179:12)
                                 0.17223
```





#### **APPENDIX 2**

Determination of other Organotin components on sample #23805; results in mg/kg

MMT = Monomethyltin
DMT = Dimethyltin
TMT = Trimethyltin
TPT = Tripropyltin
TBT = Tributyltin
TeBT = Tetrabutyltin

210	petected betected betected betected betected betected
S51   not detected	etected
623 not detected	etected
840 not detected         not detec	etected
841 < 0.1	etected Detected Detected Detected Detected Detected Detected Detected Detected Detected
2108	Detected Detected Detected Detected Detected Detected Detected Detected
2108	Detected Detected Detected Detected Detected Detected Detected Detected
2115	Detected Detected Detected Detected Detected Detected Detected Detected
2131 not detected         not dete	Detected Detected Detected Detected Detected Detected Detected Detected
2132	Detected Detected Detected Detected Detected Detected Detected Detected
2165 Not Detected	petected betected betected betected betected betected
2232 <td< td=""><td>etected etected etected etected etected</td></td<>	etected etected etected etected etected
2255         Not Detected         Not Detected         Not Detected         Not Detected         Not Detected           2265         < 0,05	etected Detected Detected
2265 < 0,05	etected Detected Detected
2293         0.0         0 <td>etected Detected Detected</td>	etected Detected Detected
2301	Petected Petected Petected
2310         not detected	Petected Petected Petected
2311 Not Detected         <0.05	Petected Petected Petected
2313         Not Detected	etected etected
2320 < 0.1	etected
2326 ND         A	
2330         Not Detected	
2347 < 0.02	
2350 < 0.5	
2352	
2357   <	
2358 not detected         not dete	
2363 not detected	
2365 < 0.05	
2366 < 0.2	
2370 < 0.01	1
2372 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02	
2375	
2378	
	etected
2380 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1	
2382 < 0.050 < 0.050 < 0.050 < 0.050 < 0.050 < 0.050	50
2386 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	
2410	
2449	
2453	
2459 ND ND ND ND ND ND	
2500 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02	
2532 Not Detected	etected
2561	
2590	
2602	
2629	
2665 < 0.2	i
· · · · · · · · · · · · · · · · · · ·	etected
2727 <0.2 <0.2	
2741 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	
2743	
	etected
2870	
2885 Not detected	etected etected
2912 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	
2976 Not Detected Not Detected Not Detected Not D	etected
	etected Petected
3015 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	etected Petected
3015 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	etected Petected
3015     <0.05	etected Petected
3015     <0.05	etected Petected
3015     <0.05	etected Petected

T	lab MMT	DMT	TMT	TPT	TBT	TeBT
_	3197 <0,01	<0,01	<0,01	<0,01	<0,01	<0,01
	3210 < 0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	3214 < 0.025	<0.025	<0.025	<0.025	<0.025	<0.025
	3218 < 0.050	< 0.050	< 0.050	<0.050	<0.050	< 0.050
	3228 not detected	not detected				
	3230 not detected	not detected				
	3237					

Determination of other Organotin components on sample #23805; results in mg/kg (continued)

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

lab	MOT	DOT	TOT	DPhT	TPhT	TCyHT
110	Not Detected					
		0.0398				
551	not detected					
623	not detected					
840	not detected					
841	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2108						
2115						
2131	not detected					
2132	< 0.05	<0.05	< 0.05	< 0.05	< 0.05	< 0.05
2165	Not Detected					
2232						
	Not Detected					
	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2293		0	0	0	0	0
	not detected					
	Not Detected					
	Not Detected					
	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2326		ND	ND	ND	ND	ND
	Not Detected	Not Detected	0.086	Not Detected	Not Detected	Not Detected
	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
	not dotacted	not detected		not detected	not detected	
	not detected not detected					
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2375						
	Not detected					
	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2382	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2449						
2453						
2459	ND	ND	ND	ND	ND	ND
2500	<0.02	<0.02	<0.02	<0.02	<0.02	< 0.02
2532	Not Detected					
2561						
2590						
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	Not detected					
	< 0.2	< 0.2			< 0.2	< 0.2
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2743						
	not detected					
	not detected	not detected			not detected	not detected
2885						

lab MOT	DOT	TOT	DPhT	TPhT	TCyHT
2912 < 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2976 Not Detected	Not Detected				
3015 < 0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3116					
3149					
3154					
3172 < 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
3176					
3197 <0,01	<0,01	<0,01	<0,01	<0,01	<0,01
3210 < 0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3214 < 0.025	<0.025	<0.025	<0.025	<0.025	<0.025
3218 < 0.050	<0.050	<0.050	<0.050	<0.050	<0.050
3228 not detected	not determined				
3230 not detected	not detected				
3237					

## Determination of other Organotin components on sample #23806; results in mg/kg

TPT = Tripropyltin
MBT = Monobutyltin
DBT = Dibutyltin
TBT = Tributyltin
TeBT = Tetrabutyltin

lah	ТРТ	MBT	DBT	ТВТ	TeBT
	Not Detected	0.050	0.018	Not Detected	Not Detected
	not detected	0.091	not detected	not detected	not detected
623	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected
841	<0.1	<0.1	<0.1	<0.1	<0.1
2108					
2115		0.041	0.017		
	not detected	0.03	0.02	not detected	not detected
	<0.05	<0.05	<0.05	<0.05	<0.05
2232	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
	< 0.05	< 0,05	< 0,05	< 0.05	< 0,05
2293	- ,	0.075	0	0	0
2301		0.12			
2310	not detected	<0.1	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
	<0.1	<0.1	<0.1	<0.1	<0.1
2326		ND	ND Not Data start	ND Not Data start	ND
	Not Detected	0.056	Not Detected	Not Detected <0.02	Not Detected
	<0.02 < 0.5	<0.02 < 0.5	<0.02 < 0.5	< 0.02	<0.02 < 0.5
2352					
2357					
	not detected	not detected	not detected	not detected	not detected
	not detected	not detected	not detected	not detected	not detected
2365	< 0.05	<0.05	<0.05	<0.05	<0.05
	<0.2	<0.2	<0.2	<0.2	<0.2
	<0.02	<0.02	<0.02	<0.02	<0.02
	<0.02	<0.02	<0.02	<0.02	<0.02
2375					
2378	Not detected	Not detected	Not detected	Not detected	Not detected
2380		<0.1	<0.1	<0.1	<0.1
	<0.050	<0.050	<0.050	< 0.050	<0.050
	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2410					
2449					
2453					
2459		ND	ND	ND	ND
	<0.02	<0.02	<0.02	<0.02	<0.02
2532 2561	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2590					
2602					
2629					
2665	not analyzed	0.076	0.063	< 0.05	< 0.05
2678	Not detected	Not detected	Not detected	Not detected	Not detected
2727		< 0.2	< 0.2	< 0.2	< 0.2
	<0.05	0.052	<0.05	<0.05	<0.05
2743		not dotacted	not dotooted	not dotacted	not detected
2864	not detected	not detected not detected	not detected not detected	not detected not detected	not detected not detected
2870		not detected	not detected	not detected	
2885		Not detected		Not detected	
	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2976		0.048	Not Detected	Not Detected	Not Detected
	<0.05	<0.05	<0.05	<0.05	<0.05
3116					
3149					
3154					
3172 3176	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
	<0,01	<0.01	<0.01	<0.01	<0.01
3210		<0.1	<0.1	<0.1	<0.1
	<0.025	<0.025	<0.025	<0.025	<0.025
	<0.050	<0.050	<0.050	<0.050	<0.050

lab TPT	MBT	DBT	TBT	TeBT
3228 not detected	not detected	not detected	not detected	not detected
3230 not detected	not detected	not detected	not detected	not detected
3237				

## Determination of other Organotin components on sample #23806; results in mg/kg (continued)

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

lab	MOT	DOT	тот	DPhT	TPhT	ТСуНТ
110	Not Detected					
210		0.0597				
551	not detected					
623	not detected					
840	not detected					
841	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2108						
2115						
	not detected					
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	Not Detected					
2232						
	Not Detected					
	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2293		0	0	0	0	0
2301						
	not detected					
	Not Detected					
	Not Detected <0.1					
2326		ND	ND	ND	ND	ND
	Not Detected					
	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
2352						
2357						
	not detected					
	not detected					
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2372	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2375						
2378						
2379	Not detected					
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2382	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
2386	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2410						
2449						
2453						
2459		ND	ND	ND	ND	ND
	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	Not Detected					
2561						
2590						
2602						
2629						
	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	Not detected					
	< 0.2 <0.05	< 0.2 <0.05	<0.05	<0.05	< 0.2 <0.05	< 0.2 <0.05
2741		<0.05 	<0.05 	<0.05 	<0.05 	
	not detected					
	not detected	not detected			not detected	not detected
2870						
2885						
	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
	Not Detected					
	<0.05	< 0.05	<0.05	<0.05	< 0.05	< 0.05
3116						

lab MOT	DOT	TOT	DPhT	TPhT	TCyHT
3149					
3154					
3172 < 0.02	2 < 0.02	< 0.02	< 0.02	< 0.02	< 0.02
3176					
3197 <0,01	<0,01	<0,01	<0,01	<0,01	<0,01
3210 < 0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3214 < 0.02	5 <0.025	<0.025	< 0.025	< 0.025	<0.025
3218 < 0.05	<0.050	< 0.050	< 0.050	< 0.050	<0.050
3228 not de	etected not detected	not detected	not detected	not detected	not detected
3230 not de	etected not detected	not detected	not detected	not detected	not detected
3237					

# **APPENDIX 3 Analytical details**

	ISO/IEC 17025			
lab	accredited	Sample preparation	Sample intake(g)	Extraction type
110	Yes	Used as received	1 gram	Ultrasonic
210	Yes			
551		Further out		I Utracania
623 840	Yes Yes	Further cut	1	Ultrasonic
841	Yes	Used as received Used as received	1 1g	Ultrasonic Ultrasonic
2108	Yes	Used as received	0,5 g	Ultrasonic
2115	Yes	Used as received	2 q	Ultrasonic
2131	Yes	Further cut	0.5 g	Ultrasonic
2132	No	Used as received	1 gram	Ultrasonic
2165	Yes	Further cut	1.0g	Mechanical shaking and Ultrasonic
2232	Yes	Used as received		
2255	Yes	Further cut	1.0	Ultrasonic
2265	Yes	Further cut	0,5 grams	Ultrasonic
2293	Yes	Used as received	1 gram	Ultrasonic
2301	No	Used as received	1 gram	Ultrasonic
2310 2311	Yes Yes	Further cut	1 0.5	Ultrasonic Ultrasonic
2313	No	Further cut Further cut	0.5 q	Ultrasonic
2320	Yes	Further cut	First trial 1g other trials 0.25g	Ultrasonic
2326	Yes	Further cut	1 GM	Ultrasonic
2330	Yes	Further cut	0.50 g	Ultrasonic
2347	No	Further cut	1.0g	Ultrasonic
2350	No	Further cut	#23805 : 1.0055 g #23806 : 1.0012 g	Ultrasonic
2352	Yes	Further cut	0.5g	Ultrasonic
2357				
2358	Yes	Used as received	1.0	Ultrasonic
2363	Yes	Further cut	3g	Ultrasonic
2365	Yes	Further cut	2.0g	Ultrasonic
2366			0.50	I Iltracania
2370 2372	Yes Yes	Further cut Further cut	0.5g 1g	Ultrasonic Ultrasonic
2375	Yes	Further cut	1g	Ultrasonic
2378	Yes	Further cut	0.5g	Ultrasonic
2379	Yes	Further cut	1 g	Ultrasonic
2380	Yes	Further cut	1.0 g	Ultrasonic
2382	Yes	Further cut	1g	Ultrasonic
2386	Yes	Further cut	0,5 g	Ultrasonic
2410	Yes	Further cut	0.01 g to 0.1 g	Ultrasonic
2449	Yes	Further cut	0.5	Ultrasonic
2453				
2459	Yes	Further cut	1.00 gram	Ultrasonic
2500	Yes	Used as received	1g	Ultrasonic
2532 2561	Yes	Further cut	0.5 grams	Ultrasonic
2590	Yes	Used as received		Ultrasonic
2602				
2629	Yes	Further cut	1.0	Ultrasonic
2665	Yes	Further cut	0,5	Ultrasonic
2678	No	Further cut	0.5 grams	Ultrasonic
2727	Yes	Further cut	1g	Ultrasonic
2741	Yes	Further cut	0.5 gram	Ultrasonic
2743	Yes	Used as received	2	Ultrasonic
2744	Yes	Used as received	0,5	Ultrasonic
2864	Yes	Further cut	0.5 g	Ultrasonic
2870	Yes	Further cut	1 gm	Ultrasonic
2885	No Voc	Further cut	0.5 g	Ultrasonic
2912 2976	Yes Yes	Used as received Used as received	0.5 g	Ultrasonic Ultrasonic
3015	Yes	Further cut	1g 1g	Ultrasonic
3116	Yes	Used as received	19 1	Ultrasonic
3149	Yes	Used as received	1 g	Ultrasonic
3154	Yes	Used as received	0,6	Ultrasonic
3172	Yes			
3176	Yes	Used as received	1	Ultrasonic
3197	Yes	Further cut	0,5 g	Ultrasonic

	ISO/IEC 17025			
lab	accredited	Sample preparation	Sample intake(g)	Extraction type
3210	Yes	Used as received	1g	Ultrasonic
3214	Yes	Further cut	1 gram	Ultrasonic
3218	Yes	Further cut	0.4g	Ultrasonic
3228	Yes	Further cut	0.5g	Ultrasonic
3230	Yes	Further cut	approximately 1.0gram	Ultrasonic
3237	Yes	Further cut	1	Ultrasonic

# **Analytical details (continued)**

		Extraction	Extraction temp	pH after adding	Acidified
lab	Extraction solvent	time (min)	(°C)	buffer	to pH 4.5
110	Acetone	60	60	4.64	No
210	<del></del>				
551	math an aliath an al		60	4 E	 Voo
623 840	methanol:ethanol MeOH:EtOH (8:2)	60	60	4.5 4.5	Yes No
841	ethanol/methanol (1:1)	60 mins	60oC		
2108	EtOH / Glacial acetic acid (95/5) + Tropolon	60 min	60°C		
2115	Ethanol /Acetic Acid	60 min	60°C	5 pH	No
2131	95% Ethanol, 5% Acetic Acid, 250 mg/l Tropolone	60 min	60 °C	5 pri	No
2132	Methanol with ethanol	60 mins	60 degree celsius	N.A.	No
2165	Methanol:ethanol (8:2) Isooctane	60mins	60°C	4.5	Yes
2232					
2255	Methanol+Ethanol & final extraction n-Hexane	60	60	4.5	No
2265	Methanol:Ethanol (80:20)	60 min	60 °C	4,5	No
2293	Methanol:Ethanol 80:20	1 hour	60°C		Yes
2301	Methanol : ethanol	60min	60 c	6.2	Yes
2310	HEXANE	60	60	4.5pH to 5.0pH	Yes
2311	Methanol + Ethanol (80:20)	60	60	4.5	Yes
2313	Isooctane	60 min	60±2°C	methanol-ethanol mixt.	Yes
2320	Methanol : Ethanol (80:20)	60 min	60°C	4.5	Yes
2326	METHANOL - ETHANOL	1 Hr	60 C	4.5	Yes
2330	MeOH:EtOH (Ratio 80:20)	60 min	60°C	4.5	Yes
2347					Yes
2350	Methanol : Ethanol (80:20)	60 minutes	60 °C	pH 4.5	No
2352	methanol/ethanol	60min	60°C	pH=4.5	Yes
2357					
2358	methanol-ethanol	60	60	4.5	Yes
2363	MeOH:EtOH=4:1	60mins	60°C	4.5	Yes
2365	ethanol:methanol=4:1	60min	60°C	4.5	Yes
2366					
2370	MeOH/EtOH	60 mins	60°C	4.5	Yes
2372	Ethanol	60 mins	RT	4.5	No
2375	Methanol/Ethanol	60 min	60 °C	pH 4,5	Yes
2378	methanol/ethanol	60min	60°C	pH=4.5	Yes
2379	Methanol : Ethanol (80/20 v/v)	60 min	60 Degree Celsius	pH 4.5	Yes
2380	Methanol-Ethanol mixture	60±5 Minute	60±2 °C	4.4	No
2382	methanol/ethanol mixture (80/20 V/V)	60±5min	60±5°C	4.5	Yes
2386	MeOH/EtOH 80/20	60 min	60 °C	not determined	No
2410	Methanol:Ethanol / Hexane	1 hr	60 °C	pH 4.5	Yes
2449	Methanol	60	70		Yes
2453					
2459	Methanol/Ethanol (80:20)	1 hour	60°C	4.5	Yes
2500					
2532	Methanol: Ethanol	60 mints	60 °C	4.5	Yes
2561					
2590	20 ml MeOH/Ethanol 80:20 v/v	60 min	60°C		No
2602					
2629	Hexane	60 minutes	60	4.5	Yes
2665	methanol/ethanol (80/20 v/v)	60 min	60°C		Yes
2678	Methanol/Ethanol mixture (80/20 v/v) + Hexane	60 minutes	60°C	4.5	No
2727	methanol/ethanol 80:20	60 minutes	60°C		No
2741	Methanol/Ethanol	60	60		No
2743	methanol:ethanol	60	60	4.5	No
2744	Methanol/Ethanol	1 hour	60	4,5	Yes
2864	MeOH/EtOH	60	60		Yes
2870	Ethanol/glacial acetic acid 95/5 + Tropolone	60 minutes	60 degree C	4.5	Yes

lab	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding buffer	Acidified to pH 4.5
2885	Methanol	60 min	60	4.5	No
2912	methanol/ethanol (80/20 in volume)	60 min	60 °C		No
2976	MeOH:EtOH (8:2)	60 min	60oC	pH = 4.5	Yes
3015	methanol/ethanol 4:1	60min	60°C	PH=4.5	No
3116	methanol/ethanol (80/20 v/v)	60	60	4.5	No
3149	Methanol/Ethanol 80/20	1 h +-5	60 +-5		
3154	Methanol	60	60		No
3172					
3176	HCI + MeOH	30	Room temperature	4.5	Yes
3197	n-hexane	60 min.	70 C		Yes
3210	MeOH/EtOH (80/20)	60	60		
3214	Methanol:Ethanol:4:1	1 hr	60	4.5	Yes
3218	n-hexane	60min	60°C	4.5	No
3228	8:2 v/v MeOH/EtOH	60min	60°C	PH=5.6	No
3230	2,2,4 Trimethylpentane	60 minutes	60 degree celcius	4.5	Yes
3237	iso-octane	60	60		No

#### **APPENDIX 4**

#### Number of participants per country

- 2 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in CAMBODIA
- 1 lab in FRANCE
- 7 labs in GERMANY
- 1 lab in GUATEMALA
- 3 labs in HONG KONG
- 5 labs in INDIA
- 2 labs in INDONESIA
- 5 labs in ITALY
- 3 labs in KOREA, Republic of
- 1 lab in MAURITIUS
- 1 lab in MOROCCO
- 13 labs in P.R. of CHINA
- 3 labs in PAKISTAN
- 2 labs in PORTUGAL
- 1 lab in SINGAPORE
- 1 lab in SRI LANKA
- 1 lab in SWITZERLAND
- 4 labs in TAIWAN
- 1 lab in THAILAND
- 1 lab in TUNISIA
- 5 labs in TURKEY
- 1 lab in U.S.A.
- 1 lab in UNITED KINGDOM
- 5 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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(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

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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