

Institute for
Interlaboratory Studies

Results of Proficiency Test Organotin Compounds in Textile December 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink

Correctors: ing. G.A. Oosterlaken-Buijs & Mrs. E.R. Montenij-Bos

Approved by: ing. A.S. Noordman-de Neef

Report: iis22T14

March 2023

CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	QUALITY SYSTEM	3
2.2	PROTOCOL.....	3
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	ANALYZES	5
3	RESULTS	6
3.1	STATISTICS	6
3.2	GRAPHICS	7
3.3	Z-SCORES	7
4	EVALUATION	8
4.1	EVALUATION PER SAMPLE AND PER COMPONENT	8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES.....	9
4.3	COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2022 WITH PREVIOUS PTS.....	10
4.4	EVALUATION OF ANALYTICAL DETAILS	11
5	DISCUSSION	11
6	CONCLUSION.....	12

Appendices:

1.	Data, statistical and graphic results	13
2.	Determination of other Organotin Compounds.....	23
3.	Analytical Details	27
4.	Number of participants per country.....	29
5.	Abbreviations and literature.....	30

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 2022/2023 it was decided to continue the proficiency test for the determination of Organotin Compounds in Textile.

In this interlaboratory study 85 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 an ISO/IEC17025 accredited laboratory.

It was decided to send two different textile samples of approximately 3 grams each, both positive on some Organotin compounds, labelled #22810 and #22811 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 gray cotton textile positive on Dioctyltin (DOT) was selected. The batch was cut into small pieces. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #22810.

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

	Dioctyltin (DOT) in mg/kg
sample #22810-1	11.66
sample #22810-2	12.59
sample #22810-3	12.65
sample #22810-4	13.16
sample #22810-5	13.17
sample #22810-6	12.82
sample #22810-7	11.13
sample #22810-8	12.65

Table 1: homogeneity test results of subsamples #22810

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

	Dioctyltin (DOT) in mg/kg
r (observed)	2.01
reference test method	ISO/TS16179:12
0.3 x R (reference test method)	2.41

Table 2: evaluation of the repeatability of subsamples #22810

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

For the second sample a batch of purple cotton textile positive on Tributyltin (TBT) was selected. The batch was cut into small pieces. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #22811. The homogeneity of the subsamples was checked by determination of Tributyltin (TBT) in accordance with ISO17353 on 8 stratified randomly selected subsamples.

	Tributyltin (TBT) in mg/kg
sample #22811-1	5.97
sample #22811-2	6.30
sample #22811-3	6.83
sample #22811-4	6.30
sample #22811-5	6.75
sample #22811-6	6.64
sample #22811-7	6.31
sample #22811-8	6.49

Table 3: homogeneity test results of subsamples #22811

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

	Tributyltin (TBT) in mg/kg
r (observed)	0.80
reference test method	ISO/TS16179:12
0.3 x R (reference test method)	1.25

Table 4: evaluation of the repeatability of subsamples #22811

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

To each of the participating laboratories one sample labelled #22810 and one sample labelled #22811 were sent on November 9, 2022.

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), Mono-octyltin (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:

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

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 some problems were encountered with the dispatch of the samples. Seven participants reported test results after the final reporting date and five other participants did not report any test results. Not all participants were able to report all components requested.

In total 80 participants reported 334 numerical test results. Observed were 15 outlying test results, which is 4.5%. 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 in textile test method ISO/TS16179 is the most used test method. Unfortunately, test method ISO/TS16179 mentions for only three Organotin components precision data that varies greatly from one another (see table B.1 of ISO/TS16179:12) with MBT having an RSD of 23%. Therefore, we decided that in iis PTs on Organotin in Textile to compare all Organotin compounds with a target value of 23% for the evaluation of the test results. This means that the target reproducibility for each Organotin component will be $2.8 * 23 * \text{mean PT} / 100$.

sample #22810

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

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

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 #22811

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

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

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

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 ($2.8 * \text{standard deviation}$) and the target reproducibility derived from reference method are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Monooctyltin (MOT)	mg/kg	70	1.12	0.72	0.72
Diocetyl tin (DOT)	mg/kg	77	12.0	5.3	7.7

Table 5: reproducibilities of tests on sample #22810

Component	unit	n	average	2.8 * sd	R(lit)
Monobutyltin (MBT)	mg/kg	43	0.15	0.10	0.10
Dibutyltin (DBT)	mg/kg	54	0.20	0.15	0.13
Tributyltin (TBT)	mg/kg	75	2.51	1.63	1.62

Table 6: reproducibilities of tests on sample #22811

Without further statistical calculations it can be concluded that for many components there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2022 WITH PREVIOUS PTS

	December 2022	December 2021	December 2020	December 2019	December 2018
Number of reporting laboratories	80	72	85	85	100
Number of test results	334	185	247	317	415
Number of statistical outliers	15	11	12	12	7
Percentage of statistical outliers	4.5%	5.9%	4.9%	3.8%	1.7%

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 2022	December 2021	December 2020	December 2019	2018 – 2016	Reference ISO16179
Monomethyltin (MMT)	---	34%	---	37%	---	23%
Dimethyltin (DMT)	---	23%	---	22%	25-40%	23%
Trimethyltin (TMT)	---	---	---	26%	---	23%
Monobutyltin (MBT)	22%	22%	20-39%	33%	37%	23%
Dibutyltin (DBT)	26%	---	18%	22%	21-35%	23%
Tributyltin (TBT)	23%	---	---	---	29-31%	23%
Monooctyltin (MOT)	23%	---	---	---	---	23%
Diocetyl tin (DOT)	16%	---	---	---	---	23%

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 iis PTs except for DOT, which is remarkably small.

4.4 EVALUATION OF ANALYTICAL DETAILS

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

- About 90% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- About 35% of the participants indicate to have used the samples as received and 65% of the participants further cut or further grinded the samples prior to analysis.
- About 60% of the participants used 1 gram as sample intake. About 35% around 0.5 grams and 5% around 2 or more grams.
- About 90% used Ultrasonic technique to extract/release the components from the samples.
- About 85% 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 50 and 70 °C.
- About 90% reported to observe a pH of 4.5 - 4.6. About 55% have adjusted the pH.

No further statistical analysis has been performed with respect to the analytical details because most of the calculated reproducibilities are in agreement with the requirements of the target reproducibility.

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 OEKO-TEX® Standard 100 (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 #22810 and based on MOT test results two participants would have rejected the sample for all classes and all other participants would have accepted the sample for all classes.

For sample #22810 and based on DOT test results one participant would have rejected the sample for all classes and all other participants would have accepted the sample for all classes.

For sample #22811 and based on MBT test results all participants would have accepted the sample for all classes.

For sample #22811 and based on DBT test results two participants would have rejected the sample for all classes and all other participants would have accepted the sample for all classes.

For sample #22811 and based on TBT test results three participants would have accepted the sample for classes 2, 3 and 4 and two of these three would also have accepted the sample for class 1. All other participants would have rejected the sample for all classes.

	Class 1 Baby clothes in mg/kg	Class 2 Clothes direct skin contact in mg/kg	Class 3 Clothes, no direct contact in mg/kg	Class 4 Decoration material in mg/kg
TBT, TPhT	0.5	1.0	1.0	1.0
Other Organotin compounds	1.0	2.0	2.0	2.0

Table 9: OEKO-TEX® Standard 100 criteria for Organotin in textiles in EU

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 Monoctyltin (MOT) and Dioctyltin (DOT) in sample #22810 and Monobutyltin (MBT), Dibutyltin (DBT) and Tributyltin (TBT) in sample #22811.

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.

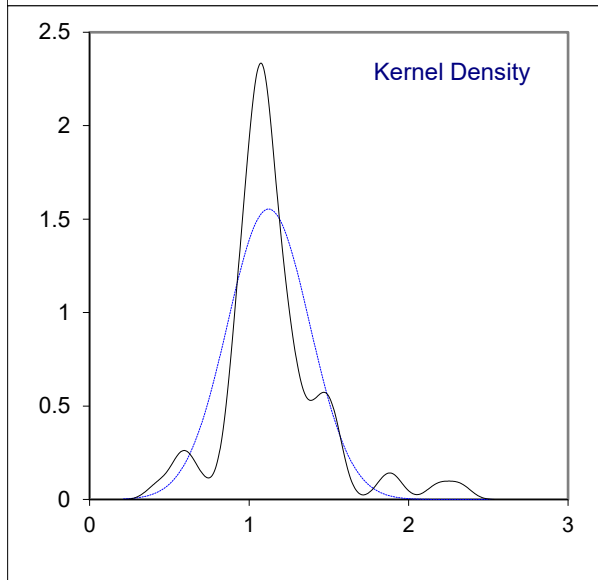
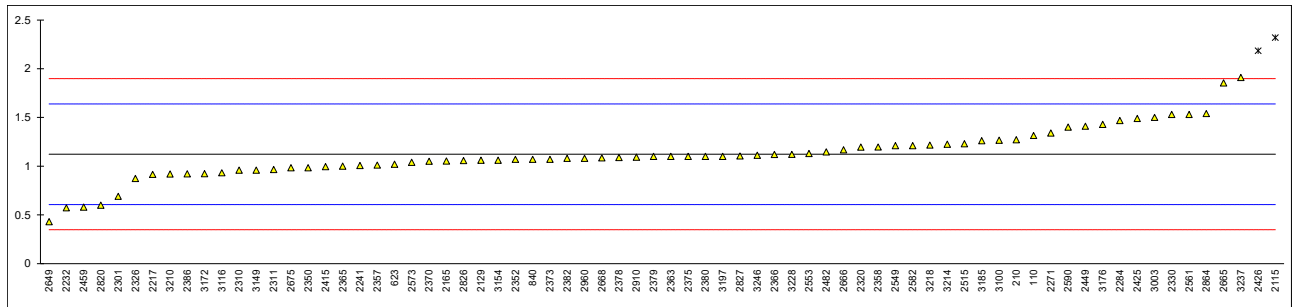
APPENDIX 1

Determination of Monoctyltin (MOT) on sample #22810; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO/TS 16179	1.315		0.74	
210	In house	1.27		0.57	
551		----		----	
623	ISO/TS 16179	1.020		-0.40	
840	ISO22744	1.07		-0.20	
2115	In house	2.32	C,R(0.01)	4.64	First reported 3.782
2129	ISO/TS 16179	1.06		-0.24	
2165	ISO/TS 16179	1.053		-0.27	
2217	ISO/TS 16179	0.916		-0.80	
2232	ISO/TS 16179	0.572		-2.13	
2241	ISO/TS 16179	1.006		-0.45	
2247	ISO/TS 16179	Not detected		----	
2265		----		----	
2271	ISO/TS 16179	1.34		0.84	
2284	ISO/TS 16179	1.468		1.34	
2300	ISO/TS 16179	<0.2	C	<-3.57	Possibly a false negative test result. First reported n.d.
2301	ISO/TS 16179	0.69		-1.68	
2310	ISO22744-1	0.96		-0.63	
2311	ISO22744-1	0.96556		-0.61	
2320	ISO22744-1	1.195		0.28	
2326	ISO22744-1	0.873		-0.97	
2330	ISO/TS 16179	1.530		1.58	
2350	ISO/TS 16179	0.984		-0.54	
2352	GB/T20385.1	1.070		-0.20	
2357	ISO/TS 16179	1.011		-0.43	
2358		1.196		0.28	
2363		1.1		-0.09	
2365	ISO/TS 16179	1.00		-0.48	
2366	ISO/TS 16179	1.12		-0.01	
2370	ISO22744-1	1.05		-0.28	
2373	ISO17353	1.07		-0.20	
2375	ISO22744-1	1.1		-0.09	
2378	ISO22744-1	1.09		-0.13	
2379	ISO/TS 16179	1.0993		-0.09	
2380	ISO/TS 16179	1.1		-0.09	
2382	ISO22744-1	1.08		-0.17	
2386	ISO/TS 16179	0.921		-0.78	
2415	ISO/TS 16179	0.995		-0.49	
2425	In house	1.49		1.42	
2426	ISO/TS 16179	2.185	R(0.01)	4.11	
2449	ISO/TS 16179	1.410		1.11	
2453		----		----	
2459	ISO/TS 16179	0.58		-2.10	
2482	ISO/TS 16179	1.147		0.09	
2515	ISO16179Mod.	1.230		0.42	
2522		----		----	
2525	ISO/TS 16179	<0.1	C	<-3.96	Possibly a false negative test result. First reported n.d.
2549	ISO/TS 16179	1.21		0.34	
2553		1.13		0.03	
2561	ISO22744-1	1.53		1.58	
2569		----		----	
2573	ISO/TS 16179	1.04		-0.32	
2582	In house	1.2100		0.34	
2590	ISO/TS 16179	1.4	C	1.07	First reported 0.57
2591		----		----	
2649	In house	0.43		-2.68	
2665	In house	1.8541		2.83	
2666	ISO/TS 16179	1.168	C	0.17	First reported 1.944
2668	ISO/TS 16179	1.085		-0.15	
2675	In house	0.9830		-0.54	
2678		----		----	
2743		----		----	
2820	ISO/TS 16179	0.6		-2.02	
2826	ISO/TS 16179	1.057		-0.25	
2827	In house	1.104		-0.07	
2864	ISO/TS 16179	1.54		1.62	
2910	ISO/TS 16179	1.093		-0.12	
2960	ISO/TS 16179	1.08		-0.17	
2977		----		----	
3003	ISO/TS 16179	1.5		1.46	
3100	ISO/TS 16179	1.266		0.55	
3116	ISO/TS 16179	0.932		-0.74	
3149	ISO22744-1	0.96		-0.63	
3154	ISO/TS 16179	1.06		-0.24	

lab	method	value	mark	z(targ)	remarks
3172	ISO/TS 16179	0.9226		-0.78	
3176	In house	1.43		1.19	
3185	ISO/TS 16179	1.262		0.54	
3197	ISO/TS 16179	1.1		-0.09	
3210		0.92		-0.79	
3214	ISO/TS 16179	1.225		0.40	
3218	ISO/TS 16179	1.216		0.36	
3228	ISO/TS 16179	1.121		-0.01	
3230		----		----	
3237		1.91		3.05	
3246	ISO/TS 16179	1.112		-0.04	

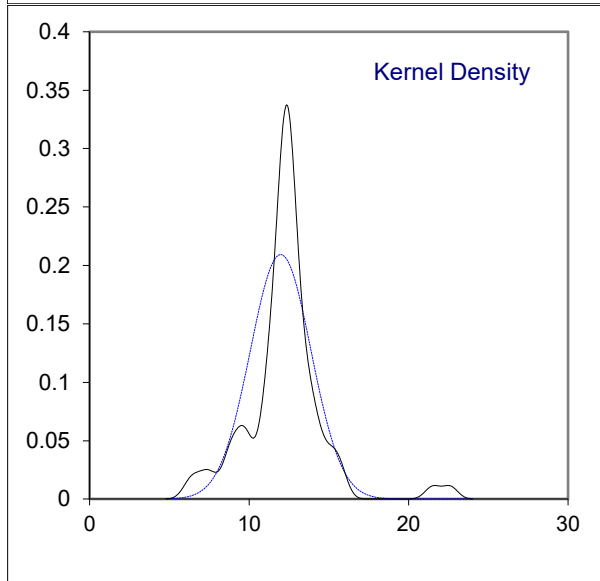
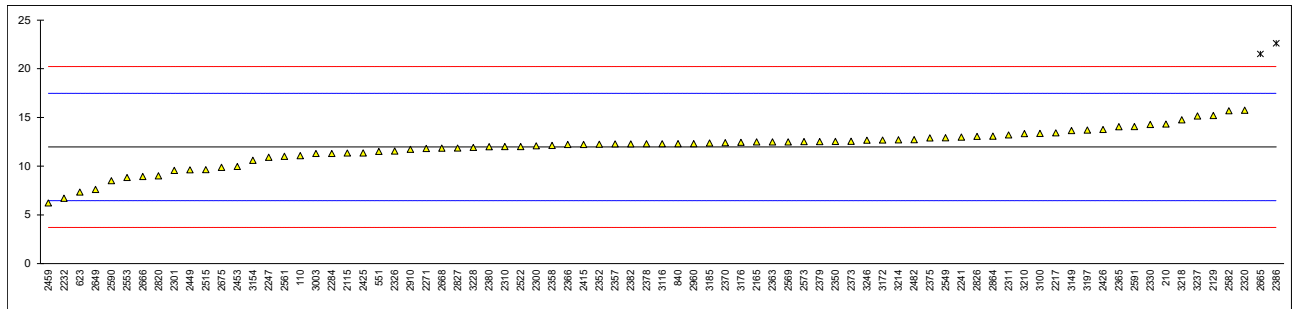
normality suspect
 n 70
 outliers 2
 mean (n) 1.1228
 st.dev. (n) 0.25678 RSD = 23%
 R(calc.) 0.7190
 st.dev.(ISO/TS16179:12) 0.25825
 R(ISO/TS16179:12) 0.7231



Determination of Dioctyltin (DOT) on sample #22810; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO/TS 16179	11.068		-0.33	
210	In house	14.33	C	0.86	First reported 17.27
551	ISO/TS 16179	11.5173		-0.17	
623	ISO/TS 16179	7.349		-1.68	
840	ISO22744	12.31		0.12	
2115	In house	11.34		-0.23	
2129	ISO/TS 16179	15.20		1.17	
2165	ISO/TS 16179	12.49		0.19	
2217	ISO/TS 16179	13.419		0.52	
2232	ISO/TS 16179	6.706		-1.91	
2241	ISO/TS 16179	12.986	C	0.37	First reported 16.340
2247	ISO/TS 16179	10.904		-0.39	
2265		-----		-----	
2271	ISO/TS 16179	11.82		-0.06	
2284	ISO/TS 16179	11.306		-0.24	
2300	ISO/TS 16179	12.09		0.04	
2301	ISO/TS 16179	9.56	C	-0.88	First reported 8.4
2310	ISO22744-1	12.01		0.01	
2311	ISO22744-1	13.211		0.45	
2320	ISO22744-1	15.737		1.37	
2326	ISO22744-1	11.564		-0.15	
2330	ISO/TS 16179	14.283		0.84	
2350	ISO/TS 16179	12.536		0.20	
2352	GB/T20385.1	12.260		0.10	
2357	ISO/TS 16179	12.290		0.11	
2358		12.133		0.06	
2363		12.5		0.19	
2365	ISO/TS 16179	14.05		0.75	
2366	ISO/TS 16179	12.22		0.09	
2370	ISO22744-1	12.4		0.15	
2373	ISO17353	12.56		0.21	
2375	ISO22744-1	12.9		0.34	
2378	ISO22744-1	12.3		0.12	
2379	ISO/TS 16179	12.5260		0.20	
2380	ISO/TS 16179	12.0		0.01	
2382	ISO22744-1	12.29		0.11	
2386	ISO/TS 16179	22.6	R(0.01)	3.86	
2415	ISO/TS 16179	12.221		0.09	
2425	In house	11.36		-0.22	
2426	ISO/TS 16179	13.77		0.65	
2449	ISO/TS 16179	9.621		-0.85	
2453		9.967		-0.73	
2459	ISO/TS 16179	6.23		-2.09	
2482	ISO/TS 16179	12.72		0.27	
2515	ISO16179Mod.	9.643		-0.85	
2522	ISO/TS 16179	12.01		0.01	
2525	ISO/TS 16179	<0.1	C	<-4.31	Possibly a false negative test result. First reported n.d.
2549	ISO/TS 16179	12.92		0.34	
2553		8.84		-1.14	
2561	ISO22744-1	11.01		-0.35	
2569	ISO/TS 16179	12.5		0.19	
2573	ISO/TS 16179	12.52		0.20	
2582	In house	15.6893		1.35	
2590	ISO/TS 16179	8.5	C	-1.26	First reported 6.66
2591	In house	14.067		0.76	
2649	In house	7.62		-1.58	
2665	In house	21.510	R(0.01)	3.46	
2666	ISO/TS 16179	8.951		-1.10	
2668	ISO/TS 16179	11.84		-0.05	
2675	In house	9.8815		-0.76	
2678		-----		-----	
2743		-----		-----	
2820	ISO/TS 16179	9.0		-1.08	
2826	ISO/TS 16179	13.047		0.39	
2827	In house	11.866		-0.04	
2864	ISO/TS 16179	13.08		0.40	
2910	ISO/TS 16179	11.72		-0.09	
2960	ISO/TS 16179	12.31		0.12	
2977		-----		-----	
3003	ISO/TS 16179	11.3		-0.24	
3100	ISO/TS 16179	13.360		0.50	
3116	ISO/TS 16179	12.3		0.12	
3149	ISO22744-1	13.66		0.61	
3154	ISO/TS 16179	10.61		-0.50	

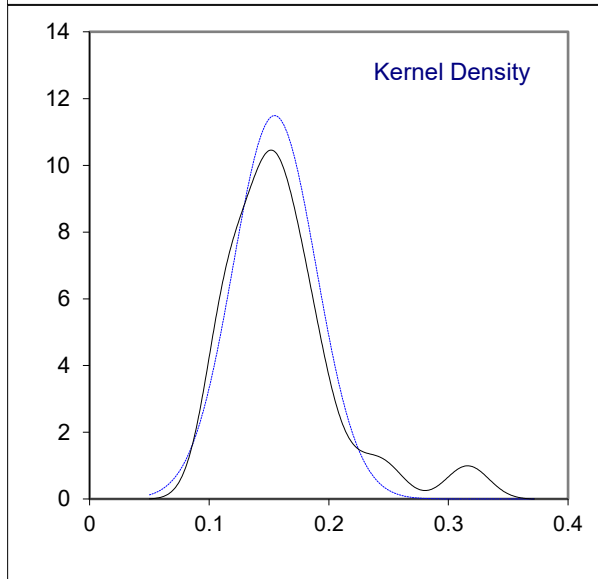
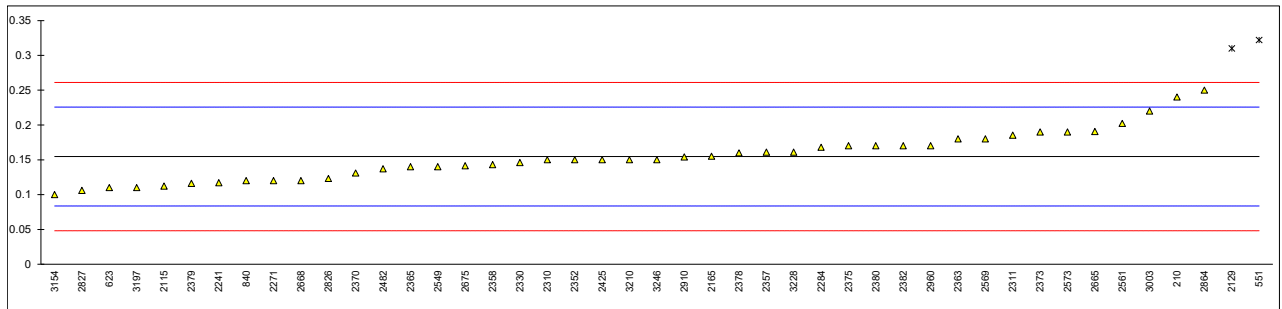
lab	method	value	mark	z(targ)	remarks
3172	ISO/TS 16179	12.677		0.25	
3176	In house	12.45	C	0.17	First reported 16.96
3185	ISO/TS 16179	12.380		0.15	
3197	ISO/TS 16179	13.7		0.63	
3210		13.34		0.50	
3214	ISO/TS 16179	12.710		0.27	
3218	ISO/TS 16179	14.765		1.01	
3228	ISO/TS 16179	11.92		-0.02	
3230		----		----	
3237		15.15		1.15	
3246	ISO/TS 16179	12.666		0.25	
normality		suspect			
n		77			
outliers		2			
mean (n)		11.9748			
st.dev. (n)		1.90614	RSD = 16%		
R(calc.)		5.3372			
st.dev.(ISO/TS16179:12)		2.75420			
R(ISO/TS16179:12)		7.7118			



Determination of Monobutyltin (MBT) on sample #22811; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO/TS 16179	<0.1		----	
210	In house	0.24		2.40	
551	ISO/TS 16179	0.3220	R(0.01)	4.71	
623	ISO/TS 16179	0.110		-1.26	
840	ISO/TS 16179Mod.	0.12	C	-0.97	First reported "not detected"
2115	In house	0.112		-1.20	
2129	ISO/TS 16179	0.31	R(0.01)	4.37	
2165	ISO/TS 16179	0.155		0.01	
2217		----		----	
2232		----		----	
2241	ISO/TS 16179	0.117		-1.06	
2247	ISO/TS 16179	not detected		----	
2265		----		----	
2271	ISO/TS 16179	0.12		-0.97	
2284	ISO/TS 16179	0.168		0.38	
2300	ISO/TS 16179	<0.2	C	----	First reported "not detected"
2301		----		----	
2310	ISO22744-1	0.15		-0.13	
2311	ISO22744-1	0.1853		0.86	
2320	ISO22744-1	<0.1	C	----	First reported "not detected"
2326	ISO22744-1	ND		----	
2330	ISO/TS 16179	0.146		-0.24	
2350	ISO/TS 16179	< 0.5		----	
2352	GB/T20385.1	0.150		-0.13	
2357	ISO/TS 16179	0.161		0.18	
2358		0.143		-0.33	
2363	ISO22744	0.18		0.71	
2365	ISO/TS 16179	0.14		-0.41	
2366	ISO/TS 16179	<0.5		----	
2370	ISO17353	0.131		-0.66	
2373	ISO17353	0.19		0.99	
2375	ISO22744-1	0.17		0.43	
2378	ISO22744-1	0.16		0.15	
2379	ISO22744-1	0.1162		-1.08	
2380	ISO/TS 16179	0.17		0.43	
2382	ISO22744-1	0.17		0.43	
2386	ISO/TS 16179	<0.20		----	
2415		----		----	
2425	In house	0.15		-0.13	
2426	ISO/TS 16179	Not detected		----	
2449		----		----	
2453		----		----	
2459	ISO/TS 16179	ND		----	
2482	ISO/TS 16179	0.1371		-0.49	
2515		----		----	
2522		----		----	
2525	ISO/TS 16179	<0.1	C	----	First reported "not detected"
2549	ISO/TS 16179	0.14		-0.41	
2553		----		----	
2561	ISO22744-1	0.2022	C	1.34	First reported "not detected"
2569	ISO17353	0.18		0.71	
2573	ISO/TS 16179	0.19		0.99	
2582	In house	Not detected		----	
2590		----		----	
2591		----		----	
2649		----		----	
2665	In house	0.1905		1.01	
2666		----		----	
2668	ISO/TS 16179	0.12		-0.97	
2675	In house	0.1413		-0.38	
2678		----		----	
2743		----		----	
2820		----		----	
2826	ISO/TS 16179	0.1231		-0.89	
2827	In house	0.106		-1.37	
2864	ISO/TS 16179	0.25	C	2.68	First reported "not detected"
2910	ISO/TS 16179	0.154		-0.02	
2960	ISO/TS 16179	0.17		0.43	
2977		----		----	
3003	ISO/TS 16179	0.22		1.84	
3100	ISO/TS 16179	<0.20		----	
3116		----		----	
3149		----		----	
3154	ISO/TS 16179	0.1	C	-1.54	First reported "not detected"

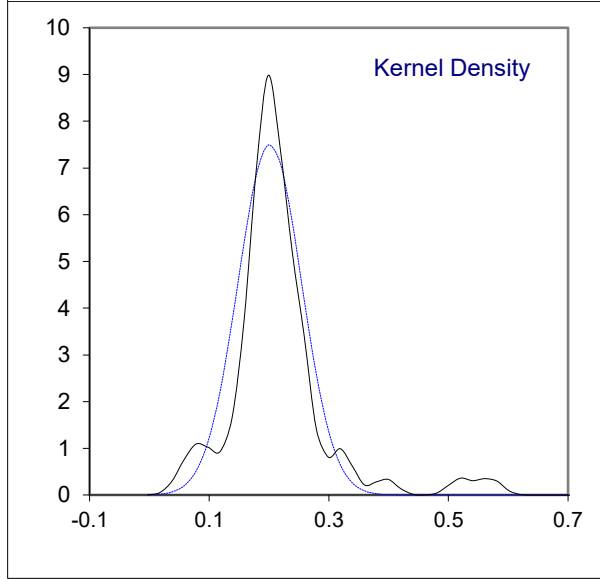
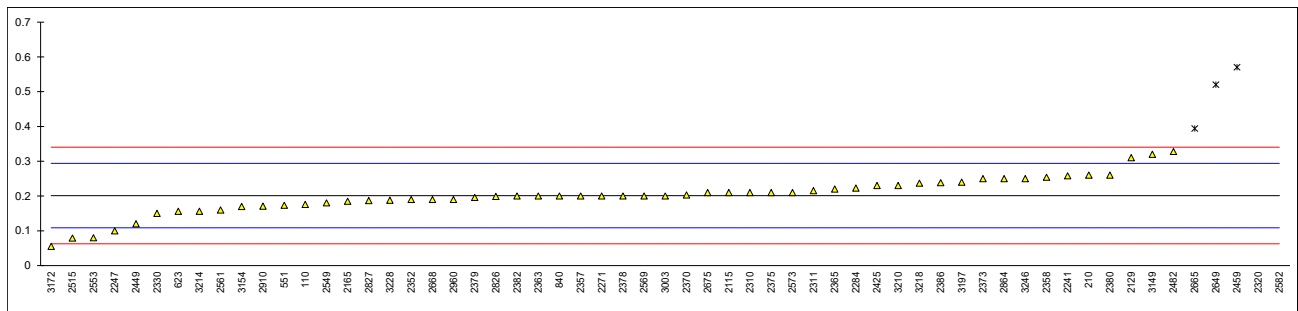
lab	method	value	mark	z(targ)	remarks
3172	ISO/TS 16179	< 0.02		----	
3176		----		----	
3185	ISO/TS 16179	<0.2		----	
3197	ISO/TS 16179	0.11		-1.26	
3210		0.15		-0.13	
3214	ISO/TS 16179	<0.1		----	
3218	ISO/TS 16179	<0.20	C	----	First reported "not detected"
3228	ISO/TS 16179	0.161		0.18	
3230		----		----	
3237		----		----	
3246	ISO/TS 16179	0.150		-0.13	
normality		OK			
n		43			
outliers		2			
mean (n)		0.15464			
st.dev. (n)		0.034708	RSD = 22%		
R(calc.)		0.09718			
st.dev.(ISO/TS16179:12)		0.035568			
R(ISO/TS16179:12)		0.09959			



Determination of Dibutyltin (DBT) on sample #22811; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO/TS 16179	0.176		-0.55	
210	In house	0.26		1.26	
551	ISO/TS 16179	0.1734		-0.61	
623	ISO/TS 16179	0.156		-0.98	
840	ISO/TS 16179Mod.	0.20	C	-0.03	First reported "not detected"
2115	In house	0.21	C	0.18	First reported 0.458
2129	ISO/TS 16179	0.31		2.34	
2165	ISO/TS 16179	0.185		-0.35	
2217		----		----	
2232		----		----	
2241	ISO/TS 16179	0.258		1.22	
2247	ISO/TS 16179	0.100		-2.19	
2265		----		----	
2271	ISO/TS 16179	0.200		-0.03	
2284	ISO/TS 16179	0.223		0.47	
2300	ISO/TS 16179	<0.2	C	----	First reported "not detected"
2301		----		----	
2310	ISO22744-1	0.21		0.18	
2311	ISO22744-1	0.2155		0.30	
2320	ISO22744-1	4.797	C,R(0.01)	99.19	First reported 2.712
2326	ISO22744-1	ND		----	
2330	ISO/TS 16179	0.150		-1.11	
2350	ISO/TS 16179	< 0.5		----	
2352	GB/T20385.1	0.190		-0.25	
2357	ISO/TS 16179	0.200		-0.03	
2358		0.254		1.13	
2363	ISO22744	0.20		-0.03	
2365	ISO/TS 16179	0.22		0.40	
2366	ISO/TS 16179	<0.5		----	
2370	ISO17353	0.203		0.03	
2373	ISO17353	0.25		1.05	
2375	ISO22744-1	0.21		0.18	
2378	ISO22744-1	0.20		-0.03	
2379	ISO22744-1	0.1960		-0.12	
2380	ISO/TS 16179	0.26		1.26	
2382	ISO22744-1	0.20		-0.03	
2386	ISO/TS 16179	0.238		0.79	
2415		----		----	
2425	In house	0.23		0.62	
2426	ISO/TS 16179	Not detected		----	
2449	ISO/TS 16179	0.12	C	-1.76	First reported 0.052
2453		----		----	
2459	ISO/TS 16179	0.57	R(0.01)	7.96	
2482	ISO/TS 16179	0.3278		2.73	
2515	ISO 16179Mod.	0.079		-2.64	
2522	ISO/TS 16179	<0.1		----	
2525	ISO/TS 16179	<0.1	C	----	First reported "not detected"
2549	ISO/TS 16179	0.18		-0.46	
2553		0.08	C	-2.62	First reported 0.05
2561	ISO22744-1	0.16		-0.89	
2569	ISO17353	0.2		-0.03	
2573	ISO/TS 16179	0.21		0.18	
2582	In house	5.0699	R(0.01)	105.08	
2590		----		----	
2591	In house	Not detected		----	
2649	In house	0.52	R(0.01)	6.88	
2665	In house	0.3937	R(0.05)	4.15	
2666		----		----	
2668	ISO/TS 16179	0.19		-0.25	
2675	In house	0.2096		0.18	
2678		----		----	
2743		----		----	
2820		----		----	
2826	ISO/TS 16179	0.1986		-0.06	
2827	In house	0.187		-0.31	
2864	ISO/TS 16179	0.25	C	1.05	First reported "not detected"
2910	ISO/TS 16179	0.171		-0.66	
2960	ISO/TS 16179	0.19		-0.25	
2977		----		----	
3003	ISO/TS 16179	0.20		-0.03	
3100	ISO/TS 16179	<0.20		----	
3116		----		----	
3149	ISO22744-1	0.32		2.56	
3154	ISO/TS 16179	0.17	C	-0.68	First reported "not detected"

lab	method	value	mark	z(targ)	remarks
3172	ISO/TS 16179	0.0555		-3.15	
3176		----		----	
3185	ISO/TS 16179	<0.2		----	
3197	ISO/TS 16179	0.24		0.83	
3210		0.23		0.62	
3214	ISO/TS 16179	0.156		-0.98	
3218	ISO/TS 16179	0.237		0.77	
3228	ISO/TS 16179	0.188		-0.29	
3230		----		----	
3237		----		----	
3246	ISO/TS 16179	0.250		1.05	
normality		suspect			
n		54			
outliers		5			
mean (n)		0.20143			
st.dev. (n)		0.053228	RSD = 26%		
R(calc.)		0.14904			
st.dev.(ISO/TS16179:12)		0.046330			
R(ISO/TS16179:12)		0.12972			

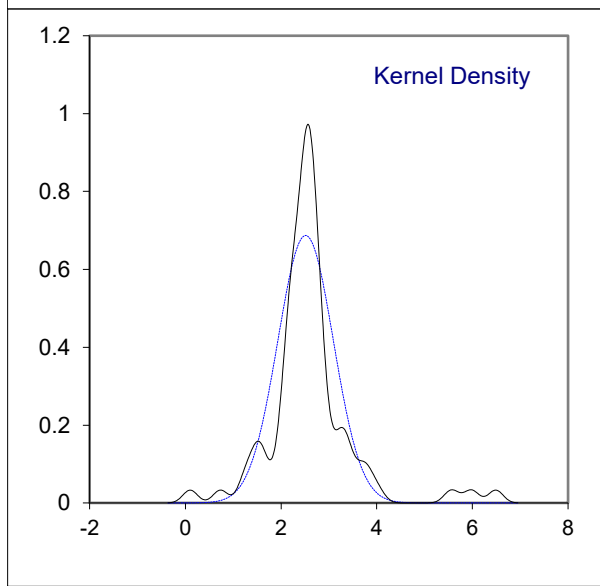
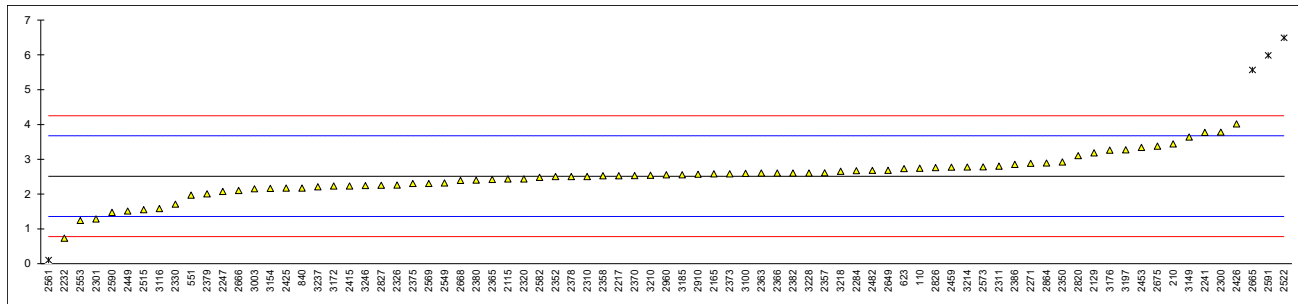


Determination of Tributyltin (TBT) on sample #22811; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO/TS 16179	2.742		0.39	
210	In house	3.44		1.60	
551	ISO/TS 16179	1.9679		-0.94	
623	ISO/TS 16179	2.727		0.37	
840	ISO/TS 16179Mod.	2.17		-0.60	
2115	In house	2.433		-0.14	
2129	ISO/TS 16179	3.18		1.15	
2165	ISO/TS 16179	2.58		0.11	
2217	ISO/TS 16179	2.527		0.02	
2232	ISO/TS 16179	0.733		-3.08	
2241	ISO/TS 16179	3.770		2.17	
2247	ISO/TS 16179	2.071		-0.77	
2265		-----		-----	
2271	ISO/TS 16179	2.88		0.63	
2284	ISO/TS 16179	2.668		0.27	
2300	ISO/TS 16179	3.78	C	2.19	First reported 4.59
2301		1.28	C	-2.13	First reported "not detected"
2310	ISO22744-1	2.50		-0.02	
2311	ISO22744-1	2.8017		0.50	
2320	ISO22744-1	2.433		-0.14	
2326	ISO22744-1	2.257		-0.44	
2330	ISO/TS 16179	1.710		-1.39	
2350	ISO/TS 16179	2.920		0.70	
2352	GB/T20385.1	2.500		-0.02	
2357	ISO/TS 16179	2.610		0.17	
2358		2.526		0.02	
2363	ISO22744	2.60		0.15	
2365	ISO/TS 16179	2.42		-0.16	
2366	ISO/TS 16179	2.60		0.15	
2370	ISO17353	2.53		0.03	
2373	ISO17353	2.58		0.11	
2375	ISO22744-1	2.3		-0.37	
2378	ISO22744-1	2.5		-0.02	
2379	ISO22744-1	2.0035		-0.88	
2380	ISO/TS 16179	2.4		-0.20	
2382	ISO22744-1	2.60		0.15	
2386	ISO/TS 16179	2.85		0.58	
2415	ISO/TS 16179	2.231		-0.49	
2425	In house	2.17		-0.60	
2426	ISO/TS 16179	4.015		2.60	
2449	ISO/TS 16179	1.51		-1.74	
2453		3.339		1.43	
2459	ISO/TS 16179	2.77		0.44	
2482	ISO/TS 16179	2.676		0.28	
2515	ISO 16179Mod.	1.55	C	-1.67	First reported 1.087
2522	ISO/TS 16179	6.49	C,R(0.01)	6.88	First reported 4.47
2525	ISO/TS 16179	<0.1	C	<-4.17	Possibly a false negative test result. First reported n.d.
2549	ISO/TS 16179	2.32		-0.34	
2553		1.24		-2.20	
2561	ISO22744-1	0.10	R(0.01)	-4.17	
2569	ISO17353	2.3		-0.37	
2573	ISO/TS 16179	2.78		0.46	
2582	In house	2.4799		-0.06	
2590	ISO/TS 16179	1.47		-1.81	
2591	In house	5.981	R(0.01)	6.00	
2649	In house	2.68		0.29	
2665	In house	5.5655	R(0.01)	5.28	
2666	ISO/TS 16179	2.101		-0.71	
2668	ISO/TS 16179	2.39		-0.21	
2675	In house	3.3780		1.49	
2678		-----		-----	
2743		-----		-----	
2820	ISO/TS 16179	3.1		1.01	
2826	ISO/TS 16179	2.760		0.43	
2827	In house	2.250		-0.46	
2864	ISO/TS 16179	2.89		0.65	
2910	ISO/TS 16179	2.566		0.09	
2960	ISO/TS 16179	2.55		0.06	
2977		-----		-----	
3003	ISO/TS 16179	2.15		-0.63	
3100	ISO/TS 16179	2.594		0.14	
3116	ISO/TS 16179	1.58		-1.62	
3149	ISO22744-1	3.64	C	1.95	First reported 4.79
3154	ISO/TS 16179	2.16		-0.61	

lab	method	value	mark	z(targ)	remarks
3172	ISO/TS 16179	2.2297		-0.49	
3176	In house	3.26		1.29	
3185	ISO/TS 16179	2.550		0.06	
3197	ISO/TS 16179	3.27		1.31	
3210		2.54		0.04	
3214	ISO/TS 16179	2.775		0.45	
3218	ISO/TS 16179	2.654		0.24	
3228	ISO/TS 16179	2.601		0.15	
3230		----		----	
3237		2.21		-0.53	
3246	ISO/TS 16179	2.242		-0.47	

normality suspect
 n 75
 outliers 4
 mean (n) 2.5142
 st.dev. (n) 0.58106 RSD = 23%
 R(calc.) 1.6270
 st.dev.(ISO/TS16179:12) 0.57826
 R(ISO/TS16179:12) 1.6191



APPENDIX 2**Determination of other Organotin components on sample #22810; results in mg/kg**

MMT = Monomethyltin
 DMT = Dimethyltin
 TMT = Trimethyltin
 TPT = Tripropyltin
 MBT = Monobutyltin
 DBT = Dibutyltin

TBT = Tributyltin
 TeBT = Tetrabutyltin
 TOT = Trioctyltin
 DPhT = Diphenyltin
 TPHT = Triphenyltin
 TCyHT = Tricyclohexyltin

lab	MMT	DMT	TMT	TPT	MBT	DBT	TBT	TeBT	TOT	DPhT	TPHT	TCyHT
110	<0.1	<0.1	<0.1	<0.1	<0.1	0.146	0.112	<0.1	<0.1	<0.1	<0.1	<0.1
210	----	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	0.1665	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected
2115	----	0.054	----	----	0.082	----	----	----	0.057	----	----	----
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2165	----	----	----	----	not detected	not detected	0.095	not detected	----	----	not detected	not detected
2217	----	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----	----
2241	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.091	not detected	not detected	not detected
2247	not detected	not detected	not detected	not detected	0.055	detected	0.101	detected	detected	detected	detected	detected
2265	----	----	----	----	----	----	----	----	----	----	----	----
2271	not detected	not detected	not detected	not detected	not detected	not detected	0.12	not detected	not detected	not detected	not detected	not detected
2284	<0.025	<0.025	<0.025	<0.025	0.048	<0.025	0.112	<0.025	0.061	<0.025	<0.025	<0.025
2300	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2301	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected
2310	not detected	not detected	not detected	not detected	not detected	not detected	0.09	not detected	not detected	not detected	not detected	not detected
2311	detected	<0.025	detected	detected	<0.025	detected	0.1149	detected	<0.025	detected	detected	detected
2320	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2326	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2330	not detected	not detected	not detected	not detected	not detected	not detected	0.127	not detected	not detected	not detected	not detected	not detected
2350	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
2352	----	----	----	----	----	----	0.100	----	----	----	----	----
2357	----	----	----	----	----	----	0.100	----	----	----	----	----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not detected	not detected	not detected	0.1	not detected	0.1	not detected	not detected	not detected
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.06	<0.05	<0.05	<0.05
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2370	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.123	<0.02	0.0688	<0.02	<0.02	<0.02
2373	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2375	----	----	----	----	----	----	0.12	----	----	----	----	----
2378	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.0530	not detected	not detected	not detected
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2382	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.10	<0.05	0.10	<0.05	<0.05	<0.05
2386	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2415	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected
2425	not detected	not detected	not detected	not detected	not detected	not detected	0.13	not detected	0.06	not detected	not detected	not detected
2426	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected
2449	----	----	----	----	0.051	----	0.152	----	0.061	----	----	----
2453	----	----	----	----	----	----	0.2550	----	----	----	----	----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 0,05	< 0,05	< 0,05	< 0,05	0.0384	< 0,025	0.0726	< 0,025	0.0697	< 0,1	< 0,1	< 0,1

lab	MMT	DMT	TMT	TPT	MBT	DBT	TBT	TeBT	TOT	DPhT	TPhT	TCyHT
2515	----	----	----	----	----	----	0.146	----	0.051	----	----	----
2522	----	----	----	----	----	<0.1	<0.1	----	----	----	<0.1	----
2525	not detected	not detected	not detected	not detected	0.2223	0.14	3.8147	not detected	detected	not detected	not detected	not detected
2549	not detected	not detected	not detected	not detected	not detected	not detected	0.11	not detected	0.07	not detected	not detected	not detected
2553	----	----	----	----	----	----	0.09	----	0.05	----	----	----
2561	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.07	not detected	not detected	not detected
2569	----	not detected	not detected	not detected	not detected	not detected	0.11	not detected	not detected	not detected	not detected	not detected
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2582	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2590	----	----	----	----	----	----	----	----	----	----	----	----
2591	----	----	not detected	not detected	----	not detected	not detected	not detected	not detected	----	not detected	not detected
2649	----	----	----	----	----	----	----	----	----	----	----	----
2665	<0.001	0.0289	0.0024	<0.001	0.0387	<0.001	0.0640	<0.001	0.0357	<0.001	<0.001	<0.001
2666	----	----	----	----	----	----	----	----	----	----	----	----
2668	not detected	not detected	not detected	not detected	not detected	not detected	0.094	not detected	0.054	not detected	not detected	not detected
2675	not detected	0.1332	not detected	0.0437	0.0370	not detected	0.1910	----	0.0610	----	not detected	not detected
2678	----	----	----	----	----	----	----	----	----	----	----	----
2743	----	----	----	----	----	----	----	----	----	----	----	----
2820	----	----	----	----	----	----	----	----	----	----	----	----
2826	----	----	----	not detected	not detected	not detected	0.1047	not detected	not detected	----	not detected	not detected
2827	not detected	not detected	not detected	not detected	not detected	not detected	0.103	not detected	0.063	not detected	not detected	not detected
2864	----	----	----	----	not detected	not detected	not detected	not detected	----	----	not detected	not detected
2910	----	----	----	----	not detected	not detected	0.113	not detected	----	----	not detected	not detected
2960	not detected	not detected	not detected	not detected	not detected	not detected	0.09	not detected	not detected	not detected	not detected	not detected
2977	----	----	----	----	----	----	----	----	----	----	----	----
3003	----	----	----	----	0.05	----	0.11	----	0.05	----	----	----
3100	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
3116	----	----	----	----	----	----	0.108	----	----	----	----	----
3149	----	----	----	----	----	----	0.12	----	----	----	----	----
3154	not detected	0.02	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3172	<0.02	0.0343	<0.02	<0.02	0.0337	<0.02	<0.02	<0.02	0.0653	<0.02	<0.02	<0.02
3176	----	----	----	----	----	----	----	----	----	----	----	----
3185	----	----	----	----	<0.2	<0.2	<0.2	<0.2	----	----	<0.2	<0.2
3197	<0,02	<0,02	<0,02	<0,02	<0,02	<0,02	0.09	<0,02	0.14	<0,02	<0,02	<0,02
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	----	<0.1	<0.1	<0.1	<0.1	<0.1
3214	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3218	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3228	----	----	----	----	not detected	not detected	0.101	not detected	----	----	not detected	not detected
3230	----	----	----	----	----	----	----	----	----	----	----	----
3237	----	----	----	----	----	----	----	----	----	----	----	----
3246	not detected	not detected	not detected	not detected	----	not detected	----	not detected	----	not detected	not detected	not detected

Lab 2301 first reported for TBT 0.018

Lab 2379 first reported for TOT 0.2041

Lab 2525 first reported for DBT not detected

Lab 2675 first reported for TBT 0.2098

Lab 3116 first reported for TBT 0.0540

Lab 3154 first reported for DMT 0.02

Determination of other Organotin components on sample #22811; results in mg/kg

MMT = Monomethyltin
 DMT = Dimethyltin
 TMT = Trimethyltin
 TPT = Tripropyltin
 TeBT = Tetrabutyltin
 MOT = Monoctyltin

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

lab	MMT	DMT	TMT	TPT	TeBT	MOT	DOT	TOT	DPHT	TPhT	TCyHT
110	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
210	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	----	0.064	----	----	----	----	----	----	----	----	----
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2165	----	----	----	----	not detected	not detected	not detected	----	----	not detected	not detected
2217	----	----	----	----	----	----	----	----	----	----	----
2232	----	----	----	----	----	----	----	----	----	----	----
2241	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2247	not detected	not detected	not detected	not detected	not detected	not detected	0.148	not detected	not detected	not detected	not detected
2265	----	----	----	----	----	----	----	----	----	----	----
2271	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2284	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2300	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2301	----	----	----	----	----	----	----	not detected	----	----	----
2310	not detected	not detected	not detected	not detected	not detected	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	not detected	not detected	not detected	not detected	not detected
2320	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2326	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2330	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2350	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
2352	----	----	----	----	----	----	----	----	----	----	----
2357	----	----	----	----	----	----	----	----	----	----	----
2358	----	----	----	----	----	----	----	----	----	----	----
2363	not detected	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	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2370	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2373	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2375	----	----	----	----	----	----	----	----	----	----	----
2378	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2382	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2386	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2415	----	----	----	----	----	----	----	----	----	----	----
2425	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2426	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2449	----	----	0.0251	----	----	----	----	----	----	----	----
2453	----	----	----	----	----	----	----	----	----	----	----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 0,05	< 0,05	< 0,05	< 0,05	< 0,025	< 0,025	< 0,025	< 0,1	< 0,1	< 0,1	< 0,1
2515	----	----	----	----	----	----	----	----	----	----	----

APPENDIX 3 Analytical details

lab	ISO/IEC 17025 accredited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding the buffer	Acidified to pH 4.5
110	Yes	Further cut	---	Ultrasonic	MeOH : EtOH (80:20 V/V)	60 min	60°C	6.10-6.15	No
210	Yes	Further cut	1g	Ultrasonic					---
551	Yes	Further cut	0.5g	Ultrasonic	MeOH:EtOH 80/20%	60 min	60 °C	4.5	No
623	Yes	Further cut	1	Ultrasonic	methanol : ethanol	60	60		---
840	No	Further cut	0.5	Ultrasonic	MEOH:ETHANOL (8:2)	60	60	4.5	No
2115	Yes	Used as received	1 g	Ultrasonic	EtOH - Acetic acid (95:5)	60 min	60 °C		Yes
2129	Yes	Used as received	0.6g	Ultrasonic	HCl solution in methanol	60 min	60 °C	4,66	No
2165	Yes	Further cut	1.0g	Mechanical shaking	methanol : ethanol (8:2) isooctane	60 min.	60 °C	4.5	Yes
2217	Yes	Used as received	0.9986 g	Ultrasonic	methanol: ethanol 80:20	30 min	60	4.5	No
2232	Yes	Further cut	1g	Ultrasonic	Methanol:Ethanol(80:20)	60 min	60°C	5.6	Yes
2241	Yes	Further cut	0.5g	Ultrasonic	The mixture solution of MeOH and Ethanol(4:1).	60 min	60 °C.	4.0-4.5.	No
2247	Yes	Further cut	---	Mechanical Shaking / Ultrasonic	80:20 (MeOH:EtOH)	60 min	60 °C	4.5	No
2265	---	---	---	---					---
2271	Yes	Further cut	0.5g	Ultrasonic	methanol/ethanol mixture (80/20 in volume).	60 min	60 °C	4.5	No
2284	Yes	Further cut	1g	Ultrasonic	Methanol: ethanol =4:1	1h	60	4.5	Yes
2300	Yes	Further cut	1 gram.	Ultrasonic	Methanol.	60 min	60°C	4.5	No
2301	No	Further cut	1 g.	Ultrasonic	Methanol/Ethanol	60 min	70c	5.6	Yes
2310	Yes	Further cut	1	Mechanical Shaking	Hexane	60	60	4.5-5.0	Yes
2311	Yes	Further cut	0.5	Ultrasonic	Methanol + Ethanol (80:20)	60	60	4.5	No
2320	Yes	Further cut	0.5g	Ultrasonic	Methanol:Ethanol (80:20)	60min	60 °C	4.5	Yes
2326	Yes	Further cut	1gm	Ultrasonic	Methanol Extraction	60 min	60°C	4.5	Yes
2330	No	Further cut	0.50g	Ultrasonic	Ethanol/Methanol	60 min	60 C	4.5	Yes
2350	No	Further cut	1 g	Ultrasonic	Methanol : Ethanol (8:2)	60 min	60 °C	4.5	No
2352	Yes	Further cut	1.0g	Ultrasonic	methanol & ethanol	60min	60°C	4.5	Yes
2357	---	---	---	---					---
2358	Yes	Used as received	1.0	Ultrasonic	Ethanol/Methanol mixture	60	60	NA	Yes
2363	Yes	Further cut	2.5g	Ultrasonic	Methanol: Ethanol=4:1	60 min	60 °C	4.5	No
2365	Yes	Further cut	1.0g	Ultrasonic	20 mL methanol-ethanol mixture	60min	60 °C	4.5	Yes
2366	No	Further cut	1g	Ultrasonic					No
2370	Yes	Further cut	1 g	Ultrasonic	#22810 ethanol/methanol 1:4, #22811 ethanol	60 min	60°C / room	4.5	Yes
2373	Yes	Further cut	1.0g	Ultrasonic	n-Hexane	1h	40°C	4.5	No
2375	Yes	Further cut	0.5 gram	Ultrasonic	Methanol/Ethanol	60 min	60 C	4.5	Yes
2378	Yes	Used as received	0.5g	Mechanical Shaking	N-Hexane	60min	60 °C	4.5	Yes
2379	Yes	Further cut	1 g	Ultrasonic	MeOH :EtOH 80:20 V/V	60 min	60 C	4.5	Yes
2380	Yes	Further cut	1.0 g	Ultrasonic	Methanol & Ethanol Mixture	60	60 °C	5.8	Yes
2382	Yes	Further cut	1.0g	Ultrasonic	Hexane	60min	60 °C	4.5	Yes
2386	Yes	Used as received	1 g	Ultrasonic	Methanol/Ethanol (80/20 V/V)	60 min	60 °C	4.5	No
2415	Yes	Used as received	0.5	Ultrasonic	MeOH:EtOH (8:2 v/v)	60	60	4.5	Yes
2425	Yes	Further cut	0.5g	Ultrasonic	80:20 (Methanol: Ethanol)	60 min	60° C	N/A	Yes
2426	Yes	Further cut	0.5 gram	Ultrasonic	Methanol (Ethanol Mixture)	60 min	60°C	4.5	No
2449	Yes	Further cut	1 GRAM	Ultrasonic	Sodium carbamate in methanol	60 min	70 C	4.5	Yes
2453	Yes	Further cut	±1g	Thermal Desorption					---
2459	Yes	Further grinded	1.00 gm	Ultrasonic	Methanol:Ethanol (80:20)	60min	60°C	4.5	No
2482	Yes	Used as received	0,5	Ultrasonic	Methanol:Ethanol 80:20	60	60		No
2515	Yes	Used as received	1gram	Ultrasonic	methanol/ethanol (80/20)	1hours	60 °C	4.5	No
2522	Yes	Used as received	1 g	Ultrasonic	isooctane	60 min	60 °C	4.5	Yes
2525	Yes	Used as received	1,0 g	Ultrasonic	Methanol/Ethanol (80:20)	60 min	60 °C	4,5	No
2549	Yes	Used as received	1.0 gm	Ultrasonic	Ethanol: Methanol	60	60	5.7	Yes
2553	Yes	Used as received	1g	Mechanical Shaking	80% methanol/20% ethanol v/v	60 min	60 °C	4.5	Yes
2561	Yes	Used as received	1g	Mechanical Shaking	80:20 mix of methanol:ethanol	60	60 °C	4.5	Yes
2569	Yes	Further cut	1gm	Ultrasonic	Methanol & Ethanol mixture	60 Min	60 °C		Yes

lab	ISO/IEC 17025 accredited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding the buffer	Acidified to pH 4.5
2573	Yes	Used as received	1g	Ultrasonic	methanol/ethanol(4:1)	60 min	60°C	4.5	Yes
2582	Yes	Further cut	1 gram	Ultrasonic	Acetone	60 min	room	4.5	No
2590	Yes	Used as received	1g	Ultrasonic	meoh:etoh 80:20	60 min	60°C		Yes
2591	No	Further cut	1.0 gram	Ultrasonic	MeOH/EtOH 80/20	60 min	60°C		No
2649	Yes	Further cut	1 g	Ultrasonic	n-Hexane	1 hour	70	4.5	Yes
2665	Yes	Used as received	0.5g	Mechanical Shaking	Ethanol, Hexane	960	22	4	Yes
2666	Yes	Further cut	1	Ultrasonic	methanol/ethanol	60	60		Yes
2668	Yes	Further cut	0.5 gms	Ultrasonic	Ethanol:Methanol	60 min	60C	4.5	Yes
2675	No	Used as received	1,0	Ultrasonic	Ethanol/acetic acid 95:5	60	60		No
2678	---	---	---	---	---	---	---	---	---
2743	---	---	---	---	---	---	---	---	---
2820	Yes	Used as received	1 g	Ultrasonic	methanol/ethanol	60	50		Yes
2826	Yes	Used as received	0.5 g	Ultrasonic	Methanol: Ethanol (80:20)	1 hour	60°C	4.5	No
2827	Yes	Further cut	0.5g	Ultrasonic	Methanol/Ethanol	60 mins	60 °C	5.4	Yes
2864	---	---	---	---	---	---	---	---	---
2910	Yes	Used as received	2.6	Soxhlet	Isooctane	60	60	4.52	Yes
2960	Yes	Used as received	0.5	Stirrer	Methanol/Ethanol	1h	60	4.5	No
2977	---	---	---	---	---	---	---	---	---
3003	Yes	Further grinded	2.5 gram	Ultrasonic	Methanol-Ethanol mixture of 80/20 ratio	>120 min	60	4.5	No
3100	Yes	Further cut	0.5g	Mechanical Shaking	Methyl ethyl alcohol	60min	60°C	4.48	Yes
3116	Yes	Used as received	0.5 gram	Ultrasonic	methanol/ethanol (80/20 v/v)	1h	60 °C	4.5	No
3149	Yes	Used as received	1	Ultrasonic	Methanol/Ethanol 80/20	60	60		---
3154	Yes	Used as received	0,6	Ultrasonic	Methanol	60 min	60 °C	4,5	No
3172	Yes	---	---	---	---	---	---	---	---
3176	Yes	Used as received	1,0	Ultrasonic	MEOH/HCl	30	room	4,5	Yes
3185	Yes	Further cut	1gram	Ultrasonic	methanol:ethanol=4:1	60 min	60°C	6.52	No
3197	Yes	Further cut	0,5 g	Ultrasonic	Methanol/Ethanol (80:20)	60 min	60 C	4,5	No
3210	Yes	Used as received	1	Ultrasonic	MeOH/EtOH (80/20)	60	60		No
3214	Yes	Further cut	1 g	Ultrasonic	Methanol:Ethanol=4:1	60 min	60°C	4.5	Yes
3218	Yes	Used as received	0.5g	Ultrasonic	n-hexane	60 min	60°C	4.5	Yes
3228	Yes	Further cut	0.5	Ultrasonic	isooctane	60	60	4.5	Yes
3230	---	---	---	---	---	---	---	---	---
3237	Yes	Further cut	0,5	Ultrasonic	isooctane	60	60	na	No
3246	Yes	Used as received	1.00g	Ultrasonic	Methanol: ethanol = 8:2	1h	60°C	4.5	No

APPENDIX 4

Number of participants per country

4 labs in BANGLADESH
1 lab in BRAZIL
1 lab in CAMBODIA
1 lab in FRANCE
9 labs in GERMANY
3 labs in HONG KONG
1 lab in HUNGARY
8 labs in INDIA
2 labs in INDONESIA
7 labs in ITALY
1 lab in KOREA, Republic of
1 lab in MAURITIUS
1 lab in MOROCCO
20 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SINGAPORE
1 lab in SPAIN
3 labs in SRI LANKA
3 labs in TAIWAN
1 lab in THAILAND
1 lab in TUNISIA
4 labs in TURKEY
1 lab in U.S.A.
1 lab in UNITED KINGDOM
4 labs in VIETNAM

APPENDIX 5

Abbreviations

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

Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)