Results of Proficiency Test
Organotin Compounds in Textile
December 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. A. Ouwerkerk
Correctors: ing. G.A. Oosterlaken-Buijs & ing. R.J. Starink
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1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known organizations are for instance: Bluesign® (Switzerland), 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 2021/2022 it was decided to continue the proficiency test for the determination of Organotin Compounds in textile.

In this interlaboratory study 80 laboratories in 28 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Organotin Compounds in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different textile samples of approximately 3 grams each, both positive on some Organotin compounds, labelled #21810 and #21811.
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 green cotton textile positive on Dimethyltin (DMT) was selected. The batch was cut into small pieces. After homogenization 110 small plastic bags were filled with approximately 3 grams each and labelled #21810.
The homogeneity of the subsamples was checked by determination of Dimethyltin (DMT) in accordance with an in house test method on 8 stratified randomly selected subsamples.

	Dimethyltin (DMT) in mg/kg
sample #21810-1	1.158
sample #21810-2	1.171
sample #21810-3	1.275
sample #21810-4	1.268
sample #21810-5	1.216
sample #21810-6	1.190
sample #21810-7	1.150
sample #21810-8	1.211

Table 1: homogeneity test results of subsamples #21810

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.

	Dimethyltin (DMT) in mg/kg
r (observed)	0.132
reference test method	ISO/TS16179:12
0.3 x R (reference test method)	0.233

Table 2: evaluation of the repeatability of subsamples #21810

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 yellow cotton textile positive on Monobutyltin (MBT) was selected. The batch was cut into small pieces. After homogenization 106 small plastic bags were filled with approximately 3 grams each and labelled #21811.
The homogeneity of the subsamples was checked by determination of Monobutyltin (MBT) in accordance with an in house test method on 8 stratified randomly selected subsamples.

	Monobutyltin (MBT) in mg/kg
sample #21811-1	6.183
sample #21811-2	5.711
sample #21811-3	5.355
sample #21811-4	5.403
sample #21811-5	5.243
sample #21811-6	5.355
sample #21811-7	5.431
sample #21811-8	5.962

Table 3: homogeneity test results of subsamples #21811

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

	Monobutyltin (MBT) in mg/kg
r (observed)	0.944
reference test method	ISO/TS16179:12
0.3 x R (reference test method)	1.078

Table 4: evaluation of the repeatability of subsamples #21811

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 #21810 and one sample labelled #21811 were sent on November 17, 2021.

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:

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

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

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

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Two participants reported test results after the reporting date and eight other participants did not report any test results. Not all participants were able to report all components requested.

In total 72 participants reported 185 numerical test results. Observed were 11 outlying test results, which is 5.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data 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 * mean PT /100.

sample #21810

Monomethyltin (MMT): 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.

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

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

sample #21811

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

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

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test method (in casu ISO/TS161719) are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(lit)
Monomethyltin (MMT)	mg/kg	42	0.169	0.162	0.109
Dimethyltin (DMT)	mg/kg	65	4.91	3.19	3.17

Table 5: reproducibilities of tests on sample #21810

Component	unit	n	average	2.8 * sd	R(lit)
Monobutyltin (MBT)	mg/kg	67	12.17	7.35	7.84

Table 6: reproducibility of tests on sample #21811

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2021 WITH PREVIOUS PTS

	December 2021	December 2020	December 2019	December 2018	December 2017
Number of reporting laboratories	72	85	85	100	27
Number of test results	185	247	317	415	67
Number of statistical outliers	11	12	12	7	6
Percentage of statistical outliers	5.9%	4.9%	3.8%	1.7%	9%

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, expressed as relative standard deviation (RSD) of the PTs, in the next table.

Component	December 2021	December 2020	December 2019	December 2018	2017 - 2016	Reference ISO16179
Monomethyltin (MMT)	34%	--	37%	--	--	23%
Dimethyltin (DMT)	23%	--	22%	25-40%	38%	23%
Trimethyltin (TMT)	--	--	26%			23%
Monobutyltin (MBT)	22%	20-39%	33%	--	37%	23%
Dibutyltin (DBT)	--	18%	22%	21%	35%	23%
Tributyltin (TBT)	--	--	--	29-31%	29%	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 at a very low Organotin concentration (MMT).

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:

- 84% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- About half of the participants indicate to have used the samples as received and the other half further cut the samples prior to analysis.
- Most of the participants used between 0.5 - 1 grams of sample intake; 39% around 0.5 grams and 50% around 1 gram.
- 97% used Ultrasonic technique to extract/release the components from the samples.
- About 70% used a mixture of Methanol and Ethanol as extraction solvent, about 10% used other mixtures and about 20% used one solvent e.g. Hexane, Acetone or Ethanol.
- Almost all participants used an extraction/release time of 60 minutes, about 85% used an extraction/release temperature of 60°C, about 10% used a lower temperature.

- About 85% reported to observe a pH of 4.5 - 4.6. About 50% have adjusted the pH.

For Dimethyltin (DMT) and Monobutyltin (MBT) the calculated reproducibility is in full agreement with the requirements of the target reproducibility, therefore 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 Oeko-Tex Standard 100 (see table 9), it could be noted that some laboratories would make a different decision about the acceptability of the textile.
Based on DMT sample #21810 would have been rejected for all classes by almost all reporting laboratories. Only one laboratory would have rejected the sample for class 1 and accepted it for the classes 2,3 and 4.
Seventy reporting laboratories would have rejected sample #21811 based on MBT for all classes. One laboratory would have rejected the sample for class 1 and accepted it for the classes 2,3 and 4.

	Class 1 Baby clothes	Class 2 Clothes direct skin contact	Class 3 Clothes, no direct contact with skin	Class 4 Decoration material
TBT, TPhT (mg/kg)	0.5	1.0	1.0	1.0
Other Organotin compounds (mg/kg)	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 Monomethyltin (MMT) and Dimethyltin (DMT) in sample #21810 and Monobutyltin (MBT) in sample #21811.

The quantification of Monomethyltin (MMT) in sample #21810 was more problematic. Please note that the consensus value is near the detection limit and thus much lower than the criteria according to the Ecolabelling.

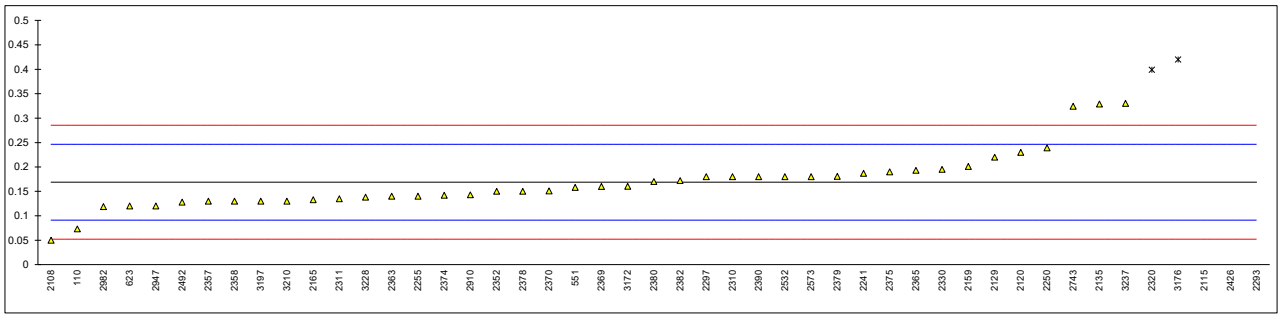
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

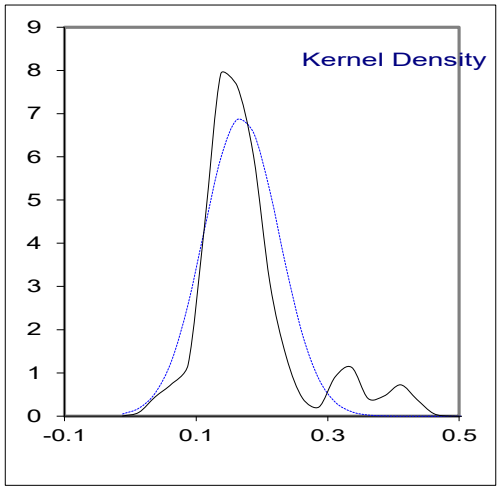
APPENDIX 1

Determination of Monomethyltin (MMT) on sample #21810; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17353	0.073		-2.47	
210		-----		-----	
339	In house	<0.1		-----	
551	ISO/TS 16179	0.158		-0.28	
623	ISO/TS 16179	0.120		-1.26	
840	ISO/TS 16179	<0.2	C	-----	First reported Not detected
841	ISO/TS 16179	<0.2		-----	
2108	In house	0.05		-3.06	
2115	In house	1	C,R(0.01)	21.40	First reported 1.41
2120	ISO22744-1	0.23		1.57	
2129		0.220		1.32	
2131	In house	not detected		-----	
2135	ISO/TS 16179	0.329		4.12	
2159	ISO/TS 16179	0.201		0.83	
2165	ISO/TS 16179	0.133		-0.92	
2241	ISO/TS 16179	0.187		0.47	
2250	ISO/TS 16179	0.239		1.81	
2255	ISO/TS 16179	0.14		-0.74	
2264		-----		-----	
2265	ISO/TS 16179	< 0,05		-----	
2289		-----		-----	
2293	ISO/TS 16179	6.268	C,R(0.01)	157.05	First reported 8.594
2297	ISO/TS 16179	0.18		0.29	
2310	ISO17353	0.18		0.29	
2311	ISO/TS 16179	0.135		-0.87	
2320	ISO22744-1	0.399	C,R(0.05)	5.93	First reported N.D.
2330	ISO22744-1	0.195		0.67	
2350	ISO/TS 16179	< 0.2		-----	
2352	ISO/TS 16179	0.15		-0.49	
2357	ISO/TS 16179	0.13		-1.00	
2358	ISO/TS 16179	0.13		-1.00	
2363	ISO/TS 16179	0.14		-0.74	
2365	ISO/TS 16179	0.193		0.62	
2366	ISO/TS 16179	<0.5		-----	
2369	ISO/TS 16179	0.16		-0.23	
2370	ISO22744-1	0.151		-0.46	
2374	ISO/TS 16179	0.142		-0.69	
2375	ISO22744-1	0.19		0.54	
2378	ISO22744-1	0.15		-0.49	
2379	ISO/TS 16179	0.1808		0.31	
2380	ISO/TS 16179	0.17		0.03	
2382	ISO/TS 16179	0.172		0.08	
2386	ISO/TS 16179	<0.2		-----	
2390	ISO17353	0.18	C	0.29	First reported Not Detected
2426	ISO/TS 16179	1.132	R(0.01)	24.80	
2429		Not determined	C	-----	First reported Not Detected
2452	ISO/TS 16179	not determined		-----	
2453		-----		-----	
2492	In house	0.128		-1.05	
2511		-----		-----	
2532	ISO/TS 16179	0.18		0.29	
2561		-----		-----	
2573	ISO/TS 16179	0.18		0.29	
2590		-----		-----	
2591		-----		-----	
2671		-----		-----	
2674	ISO/TS 16179	not applicable		-----	
2678		-----		-----	
2737		-----		-----	
2743	ISO/TS 16179	0.3244		4.01	
2864		-----		-----	
2892		-----		-----	
2910	ISO/TS 16179	0.143		-0.67	
2912		-----		-----	
2947	In house	0.12	C	-1.26	First reported not detected
2953		-----	W	-----	Test result withdrawn, reported 1.76
2959		-----		-----	
2976		-----		-----	
2982	ISO/TS 16179	0.119		-1.28	
3116		-----		-----	
3154		-----		-----	
3172	ISO/TS 16179	0.16044		-0.22	
3176	In house	0.42	R(0.05)	6.47	
3197	ISO17353	0.13		-1.00	

lab	method	value	mark	z(targ)	remarks
3210	In house	0.13		-1.00	
3214	ISO/TS 16179	<0.1		-----	
3220		-----		-----	
3228	ISO/TS 16179	0.138		-0.79	
3230		-----		-----	
3237	ISO/TS 16179	0.33		4.15	
	normality	not OK			
	n	42			
	outliers	5			
	mean (n)	0.1688			
	st.dev. (n)	0.05780	RSD=34%		
	R(calc.)	0.1618			
	st.dev.(ISO/TS16179:12)	0.03884			
	R(ISO/TS16179:12)	0.1087			



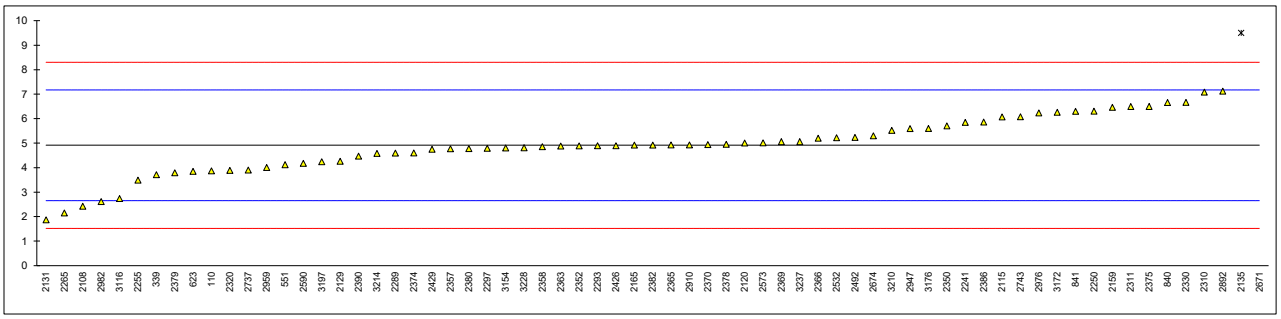


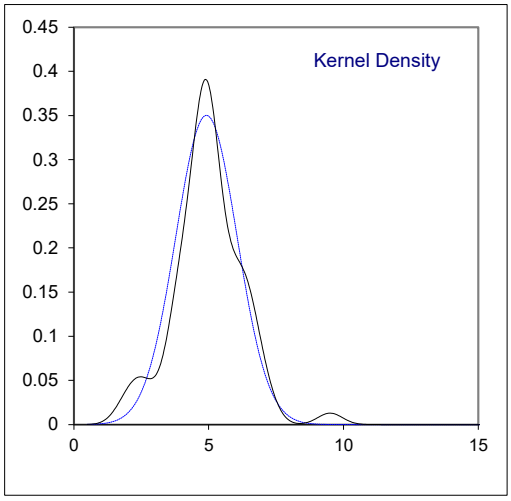
Determination of Dimethyltin (DMT) on sample #21810; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17353	3.867	C	-0.93	First reported 1.289
210		-----		-----	
339	In house	3.71		-1.07	
551	ISO/TS 16179	4.12		-0.70	
623	ISO/TS 16179	3.850		-0.94	
840	ISO/TS 16179	6.66		1.54	
841	ISO/TS 16179	6.30		1.23	
2108	In house	2.42		-2.21	
2115	In house	6.07		1.02	
2120	ISO22744-1	5.0		0.08	
2129		4.260		-0.58	
2131	In house	1.87		-2.69	
2135	ISO/TS 16179	9.50	R(0.05)	4.06	
2159	ISO/TS 16179	6.464		1.37	
2165	ISO/TS 16179	4.921		0.01	
2241	ISO/TS 16179	5.846		0.82	
2250	ISO/TS 16179	6.309		1.23	
2255	ISO/TS 16179	3.49		-1.26	
2264		-----		-----	
2265	ISO/TS 16179	2.15		-2.45	
2289	ISO/TS 16179	4.59		-0.29	
2293	ISO/TS 16179	4.894		-0.02	
2297	ISO/TS 16179	4.79		-0.11	
2310	ISO17353	7.08		1.92	
2311	ISO/TS 16179	6.494		1.40	
2320	ISO22744-1	3.890		-0.91	
2330	ISO22744-1	6.663		1.55	
2350	ISO/TS 16179	5.703		0.70	
2352	ISO/TS 16179	4.89		-0.02	
2357	ISO/TS 16179	4.77		-0.13	
2358	ISO/TS 16179	4.852		-0.06	
2363	ISO/TS 16179	4.88		-0.03	
2365	ISO/TS 16179	4.928		0.01	
2366	ISO/TS 16179	5.2		0.25	
2369	ISO/TS 16179	5.06		0.13	
2370	ISO22744-1	4.94		0.02	
2374	ISO/TS 16179	4.601		-0.28	
2375	ISO22744-1	6.5		1.40	
2378	ISO22744-1	4.95		0.03	
2379	ISO/TS 16179	3.7886		-1.00	
2380	ISO/TS 16179	4.78		-0.12	
2382	ISO/TS 16179	4.923		0.01	
2386	ISO/TS 16179	5.860		0.84	
2390	ISO17353	4.469	C	-0.39	First reported 1.155
2426	ISO/TS 16179	4.895		-0.02	
2429		4.751		-0.14	
2452	ISO/TS 16179	-----		-----	
2453		-----		-----	
2492	In house	5.233		0.28	
2511		-----		-----	
2532	ISO/TS 16179	5.22		0.27	
2561		-----		-----	
2573	ISO/TS 16179	5.01		0.08	
2590	ISO/TS 16179	4.180		-0.65	
2591		-----		-----	
2671	ISO/TS 16179	20.52	R(0.01)	13.80	
2674	ISO/TS 16179	5.296		0.34	
2678		-----		-----	
2737	ISO/TS 16179	3.9		-0.90	
2743	ISO/TS 16179	6.0778		1.03	
2864		-----		-----	
2892	ISO/TS 16179	7.122		1.95	
2910	ISO/TS 16179	4.93		0.01	
2912		-----		-----	
2947	In house	5.59		0.60	
2953		-----		-----	
2959	ISO/TS 16179	4.004		-0.81	
2976	ISO/TS 16179	6.2300		1.16	
2982	ISO/TS 16179	2.611		-2.04	
3116	ISO/TS 16179	2.736		-1.93	
3154	ISO/TS 16179	4.80		-0.10	
3172	ISO/TS 16179	6.2631		1.19	
3176	In house	5.6	C	0.61	First reported 12.54
3197	ISO17353	4.24		-0.60	
3210	In house	5.52		0.54	

lab	method	value	mark	z(targ)	remarks
3214	ISO/TS 16179	4.583		-0.29	
3220		-----		-----	
3228	ISO/TS 16179	4.81		-0.09	
3230		-----		-----	
3237	ISO/TS 16179	5.06	C	0.13	First reported 7.22

normality	OK
n	65
outliers	2
mean (n)	4.9148
st.dev. (n)	1.13940 RSD=23%
R(calc.)	3.1903
st.dev.(ISO/TS16179:12)	1.13041
R(ISO/TS16179:12)	3.1652



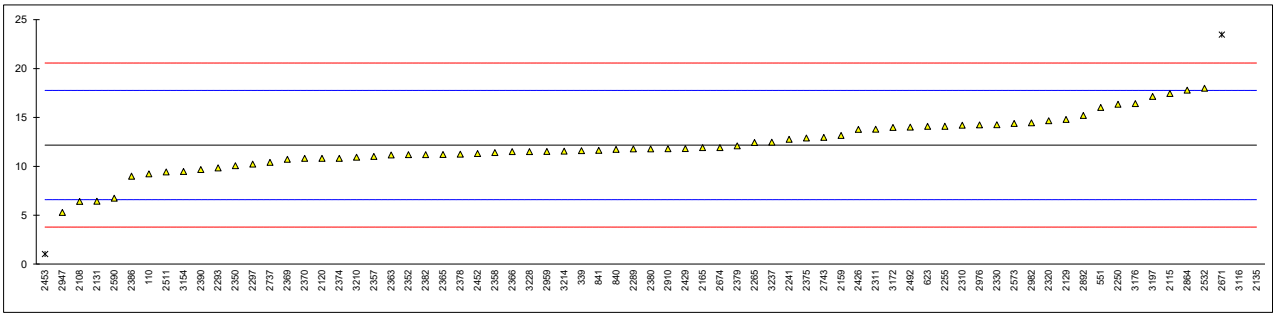


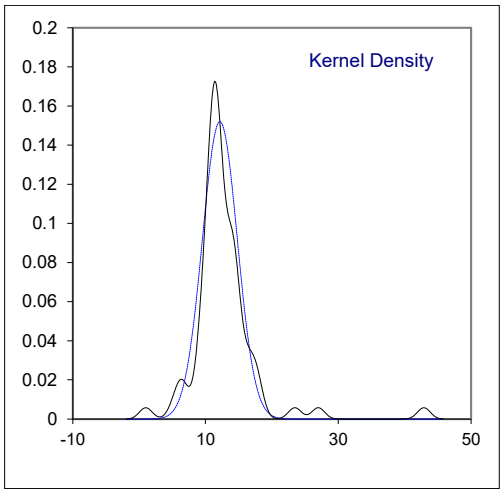
Determination of Monobutyltin (MBT) on sample #21811; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO17353	9.236		-1.05	
210		-----		-----	
339	In house	11.6		-0.20	
551	ISO/TS 16179	16.01		1.37	
623		14.075		0.68	
840	ISO/TS 16179	11.73		-0.16	
841	ISO/TS 16179	11.63		-0.19	
2108	In house	6.40		-2.06	
2115	In house	17.44		1.88	
2120	ISO22744-1	10.8		-0.49	
2129		14.785		0.93	
2131	In house	6.42		-2.05	
2135	ISO/TS 16179	42.91	R(0.01)	10.98	
2159	ISO/TS 16179	13.157		0.35	
2165	ISO/TS 16179	11.92		-0.09	
2241	ISO/TS 16179	12.755		0.21	
2250	ISO/TS 16179	16.345		1.49	
2255	ISO/TS 16179	14.1		0.69	
2264		-----		-----	
2265	ISO/TS 16179	12.43		0.09	
2289	ISO/TS 16179	11.78		-0.14	
2293	ISO/TS 16179	9.831		-0.84	
2297	ISO/TS 16179	10.23		-0.69	
2310	ISO17353	14.2		0.72	
2311	ISO/TS 16179	13.798		0.58	
2320	ISO22744-1	14.662		0.89	
2330	ISO22744-1	14.246		0.74	
2350	ISO/TS 16179	10.057		-0.76	
2352	ISO/TS 16179	11.19		-0.35	
2357	ISO/TS 16179	11.01		-0.42	
2358	ISO/TS 16179	11.39		-0.28	
2363	ISO/TS 16179	11.15		-0.37	
2365	ISO/TS 16179	11.207		-0.35	
2366	ISO/TS 16179	11.5	C	-0.24	First reported <0.5
2369	ISO/TS 16179	10.71		-0.52	
2370	ISO17353	10.8		-0.49	
2374	ISO/TS 16179	10.802		-0.49	
2375	ISO22744-1	12.9		0.26	
2378	ISO22744-1	11.24		-0.33	
2379	ISO/TS 16179	12.0818		-0.03	
2380	ISO/TS 16179	11.78		-0.14	
2382	ISO/TS 16179	11.196		-0.35	
2386		8.992		-1.14	
2390	ISO17353	9.66		-0.90	
2426	ISO/TS 16179	13.770		0.57	
2429		11.808		-0.13	
2452	ISO/TS 16179	11.289		-0.32	
2453	In house	1.014	C,R(0.01)	-3.99	First reported 221.03
2492	In house	14.013		0.66	
2511	ISO/TS 16179	9.4142		-0.99	
2532	ISO/TS 16179	17.99		2.08	
2561		-----		-----	
2573	ISO/TS 16179	14.39		0.79	
2590	ISO/TS 16179	6.740		-1.94	
2591		-----		-----	
2671	ISO/TS 16179	23.47	R(0.01)	4.03	
2674	ISO/TS 16179	11.922		-0.09	
2678		-----		-----	
2737	ISO/TS 16179	10.4		-0.63	
2743	ISO/TS 16179	12.9554		0.28	
2864	ISO/TS 16179	17.79		2.01	
2892	ISO/TS 16179	15.200		1.08	
2910	ISO/TS 16179	11.80		-0.13	
2912		-----		-----	
2947	In house	5.28		-2.46	
2953		-----		-----	
2959	ISO/TS 16179	11.51		-0.24	
2976	ISO/TS 16179	14.2457		0.74	
2982	ISO/TS 16179	14.435		0.81	
3116	ISO/TS 16179	27.0	C,R(0.01)	5.30	First reported 24.01
3154	ISO/TS 16179	9.46		-0.97	
3172	ISO/TS 16179	13.982		0.65	
3176	ISO22744-1	16.4	C	1.51	First reported 22.66
3197	ISO17353	17.15		1.78	
3210	In house	10.92		-0.45	

lab	method	value	mark	z(targ)	remarks
3214	ISO/TS 16179	11.552		-0.22	
3220		-----		-----	
3228	ISO/TS 16179	11.5		-0.24	
3230		-----		-----	
3237	ISO/TS 16179	12.46	C	0.10	First reported 21.78

normality	OK
n	67
outliers	4
mean (n)	12.1735
st.dev. (n)	2.62592
RSD=22%	
R(calc.)	7.3526
st.dev.(ISO/TS16179:12)	2.79990
R(ISO/TS16179:12)	7.8397





APPENDIX 2

Determination of other Organotin components on sample #21810; results in mg/kg

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

lab	TMT	TPT	MBT	DBT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
110	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
551	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
623	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
840	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
841	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2108	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2120	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20	< 0,20
2129	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2131	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2159	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2165	-----	-----	not det	not det	not det	not det	not det	not det	-----	-----	not det	not det
2241	-----	-----	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-----	<0.05	<0.05	<0.05
2250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2255	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2264	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2289	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2293	6.541	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2297	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2310	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2311	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2320	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2330	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2350	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2363	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2365	<0.05	<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	<0.5
2369	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2374	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2379	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2380	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2382	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02
2386	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
2390	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2426	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2429	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2452	-----	-----	not det	not det	not det	not det	not det	not det	-----	-----	not det	not det
2453	-----	-----	not det	not det	not det	not det	not det	not det	-----	-----	not det	not det
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2573	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2590	5.739	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2591	not det	not det	-----	not det	not det	not det	-----	not det	not det	-----	not det	not det
2671	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2674	-----	-----	not det	not det	-----	not det	not det	not det	-----	-----	not det	not det
2678	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	0.0538	-----	-----	-----	-----	-----	-----	-----	-----	-----
2864	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2910	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2912	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2947	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2953	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2959	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2976	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2982	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det

lab	TMT	TPT	MBT	DBT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3210	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
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
3220	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3228	-----	-----	not det	not det	not det	not det	not det	not det	-----	-----	not det	not det
3230	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

Determination of other Organotin components on sample #21811; results in mg/kg

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

lab	MMT	DMT	TMT	TPT	DBT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
110	not det	0.031	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.1	<0.1	-----	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
551	-----	0.053	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
623	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
840	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
841	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2108	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	0.06	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2120	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20	<0,20
2129	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2131	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2159	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2165	not det	0.073	-----	-----	not det	not det	not det	not det	not det	-----	-----	not det	not det
2241	<0.05	0.0611	-----	-----	<0.05	<0.05	<0.05	<0.05	<0.05	-----	<0.05	<0.05	<0.05
2250	-----	0.032	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2255	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2264	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2289	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2293	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2297	nd	nd	nd	nd	Nd	nd	Nd	nd	nd	nd	nd	nd	nd
2310	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2311	Not det	<0.05	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2320	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2330	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2350	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
2352	-----	0.06	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	0.06	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	not det	0.070	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2363	not det	0.06	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5 C	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2369	<0.05	0.06	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2374	not det	0.0702	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<0.05	0.06	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2379	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2380	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2382	＜0.02	0.073	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02	＜0.02
2386	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
2390	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2426	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2429	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2452	-----	-----	-----	-----	not det	not det	not det	not det	not det	-----	-----	not det	not det
2453	-----	-----	-----	-----	not det	not det	not det	not det	not det	-----	-----	not det	not det
2492	-----	0.068	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not det	0.08	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2573	Not det	0.07	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2591	-----	-----	not det	not det	not det	not det	not det	-----	not det	not det	-----	not det	not det
2671	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2674	-----	-----	-----	-----	not det	-----	not det	not det	not det	-----	-----	not det	not det
2678	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2743	-----	0.0723	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2864	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2910	not det	0.075	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2912	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2947	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det	not det
2953	17.69	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2959	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2976	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2982	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det	Not det
3116	-----	0.1188	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	MMT	DMT	TMT	TPT	DBT	TBT	TeBT	MOT	DOT	TOT	DPhT	TPhT	TCyHT
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.02	0.069296	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3210	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
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	<0.1
3220	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3228	not det	0.065	-----	-----	not det	not det	not det	not det	not det	-----	-----	not det	not det
3230	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

Lab 2366 first reported 11.5

APPENDIX 3 Analytical details

lab	ISO/IEC 17025 accre-dited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extrac-tion time (min)	Extrac-tion temp (°C)	pH after adding the buffer	Aci-dified to pH 4.5
110	Yes	Further cut	0.5 g	Ultrasonic	Acetone	60 min	40	pH 4.5±0.3	Yes
210	---	---		---					---
339	No	---		---					---
551	Yes	Further cut	1g	Ultrasonic	Methanol-ethanol	60	60		---
623	Yes	Further cut	1	Ultrasonic	methanol-ethanol	60	60		---
840	Yes	Further cut	0.5	Ultrasonic	methanol-ethanol 8:2	60	60		No
841	Yes	Used as received	1 grams	Ultrasonic	Methanol + Ethanol	60 min	60°C		---
2108	Yes	Used as received	0,5g	Ultrasonic	EtOH/Acetic acid+Tropolon	60 min	60°C	---	---
2115	Yes	Used as received	1 g	Ultrasonic	Ethanol/Acetic Acid	60 min	60 °C		---
2120	No	Used as received	1 g	Ultrasonic	Methanol/ethanol (80:20)	60 min	60ºC	---	No
2129	Yes	Used as received	0,6 g	Ultrasonic	hydrochloric acid solution in methanol	60 min	60°C	4,66	No
2131	Yes	Used as received	0.5g	Ultrasonic	Ethanol/glacial acetic acid 95:5	60 Min	40	5	No
2135	Yes	Used as received	1	Ultrasonic	Ethanol/Methanol 20/80	60	60	4,5	No
2159	Yes	Used as received	1,0 gram	Ultrasonic	hexane	60 min	70°C	4,5	Yes
2165	Yes	Further cut	1g	Ultrasonic	methanol : ethanol (8:2) isooctane	60min.	60°C	4.5	No
2241	Yes	Used as received	0.5g	Ultrasonic	methanol-ethanol mixture	60min	60°C	not test	No
2250	Yes	Used as received	0,1	Ultrasonic	Methanol/Ethanol (4:1)	60	60	4,5	No
2255	Yes	Further cut	0.5	Ultrasonic	n-Hexane	60	60	4.5	Yes
2264	---	---		---					---
2265	Yes	Used as received	0,5	Ultrasonic	MeOH 80% - EtOH 20%	60	60		No
2289	Yes	Further cut	1.0g	Ultrasonic	methanol/ethanol	60min	60°C	4.5	No
2293	Yes	Further cut	0.5	Ultrasonic	Methanol/ ethanol	60 min	60°C	No	No
2297	---	---		---					---
2310	Yes	Further cut	1	Ultrasonic	Acetone	60 min	40	4.5 to 5.0	Yes
2311	Yes	Further cut	1	Ultrasonic	Acetone	60	40	5.4	Yes
2320	Yes	Further cut	0.3g	Ultrasonic	Methanol:Ethanol 80/20 (v/v)	1Hour	60°C	4.5	Yes
2330	No	Further cut	0.5 gram	Ultrasonic	Methanol : Ethanol (ratio 8:2)	60 min	60 ± 2°C	NA	---
2350	No	Further cut	#21810 : 0.5004 g #21811 : 1.0027 g	Ultrasonic	Methanol : Ethanol (8:2)	60 min	60 °C	pH 4.5	No
2352	Yes	Further cut	1g	Ultrasonic	Methanol Ethanol	60	60	4.50	Yes
2357	---	---		---					---
2358	Yes	Used as received	1 g	Ultrasonic	Methanol-ethanol mixture, liquid-liquid extraction with isooctane	60 min	60°C	pH 4.5	Yes
2363	Yes	Further cut	2g	Ultrasonic	Methanol: Ethanol=4:1	60min	60℃		Yes
2365	Yes	Used as received	1.5g	Ultrasonic	MeOH:HeX=4:1	60min	60℃	Ph=4.6	Yes
2366	No	Further cut	0.5g	Mechanical Shaking	methanol: ethanol= 80:20	60min	60	4.5	No
2369	---	---		---					---
2370	Yes	Further cut	1 g	Ultrasonic	ISO 22744:Ethanol/Methanol ISO 17353:Ethanol	60 min	ISO 17353: 40℃/ ISO 22744: 60℃	pH=4.5	Yes
2374	Yes	Used as received	1g	Ultrasonic	Hexane	1 hour	60 °C	pH 4.5	No
2375	Yes	Further cut	0.5g	Ultrasonic	Methanol/Ethanol	60 min	60 °C	pH 4,5	Yes
2378	No	Further cut	0.5	Ultrasonic	n-hexan	60min	60	4.5	Yes
2379	Yes	Further cut	0.5 gram	Ultrasonic	MeOH : EtOH 80 : 20	60 min	60°C	pH 4.5	Yes
2380	Yes	Further cut	0.5 g	Ultrasonic	Methanol-Ethanol	60 Min	60+/-2°C	7.0	Yes
2382	Yes	Used as received	0.5g	Ultrasonic	16ml methanol + 4ml ethanol ultrasonic, Isooctane extract	60 mins	60℃	4.5	Yes
2386	Yes	Further cut	0,5	Ultrasonic	Methanol/Ethanol 80/20 v/v	60	60	4,5	No
2390	Yes	Further cut	1.0 gram	Mechanical Shaking	Acetone	60 min	40 °C	4.5	Yes
2426	Yes	Further cut	1.00g	Ultrasonic	Methanol-Ethanol mixture	60min	60°C	4.5	No

lab	ISO/IEC 17025 accre-dited	Sample preparation	Sample intake (g)	Extraction type	Extraction solvent	Extraction time (min)	Extraction temp (°C)	pH after adding the buffer	Aci-dified to pH 4.5
2429	Yes	Used as received	0.5g	Ultrasonic	Hexane	60	60	4.5	No
2452	No	Used as received	0.5	Ultrasonic	Ethanol/Methanol (20/80 V/V)	60	60		Yes
2453	No	Used as received	±1.5g	---					---
2492	Yes	Used as received	0.5g	Ultrasonic	250ppm Tropolone in Ethanol with 5% Acetic Acid	60	40	4.5	No
2511	---	---		---					---
2532	Yes	Further cut	0.5g	Ultrasonic	Methanol : Ethanol	60 min	60 °C		No
2561	---	---		---					---
2573	Yes	Used as received	1g	Ultrasonic	methanol/ethanol(4:1)	60 min	60 °C	4.5	Yes
2590	Yes	Used as received	1G	Ultrasonic	MeOH: EtOH 80:20	60 min	60°C	4.5	No
2591	No	Further cut	1.0 g	Ultrasonic	MeOH/EtOH 80/20	60 min	60ºC		No
2671	Yes	Used as received	1 g	Ultrasonic	Hexane	1 hr	60	4.5	Yes
2674	Yes		1.0g	Ultrasonic	methanol and ethanol	60	60	PH=4.5	No
2678	---	---		---					---
2737	Yes	Used as received	1g	Ultrasonic	Methanol/Ethanol=4:1	60min	60 °C	4.5	Yes
2743	Yes	Used as received	1g	Ultrasonic	Methanol/Ethanol 4:1	60min	60C	4,5	No
2864	Yes	Used as received	0.5 g	Ultrasonic	MeoH:EtoH=80:20	60 min	60 °C	4.5	Yes
2892	Yes	Further cut	1.0	Ultrasonic	Methanol/Ethanol : 80/20	60	60	4.5	No
2910	Yes	Used as received	0.5g	Ultrasonic	Ethanol:Methanol=1:4	60min	60℃	4.5	No
2912	---	---		---					---
2947	No	Used as received	0.5	Ultrasonic	Ethanol/acetic acid	60	RT	No	
2953	---	---		---					---
2959	Yes	Used as received	1g	Ultrasonic	methanol/ ethanol	60min	60℃		---
2976	No	Used as received	1.5gr	Ultrasonic	Methanol/Ethanol (80:20)	60 min	60°C	4.54	No
2982	Yes	Used as received	1 gm	Ultrasonic	Mixture of Methanol & Ethanol	60 min	60°C	pH: 4.5	Yes
3116	Yes	Used as received	1 gram	Ultrasonic	methanol/ethanol (80/20 v/v)	60 min	60°C	pH4.5	Yes
3154	Yes	Used as received	1	Ultrasonic	MeOH/EtOH	60	70		---
3172	Yes	---		---					---
3176	Yes	Further cut	1	Ultrasonic	For in house method: HCl/Methanol For ISO 22744-1 :Methanol/Ethanol	INH: 30 min ISO 22744-1 : 1hr	INH: RT ISO 22744-1 : 60°C	4.5	Yes
3197	Yes	Further cut	1	Ultrasonic	Ethanol	120min	22C	4,5	No
3210	Yes	Further cut	1g	Ultrasonic	Methanol/Ethanol and Hexane	60min	60°C		Yes
3214	Yes	Further cut	1 g	Ultrasonic	Methanol / Ethanol (4:1 V/V)	60 min	60°C	4.5	Yes
3220	---	---		---					---
3228	Yes	Further cut	2	Ultrasonic	methanol/ethanol=80/20	60	60	4.5	No
3230	---	---		---					---
3237	Yes	Further cut	0,5	Ultrasonic	Methanol-Ethanol (80:20)	60	60	-	No

APPENDIX 4

Number of participants per country

1 lab in	AUSTRIA
3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA
2 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
3 labs in	HONG KONG
5 labs in	INDIA
1 lab in	INDONESIA
6 labs in	ITALY
1 lab in	MAURITIUS
1 lab in	MOROCCO
20 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	PERU
2 labs in	PORTUGAL
1 lab in	SOUTH KOREA
1 lab in	SPAIN
1 lab in	SRI LANKA
1 lab in	SWITZERLAND
3 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TUNISIA
5 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

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