Results of Proficiency Test
Organotin in textile
December 2016

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing.
Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile & leather products on a voluntary basis. Well known organisations are for instance: Bluesign® (Switzerland), which has created a Bluesign® system substances list (BSSL) and Öko-Tex Standard 100 (Germany).
On request of several laboratories, the Institute for Interlaboratory Studies decided to organize a proficiency test for Organotin components in textile. In the annual proficiency test program of 2016/2017, this proficiency test was set up for the first time.
In this interlaboratory study 58 laboratories in 20 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2016 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 organiser of the proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to use one textile sample, positive on Organotin. The sample was labelled #16650. 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 IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). 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

One batch of textile was donated by a third party laboratory. The bulk sample, a hosiery fabric positive on Organotin, was cut into pieces. Out of this batch, after mixing well, 76 subsamples of 1.25 grams each were packed and labelled #16650.
The homogeneity of 8 stratified randomly selected samples was checked by determination of Monobutyltin (MBT) by an ISO/IEC 17025 accredited laboratory. The determination is performed in accordance with an in-house test method for Organotin. See the following table for the test results.

	MBT in mg/kg
Sample #16650-1	0.37
Sample #16650-2	0.36
Sample #16650-3	0.33
Sample #16650-4	0.34
Sample #16650-5	0.36
Sample #16650-6	0.35
Sample #16650-7	0.38
Sample #16650-8	0.33

Table 1: Homogeneity test results of subsample #16650

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the target test method, in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	MBT in mg/kg
r (observed)	0.06
reference test method	ISO-TS 16179:12
0.3 x R (reference test method)	0.09

Table 2: Repeatability of subsamples #16650.

The repeatability of Monobutyltin (MBT) was in agreement with 0.3 times the target requirements of ISO-TS 16179:12. Therefore, homogeneity of the subsamples was assumed.
To each participating laboratory one sample of 1.25 grams, labelled #16650, was sent on November 16, 2016. A letter of instructions was added to the sample package.

2.5 ANALYSES

The participants were asked to determine the concentrations of the following Organotin components: Monomethyltin (MMT), Dimethyltin (DMT), Trimethyltin (TMT), Tripropyltin (TPT), Monobutyltin (MBT), Dibutyltin (BDT), Tributyltin (TBT), Tetrabutyltin (TeBT), Monooctyltin (MOT), Dioctyltin (DOT), Trioctyltin (TOT), Diphenyltin (DPhT), Triphenyltin (TPhT) and Tricyclohexyltin (TCyHT) on sample #16650 applying the analysis procedure that is routinely used in the

laboratory. A special instruction was given to treat the sample as a monocolored sample and not to analyze the colours separately. Also some method details were requested to be reported.
It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but to report as much significant figures as possible. It was also requested not to report “less than’ results, which are above the detection limit, because such results cannot be used for meaningful statistical evaluation.

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that did not report test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results. Additional or corrected test results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation’ of April 2014 (iis-protocol, version 3.3).

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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs’ and or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation, it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

3.3 Z-SCORES

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

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8. In case no literature reproducibility was available, other target values were used.

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 result tables in appendix 1.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This should be done in order to evaluate whether the reported test results are fit-for-purpose.

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

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

4 EVALUATION

During the execution of this proficiency test, no problems occurred with the delivery of the samples. Six laboratories did not report any test results and two laboratories reported results after the final reporting date.
In total 48 numerical test results were received. Observed was 1 outlying test result, which is 2.1% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Organotin a number of the test methods with precision data are available (eg. ISO/TS 16179 and ISO 17353). Regretfully, none of the test methods describes the Organotin determination in textile only. In this proficiency test the majority of the participants used ISO 17353 as test method, which is applicable for water samples. A number of other participants used ISO/TS 16179 as test method, which is applicable for footwear materials.
As the ISO/TS 16179 seems more suitable, it was therefore decided to use the target reproducibility, estimated from the precision data as mentioned in the annex of ISO/TS 16179.

4.1 EVALUATION ANALYTICAL DETAILS

For this PT some analysis details were requested (see appendix 2 and 3). Questions like: was the laboratory accredited, what type of extraction was used, which solvent for extraction, etc.?

From the answers given by the participants, the following can be summarized:
About 79% of the participants is accredited according to ISO/IEC 17025.

25% of the participants, that reported to have used ISO 17353 as test method, did not follow the test method completely. These participants mentioned to have used acetone as extraction solvent instead of hexane. No relation was found between the test results from each of the two extraction solvents, when these test results were evaluated separately.

4.2 EVALUATION PER TEST

In this section the reported results are discussed per test. All statistical results reported on the textile sample is summarised in appendix 1.

The original data set of MBT proved to have a normal Gaussian distribution.

Sample #16650:

Monobutyltin (MBT): This determination is problematic. No statistical outliers were observed. However, the calculated reproducibility of the total group is not in agreement with the ISO/TS 16179:12, nor with ISO 17353:04 (see also chapter 6).

4.3 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility (see 4.1) and the reproducibility as found for the group of participating laboratories.
The number of significant results, the average test result, the calculated reproducibility (standard deviation*2.8) and the target reproducibility are compared in the next table:

	unit	*n*	*Average*	*2.8 * sd*	*R (target)*
Monobutyltin (MBT)	mg/kg	47	1.21	1.26	0.78

Table 3: Reproducibility of Organotin, sample #16650

Without further statistical calculations, it can be concluded that for MBT, the total group of participating laboratories may have difficulties with the analysis. See also the discussion in paragraphs 4.1 and 6.

5 COMPARISON INTERLABORATORY RESULT

The variation found for this first organized proficiency test is large in comparison with the requirements mentioned in the standard.

	December 2016	*ISO/TS16179:12*
MBT	37%	23%

Table 4: Comparison of uncertainties in iis proficiency tests

6 DISCUSSION

In this proficiency test for the determination of Organotin in textile, it was noticed that the majority of the participants was able to detect and quantify correctly MBT in sample #16650.

A number of different test methods were reported to have been used. Most often ISO17353 (27 laboratories) was mentioned as test method used, followed by ISO/TS16179 (11 laboratories) and seven laboratories reported to have used an in house test method.

The test results of the Monobutyltin determination were evaluated separately per test method. The mean of the ISO/TS16179 test results is high in comparison with the mean of all test results. However, the calculated reproducibility of the ISO/TS16179 test results is smaller than the reproducibility of all test results, but still not in agreement with the precision requirements of ISO/TS16179 (see page 10 and 11).
The mean of the ISO17353 test results is equal to the mean of all test results. However, the calculated reproducibility is small in comparison with the reproducibility of all test results and in good agreement with the precision requirements of ISO17353 (see page 10 and 10).

When the test results of this interlaboratory study were compared to the Öko-Tex Standard 100, it could be noted that a number of laboratories would make a different decision about the acceptability of the textile. Eleven reporting laboratories would accept the sample for all classes (less than 1 mg/kg) and in total forty-four reporting laboratories would accept the sample only for class 2, 3 and 4. Five reporting laboratories would reject the sample for all classes (exceed 2 mg/kg).

Öko-Tex Standard 100	Class 1 Baby clothes (mg/kg)	Class 2 Clothes direct skin contact (mg/kg)	Class 3 Clothes, no direct contact with skin (mg/kg)	Class 4 Decoration material (mg/kg)
Monobutyltin (MBT)	1.0	2.0	2.0	2.0

Table 5: Ecolabelling Standard and Requirements for Textiles in EU

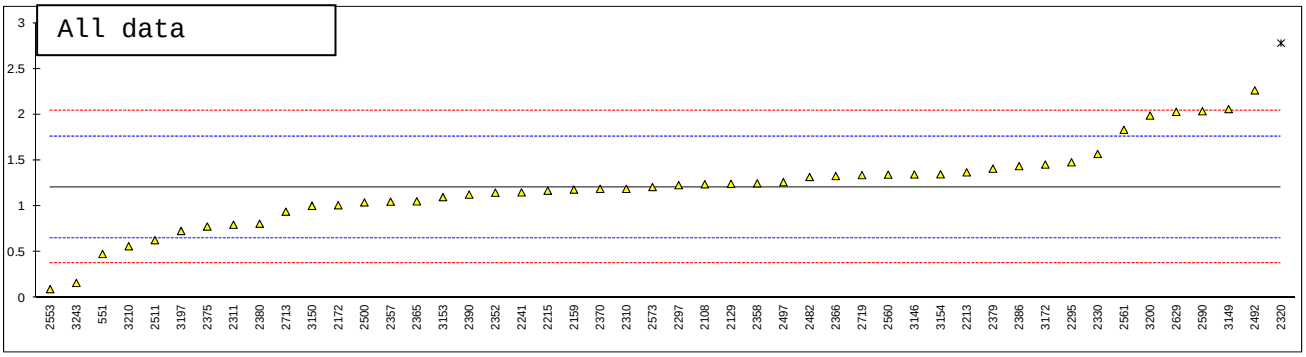
From the specific details which were requested to report it was difficult to draw significant conclusions to judge the performance of each laboratory on this Organotin determination. Although it is clear that not all laboratories followed the reported test method completely, it can be concluded that the observed variation in this interlaboratory study may not be caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1

Determination of Monobutyltin (MBT) on sample #16650; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551	In house	0.4791		-2.62	
2108	In house	1.24		0.11	
2129		1.245		0.13	
2159	ISO17353	1.182		-0.10	
2172	ISO17353	1.011		-0.71	
2213	ISO17353	1.37		0.58	
2215	ISO17353	1.17		-0.14	
2241	ISO17353	1.151		-0.21	
2264	ISO16179	< 0.55		-----	
2295	ISO17353	1.48		0.97	
2297	ISO17353	1.23		0.08	
2310	ISO17353	1.19		-0.07	
2311	ISO17353	0.799		-1.47	
2320	In house	2.780	C,R(0.05)	5.65	First reported 2.998
2330	ISO17353	1.571		1.30	
2352	ISO17353	1.148		-0.22	
2357	ISO17353	1.05		-0.57	
2358	ISO17353	1.2502		0.15	
2365	ISO17353	1.054		-0.56	
2366	ISO17353	1.33		0.44	
2370	ISO17353	1.19		-0.07	
2375	ISO17353	0.779		-1.55	
2379	ISO17353	1.41		0.72	
2380	ISO17353	0.81		-1.43	
2386	ISO17353	1.438		0.82	
2390	ISO17353	1.127		-0.29	
2475		-----		-----	
2482	ISO/TS16179	1.32		0.40	
2492	In house	2.265		3.80	
2497		1.263		0.19	
2500	ISO17353	1.0423		-0.60	
2511	ISO17353	0.63		-2.08	
2553	In house	0.096		-4.00	
2560	ISO17353	1.345		0.49	
2561	ISO/TS16179	1.834		2.25	
2573	ISO/TS16179	1.21		0.00	
2590	ISO/TS16179	2.037		2.98	
2629	ISO17353	2.03	C	2.95	First reported 12.5
2658		-----		-----	
2672		-----		-----	
2713	In house	0.94090		-0.96	
2719	ISO/TS16179	1.34		0.47	
2749		-----		-----	
2770		-----		-----	
3146	ISO/TS16179	1.3467		0.50	
3149	ISO/TS16179	2.06	C	3.06	First reported 2.29
3150	ISO17353	1.005		-0.73	
3153	DIN38407	1.1		-0.39	
3154	ISO/TS16179	1.349		0.50	
3172	ISO/TS16179	1.455		0.88	
3197	ISO/TS16179	0.73		-1.72	
3199		-----		-----	
3200	ISO17353	1.99		2.81	
3207		-----		-----	
3209		-----		-----	
3210	In house	0.564		-2.32	
3237		-----		-----	
3243	In house	0.163	C	-3.76	First reported 0.368

			Only ISO16179 data	Only ISO17353 data
	normality	OK	OK	suspect
	n	47	10	27
	outliers	1	0	0
	mean (n)	1.2089	1.4682	1.2142
	st.dev. (n)	0.44985	0.40644	0.32009
	R(calc.)	1.2596	1.1380	0.8963
	R(ISO16179:12)	0.7786	0.9455	--
	For comparison			
	R(ISO17353:04)	1.0291	--	1.0335
	R(Horwitz)	0.5264	--	--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2
1.5
1
0.5
0
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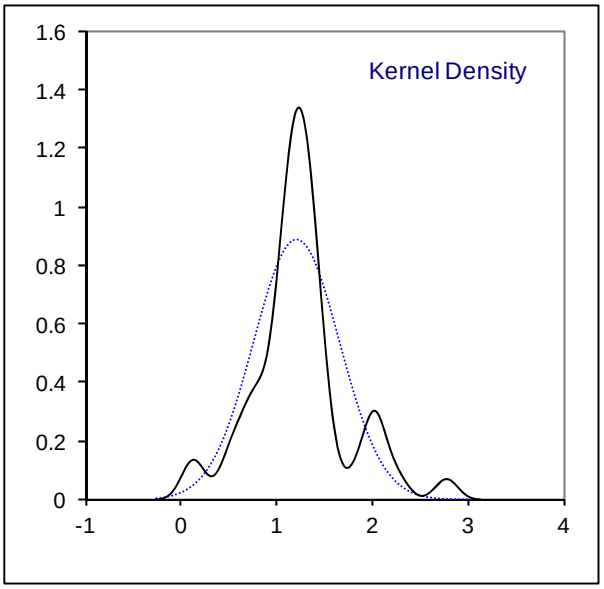
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1.2
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0.8
0.6
0.4
0.2
0
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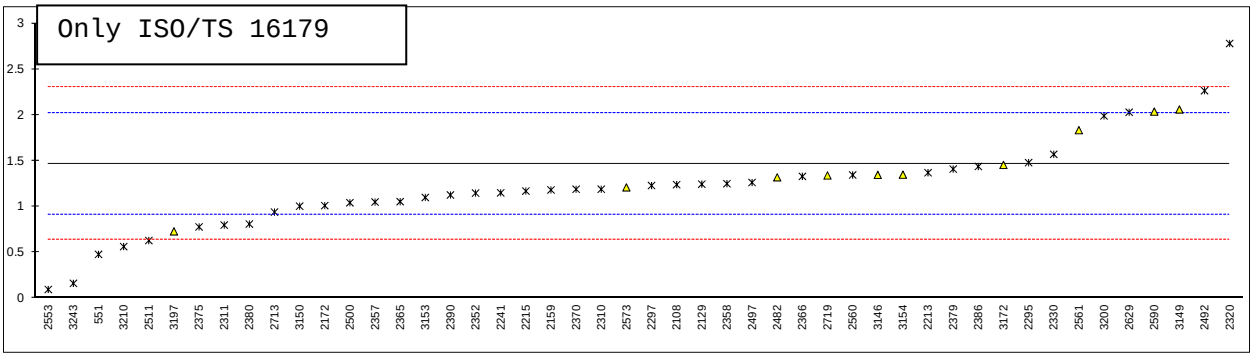
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2.5
2
1.5
1
0.5
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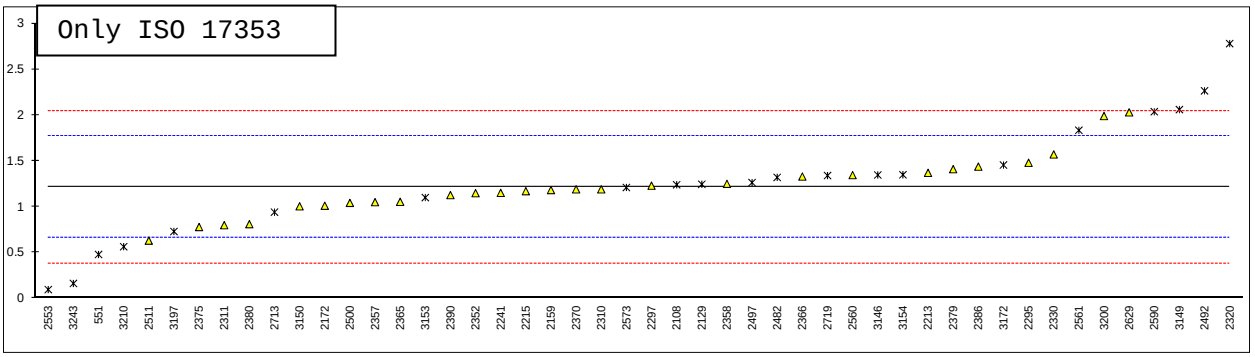
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2
1.5
1
0.5
0
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Determination of other Organotin components on sample #16650; results in mg/kg

lab	method	MMT	mark	DMT	mark	TMT	mark	TPT	mark	DBT	mark
551		-----		-----		-----		-----		-----	
2108		-----		-----		-----		-----		-----	
2129		-----		-----		-----		-----		-----	
2159	ISO17353	<0.025		<0.025		<0.025		<0.025		<0.025	
2172		-----		-----		-----		-----		-----	
2213	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2215	ISO17353	<0.05		<0.05		<0.05		<0.05		<0.05	
2241	ISO17353	N.D.		-----		-----		-----		N.D.	
2264	ISO16179	-----		-----		-----		-----		< 0.55	
2295		-----		-----		-----		-----		-----	
2297	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2310	ISO17353	Not detected		Not detected		Not detected		Not detected		Not detected	
2311	ISO17353	Not detected		Not detected		Not detected		Not detected		Not detected	
2320	In house	Under devel.		Under devel.		Under devel.		Not detected		Not detected	
2330	ISO17353	ND		ND		ND		ND		ND	
2352	ISO17353	Nd		nd		nd		nd		nd	
2357	ISO17353	ND		ND		ND		ND		ND	
2358	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2365	ISO17353	ND		ND		ND		ND		ND	
2366	ISO17353	Out Cap		Out Cap		Out Cap		<0.2		<0.2	
2370	ISO17353	n.d.		n.d.		n.d.		n.d.		n.d.	
2375		-----		-----		-----		-----		-----	
2379	ISO17353	-----		-----		-----		-----		not detected	
2380	ISO17353	N.D		N.D		N.D		N.D		N.D	
2386	ISO17353	<0,01		<0,01		<0,01		<0,01		<0,01	
2390	ISO17353	ND		ND		ND		ND		ND	
2475		-----		-----		-----		-----		-----	
2482		-----		-----		-----		-----		-----	
2492		-----		-----		-----		-----		-----	
2497		-----		-----		-----		-----		-----	
2500	ISO17353	N.D.		N.D.		N.D.		N.D.		N.D.	
2511		-----		-----		-----		-----		-----	
2553	In house	ND		ND		ND		ND		ND	
2560	ISO17353	<0.02		<0.02		<0.02		<0.02		<0.02	
2561	ISO/TS16179	-----		-----		-----		-----		0.0	
2573	ISO/TS16179	Not detected		Not detected		Not detected		Not detected		Not detected	
2590	ISO/TS16179	-----		< L.O.Q.		-----		< L.O.Q.		< L.O.Q.	
2629	ISO17353	-----		-----		-----		-----		ND	
2658		-----		-----		-----		-----		-----	
2672		-----		-----		-----		-----		-----	
2713	In house	-----		-----		-----		-----		n.d	
2719	ISO/TS16179	-----		-----		-----		-----		<0.05	
2749		-----		-----		-----		-----		-----	
2770		-----		-----		-----		-----		-----	
3146		-----		-----		-----		-----		-----	
3149	ISO/TS16179	-----		-----		-----		-----		< 0,05	
3150	ISO17353	<0,05		<0,05		<0,05		<0,05		<0,05	
3153	DIN38407	<0.05		<0.05		<0.05		<0.05		<0.05	
3154		-----		-----		-----		-----		-----	
3172	ISO/TS16179	< 0.05		< 0.05		< 0.05		< 0.05		< 0.05	
3197	ISO/TS16179	ND		ND		ND		ND		ND	
3199		-----		-----		-----		-----		-----	
3200		-----		-----		-----		-----		-----	
3207		-----		-----		-----		-----		-----	
3209		-----		-----		-----		-----		-----	
3210	In house	-----		-----		-----		-----		<0.50	
3237		-----		-----		-----		-----		Not detected	
3243	In house	n.d.		n.d.		n.d.		n.d.		n.d.	
	normality	n.a.		n.a.		n.a.		n.a.		n.a.	
	n	25		25		24		27		36	
	outliers	n.a.		n.a.		n.a.		n.a.		n.a.	
	mean (n)	<0.1		<0.1		<0.1		<0.2		<0.2	
	st.dev. (n)	n.a.		n.a.		n.a.		n.a.		n.a.	
	R(calc.)	n.a.		n.a.		n.a.		n.a.		n.a.	
	R(Horwitz)	n.a.		n.a.		n.a.		n.a.		n.a.	

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

Determination of other Organotin components on sample #16650; results in mg/kg == continued ==

lab	method	TBT	mark	TeBT	mark	MOT	mark	DOT	mark	TOT	mark
551		-----		-----		-----		-----		-----	
2108		-----		-----		-----		-----		-----	
2129		-----		-----		-----		-----		-----	
2159	ISO17353	<0.025		<0.025		<0.025		<0.025		<0.025	
2172		-----		-----		-----		-----		-----	
2213	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2215	ISO17353	<0.05		<0.05		<0.05		<0.05		<0.05	
2241	ISO17353	N.D.		N.D.		N.D.		N.D.		-----	
2264	ISO16179	< 0.55		-----		-----		< 0.55		< 0.55	
2295		-----		-----		-----		-----		-----	
2297	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2310	ISO17353	Not detected		Not detected		Not detected		Not detected		Not detected	
2311	ISO17353	Not detected		Not detected		Not detected		Not detected		Not detected	
2320	In house	Not detected		Not detected		Not detected		Not detected		Not detected	
2330	ISO17353	ND		ND		ND		ND		ND	
2352	ISO17353	nd		nd		nd		nd		nd	
2357	ISO17353	ND		ND		ND		ND		ND	
2358	ISO17353	<0.1		<0.1		<0.1		<0.1		<0.1	
2365	ISO17353	ND		ND		ND		ND		ND	
2366	ISO17353	<0.2		<0.2		<0.2		<0.2		<0.2	
2370	ISO17353	n.d.		n.d.		n.d.		n.d.		n.d.	
2375		-----		-----		-----		-----		-----	
2379	ISO17353	not detected		not detected		not detected		***2.01***	F+?	-----	
2380	ISO17353	N.D		N.D		N.D		N.D		N.D	
2386	ISO17353	<0,01		<0,01		<0,01		<0,01		<0,01	
2390	ISO17353	ND		ND		ND		ND		ND	
2475		-----		-----		-----		-----		-----	
2482		-----		-----		-----		-----		-----	
2492		-----		-----		-----		-----		-----	
2497		-----		-----		-----		-----		-----	
2500	ISO17353	N.D.		N.D.		N.D.		N.D.		N.D.	
2511		-----		-----		-----		-----		-----	
2553	In house	ND		ND		ND		ND		ND	
2560	ISO17353	<0.02		<0.02		<0.02		<0.02		<0.02	
2561	ISO/TS16179	0.0		0.0		0.0		0.0		-----	
2573	ISO/TS16179	Not detected		Not detected		Not detected		Not detected		Not detected	
2590	ISO/TS16179	< L.O.Q.		< L.O.Q.		< L.O.Q.		< L.O.Q.		-----	
2629	ISO17353	ND		ND		ND		ND		ND	
2658		-----		-----		-----		***0.22***	F+?	-----	
2672		-----		-----		-----		-----		-----	
2713	In house	n.d		-----		-----		n.d		-----	
2719	ISO/TS16179	<0.05		<0.05		<0.05		<0.05		-----	
2749		-----		-----		-----		-----		-----	
2770		-----		-----		-----		-----		-----	
3146		-----		-----		-----		-----		-----	
3149	ISO/TS16179	< 0,05		< 0,05		< 0,05		< 0,05		-----	
3150	ISO17353	<0,05		<0,05		<0,05		<0,05		<0,05	
3153	DIN38407	<0.05		<0.05		<0.05		<0.05		<0.05	
3154		-----		-----		-----		-----		-----	
3172	ISO/TS16179	< 0.05		< 0.05		< 0.05		< 0.05		< 0.05	
3197	ISO/TS16179	ND		ND		ND		ND		ND	
3199		-----		-----		-----		-----		-----	
3200		-----		-----		-----		-----		-----	
3207		-----		-----		-----		-----		-----	
3209		-----		-----		-----		-----		-----	
3210	In house	<0.50		<0.50		<0.50		<0.50		-----	
3237		Not detected		-----		-----		Not detected		-----	
3243	In house	n.d.		n.d.		n.d.		n.d.		n.d.	
	normality	n.a.		n.a.		n.a.		n.a.		n.a.	
	n	35		33		33		34		27	
	outliers	n.a.		n.a.		n.a.		n.a.		n.a.	
	mean (n)	<0.2		<0.2		<0.2		<0.2		<0.2	
	st.dev. (n)	n.a.		n.a.		n.a.		n.a.		n.a.	
	R(calc.)	n.a.		n.a.		n.a.		n.a.		n.a.	
	R(Horwitz)	n.a.		n.a.		n.a.		n.a.		n.a.	

TBT = Tributyltin
TeBT = Tetrabutyltin
MOT = Monooctyltin
DOT = Dioctyltin
TOT = Trioctyltin

Determination of other Organotin components on sample #16650; results in mg/kg == continued ==

lab	method	DPhT	mark	TPhT	mark	TCyHT	mark
551		-----		-----		-----	
2108		-----		-----		-----	
2129		-----		-----		-----	
2159	ISO17353	<0.025		<0.025		<0.025	
2172		-----		-----		-----	
2213	ISO17353	<0.1		<0.1		<0.1	
2215	ISO17353	<0.05		<0.05		<0.05	
2241	ISO17353	N.D.		N.D.		N.D.	
2264	ISO16179	-----		< 0.55		-----	
2295		-----		-----		-----	
2297	ISO17353	<0.1		<0.1		<0.1	
2310	ISO17353	Not detected		Not detected		Not detected	
2311	ISO17353	Not detected		Not detected		Not detected	
2320	In house	Not detected		Not detected		Not detected	
2330	ISO17353	ND		ND		ND	
2352	ISO17353	nd		nd		nd	
2357	ISO17353	ND		ND		ND	
2358	ISO17353	<0.1		<0.1		<0.1	
2365	ISO17353	ND		ND		ND	
2366	ISO17353	Out Cap		<0.2		<0.2	
2370	ISO17353	n.d.		n.d.		n.d.	
2375		-----		-----		-----	
2379	ISO17353	-----		not detected		not detected	
2380	ISO17353	N.D		N.D		N.D	
2386	ISO17353	<0,01		<0,01		<0,01	
2390	ISO17353	ND		ND		ND	
2475		-----		-----		-----	
2482		-----		-----		-----	
2492		-----		-----		-----	
2497		-----		-----		-----	
2500	ISO17353	N.D.		N.D.		N.D.	
2511		-----		-----		-----	
2553	In house	ND		ND		ND	
2560	ISO17353	<0.02		<0.02		<0.02	
2561	ISO/TS16179	0.0		0.0		0.0	
2573	ISO/TS16179	Not detected		Not detected		Not detected	
2590	ISO/TS16179	< L.O.Q.		< L.O.Q.		< L.O.Q.	
2629	ISO17353	ND		ND		ND	
2658		-----		-----		-----	
2672		-----		-----		-----	
2713	In house	-----		n.d		-----	
2719	ISO/TS16179	-----		<0.05		<0.05	
2749		-----		-----		-----	
2770		-----		-----		-----	
3146		-----		-----		-----	
3149	ISO/TS16179	-----		< 0,05		< 0,05	
3150	ISO17353	<0,05		<0,05		<0,05	
3153	DIN38407	<0.05		<0.05		<0.05	
3154		-----		-----		-----	
3172	ISO/TS16179	< 0.05		< 0.05		< 0.05	
3197	ISO/TS16179	ND		ND		ND	
3199		-----		-----		-----	
3200		-----		-----		-----	
3207		-----		-----		-----	
3209		-----		-----		-----	
3210	In house	-----		<0.50		<0.50	
3237		-----		Not detected		-----	
3243	In house	n.d.		n.d.		n.d.	
	normality	n.a.		n.a.		n.a.	
	n	29		35		33	
	outliers	n.a.		n.a.		n.a.	
	mean (n)	<0.2		<0.2		<0.2	
	st.dev. (n)	n.a.		n.a.		n.a.	
	R(calc.)	n.a.		n.a.		n.a.	
	R(Horwitz)	n.a.		n.a.		n.a.	

DPhT = Diphenyltin
TPhT = Triphenyltin
TCyHT = Tricyclohexyltin

APPENDIX 2

Accreditation details of the participants

labnrs	Is your laboratory accredited for this determination	Labnrs	Is your laboratory accredited for this determination
551	---	2500	Yes
2108	Yes	2511	No
2129	Yes	2553	Yes
2159	Yes	2560	Yes
2172	Yes	2561	Yes
2213	Yes	2573	Yes
2215	Yes	2590	Yes
2241	Yes	2629	Yes
2264	No	2658	Yes
2295	Yes	2672	---
2297	Yes	2713	Yes
2310	Yes	2719	Yes
2311	Yes	2749	---
2320	Yes	2770	---
2330	No	3146	Yes
2352	Yes	3149	No
2357	Yes	3150	Yes
2358	Yes	3153	Yes
2365	Yes	3154	Yes
2366	Yes	3172	Yes
2370	Yes	3197	Yes
2375	Yes	3199	---
2379	No	3200	---
2380	Yes	3207	---
2386	Yes	3209	---
2390	Yes	3210	No
2475	---	3237	No
2482	Yes	3243	No
2492	Yes		
2497	Yes		

APPENDIX 3

Details of the methods used by the participants

labnrs	1. Intake sample determination	2. What type of extraction was used?	3. Which solvent was used to extract the components from the textile?	4. What was the extraction time (minutes) and temperature (°C)?	5. What was the pH after adding the buffer to the extraction solvent?	6. Was the extraction solution after adding the buffer acidified until pH 4.5	Remarks on Additional Questions:
551	-----	-----	-----	-----	-----	-----	-----
2108	0,5g	Ultrasonic	Ethanol / Glacial acetic acid	-----	-----	-----	-----
2129	0,6 g each, double det.	Ultrasonic	acidic methanol	60 min room temperature	not checked	no	-----
2159	1 gram	Ultrasonic	Methanol	1 hour / 70°C	4.5	Yes	-----
2172	0.5g	-----	methanol	70 °C for 60 minutes	4.5	yes	-----
2213	0.5 gm	-----	Ethanol	60 minutes at room temperature.	8.5	Yes	-----
2215	0.5	Ultrasonic	acetate buffer solution	70 oC for 60 minutes	4.5	-----	-----
2241	0.2g	Ultrasonic	Ethanol	2h & room temperature	4.5	Yes	-----
2264	0.7 g	Ultrasonic	Methanol + ethanol	60 minutes at 60°C	-	4.5	-----
2295	1.0	Ultrasonic	95%methanol 5%ethanol	60 min at room temp	4.5	yes	-----
2297	1.0023g	Ultrasonic	acidic methanol	60°C for 1Hr	4.5	yes	-----
2310	0.5 gram	solvent extraction	Acetone	1 hour and 40°C	4.5pH - 5.0pH	yes	-----
2311	1gm	Ultrasonic extraction	Acetone	Time 60 minutes and temperature 40°C	5.4	YES	-----
2320	0.60800 g	solvent extraction	acetone	60 minutes, 40 °C	4.6	no	-----
2330	0.2g	Derivatization and liquid-liquid extraction	Acetone	40 °C 1hr	4.5	Yes	-----
2352	0.2g	Ultrasonic	Acetone	60min 40°C	4.50	n-Hexane	No
2357	0.5g	Ultra sonic	Acetone	60min 40°C	4.7	Hexane	-----
2358	0.2 g	Sonication	Acetone	60 minutes and 40 degree C	5.1	Yes	-----
2365	0.1g	Ultrasonic	acetone	60 minutes,40Celsius degree	4.4	NA	-----
2366	0.25g	Ultrasonic	Acetone	40°C,1 Hour	About 5.0	Yes	None
2370	0.25g	Ultrasonic	Ethanol	60minutes,30°C	pH=4.5	Acetic acid	-----
2375	0.5 gr	Ultrasonic	Acetone	40 C - 60 minutes	pH=4,5	Hexane	-----
2379	-----	-----	-----	-----	-----	-----	-----
2380	0.5081	Solvent Extraction	Acetone	60 minute & 40 °C	4.5	yes	-----
2386	0,500 g	solid/liquid	Acetone	60 min, 40 °C	4,5	No	-----
2390	0.3 gm	Sonication	Acetone	60 minutes, 42°C	5.8	Yes	-----
2475	-----	-----	-----	-----	-----	-----	-----
2482	0.5 g	Ultrasonic	Methanol/Ethanol 80:20	60 minutes, 60°C	4.5	no	-----
2492	0.3g	Ultrasonic	Ethanol/acetic acid solution 95/5 (v/v) + Sodium diethyl dithiocarbamate-solution (NaDDC)	60min and 40°C	pH 5	No	-----
2497	0.5g	-----	methanol/ethanol 80/20	60 min - 60°C	not determined	not determined	-----
2500	4	-----	methanol	70 oC for 60 minutes	4.5	YES	-----
2511	-----	-----	-----	-----	-----	-----	-----

2553	1.000 g	Ultrasonic	Solvent of 1 g sodium diethyldithio carbamate trihydrate in 500 mL methanol.	60 min in 70°C	4.5	yes	-----
2560	0.7248g		0.03% Sodium diethyl Dithiocarbonate in methanol & ethanol	60mins at room temp.	5. 4.5±0.3		-----
2561	0.1g	Ultrasonic	80:20 methanol:ethanol mix	60 mina @ 60C		no	-----
2573	0.5g	Ultrasonic	methanol/ethanol mixture (80/20 v/v)	1 h at 60 °C	around pH 5.0	No	-----
2590	1 g	Ultrasonic	Methanol/Ethanol 80:20 -	60 minutes and 60 °C	6.5	Yes it was	-----
2629	0.5 gram	Liquid - Liquid extraction	n-Hexane/Methanol	60 minutes at 60 0C	4.5	4.5	-----
2658	1 gram	Ultrasonic	methanol/ethanol	1 h 60°C	4,5	yes	-----
2672	-----	-----	-----	-----	-----	-----	-----
2713	˜ 0.5 gr	liquid liquid extraction	Methanol with Sodium diethyldithio carbamate trihydrate	60 minutes and 60 °C by ultrasonic bath	not measured	Yes	MBT is not accredited in accordance with ISO/IEC17025
2719	1.0088	-----	Methanol/ethanol (80:20)	60 minutes, 60 °C		yes	-----
2749	-----	-----	-----	-----	-----	-----	-----
2770	-----	-----	-----	-----	-----	-----	-----
3146	0,5g	Ultrasonic	Methanol/ethanol 80/20 V/V	1h 60°C ultrasonic bath	-----	-----	-----
3149	1 g	Ultrasonic	Methanol/Ethanol 80/20 + Tropolon	1 h, 60 °C	-----	-----	-----
3150	2 g	Reflux	0,1 % methanolic hydrochloric acid	30 min	-----	-----	-----
3153	0.2g	Ultrasonic	Ethanol	Room temperature for 60 mins x 2	4.7	Yes	-----
3154	0,5 g	Ultrasonic	methanol / ethanol	60 min 60 °C	-----	-----	-----
3172	0.5 grams	Ultrasonic	methanol-ethanol 8:2	60min - 60°C	4.5	--	-----
3197	1 g	Ultrasonic	Methanol:Ethanol 80:20	60 minutes and 60C	5,5	Yes	-----
3199	-----	-----	-----	-----	-----	-----	-----
3200	0.5g	Shake	Methanl:Ethanol = 2:8	60°C, 1 hour	5.5	-----	-----
3207	-----	-----	-----	-----	-----	-----	-----
3209	-----	-----	-----	-----	-----	-----	-----
3210	0.998 grams	Ultrasonic/ liquid liquid extraction	methanol (12 ml) and ethanol (3 ml)	60 °C for 60 minutes	not measured	not measured	-----
3237	0,5 grams	Ultrasonic	Ethanol	60 C, 60 minutes	-----	Acetic acid	-----
3243	0.6 g	Ultrasonic	Ethanol	1 hour, RT	4.5	-----	-----

APPENDIX 4

Number of participants per country

2 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA, Kingdom of
2 labs in	FRANCE
10 labs in	GERMANY
3 labs in	HONG KONG
3 labs in	INDIA
3 labs in	ITALY
13 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	PERU
2 labs in	SRI LANKA
1 lab in	SWITZERLAND
1 lab in	TAIWAN R.O.C.
2 labs in	THAILAND
1 lab in	TUNESIA
7 labs in	TURKEY
1 lab in	UNITED STATES OF AMERICA
1 lab in	UNITED KINGDOM
2 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
n.a. = not applicable
n.d. = not detected
W = test result withdrawn on request of participant

Literature:

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7 P.L. Davies. Fr Z. Anal. Chem. 351. 513. (1988)
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9 ISO 5725. (1986)
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