Results of Proficiency Test Heavy Metals by perspiration in textile November 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis18A10

January 2019

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1 INTRODUCTION

Since the 1990's, 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 textiles, there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known programs are for instance Milieukeur (the Netherlands), Oeko-Tex Standard 100 (Germany), BlueSign (Europe) and AAFA (United States).

Since 2002, the Institute of Interlaboratory Studies (iis) organizes a proficiency scheme for perspirated metals in textile every year. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the analysis of perspirated metals in textile.
In this interlaboratory study, 92 laboratories from 26 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different textile samples, which both were artificially fortified with different metal dyes. The participants were also requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

Two different textile samples were obtained from third party laboratories. The first batch was an one side red coloured polyester, fortified with Antimony. The second batch was a blue-green cotton, which was fortified with Chromium and Mercury.
Both batches were cut finely, well mixed and divided over 110 subsamples of 3 grams each and respectively labelled #18360 and #18361. The homogeneity of subsamples #18630 and #18631 was checked by the determination of perspirated Antimony on sample #18630 and by the determination of perspirated Chromium and Mercury on sample #18631 on 8 stratified randomly selected subsamples of each set.

	Perspirated Antimony in mg/kg
Sample #18630-1	37.4
Sample #18630-2	36.2
Sample #18630-3	38.5
Sample #18630-4	39.5
Sample #18630-5	36.3
Sample #18630-6	38.3
Sample #18630-7	36.4
Sample #18630-8	37.3

Table 1: homogeneity test results of subsamples #18630

	Perspirated Chromium in mg/kg	*Perspirated Mercury in mg/kg*
Sample #18631-1	9.01	0.209
Sample #18631-2	8.99	0.220
Sample #18631-3	8.84	0.192
Sample #18631-4	8.99	0.206
Sample #18631-5	9.27	0.189
Sample #18631-6	9.39	0.203
Sample #18631-7	9.62	0.193
Sample #18631-8	9.44	0.213

Table 2: homogeneity test results of subsamples #18631

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibilities of the reference test method, in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Perspirated Antimony in mg/kg	*Perspirated Chromium in mg/kg*	*Perspirated Mercury in mg/kg*
r (observed) #18630	3.4	--	--
r (observed) #18631	--	0.77	0.031
reference test method	EN16711-2:15	EN16711-2:15	EN16711-2:15
0.3 x R (ref. test method)	6.3	1.16	0.053

Table 3: evaluation of the repeatabilities of subsamples #18630 and #18631

The calculated repeatabilities of each metal were in good agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18630 and #18631 was assumed.

To each of the participating laboratories, one sample of #18630 and one sample of #18631 were sent on October 10, 2018.

2.5 ANALYSES

The participants were requested to determine on both samples: perspirated heavy metals: Antimony, Arsenic, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel and Zinc applying the analysis procedure that is routinely used in the laboratory, but also to use preferably a solid/liquid ratio of 1/50 g/ml as prescribed in EN16711-2:15 (DIN 54233-3:10). 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 results, but 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 evaluations.

To get comparable results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test method 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 appendix 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 reanalyses). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test 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 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 dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. 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 as met for all evaluated tests, therefore, the uncertainty of all assigned values maybe 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 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, e.g. EN reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this 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.
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 interlaboratory study, no problems were encountered with the dispatch of the samples. Only one participant did not report any test results at all. Not all laboratories were able to report all metals requested.
Finally, the 91 reporting laboratories reported 254 numerical test results. Observed were 8 outlying results, which is 3.1% of all reported numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original 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 METAL

In this section, the results are discussed per sample and per metal.
The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 5.

In 2010 the draft method DIN 54233-3 was issued. This method mentions the standard deviation and variation coefficient per metal between laboratories (see table A.1). The reproducibility of each metal was calculated by multiplying the standard deviation (or variation coefficient) of the metal with 2.8. In 2015 this test method was finalized and published as EN16711-2.

Sample #18630:

Antimony: The determination of this metal was not problematic at a perspiration level of 44.8 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN16711-2:15.

Other metals: The majority of the participants agreed on a content close to or below the quantification limit of Arsenic, Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel and Zinc. Therefore, no significant conclusions were drawn.

Sample #18631

Chromium: The determination of this metal was not problematic at a perspiration level of 9.1 mg/kg. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility of EN16711-2:15.

Mercury: The determination of this metal was problematic at a perspiration level of 0.26 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility of EN16711-2:15.

Other metals: The majority of the participants agreed on a content close to or below the quantification limit of Antimony, Arsenic, Cadmium, Cobalt, Copper, Lead, Manganese, Nickel and Zinc. Therefore, no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as declared by the relevant reference test method EN16711-2 and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * sd) and the target reproducibility derived from the reference test method (in casu EN16711-2) are presented in the next two tables.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Antimony as Sb	mg/kg	87	44.8	10.5	25.1

Table 4: reproducibilities of perspirated metals in sample #18630

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Chromium as Cr	mg/kg	88	9.1	2.5	3.8
Mercury as Hg	mg/kg	71	0.26	0.25	0.23

Table 5: reproducibilities of perspirated metals in sample #18631

From the above tables, it can be concluded that, without statistical calculations, the group of participating laboratories do not have difficulties with the analysis when compared with the target reproducibility of the reference test method EN16711-2:15, except for the determination of Mercury. See also the discussions in paragraphs 4.1 and 6.

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

The uncertainties that were found in the results during the present PT are in line with the uncertainties as observed in previous rounds and with the target requirements (see below table).

Parameter	*Nov. 2018*	*Nov. 2017*	*Oct. 2016*	*Oct. 2015*	*2010-2014*	*EN16711-2*
Arsenic as As	--	--	--	--	--	20%
Antimony as Sb	8%	--	--	--	16-19%	20%
Cadmium as Cd	--	18%	(24%)	12%	9-14%	10%
Chromium as Cr	10%	13%	12%	--	15-19%	15%
Cobalt as Co	--	9%	13%	--	8-14%	13%
Copper as Cu	--	--	10%	9-11%	10-22%	16%
Lead as Pb	--	40%	35%	--	--	40%
Manganese as Mn	--	--	--	--	--	--
Mercury as Hg	34%	--	(45%)	--	41%	31%
Nickel as Ni	--	--	--	11%	7-14%	10%
Zinc as Zn	--	(25%)	--	--	--	--

Table 6: development of uncertainties over the last years

*) results between brackets may be near or below the detection limit

4.4 EVALUATION OF ANALYTICAL DETAILS

The reported details of the analytical test methods that were used by the participants are listed in appendix 3. About 73% of the participating laboratories reported to be accredited for the determination of perspirated metals in textile.

For this PT, it was requested to report if the sample was further cut/grinded, the sample intake and what ratio (grams per ml textile) was used. It appeared that no effect was observed on the reported test results for the determined metals in sample #18630 nor in sample #18631.

5 DISCUSSION

The participants were requested to report eleven different metals. The majority of participants did only detect Antimony on sample #18630 and Chromium and Mercury on sample #18631.

Some participants would make different decisions about the acceptability of the textiles for the determined parameters, when the test results of this interlaboratory study are compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 7).
The detection limit reported by some laboratories does not meet the requirements of the Standards (reported detection limit is larger than the maximum required concentration by the Ecolabelling standard).

Ecolabel	Class 1: baby clothes	Class 2: in direct skin contact	Class 3: with no direct skin contact	Class 4: Decoration material
Antimony (Sb) mg/kg	30.0	30.0	30.0	--
Arsenic (As) mg/kg	0.2	1.0	1.0	1.0
Cadmium (Cd) mg/kg	0.1	0.1	0.1	0.1
Chromium (Cr) mg/kg	1.0	2.0	2.0	2.0
Cobalt (Co) mg/kg	1.0	4.0	4.0	4.0
Copper (Cu) mg/kg	25.0	50.0	50.0	50.0
Lead (Pb) mg/kg	0.2	1.0	1.0	1.0
Manganese (Mn) mg/kg	--	--	--	--
Mercury (Hg) mg/kg	0.02	0.02	0.02	0.02
Nickel (Ni) mg/kg	1.0	4.0	4.0	4.0
Zinc (Zn) mg/kg	--	--	--	--

Table 7: Ecolabelling Standards and Requirements for Textiles in EU

Methods for determination of these Heavy Metals via perspiration are specified in the Standards of the Ecolabelling Institutes. The method for detection of the metals is specified as “Detection via AAS or ICP”.
It should be noticed that for the results reported in this proficiency test, the majority of the participants have probably performed the acid perspiration step according to the same conditions. Differences in sample intake and perspiration time and temperature may be

parameters of importance. In the past, the solid/liquid ratio (grams of textile per ml perspiration liquid) appeared to be a parameter of utmost importance (see reports iis07A05 and iis08A05 on “Perspirated Metals in Textile”). Therefore, in this proficiency test the laboratories were advised to use preferably a ratio of 1:50 as in the test method EN16711-2:15. The majority of the participants reported to have used this ratio.

6 CONCLUSION

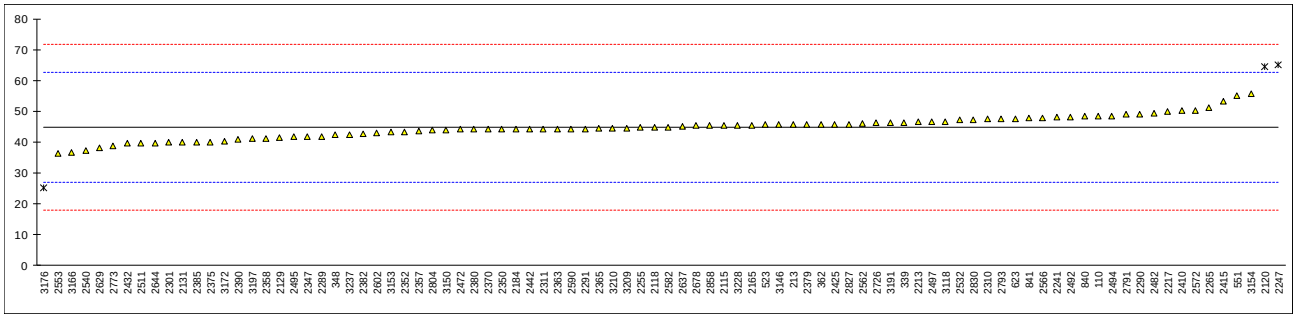
In this proficiency test, the perspirated metal content were determined. The variation observed for the perspirated metals in this interlaboratory study are in line with (or even better than) the observations in the previous proficiency tests. A possible explanation for the variation could be the preparation or the conditioning of the sample and/or by the performance of the analysis by the laboratory. Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

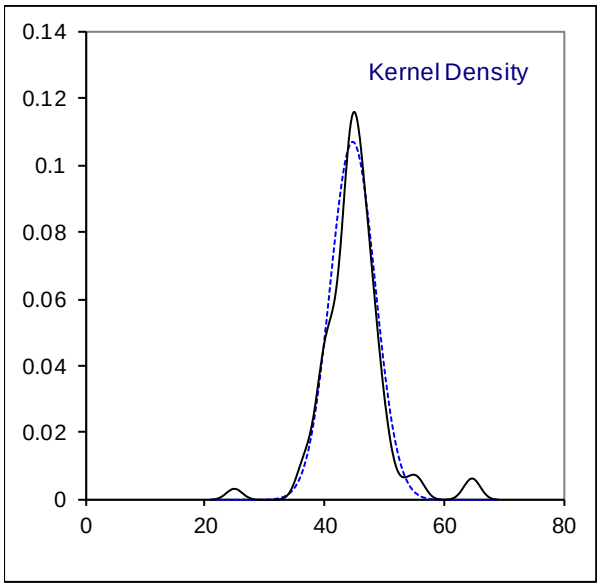
APPENDIX 1

Determination of Antimony as Sb on sample #18630; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
110	In house	48.35895		0.40	
213	ISO105E04	45.76		0.11	
339	ISO206-E04	46.4	C	0.18	First reported 82.8
348	ISO105E04	42.33		-0.28	
362	In house	45.8		0.11	
523	EN16711-2	45.59		0.09	
551	EN16711-2	55.09		1.15	
623	EN16711-2	47.57		0.31	
840	EN16711-2	48.31		0.39	
841	ISO105E04	47.7		0.32	
2115	EN16711-2	45.5		0.08	
2118	EN16711-2	44.7802		0.00	
2120	EN16711-2	64.4	R(0.01)	2.19	
2129	EN16711-2	41.4		-0.38	
2131	In house	40.019		-0.53	
2165	EN16711-2	45.51		0.08	
2184	DIN54233-3	44.16		-0.07	
2213	EN16711-2	46.52		0.19	
2217	EN16711-2	50.008		0.58	
2241	EN16711-2	47.97		0.35	
2247	EN16711-2	65.08	R(0.01)	2.26	
2255	EN16711-2	44.7		-0.01	
2265	EN16711-2	51.00		0.69	
2289	DIN54233-3	41.7		-0.35	
2290	DIN54233-3	48.99		0.47	
2291	OEKO TEX 100	44.30		-0.06	
2301	EN16711-2	40.00		-0.54	
2310	EN16711-2	47.501		0.30	
2311	EN16711-2	44.207		-0.07	
2347	ISO105E04	41.7		-0.35	
2350	EN16711-2	44.128		-0.08	
2352	EN16711-2	43.321		-0.17	
2357	EN16711-2	43.50		-0.15	
2358	EN16711-2	41.2		-0.40	
2363	EN16711-2	44.24		-0.06	
2365	EN16711-2	44.445		-0.04	
2370	EN16711-2	44.1		-0.08	
2375	EN16711-2	40.1		-0.53	
2379	ISO105E04	45.771		0.11	
2380	EN16711-2	44.083		-0.08	
2382	EN16711-2	42.824		-0.22	
2385	EN16711-2	40.1		-0.53	
2390	ISO105E04	40.98		-0.43	
2410	EN16711-2	50.2		0.60	
2415	EN16711-2	53.26		0.94	
2425	EN16711-2	45.8		0.11	
2432	ISO105E04	39.646		-0.58	
2442	EN16711-2	44.189		-0.07	
2472	GB/T17593	44.04		-0.09	
2482	EN16711-2	49.25		0.50	
2492	In house	48.090		0.37	
2494	ISO105E04	48.407		0.40	
2495	EN16711-2	41.68		-0.35	
2497	EN16711-2	46.642		0.21	
2511	EN16711-2	39.654		-0.57	
2514		-----		-----	
2532	EN16711-2	47.129		0.26	
2540	EN16711-2	37.1176		-0.86	
2553	ISO105E04	36.23		-0.96	
2562	GB/T17593	46.010		0.13	
2566	EN16711-2	47.9		0.35	
2572	DIN54233-3	50.3		0.61	
2582	EN16711-2	44.87	C	0.01	First reported 4237.38
2590	EN16711-2	44.254		-0.06	
2602	EN16711-2	43.12		-0.19	
2629	EN16711-2	38.24		-0.73	
2637	EN16711-2	45		0.02	
2638		-----		-----	
2644	EN16711-2	39.68		-0.57	
2678	EN16711-2	45.337		0.06	
2726	EN16711-2	46.281		0.16	
2773	EN16711-2	38.72		-0.68	
2791	EN16711-2	48.88		0.45	
2793	EN16711-2	47.5435		0.31	
2804	EN16711-2	43.7446		-0.12	

lab	method	value	mark	z(targ)	Remarks
2827	EN16711-2	45.84		0.12	
2830	EN16711-2	47.35		0.28	
2858	In house	45.48		0.08	
3118	EN16711-2	46.7233		0.21	
3146	EN16711-2	45.6		0.09	
3150	EN16711-2	43.759		-0.12	
3153	EN16711-2	43.234		-0.18	
3154	EN16711-2	55.81		1.23	
3166	EN16711-2	36.6		-0.92	
3172	EN16711-2	40.3		-0.50	
3176	EN16711-2	25.030	C,R(0.01)	-2.21	First reported 89.468
3191	EN16711-2	46.301		0.17	
3197	EN16711-2	41.1		-0.41	
3209	EN16711-2	44.563		-0.03	
3210	EN16711-2	44.47		-0.04	
3228	EN16711-2	45.50		0.08	
3237	ISO105E04	42.5		-0.26	
	normality	OK			
	n	87			
	outliers	3			
	mean (n)	44.805			
	st.dev. (n)	3.7323	RSD% = 8%		
	R(calc.)	10.450			
	st.dev.(EN16711-2:15)	8.9609			
	R(EN16711-2:15)	25.091			

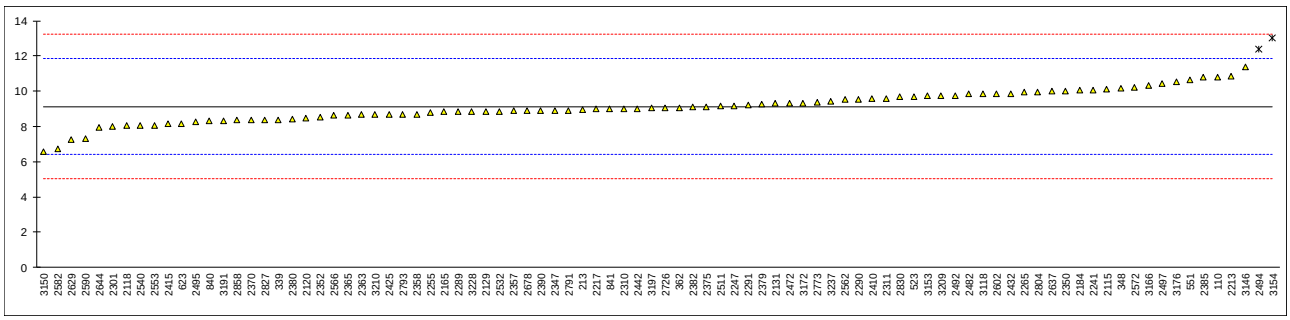


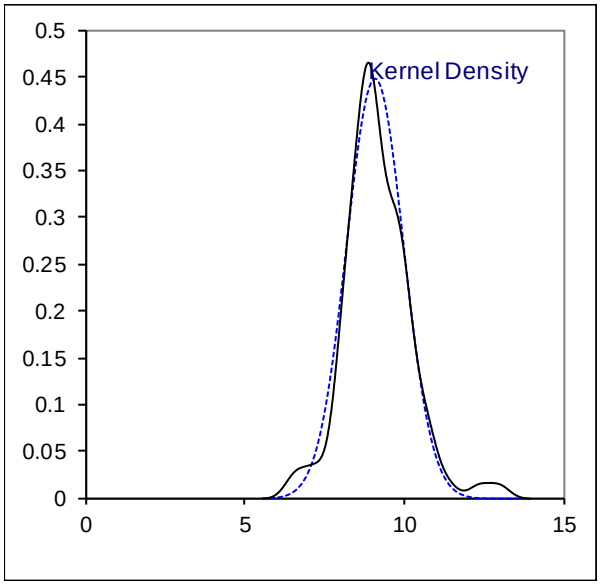


Determination of Chromium as Cr on sample #18631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	10.8242		1.25	
213	ISO105E04	8.93		-0.14	
339	ISO206-E04	8.393		-0.53	
348	ISO105E04	10.17		0.77	
362	In house	9.08		-0.03	
523	EN16711-2	9.71		0.43	
551	EN16711-2	10.65		1.12	
623	EN16711-2	8.14		-0.72	
840	EN16711-2	8.29		-0.61	
841	ISO105E04	9.0		-0.09	
2115	EN16711-2	10.1		0.72	
2118	EN16711-2	8.0340		-0.79	
2120	EN16711-2	8.49		-0.46	
2129	EN16711-2	8.86		-0.19	
2131	In house	9.310		0.14	
2165	EN16711-2	8.83		-0.21	
2184	DIN54233-3	10.05		0.68	
2213	EN16711-2	10.83		1.25	
2217	EN16711-2	8.9962		-0.09	
2241	EN16711-2	10.05		0.68	
2247	EN16711-2	9.14		0.01	
2255	EN16711-2	8.78		-0.25	
2265	EN16711-2	9.933		0.59	
2289	DIN54233-3	8.85		-0.20	
2290	DIN54233-3	9.52		0.29	
2291	OEKO TEX 100	9.20		0.06	
2301	EN16711-2	8.00		-0.82	
2310	EN16711-2	9.001		-0.09	
2311	EN16711-2	9.6097		0.36	
2347	EN16711-2	8.9		-0.16	
2350	EN16711-2	10.013		0.65	
2352	EN16711-2	8.522		-0.44	
2357	EN16711-2	8.87		-0.18	
2358	EN16711-2	8.71		-0.30	
2363	EN16711-2	8.66		-0.34	
2365	EN16711-2	8.657		-0.34	
2370	EN16711-2	8.37		-0.55	
2375	EN16711-2	9.1		-0.01	
2379	ISO105E04	9.253		0.10	
2380	EN16711-2	8.421		-0.51	
2382	EN16711-2	9.090		-0.02	
2385	EN16711-2	10.81		1.24	
2390	ISO105E04	8.89		-0.17	
2410	EN16711-2	9.6		0.35	
2415	EN16711-2	8.14		-0.72	
2425	EN16711-2	8.68		-0.32	
2432	ISO105E04	9.859		0.54	
2442	EN16711-2	9.017		-0.08	
2472	GB/T17593	9.333		0.16	
2482	EN16711-2	9.840		0.53	
2492	In house	9.743		0.46	
2494	ISO105E04	12.384	R(0.05)	2.39	
2495	EN16711-2	8.245		-0.64	
2497	EN16711-2	10.456		0.98	
2511	EN16711-2	9.136		0.01	
2514		-----		-----	
2532	EN16711-2	8.865		-0.19	
2540	EN16711-2	8.0424		-0.79	
2553	ISO105E04	8.073		-0.77	
2562	GB/T17593	9.505		0.28	
2566	EN16711-2	8.65		-0.34	
2572	DIN54233-3	10.21		0.80	
2582	EN16711-2	6.75		-1.73	
2590	EN16711-2	7.283		-1.34	
2602	EN16711-2	9.846		0.53	
2629	EN16711-2	7.257		-1.36	
2637	EN16711-2	10		0.64	
2638		-----		-----	
2644	EN16711-2	7.94		-0.86	
2678	EN16711-2	8.880		-0.18	
2726	EN16711-2	9.07		-0.04	
2773	EN16711-2	9.358		0.17	
2791	EN16711-2	8.91		-0.15	
2793	EN16711-2	8.7080		-0.30	
2804	EN16711-2	9.9690		0.62	

lab	method	value	mark	z(targ)	remarks
2827	EN16711-2	8.39		-0.53	
2830	EN16711-2	9.675		0.41	
2858	In house	8.36		-0.56	
3118	EN16711-2	9.8407		0.53	
3146	EN16711-2	11.4		1.67	
3150	EN16711-2	6.548		-1.88	
3153	EN16711-2	9.724		0.44	
3154	EN16711-2	13.02	R(0.05)	2.85	
3166	EN16711-2	10.3		0.86	
3172	EN16711-2	9.34		0.16	
3176	EN16711-2	10.520		1.02	
3191	EN16711-2	8.312		-0.59	
3197	EN16711-2	9.03		-0.07	
3209	EN16711-2	9.732		0.45	
3210	EN16711-2	8.675		-0.33	
3228	EN16711-2	8.85		-0.20	
3237	ISO105E04	9.43		0.23	
	normality	OK			
	n	88			
	outliers	2			
	mean (n)	9.120			
	st.dev. (n)	0.8914	RSD% = 10%		
	R(calc.)	2.496			
	st.dev.(EN16711-2:15)	1.3679			
	R(EN16711-2:15)	3.830			





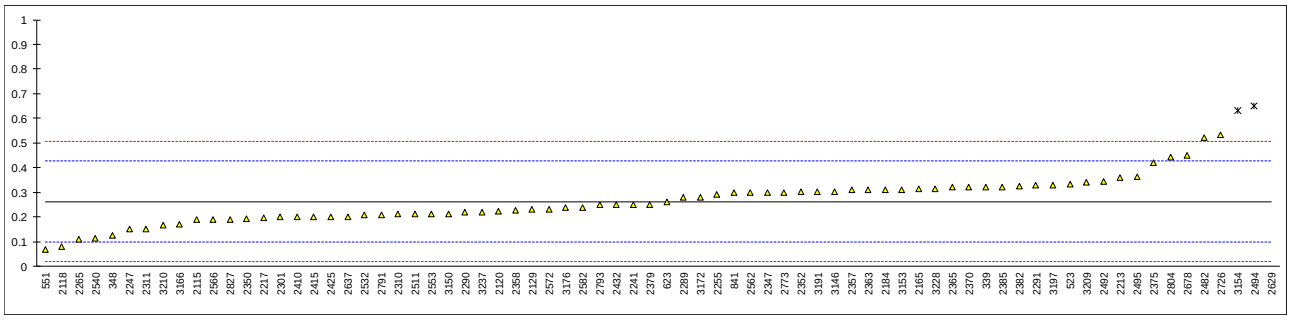
Determination of Mercury as Hg on sample #18631; results in mg/kg

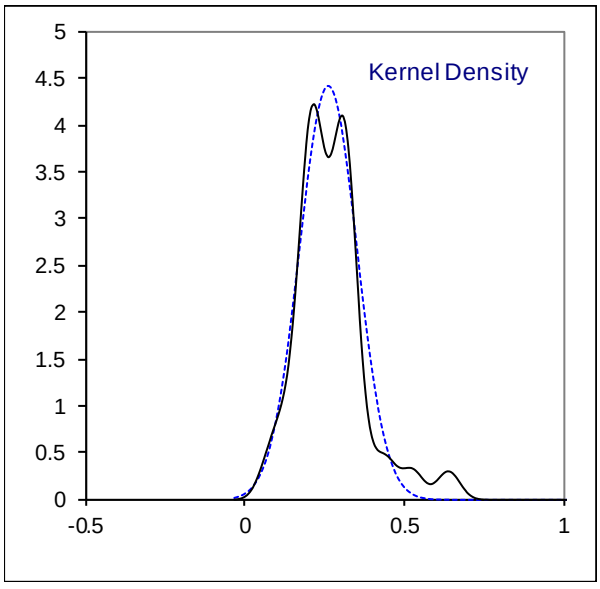
lab	method	value	mark	z(targ)	remarks
110	In house	ND		-----	
213		-----		-----	
339	ISO206-E04	0.32		0.70	
348	ISO105E04	0.124		-1.70	
362	In house	< 0.010		<-3.10	Possibly false negative test result?
523	EN16711-2	0.3341		0.87	
551	EN16711-2	0.07	C	-2.37	First reported <0.01
623	EN16711-2	0.26		-0.03	
840	EN16711-2	<0.01		<-3.10	Possibly false negative test result?
841	ISO105E04	0.299		0.44	
2115	EN16711-2	0.19		-0.89	
2118	EN16711-2	0.0801		-2.24	
2120	EN16711-2	0.223		-0.49	
2129	EN16711-2	0.230		-0.40	
2131		nd		-----	
2165	EN16711-2	0.314		0.63	
2184	DIN54233-3	0.311		0.59	
2213	EN16711-2	0.36		1.19	
2217	EN16711-2	0.1979		-0.80	
2241	EN16711-2	0.25		-0.16	
2247	EN16711-2	0.15		-1.38	
2255	EN16711-2	0.29		0.33	
2265	EN16711-2	0.109		-1.89	
2289	DIN54233-3	0.28		0.21	
2290	DIN54233-3	0.219		-0.54	
2291	OEKO TEX 100	0.33		0.82	
2301	EN16711-2	0.20		-0.77	
2310	EN16711-2	0.211		-0.64	
2311	EN16711-2	0.1527		-1.35	
2347	EN16711-2	0.30		0.46	
2350	EN16711-2	0.195		-0.83	
2352	EN16711-2	0.303		0.49	
2357	EN16711-2	0.31		0.58	
2358	EN16711-2	0.226		-0.45	
2363	EN16711-2	0.31		0.58	
2365	EN16711-2	0.320		0.70	
2370	EN16711-2	0.32		0.70	
2375	EN16711-2	0.42		1.93	
2379	ISO105E04	0.250		-0.16	
2380		-----		-----	
2382	EN16711-2	0.325		0.76	
2385	EN16711-2	0.32		0.70	
2390		N.D		-----	
2410	EN16711-2	0.20		-0.77	
2415	EN16711-2	0.200		-0.77	
2425	EN16711-2	0.2		-0.77	
2432	ISO105E04	0.249		-0.17	
2442		-----		-----	
2472		-----		-----	
2482	EN16711-2	0.5205		3.16	
2492	In house	0.3429		0.98	
2494	ISO105E04	0.65	C,R(0.01)	4.75	First reported Not detected
2495	EN16711-2	0.363		1.23	
2497		-----		-----	
2511	EN16711-2	0.213		-0.61	
2514		-----		-----	
2532	EN16711-2	0.21		-0.65	
2540	EN16711-2	0.1133		-1.84	
2553	ISO105E04	0.213		-0.61	
2562	GB/T17593	0.299		0.44	
2566	EN16711-2	0.19		-0.89	
2572	DIN54233-3	0.232		-0.38	
2582	EN16711-2	0.24	C	-0.28	First reported Not detected
2590	EN16711-2	<L.O.Q.		-----	
2602		-----		-----	
2629	EN16711-2	2.15	C,R(0.01)	23.16	First reported Not detected
2637	EN16711-2	0.2		-0.77	
2638		-----		-----	
2644		-----		-----	
2678	EN16711-2	0.45		2.30	
2726	EN16711-2	0.533		3.32	
2773	EN16711-2	0.30		0.46	
2791	EN16711-2	0.21		-0.65	
2793	EN16711-2	0.2487	C	-0.17	First reported Not detected
2804	EN16711-2	0.4442		2.23	

lab	method	value	mark	z(targ)	remarks
2827	EN16711-2	0.19		-0.89	
2830		nd		-----	
2858		---		-----	
3118	EN16711-2	ND		-----	
3146	EN16711-2	0.304		0.50	
3150	EN16711-2	0.214	C	-0.60	First reported 0.664
3153	EN16711-2	0.311		0.59	
3154	EN16711-2	0.63	R(0.01)	4.51	
3166	EN16711-2	0.172		-1.11	
3172	EN16711-2	0.28		0.21	
3176	EN16711-2	0.238		-0.30	
3191	EN16711-2	0.303		0.49	
3197	EN16711-2	0.33		0.82	
3209	EN16711-2	0.342		0.97	
3210	EN16711-2	0.168		-1.16	
3228	EN16711-2	0.315		0.64	
3237	ISO105E04	0.22		-0.53	

	normality	suspect
	n	71
	outliers	3
	mean (n)	0.2629
	st.dev. (n)	0.09023
	R(calc.)	0.2526
	st.dev.(EN16711-2:15)	0.08148
	R(EN16711-2:15)	0.2282

RSD% = 34%





APPENDIX 2

Reported test results of Arsenic (As), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Copper (Cu), Lead (Pb), Manganese (Mn), Mercury (Hg), Nickel (Ni) and Zinc (Zn) on sample #18630; results in mg/kg

lab	As	Cd	Cr	Co	Cu	Pb	Mn	Hg	Ni	Zn
110	ND	ND	ND	ND	ND	ND	ND	ND	ND	-----
213	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.05	<0.05	<0.25	<0.5	<0.5	<0.05	-----	<0.005	<0.5	-----
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	-----
362	< 0.10	< 0.05	< 0.50	< 0.50	0.80	< 0.10	-----	< 0.010	< 0.50	-----
523	< 0.05	< 0.05	< 0.05	< 0.5	< 0.5	< 0.05	-----	< 0.05	< 0.5	-----
551	<0.1	<0.05	<0.5	<0.5	<5.0	<0.1	<1.0	<0.01	<0.5	<1.0
623	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
840	<0.1	<0.05	<0.5	<0.5	<5	<0.1	<1	<0.01	<0.5	<1
841	ND	ND	ND	ND	ND	ND	-----	ND	ND	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	1.14
2118	0	0.0060	0.0080	0.0108	0	0	0.9152	0	0.0518	0.3765
2120	< 0,10	< 0,05	< 0,5	< 0,5	< 2,5	< 0,1	< 2,5	< 0,013	< 0,5	< 2,5
2129	<0,10	<0,050	<0,20	<0,20	<5,0	<0,10	-----	<0,010	<0,40	-----
2131	nd	nd	nd	nd	1.108	nd	nd	nd	nd	1.137
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2184	<0.02	<0.02	<0.5	<0.5	<0.5	<0.02	<0.5	<0.02	<0.5	-----
2213	<0.2	<0.1	<1	<1	<2	<0.2	<1	<0.01	0.16	<2
2217	0.132	0.0103	0.0145	0.0147	0.024	0.0068	-----	0.0017	0.0661	-----
2241	0.00	0.00	0.03	0.00	0.04	0.00	-----	0.00	0.01	-----
2247	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2255	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2265	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2289	<0.3	<0.03	<0.5	<1.0	<1.0	<0.3	<1.0	<0.01	<1.0	<1.0
2290	<0.1	<0.03	<0.5	<0.3	<1.0	<0.1	<5.0	<0.01	<0.3	<5.0
2291	<0.20	<0.10	<0.50	<1.00	<1.00	<0.20	<1.00	<0.02	<1.00	<3.00
2301	ND	ND	ND	ND	ND	ND	ND	ND	ND	-----
2310	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	<1.0	Not detect	Not detect	<2.0
2311	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	<2
2347	<0.1	<0.05	<0.5	<0.5	<5	<0.1	<10	<0.01	<0.5	<10
2350	<0.02	<0.02	<0.1	<0.1	<5	<0.1	-----	<0.005	<0.1	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	<0.5	<0.05	<0.5	<0.5	<5	<0.1	-----	<0.01	<0.5	-----
2363	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2365	<0.1	<0.05	<0.5	<0.5	<5	<0.1	<0.5	<0.01	<0.5	<1
2370	<0.2	<0.1	<0.5	<0.1	<5	<0.2	<1	<0.02	<0.5	<1
2375	<0.1	<0.05	<0.5	<0.5	<5	<0.1	-----	<0.01	<0.5	<0.5
2379	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	-----	Not detect	Not detect	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2385	<0,1	<0,01	<0,1	<0,1	<0,1	<0,1	<1	<0,01	<0,1	<2
2390	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	1.30
2410	<0.2	<0.1	<1.0	<1.0	<5.0	<0.2	-----	<0.02	<1.0	-----
2415	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2432	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	-----	<0.06	<0.06	<0.10	<0.6	<0.35	-----	-----	<0.05	-----
2482	-----	-----	-----	-----	-----	-----	-----	-----	-----	1.054
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2494	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect
2495	<0.02	<0.02	<0.1	<0.1	<1	<0.02	<1	<0.01	<0.1	<1
2497	-----	-----	0.181	-----	0.389	-----	-----	-----	-----	2.926
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect
2540	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	N/D	N/D	N/D	N/D	N/D	N/D	Not report	N/D	N/D	Not report
2562	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2566	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2572	<0.1	<0.03	<0.5	<0.3	<1.0	<0.1	<5.0	<0.01	<0.3	<5.0
2582	Not detect	Not detect	Not detect	Not detect	1.54	Not detect	Not detect	Not detect	0.24	0.18
2590	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.
2602	-----	0.011	-----	-----	0.125	-----	-----	-----	0.183	1.073
2629	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2637	<0,05	<0,01	<0,2	<0,01	<0,2	<0,05	<0,2	<0,01	<0,05	0.8
2638	-----	n.d	-----	n.d	n.d	n.d	n.d	-----	n.d	n.d
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2678	n.d	n.d	n.d	n.d	n.d	n.d	-----	n.d	n.d	-----
2726	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2773	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2791	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	1.24

lab	As	Cd	Cr	Co	Cu	Pb	Mn	Hg	Ni	Zn
2793	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2804	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2827	ND	ND	ND	ND	ND	ND	-----	ND	ND	-----
2830	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.63
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	---	n.d	n.d
3118	ND	ND	ND	ND	ND	ND	-----	ND	ND	-----
3146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.10
3150	<0,05	<0,1	<0,1	<0,1	<0,1	<0,1	-----	<0,02	<0,2	-----
3153	<0.3	<0.03	<0.5	<1	<1	<1	-----	<0.01	<1	-----
3154	-----	-----	0.17	-----	0.23	-----	-----	-----	-----	-----
3166	<0.01	<0.007	0.08	0.010	0.06	<0.003	0.057	<0.003	0.05	1.5
3172	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	1.488
3191	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.7181
3197	<0,1	<0,1	<0,1	<0,1	<1	<0,1	<1	<0,02	<0,1	<1
3209	<0.10	<0.05	<1.00	<1.00	<1.00	<1.00	<1.00	<0.02	<1.00	<1.00
3210	<0.2	<0.1	<1	<1	<5	<0.2	<5	<0.02	<1	<5
3228	<0.02	<0.02	<0.5	<0.5	<0.5	<0.02	<0.5	<0.02	<0.5	<2
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

Reported test results of Antimony (Sb), Arsenic (As), Cadmium (Cd), Cobalt (Co) and Copper (Cu), Lead (Pb), Manganese (Mn), Nickel (Ni) and Zinc (Zn) on sample #18631; results in mg/kg

lab	Sb	As	Cd	Co	Cu	Pb	Mn	Ni	Zn
110	ND	ND	ND	ND	ND	ND	ND	ND	-----
213	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.5	<0.05	<0.05	<0.5	<0.5	<0.05	-----	<0.5	-----
348	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----
362	< 1.00	< 0.10	< 0.050	< 0.50	0.96	< 0.10	-----	< 0.50	-----
523	< 0.05	< 0.05	< 0.05	< 0.5	< 0.5	< 0.05	-----	< 0.5	-----
551	1.24	<0.1	<0.05	<0.5	<5.0	<0.1	<1.0	<0.5	<1.0
623	ND	ND	ND	ND	ND	ND	ND	ND	ND
840	<1	<0.1	<0.05	<0.5	<5	<0.1	<1	<0.5	<1
841	ND	ND	ND	ND	ND	ND	-----	ND	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----	1.27
2118	0.1462	0.0031	0.0069	0.0057	0	0	1.0525	0.1789	0.8650
2120	< 2,5	< 0,1	< 0,05	< 0,5	< 2,5	< 0,1	< 2,5	< 0,5	< 2,5
2129	<0,20	<0,10	<0,050	<0,20	<5,0	<0,10	-----	<0,40	-----
2131	nd	nd	nd	nd	nd	nd	nd	nd	1.061
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2184	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.5	<0.5	-----
2213	<5	<0.2	<0.1	<1	<5	0.15	<2	0.28	<5
2217	0.1073	0.0392	0.0047	0.009	0.1817	0.015	-----	0.1524	-----
2241	0.19	0.00	0.00	0.00	0.23	0.00	-----	0.07	-----
2247	ND	ND	ND	ND	ND	ND	ND	ND	3.48
2255	nd	nd	nd	nd	nd	nd	nd	nd	nd
2265	-----	-----	-----	-----	-----	-----	-----	0.144	-----
2289	<1.0	<0.3	<0.03	<1.0	<1.0	<0.3	<1.0	<1.0	<1.0
2290	<1.0	<0.1	<0.03	<0.3	<1.0	<0.1	<5.0	<0.3	<5.0
2291	<3.00	<0.20	<0.10	<1.00	<1.00	<0.20	<1.00	<1.00	<3.00
2301	ND	ND	ND	ND	0.12	ND	ND	0.21	-----
2310	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	<1.0	Not detect	<2.0
2311	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	<2
2347	<1	<0.1	<0.05	<0.5	<5	<0.1	<0.5	<0.5	<1
2350	<0.5	<0.02	<0.02	<0.1	<5	<0.1	-----	0.135	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	<1.0	<0.1	<0.05	<0.5	<5	<0.1	-----	<0.5	-----
2363	ND	ND	ND	ND	ND	ND	ND	ND	ND
2365	<1	<0.1	<0.05	<0.5	<5	<0.1	<0.5	<0.5	<1
2370	<1	<0.2	<0.1	<0.1	<5	<0.2	<1	<0.5	<1
2375	<1.0	<0.1	<0.05	<0.5	<5.0	<0.1	-----	<0.5	<0.5
2379	Not detect	Not detect	Not detect	Not detect	Not detect	-----	-----	Not detect	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----	-----	-----
2385	<0,1	<0,1	<0,01	<0,1	<1	<0,1	<1	0.22	<5
2390	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	0.47
2410	<5.0	<0.2	<0.1	<1.0	<5.0	<0.2	-----	<1.0	-----
2415	-----	-----	-----	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND	ND	ND	ND
2432	-----	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	<0.35	-----	<0.06	<0.10	<0.6	<0.35	-----	<0.05	-----
2482	-----	-----	-----	-----	-----	-----	-----	0.1390	1.406
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----
2494	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2495	<0.5	<0.02	<0.02	<0.1	<1	<0.02	<1	0.150	<1
2497	0.138	-----	-----	-----	0.718	-----	-----	0.191	2.931
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect
2540	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	N/D	N/D	N/D	N/D	0.133	N/D	Not report	0.240	Not report
2562	-----	-----	-----	-----	-----	-----	-----	-----	-----
2566	ND	ND	ND	ND	ND	ND	0.34	0.25	ND
2572	<1.0	<0.1	<0.03	<0.3	<1.0	<0.1	<5.0	<0.3	<5.0
2582	Not detect	Not detect	Not detect	Not detect	1.11	Not detect	0.33	0.53	12.32
2590	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	0.757	<L.O.Q.	<L.O.Q.
2602	-----	-----	-----	-----	0.255	-----	-----	0.152	1.416
2629	nd	ND	ND	ND	ND	ND	ND	ND	nd
2637	0.3	<0,05	<0,01	0.02	<0,2	<0,05	0.4	0.15	1.8
2638	-----	-----	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2644	0.27	-----	-----	-----	0.25	-----	-----	-----	-----
2678	N.D	N.D	N.D	N.D	N.D	N.D	-----	N.D	-----
2726	-----	-----	-----	-----	0.189	-----	0.287	0.123	-----
2773	ND	ND	ND	ND	ND	ND	ND	ND	ND
2791	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	Not detect	1.61
2793	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2804	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	Sb	As	Cd	Co	Cu	Pb	Mn	Ni	Zn
2827	ND	ND	ND	ND	ND	ND	-----	ND	-----
2830	nd	nd	nd	nd	nd	nd	nd	nd	3.77
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
3118	ND	ND	ND	ND	ND	ND	-----	ND	-----
3146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.33
3150	<2	<0,05	<0,1	<0,1	<0,1	<0,1	-----	<0,2	-----
3153	<1	<0.3	<0.03	<1	<1	<0.3	-----	<1	-----
3154	0.42	-----	0.014	-----	0.53	-----	0.22	0.13	0.53
3166	<0.04	<0.01	<0.007	0.021	0.19	<0.003	0.326	0.17	1.3
3172	-----	-----	-----	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----	1.484
3191	<0.1	<0.1	<0.1	<0.1	0.1923	<0.1	0.331	0.123	1.201
3197	<1	<0,1	<0,1	<0,1	<1	<0,1	<1	0.15	<1
3209	<1.00	<0.10	<0.05	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00
3210	<5	<0.2	<0.1	<1	<5	<0.2	<5	<1	<5
3228	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.5	<0.5	<2
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3

Analytical Details

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml	remarks
110	Yes	Used as received	1 gram	1 gram / 50 ml pers. liquid	
213	No	---		---	
339	No	Further Cut	1 g	1 gram / 50 ml pers. liquid	
348	Yes	Further Cut	1	1 gram / 50 ml pers. liquid	
362	Yes	Used as received	1.0063; 1.0058	1 gram / 50 ml pers. liquid	
523	No	Further Cut	1	1 gram / 50 ml pers. liquid	
551	No	Further Cut	Yes	1 gram / 50 ml pers. liquid	
623	---	---		---	
840	Yes	Further Cut	1g	1 gram / 50 ml pers. liquid	
841	Yes	Further Cut	1	1 gram / 50 ml pers. liquid	
2115	Yes	Further Cut	0.5 g	1 gram / 50 ml pers. liquid	
2118	Yes	Used as received	1g	1 gram / 50 ml pers. liquid	
2120	Yes	---	0,5 g	1 gram / 50 ml pers. liquid	No acreditation for Hg, Zn and Mn
2129	Yes	Used as received		1 gram / 50 ml pers. liquid	
2131	Yes	Used as received	2.5	1 gram / 50 ml pers. liquid	
2165	Yes	Used as received	1g	1 gram / 50 ml pers. liquid	
2184	No	Used as received	1	1 gram / 50 ml pers. liquid	
2213	Yes	Further Cut	0.5 grams	1 gram / 50 ml pers. liquid	
2217	Yes	<1 cm2 pieces		1 gram / 50 ml pers. liquid	
2241	No	Used as received	1.0g	1 gram / 50 ml pers. liquid	
2247	Yes	Further Cut	1.0	1 gram / 50 ml pers. liquid	
2255	Yes	Further Cut	1.0 gm	1 gram / 50 ml pers. liquid	
2265	Yes	Used as received	1,0	1 gram / 50 ml pers. liquid	
2289	Yes	Further Cut	1.0	1 gram / 50 ml pers. liquid	
2290	---	---		---	
2291	Yes	Further Cut	1.00g	1 gram / 50 ml pers. liquid	/
2301	---	---		---	
2310	Yes	Further Cut	1 gram	1 gram / 50 ml pers. liquid	No accreditation for Mn and Zn
2311	Yes	Further Cut	1	1 gram / 50 ml pers. liquid	
2347	Yes	---		1 gram / 50 ml pers. liquid	
2350	No	Further Cut	1 g	1 gram / 50 ml pers. liquid	
2352	Yes	Further Cut	1g	1 gram / 50 ml pers. liquid	
2357	Yes	Further Cut		1 gram / 50 ml pers. liquid	No
2358	Yes	Further Cut	1 g	1 gram / 50 ml pers. liquid	
2363	No	Further Cut	1g	1 gram / 50 ml pers. liquid	
2365	Yes	Further Cut	0.6g	1 gram / 50 ml pers. liquid	
2370	Yes	Further Cut	1 g	1 gram / 50 ml pers. liquid	
2375	Yes	Further Cut	1 gr	1 gram / 50 ml pers. liquid	
2379	Yes	Further Cut	3 g	1 gram / 50 ml pers. liquid	
2380	Yes	Used as received	0.50 g	1 gram / 50 ml pers. liquid	
2382	No	Further Cut	1g	1 gram / 50 ml pers. liquid	
2385	Yes	Used as received	1 g	1 gram / 50 ml pers. liquid	
2390	Yes	Further Cut	1.0019, 1.0014	1 gram / 50 ml pers. liquid	
2410	Yes	Used as received	1 g	1 gram / 50 ml pers. liquid	
2415	Yes	Used as received	0.5	1 gram / 50 ml pers. liquid	
2425	Yes	Further Cut	0.5	1 gram / 50 ml pers. liquid	
2432	No	Used as received	1g	1 gram / 50 ml pers. liquid	
2442	Yes	Further Cut	2.5g	1 gram / 50 ml pers. liquid	N/A
2472	Yes	Further Cut	1gram	1 gram / 50 ml pers. liquid	
2482	Yes	Used as received	0,5	1 gram / 50 ml pers. liquid	

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml	remarks
2492	Yes	Used as received	0.5	1 gram / 20 ml pers. liquid	
2494	Yes	Used as received	0.5 gram	1 gram / 50 ml pers. liquid	
2495	Yes	Further Cut	1	1 gram / 50 ml pers. liquid	
2497	Yes	Used as received	1	1 gram / 50 ml pers. liquid	
2511	No	Further Cut	3.0 GRAM	1 gram / 50 ml pers. liquid	
2514	---	---		---	
2532	No	Further Cut	0.5grams	1 gram / 50 ml pers. liquid	
2540	Yes	Used as received	1 g	1 gram / 50 ml pers. liquid	
2553	Yes	Further Cut	1g	1 gram / 50 ml pers. liquid	N/A
2562	No	Further Cut		1 gram / 50 ml pers. liquid	
2566	Yes	Further Cut	1.0 grams	1 gram / 50 ml pers. liquid	
2572	---	---		---	
2582	Yes	Further Cut, only #18630	1.0026g, 1.0011g	1 gram / 50 ml pers. liquid	
2590	Yes	Used as received	1	1 gram / 50 ml pers. liquid	
2602	Yes	Used as received	1,000 g	1 gram / 50 ml pers. liquid	
2629	Yes	Used as received	1.0g	1 gram / 50 ml pers. liquid	Horizontally shaking at 37°C for 1 hour at 60 cycles per minutes
2637	Yes	Used as received		1 gram / 50 ml pers. liquid	
2638	No	Further Cut	1 gm	1 gram / 50 ml pers. liquid	
2644	Yes	Used as received	3 g	1 gram / 50 ml pers. liquid	
2678	No	Used as received	1g	1 gram / 50 ml pers. liquid	
2726	Yes	Used as received		1 gram / 50 ml pers. liquid	
2773	Yes	Further Cut		1 gram / 50 ml pers. liquid	
2791	Yes	Used as received	0.5020 gram	1 gram / 50 ml pers. liquid	
2793	No	Used as received	1.0032 g / 0.9998 g	1 gram / 50 ml pers. liquid	
2804	No	Used as received		1 gram / 50 ml pers. liquid	
2827	Yes	Further Cut	1g	1 gram / 50 ml pers. liquid	
2830	---	---		---	
2858	Yes	Further Cut	1.000 gm	1 gram / 20 ml pers. liquid	
3118	---	---		---	
3146	Yes	Used as received	0.50 gram	1 gram / 50 ml pers. liquid	
3150	Yes	Further Cut		1 gram / 50 ml pers. liquid	
3153	Yes	Further Cut	1.0	1 gram / 50 ml pers. liquid	
3154	Yes	Used as received		1 gram / 50 ml pers. liquid	
3166	Yes	Further Cut	0.5	1 gram / 50 ml pers. liquid	
3172	Yes	---		---	
3176	No	Used as received	0,5	1 gram / 50 ml pers. liquid	Accredited acc.to DIN 54233-3
3191	Yes	Used as received	0.5003g; 0.5001g	1 gram / 50 ml pers. liquid	
3197	Yes	Used as received	1 gram	1 gram / 50 ml pers. liquid	
3209	Yes	Used as received		1 gram / 50 ml pers. liquid	
3210	Yes	Used as received	1	1 gram / 50 ml pers. liquid	
3228	Yes	Further Cut	1.0g	1 gram / 50 ml pers. liquid	
3237	Yes	Used as received	1 gram	1 gram / 50 ml pers. liquid	

APPENDIX 4

Number of participants per country:

6 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in BULGARIA
2 labs in FRANCE
9 labs in GERMANY
5 labs in HONG KONG
1 lab in HUNGARY
9 labs in INDIA
4 labs in INDONESIA
6 labs in ITALY
3 labs in KOREA
1 lab in MEXICO
2 labs in MOROCCO
16 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SPAIN
2 labs in SRI LANKA
1 lab in SWITZERLAND
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
3 labs in TUNISIA
5 labs in TURKEY
2 labs in U.S.A.
6 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
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluations
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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