Results of Proficiency Test Heavy Metals by perspiration in textile November 2017

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

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Report: iis17A08

January 2018

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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 test scheme for perspirated metals in textile. During the annual proficiency testing program 2017/2018, it was decided to continue the proficiency test for the determination of perspirated metals in textile.
In the interlaboratory study of November 2017, 94 laboratories from 25 different countries did register for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2017 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 were artificially fortified with different metal dyes and to use a solid/liquid extraction ratio of 1/50 by preference. 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 in 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 March 2017 (iis-protocol, version 3.4). 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 prepared. The first batch (#17630) was a cotton, fortified with Cadmium. The second batch (#17631), also a cotton, was fortified with Chromium and Cobalt.
Samples #17630 and #17631 were finely cut, well mixed and divided over 100 subsamples. Sample #17630 was 2.5 g and sample #17631 was 3 g. The homogeneity of subsamples #17630 and #17631 was checked by the determination of perspirated Cadmium on sample #17630 and by the determination of perspirated Chromium and Cobalt on sample #17631 on respectively 10 and 7 stratified randomly selected subsamples.

	Perspirated Cadmium in mg/kg
Sample #17630-1	3.96
Sample #17630-2	3.86
Sample #17630-3	3.68
Sample #17630-4	3.67
Sample #17630-5	3.95
Sample #17630-6	3.83
Sample #17630-7	3.73
Sample #17630-8	3.63
Sample #17630-9	3.70
Sample #17630-10	3.83

Table 1: homogeneity test results of subsamples #17630

	Perspirated Chromium in mg/kg	*Perspirated Cobalt in mg/kg*
Sample #17631-1	2.77	8.42
Sample #17631-2	2.83	8.44
Sample #17631-3	2.92	8.92
Sample #17631-4	3.01	8.87
Sample #17631-5	2.71	8.65
Sample #17631-6	2.76	9.22
Sample #17631-7	2.82	8.54

Table 2: homogeneity test results of subsamples #17631

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 Cadmium in mg/kg	*Perspirated Chromium in mg/kg*	*Perspirated Cobalt in mg/kg*
r (observed) #17630	0.33	--	--
r (observed) #17631	--	0.28	0.82
reference test method	EN16711-2:15	EN16711-2:15	EN16711-2:15
0.3 x R (ref. test method)	0.32	0.36	0.95

Table 3: repeatabilities of subsamples #17630 and #17631

The calculated repeatabilities of each metal is in good agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #17630 and #17631 was assumed.

To each of the participating laboratories, one sample of #17630 and one sample of #17631 were sent on October 11, 2017.

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 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 can’t be used for meaningful statistical calculations.

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 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.iisn.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 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 reanalysis). 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 Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
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. 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 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 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.

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 result at all. Not all laboratories were able to report all metals requested.
Finally, the 93 reporting laboratories reported 674 numerical test results. Observed were 15 outlying results, which is 2.2% 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.

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.

4.1 EVALUATION PER SAMPLE AND PER METAL

In this section, the results are discussed per sample and per metal.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all metal determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

The participants were requested to report eleven different metals. The majority of participants did only detect Cadmium, Lead and Zinc for sample #17630 and Chromium and Cobalt for sample #17631.

The participants were also requested to report the ratio used (see appendix 2) and whether or not they are accredited to perform these tests. Of all reporting laboratories, 73% is ISO/IEC 17025 accredited.

Sample #17630:

Cadmium: The determination of this metal was problematic at a perspiration level of 4.9 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN16711-2:15.

Lead: Although this metal was not added to the textile, it is remarkable that more than sixty-six participants reported a test result above 0.2 mg/kg. The determination of this metal was not problematic at a perspiration level of 0.74 mg/kg. Four 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.

Zinc: Although this metal was not added to the textile, it is remarkable that forty participants reported a test result that was on average 5-6 mg/kg. Since the specification limits of this metal are not known, it is difficult to determine whether this level is close to or below this limit. Therefore, no z-scores were calculated.

Other metals: The majority of the participants agreed on a content close to or below the quantification limit of Antimony, Arsenic, Chromium, Cobalt, Copper, Manganese, Mercury and Nickel.

Sample #17631

Chromium: The determination of this metal was problematic for a number of laboratories at a perspiration level of 2.7 mg/kg. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility of EN16711-2:15.

Cobalt: The determination of this metal was not problematic at a perspiration level of 7.6 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good 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, Copper, Lead, Manganese, Mercury, Nickel and Zinc.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities from the reference test method EN16711-2:15 and the reproducibilities as found for the group of participating laboratories. The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities from the reference test method EN16711-2:15 are compared in the next two tables.

Parameter	*Unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Cadmium as Cd	mg/kg	90	4.88	2.50	1.37
Lead as Pb	mg/kg	66	0.74	0.83	0.83
Zinc as Zn	mg/kg	40	5.65	3.97	(1.95)

Table 4: reproducibilities of perspirated metals in sample #17630

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

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Chromium as Cr	mg/kg	85	2.72	0.97	1.14
Cobalt as Co	mg/kg	89	7.59	1.99	2.76

Table 5: reproducibilities of perspirated metals in sample #17631

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 Cadmium. See also the discussions in paragraphs 4.1 and 6.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2017 WITH THE 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. 2017*	*Oct. 2016*	*Oct. 2015*	*Oct. 2014*	*Oct. 2013*	*Oct. 2012*	*Oct. 2011*	*Oct. 2010*	*EN16711 -2:2015*
Arsenic	--	--	--	--	--	--	--	--	20%
Antimony	--	--	--	16%	--	--	19%	--	20%
Cadmium	18%	(24%)	12%	--	9%	14%	--	14%	10%
Chromium	13%	12%	--	--	15%	--	19%	--	15%
Cobalt	9%	13%	--	14%	--	11%	8%	11%	13%
Copper	--	10%	9-11%	10%	10%	--	22%	--	16%
Lead	40%	35%	--	--	--	--	--	--	40%
Manganese	--	--	--	--	--	--	--	--	--
Mercury	--	(45%)	--	--	--	41%	--	--	31%
Nickel	--	--	11%	--	11-13%	--	10-14%	7%	10%
Zinc	(25%)	--	--	--	--	--	--	--	--

Table 6: development of uncertainties over the last years

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

5 DISCUSSION

When the results of this interlaboratory study are compared to the Ecolabelling Standards and Requirements for Textiles in EU (table 7), it is noticed that some participants would make different decisions about the acceptability of the textiles for the determined parameters, to the majority of the group. 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
Arsenic (As) mg/kg	0.2	1.0	1.0	1.0
Antimony (Sb) mg/kg	30.0	30.0	30.0	--
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
Mercury (Hg) mg/kg	0.02	0.02	0.02	0.02
Nickel (Ni) mg/kg	1.0	4.0	4.0	4.0

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.

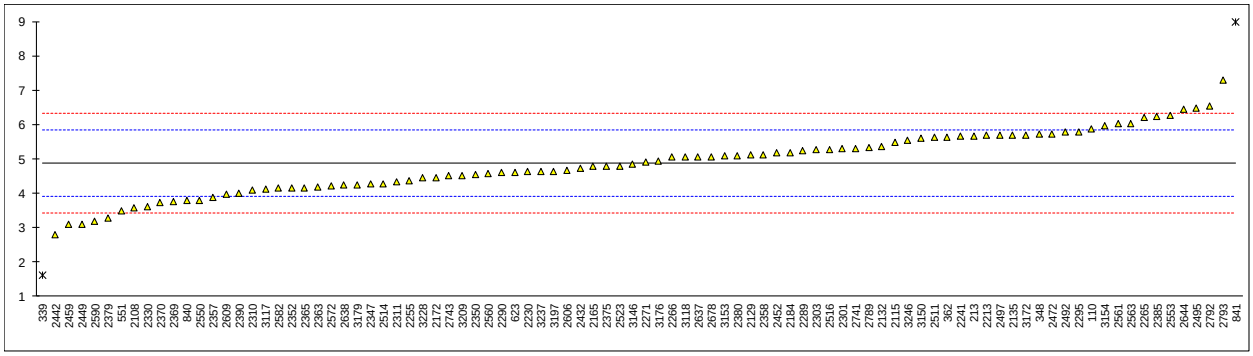
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

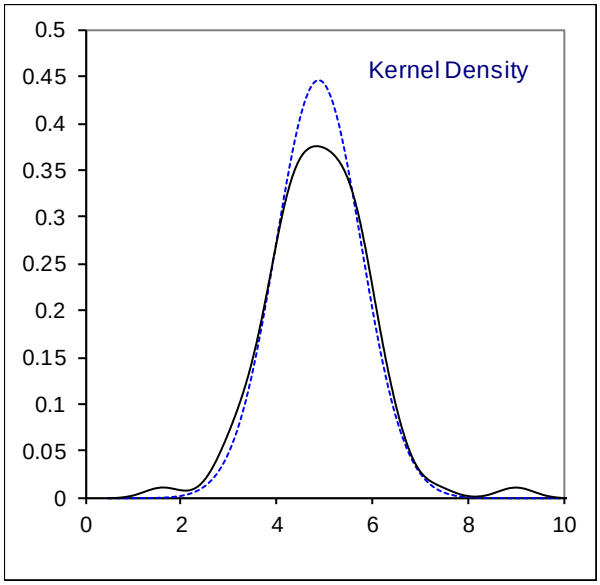
APPENDIX 1

Determination of Cadmium as Cd on sample #17630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	5.88		2.05	
213	ISO105E04	5.675		1.63	
339	ISO105E04	1.619	R(0.05)	-6.68	
348	ISO105E04	5.716		1.71	
362	In house	5.637		1.55	
551	ISO105E04	3.5021		-2.82	
623	EN16711-2	4.62		-0.53	
840	ISO105E04	3.7821		-2.25	
841	EPA3052/3051A	9.0	R(0.01)	8.44	
2108	In house	3.59		-2.64	
2115	EN16711-2	5.489		1.25	
2129	EN16711-2	5.12		0.49	
2132	EN16711-2	5.376		1.02	
2135	EN16711-2	5.694		1.67	
2165	EN16711-2	4.79		-0.18	
2172	EN16711-2	4.461		-0.86	
2184	EN16711-2	5.19		0.64	
2213	EN16711-2	5.68		1.64	
2230	EN16711-2	4.625		-0.52	
2241	ISO105E04	5.664		1.61	
2255	EN16711-2	4.38		-1.02	
2265		6.204		2.71	
2266	EN16711-2	5.046		0.34	
2271	EN16711-2	4.90		0.04	
2289	DIN54233-3	5.243		0.75	
2290	DIN54233-3	4.60		-0.57	
2295	EN16711-2	5.8		1.89	
2301	ISO105E04	5.300		0.86	
2303	EN16711-2	5.260		0.78	
2310	EN16711-2	4.08		-1.64	
2311	EN16711-2	4.34		-1.11	
2330	ISO105E04	3.6083		-2.61	
2347	EN16711-2	4.28		-1.23	
2350	EN16711-2	4.555		-0.66	
2352	EN16711-2	4.158		-1.48	
2357		3.892		-2.02	
2358	EN16711-2	5.13		0.51	
2363	EN16711-2	4.18		-1.43	
2365	EN16711-2	4.160		-1.47	
2369	ISO105E04	3.769		-2.28	
2370	EN16711-2	3.74		-2.34	
2375	EN16711-2	4.8		-0.16	
2379	ISO105E04	3.290		-3.26	
2380	ISO105E04	5.0788		0.41	
2385	EN16711-2	6.25		2.81	
2390	EN16711-2	4.00		-1.80	
2432	ISO105E04	4.732		-0.30	
2442	In house	2.8		-4.26	
2449	DIN54233-3	3.11	C	-3.63	first reported: 3708
2452	DIN54233-3	5.170		0.60	
2459	EN16711-2	3.084		-3.68	
2472	GB/T17593	5.72		1.72	
2492	In house	5.794		1.87	
2495	EN16711-2	6.482		3.28	
2497	EN16711-2	5.687		1.66	
2511	EN16711-2	5.626		1.53	
2514	EN16711-2	4.28		-1.23	
2516	EN16711-2	5.26		0.78	
2523	ISO105E04/EPA200.7	4.800		-0.16	
2550	EN16711-2	3.785		-2.24	
2553	EN16711-2	6.26	C	2.83	first reported: 125.69
2560		4.5853		-0.60	
2561	EN16711-2	6.03		2.36	
2563	EN16711-2	6.04		2.38	
2572	DIN54233-3	4.21		-1.37	
2582	EN16711-2	4.14		-1.52	
2590	EN16711-2	3.201		-3.44	
2606	In house	4.67		-0.43	
2609	EN16711-2	3.980		-1.84	
2637	EN16711-2	5.06		0.37	
2638	EN16711-2	4.235		-1.32	
2644	EN16711-2	6.46		3.24	
2678	DIN54233-3	5.075		0.40	
2741	EN16711-2	5.3		0.86	
2743	EN16711-2	4.53067		-0.71	

lab	method	value	mark	z(targ)	remarks
2789	EN16711-2	5.330		0.92	
2792	EN16711-2	6.532		3.39	
2793	EN16711-2	7.294340656		4.95	
3117	GB/T17593	4.11		-1.58	
3118	In house	5.047		0.34	
3146	EN16711-2	4.85		-0.06	
3150	EN16711-2	5.6		1.48	
3153	EN16711-2	5.076		0.40	
3154	DIN54233-3	5.97		2.24	
3166		-----		-----	
3172	DIN54233-3	5.7		1.68	
3176	DIN54233-3	4.95		0.14	
3179	EN16711-2	4.253		-1.28	
3197	EN16711-2	4.64		-0.49	
3209	EN16711-2	4.531		-0.71	
3210	EN16711-2	<0.1		<-9.80	possible false negative test result?
3228	EN16711-2	4.45		-0.88	
3237		4.63		-0.51	
3246	In house	5.540		1.35	
	normality	OK			
	n	90			
	outliers	2			
	mean (n)	4.879			
	st.dev. (n)	0.8926	RSD = 18%		
	R(calc.)	2.499			
	st.dev.(EN16711-2:15)	0.4879			
	R(EN16711-2:15)	1.366			

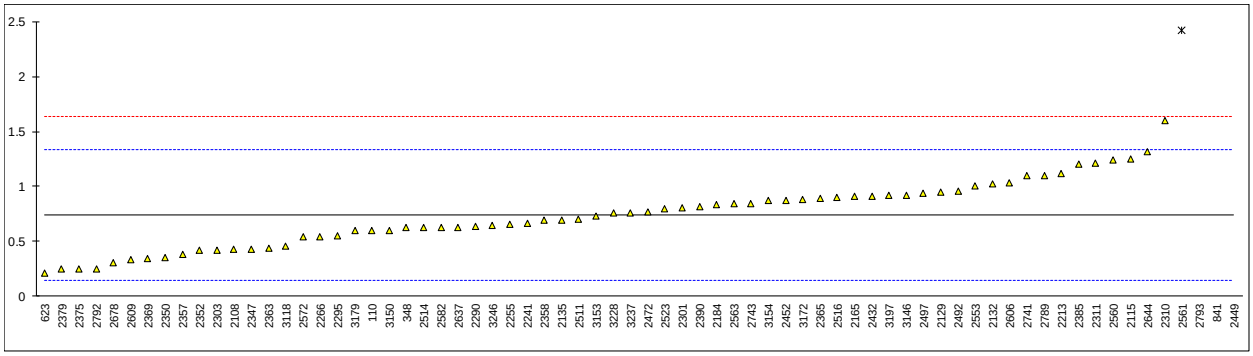


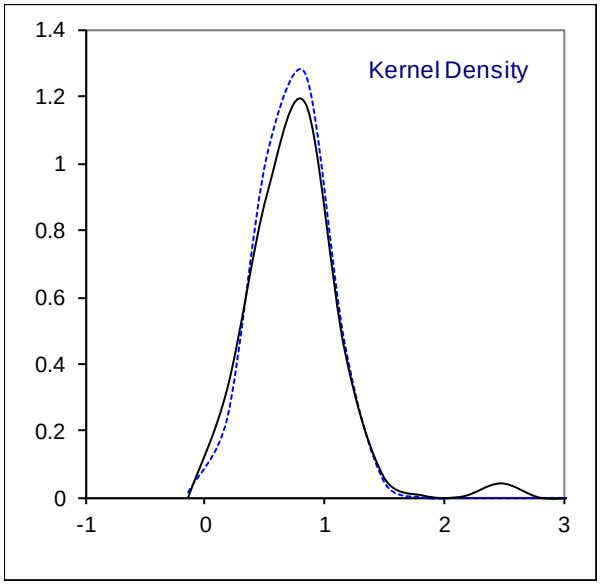


Determination of Lead as Pb on sample #17630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	0.600		-0.48	
213		-----		-----	
339	ISO105E04	<0.2		-----	
348	ISO105E04	0.63		-0.38	
362		-----		-----	
551	ISO105E04	ND		-----	
623	EN16711-2	0.21		-1.79	
840	ISO105E04	<0.2		-----	
841	EPA3052/3051A	83.0	R(0.01)	276.91	
2108	In house	0.426		-1.07	
2115	EN16711-2	1.254		1.72	
2129	EN16711-2	0.95		0.70	
2132	EN16711-2	1.027		0.96	
2135	EN16711-2	0.6912		-0.17	
2165	EN16711-2	0.909		0.56	
2172		-----		-----	
2184	EN16711-2	0.832		0.30	
2213	EN16711-2	1.12		1.27	
2230	EN16711-2	<0.1		-----	
2241	ISO105E04	0.662		-0.27	
2255	EN16711-2	0.66		-0.28	
2265		< 0,1		-----	
2266	EN16711-2	0.542		-0.68	
2271	EN16711-2	N.D.		-----	
2289		-----		-----	
2290	DIN54233-3	0.64		-0.35	
2295	EN16711-2	0.55		-0.65	
2301	ISO105E04	0.810		0.23	
2303	EN16711-2	0.4218		-1.08	
2310	EN16711-2	1.60		2.89	
2311	EN16711-2	1.21		1.57	
2330	ISO105E04	ND		-----	
2347	EN16711-2	0.43		-1.05	
2350	EN16711-2	0.353		-1.31	
2352	EN16711-2	0.415		-1.10	
2357		0.384		-1.21	
2358	EN16711-2	0.69		-0.18	
2363	EN16711-2	0.438		-1.03	
2365	EN16711-2	0.892		0.50	
2369	ISO105E04	0.346		-1.34	
2370	EN16711-2	<0.2		-----	
2375	EN16711-2	0.25		-1.66	
2379	ISO105E04	0.247		-1.67	
2380		-----		-----	
2385	EN16711-2	1.20		1.54	
2390	EN16711-2	0.82		0.26	
2432	ISO105E04	0.913		0.57	
2442		-----		-----	
2449	DIN54233-3	421	R(0.01)	1414.74	
2452	DIN54233-3	0.877		0.45	
2459	EN16711-2	<0.2		-----	
2472	GB/T17593	0.77		0.09	
2492	In house	0.953		0.71	
2495	EN16711-2	<5		-----	
2497	EN16711-2	0.939		0.66	
2511	EN16711-2	0.702		-0.14	
2514	EN16711-2	0.63		-0.38	
2516	EN16711-2	0.90		0.53	
2523	ISO105E04/EPA200.7	0.7982		0.19	
2550		-----		-----	
2553	EN16711-2	1.00	C	0.87	first reported: 20.05
2560		1.2411		1.68	
2561	EN16711-2	2.42	R(0.01)	5.65	
2563	EN16711-2	0.84		0.33	
2572	DIN54233-3	0.54		-0.68	
2582	EN16711-2	0.63		-0.38	
2590	EN16711-2	<L.O.Q.		-----	
2606	In house	1.03		0.97	
2609	EN16711-2	0.335		-1.37	
2637	EN16711-2	0.63		-0.38	
2638	EN16711-2	n.d		-----	
2644	EN16711-2	1.32		1.94	
2678	DIN54233-3	0.311		-1.45	
2741	EN16711-2	1.1		1.20	
2743	EN16711-2	0.84400		0.34	

lab	method	value	mark	z(targ)	remarks
2789	EN16711-2	1.100		1.20	
2792	EN16711-2	0.251		-1.66	
2793	EN16711-2	45.43263448	R(0.01)	150.44	
3117		-----		-----	
3118	In house	0.460		-0.95	
3146	EN16711-2	0.921		0.60	
3150	EN16711-2	0.6		-0.48	
3153	EN16711-2	0.732		-0.04	
3154	DIN54233-3	0.87		0.43	
3166		-----		-----	
3172	DIN54233-3	0.88		0.46	
3176		-----		-----	
3179	EN16711-2	0.598		-0.49	
3197	EN16711-2	0.92		0.60	
3209	EN16711-2	<1.00		-----	
3210	EN16711-2	<0.1		-----	
3228	EN16711-2	0.76		0.06	
3237		0.761		0.06	
3246	In house	0.648		-0.32	
	normality	OK			
	n	66			
	outliers	4			
	mean (n)	0.743			
	st.dev. (n)	0.2956	RSD = 40%		
	R(calc.)	0.828			
	st.dev.(EN16711-2:15)	0.2971			
	R(EN16711-2:15)	0.832			



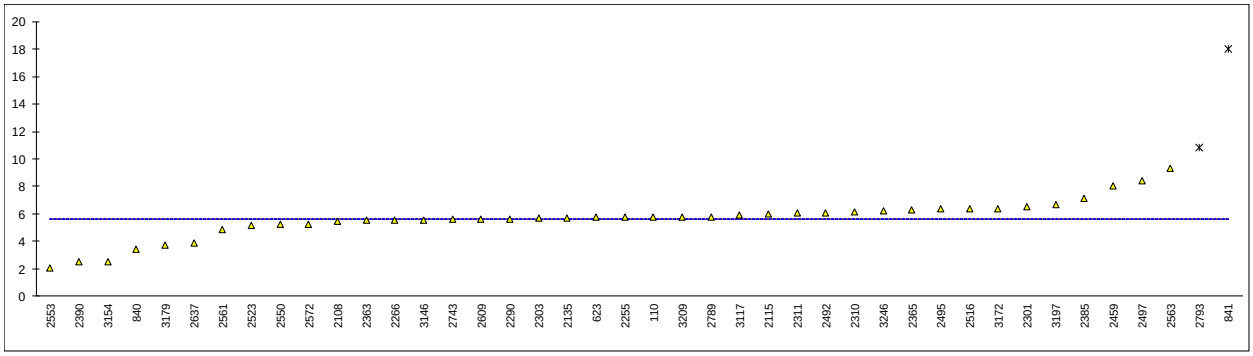


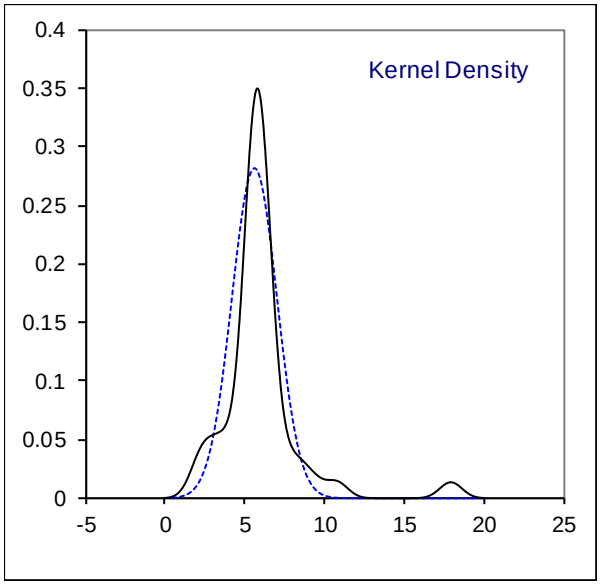
Determination of Zinc as Zn on sample #17630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	5.80		-----	
213		-----		-----	
339		-----		-----	
348		-----		-----	
362		-----		-----	
551	ISO105E04	Not App.		-----	
623	EN16711-2	5.77		-----	
840	ISO105E04	3.4667		-----	
841	EPA3052/3051A	18.0	R(0.01)	-----	
2108	In house	5.45		-----	
2115	EN16711-2	6.013	C	-----	first reported 0.013
2129		-----		-----	
2132		-----		-----	
2135	EN16711-2	5.694		-----	
2165		-----		-----	
2172		-----		-----	
2184		-----		-----	
2213	EN16711-2	<10		-----	
2230	EN16711-2	<6		-----	
2241		-----		-----	
2255	EN16711-2	5.77		-----	
2265		< 5,0		-----	
2266	EN16711-2	5.525		-----	
2271		-----		-----	
2289		-----		-----	
2290	DIN54233-3	5.62		-----	
2295		-----		-----	
2301	ISO105E04	6.520		-----	
2303	EN16711-2	5.6631		-----	
2310	EN16711-2	6.18		-----	
2311	EN16711-2	6.05		-----	
2330		-----		-----	
2347	EN16711-2	<1		-----	
2350		-----		-----	
2352		-----		-----	
2357		-----		-----	
2358		-----		-----	
2363	EN16711-2	5.51		-----	
2365	EN16711-2	6.300		-----	
2369	ISO105E04	<1		-----	
2370	EN16711-2	--		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2385	EN16711-2	7.10		-----	
2390	EN16711-2	2.55		-----	
2432		-----		-----	
2442		-----		-----	
2449		-----		-----	
2452		-----		-----	
2459	EN16711-2	8.018		-----	
2472		-----		-----	
2492	In house	6.050		-----	
2495	EN16711-2	6.350		-----	
2497	EN16711-2	8.389		-----	
2511		-----		-----	
2514		-----		-----	
2516	EN16711-2	6.38		-----	
2523	ISO105E04/EPA200.7	5.1410		-----	
2550	EN16711-2	5.254		-----	
2553	EN16711-2	2.07	C	-----	first reported: 41.55
2560		<10		-----	
2561	EN16711-2	4.87		-----	
2563	EN16711-2	9.3	C	-----	first reported: 12.1
2572	DIN54233-3	5.26		-----	
2582	EN16711-2	NA		-----	
2590	EN16711-2	<L.O.Q.		-----	
2606		-----		-----	
2609	EN16711-2	5.617		-----	
2637	EN16711-2	3.85		-----	
2638	EN16711-2	N/A		-----	
2644		-----		-----	
2678		-----		-----	
2741	EN16711-2	<6		-----	
2743	EN16711-2	5.59546		-----	

lab	method	value	mark	z(targ)	remarks
2789	EN16711-2	5.805		-----	
2792		-----		-----	
2793	EN16711-2	10.81997629	R(0.05)	-----	
3117	GB/T17593	5.93		-----	
3118		-----		-----	
3146	EN16711-2	5.57		-----	
3150		-----		-----	
3153		-----		-----	
3154	DIN54233-3	2.56		-----	
3166		-----		-----	
3172	DIN54233-3	6.4		-----	
3176		-----		-----	
3179	EN16711-2	3.733		-----	
3197	EN16711-2	6.69		-----	
3209	EN16711-2	5.803		-----	
3210	EN16711-2	<5		-----	
3228		-----		-----	
3237		-----		-----	
3246	In house	6.229		-----	

normality	suspect	
n	40	
outliers	2	
mean (n)	5.646	
st.dev. (n)	1.4192	RSD = 25%
R(calc.)	3.974	
st.dev.(Horwitz)	(0.6962)	
R(Horwitz)	(1.949)	



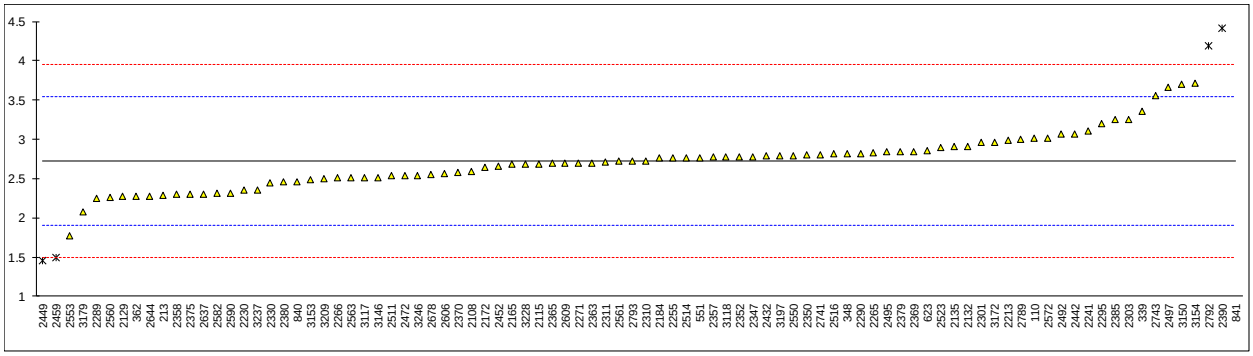


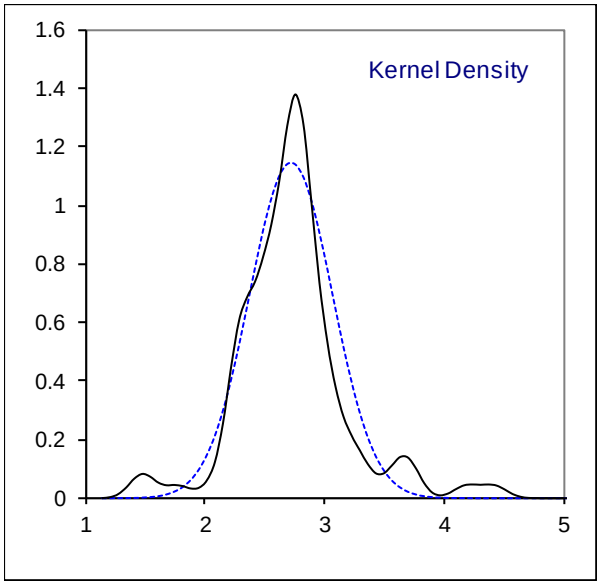
Determination of Chromium as Cr on sample #17631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	3.01		0.71	
213	ISO105E04	2.2896		-1.06	
339	ISO105E04	3.353		1.55	
348	ISO105E04	2.82		0.24	
362	In house	2.280		-1.08	
551	ISO105E04	2.7661		0.11	
623	EN16711-2	2.85		0.31	
840	ISO105E04	2.4557		-0.65	
841	EPA3052/3051A	18.0	R(0.01)	37.42	
2108	In house	2.59		-0.32	
2115	EN16711-2	2.683		-0.10	
2129		2.28		-1.08	
2132	EN16711-2	2.908		0.46	
2135	EN16711-2	2.9065		0.45	
2165		2.68		-0.10	
2172	EN16711-2	2.649		-0.18	
2184	EN16711-2	2.76		0.09	
2213	EN16711-2	2.99		0.66	
2230	EN16711-2	2.35		-0.91	
2241	ISO105E04	3.102		0.93	
2255	EN16711-2	2.76		0.09	
2265		2.825		0.25	
2266		2.510		-0.52	
2271	EN16711-2	2.70		-0.05	
2289	DIN54233-3	2.246		-1.17	
2290	DIN54233-3	2.82		0.24	
2295	EN16711-2	3.2		1.17	
2301	ISO105E04	2.960		0.58	
2303	EN16711-2	3.2561		1.31	
2310	EN16711-2	2.73		0.02	
2311	EN16711-2	2.71		-0.03	
2330	ISO105E04	2.4480		-0.67	
2347	EN16711-2	2.78		0.14	
2350	EN16711-2	2.797		0.18	
2352	EN16711-2	2.780		0.14	
2357		2.777		0.13	
2358	EN16711-2	2.30		-1.03	
2363		2.7		-0.05	
2365	EN16711-2	2.692		-0.07	
2369	ISO105E04	2.847		0.31	
2370	EN16711-2	2.58		-0.35	
2375	EN16711-2	2.3		-1.03	
2379	ISO105E04	2.844		0.30	
2380	ISO105E04	2.4551		-0.65	
2385	EN16711-2	3.25		1.29	
2390	EN16711-2	4.42	R(0.01)	4.16	
2432	ISO105E04	2.790		0.17	
2442	In house	3.07		0.85	
2449	DIN54233-3	1.46	C,R(0.01)	-3.09	first reported: 2466
2452	DIN54233-3	2.6545		-0.17	
2459	EN16711-2	1.500	R(0.01)	-2.99	
2472	GB/T17593	2.54		-0.45	
2492	In house	3.068		0.85	
2495	EN16711-2	2.837		0.28	
2497	EN16711-2	3.662		2.30	
2511	EN16711-2	2.535		-0.46	
2514	EN16711-2	2.76		0.09	
2516	EN16711-2	2.81		0.22	
2523	ISO105E04/EPA200.7	2.894		0.42	
2550	EN16711-2	2.794		0.18	
2553	EN16711-2	1.77	C	-2.33	first reported: 35.51
2560	EN16711-2	2.2607		-1.13	
2561	EN16711-2	2.72		0.00	
2563		2.51		-0.52	
2572	DIN54233-3	3.01		0.71	
2582	EN16711-2	2.31		-1.01	
2590	EN16711-2	2.3110		-1.01	
2606	In house	2.56		-0.40	
2609	EN16711-2	2.692		-0.07	
2637	EN16711-2	2.3		-1.03	
2638	EN16711-2	N/A		-----	
2644	EN16711-2	2.28		-1.08	
2678	DIN54233-3	2.547		-0.43	
2741	EN16711-2	2.8		0.19	
2743		3.55476		2.04	

lab	method	value	mark	z(targ)	remarks
2789	EN16711-2	3.005		0.69	
2792	EN16711-2	4.187	R(0.01)	3.59	
2793	EN16711-2	2.729927814		0.02	
3117	GB/T17593	2.51		-0.52	
3118	In house	2.779		0.14	
3146	EN16711-2	2.51		-0.52	
3150	EN16711-2	3.7		2.40	
3153	EN16711-2	2.481		-0.59	
3154	DIN54233-3	3.71		2.42	
3166		-----		-----	
3172	DIN54233-3	2.96		0.58	
3176		-----		-----	
3179	EN16711-2	2.07		-1.60	
3197	EN16711-2	2.79		0.17	
3209	EN16711-2	2.502		-0.54	
3210	EN16711-2	<0.5		<-5.44	possible false negative test result?
3228	EN16711-2	2.68		-0.10	
3237		2.36		-0.89	
3246	In house	2.543		-0.44	

normality	suspect	
n	85	
outliers	5	
mean (n)	2.722	
st.dev. (n)	0.3478	RSD = 13%
R(calc.)	0.974	
st.dev.(EN16711-2:15)	0.4083	
R(EN16711-2:15)	1.143	



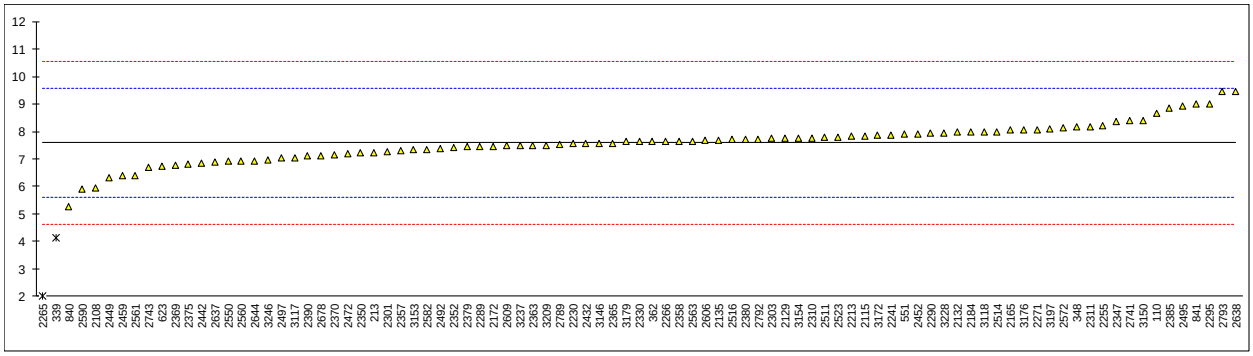


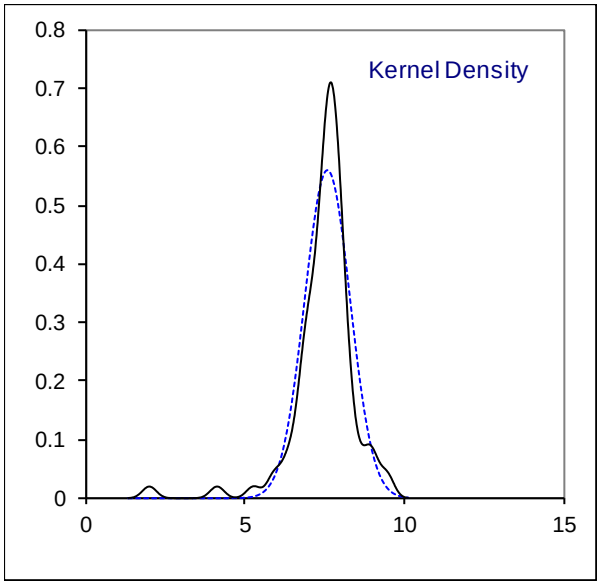
Determination of Cobalt as Co on sample #17631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	8.65		1.08	
213	ISO105E04	7.229		-0.36	
339	ISO105E04	4.140	R(0.01)	-3.50	
348	ISO105E04	8.16		0.58	
362	In house	7.645		0.06	
551	ISO105E04	7.89		0.31	
623	EN16711-2	6.72		-0.88	
840	ISO105E04	5.2789		-2.34	
841	EPA3052/3051A	9.0		1.43	
2108	In house	5.94		-1.67	
2115	EN16711-2	7.848		0.26	
2129		7.76		0.17	
2132	EN16711-2	7.966		0.38	
2135	EN16711-2	7.6805		0.09	
2165		8.05		0.47	
2172	EN16711-2	7.464		-0.13	
2184	EN16711-2	7.97		0.39	
2213	EN16711-2	7.82		0.23	
2230	EN16711-2	7.56		-0.03	
2241	ISO105E04	7.868		0.28	
2255	EN16711-2	8.22		0.64	
2265		2.021	R(0.01)	-5.64	
2266		7.649		0.06	
2271	EN16711-2	8.06		0.48	
2289	DIN54233-3	7.445		-0.15	
2290	DIN54233-3	7.94		0.36	
2295	EN16711-2	9		1.43	
2301	ISO105E04	7.280		-0.31	
2303	EN16711-2	7.7406		0.15	
2310	EN16711-2	7.77		0.18	
2311	EN16711-2	8.17		0.59	
2330	ISO105E04	7.6439		0.06	
2347	EN16711-2	8.37		0.79	
2350	EN16711-2	7.228		-0.37	
2352	EN16711-2	7.431		-0.16	
2357		7.308		-0.28	
2358	EN16711-2	7.65		0.06	
2363		7.5		-0.09	
2365	EN16711-2	7.581		-0.01	
2369	ISO105E04	6.755		-0.85	
2370	EN16711-2	7.15		-0.44	
2375	EN16711-2	6.8		-0.80	
2379	ISO105E04	7.435		-0.16	
2380	ISO105E04	7.7137		0.13	
2385	EN16711-2	8.85		1.28	
2390	EN16711-2	7.10		-0.50	
2432	ISO105E04	7.576		-0.01	
2442	In house	6.86		-0.74	
2449	DIN54233-3	6.32	C	-1.29	first reported: 7321
2452	DIN54233-3	7.924		0.34	
2459	EN16711-2	6.391		-1.21	
2472	GB/T17593	7.17		-0.42	
2492	In house	7.364		-0.23	
2495	EN16711-2	8.938		1.37	
2497	EN16711-2	7.029		-0.57	
2511	EN16711-2	7.792		0.21	
2514	EN16711-2	7.98		0.40	
2516	EN16711-2	7.71		0.12	
2523	ISO105E04/EPA200.7	7.811		0.23	
2550	EN16711-2	6.906		-0.69	
2553	EN16711-2	ND		-----	
2560	EN16711-2	6.9124		-0.69	
2561	EN16711-2	6.41		-1.19	
2563		7.65		0.06	
2572	DIN54233-3	8.13		0.55	
2582	EN16711-2	7.35		-0.24	
2590	EN16711-2	5.9102		-1.70	
2606	In house	7.68		0.09	
2609	EN16711-2	7.481		-0.11	
2637	EN16711-2	6.9		-0.70	
2638	EN16711-2	9.455		1.89	
2644	EN16711-2	6.93		-0.67	
2678	DIN54233-3	7.108		-0.49	
2741	EN16711-2	8.4		0.82	
2743		6.68815		-0.91	

lab	method	value	mark	z(targ)	remarks
2789	EN16711-2	7.530		-0.06	
2792	EN16711-2	7.719		0.13	
2793	EN16711-2	9.445899926		1.88	
3117	GB/T17593	7.05		-0.55	
3118	In house	7.978		0.39	
3146	EN16711-2	7.58		-0.01	
3150	EN16711-2	8.4		0.82	
3153	EN16711-2	7.328		-0.26	
3154	DIN54233-3	7.76		0.17	
3166		-----		-----	
3172	DIN54233-3	7.85		0.26	
3176	DIN54233-3	8.05		0.47	
3179	EN16711-2	7.643		0.05	
3197	EN16711-2	8.10		0.52	
3209	EN16711-2	7.503		-0.09	
3210	EN16711-2	<0.5		<-7.19	possible false negative test result?
3228	EN16711-2	7.96		0.38	
3237		7.49		-0.10	
3246	In house	6.949		-0.65	

	normality	suspect	
	n	89	
	outliers	2	
	mean (n)	7.589	
	st.dev. (n)	0.7103	RSD = 9%
	R(calc.)	1.989	
	st.dev.(EN16711-2:15)	0.9865	
	R(EN16711-2:15)	2.762	





APPENDIX 2

Reported test results of Antimony (Sb), Arsenic (As), Chromium (Cr), Cobalt (Co), Copper (Cu), Manganese (Mn), Mercury (Hg) and Nickel (Ni) on sample #17630; results in mg/kg

lab	Sb	As	Cr	Co	Cu	Mn	Hg	Ni
110	0.112	<0.01	0.121	<1	<1	0.285	<0.01	0.044
213	-----	-----	-----	-----	-----	-----	-----	-----
339	<30	<0.2	<0.5	<1	<25	-----	<0.02	<1
348	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.
362	-----	-----	-----	-----	0.654	-----	-----	-----
551	ND	ND	ND	ND	ND	Not Aplicable	ND	ND
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	<0.5	<0.02	<0.1	<0.1	<5	<1	<0.01	<0.5
841	n.d	n.d	3.0	n.d	2.0	n.d	n.d	n.d
2108	<4	<0,1	<0,1	<0,1	<4	<4	<0,01	<0,1
2115	-----	-----	-----	-----	-----	0.605	-----	-----
2129	<0,2	<0,1	<0,20	<0,20	<5,0	-----	<0,010	<0,40
2132	<2	<0.05	<0.1	<0.3	<2	-----	<0.1	<0.3
2135	<0,2	<0,2	<0,5	<1	<2	<1	<0,02	<0,2
2165	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.
2172	<0.5	<0.02	<0.1	<0.1	<5	-----	<0.005	<0.1
2184	< 0.5	< 0.02	< 0.5	< 0.5	< 0.5	-----	< 0.02	< 0.5
2213	<5	<0.2	<0.5	<1	<5	<10	<0.02	<0.5
2230	<0.5	<0.02	<0.1	<0.1	<5	<0.25	<0.005	<0.1
2241	0.005	0.001	0.019	0.003	0.364	-----	0.002	0.089
2255	ND	ND	ND	ND	ND	ND	ND	ND
2265	< 2,0	< 0,1	< 0,3	< 0,3	< 5,0	< 5,0	< 0,02	< 0,2
2266	0	0	0	0	0.401	0.205	0.050	0.109
2271	N.D.	N.D.	N.D.	N.D.	N.D.	-----	N.D.	N.D.
2289	-----	-----	-----	-----	-----	-----	-----	-----
2290	<1	<0.1	<0.5	<0.3	<1	<1	<0.01	<0.3
2295	<10	<0.1	<0.5	<0.5	<10	-----	<0.02	<0.5
2301	<0.5	0.090	<0.1	<0.1	<5.0	0.280	<0.005	<0.1
2303	0.0188	0.0059	0.0267	0.0038	0.7868	0.2012	0.0126	0.8458
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ND	ND	ND	ND	ND	-----	ND	ND
2347	<1	<0.1	<1	<0.5	<5	<0.5	<0.01	<0.5
2350	<0.5	<0.02	<0.1	<0.1	<5	-----	<0.005	<0.1
2352	nd	nd	nd	nd	nd	-----	nd	nd
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	<1.0	<0.1	<0.5	<0.5	<5.0	-----	<0.01	<0.5
2363	nd	nd	nd	nd	nd	nd	nd	nd
2365	<1	<0.1	<1	<0.5	<5	<0.5	<0.01	<0.5
2369	<1	<0.2	<1	<1	<5	<1	<0.02	<0.5
2370	<1	<0.2	<0.5	<0.1	<5	--	<0.02	<0.5
2375	-----	-----	-----	-----	-----	-----	-----	-----
2379	<0.5	<0.05	<5.00	<5.00	<5.00	-----	<0.02	<5.0
2380	-----	-----	-----	-----	-----	-----	-----	-----
2385	<1	<0,1	<0,5	<0,5	<0,5	<0,5	<0,01	<0,5
2390	ND	ND	ND	ND	ND	ND	ND	ND
2432	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	1.52	-----	-----	0.43
2452	0.000	0.0275	0.094	0.002	0.1645	-----	0	0
2459	<1.0	<0.1	<1.0	<1.0	1.467	<1.0	<0.02	0.467
2472	<0.35	-----	<0.06	<0.10	<0.6	-----	-----	<0.05
2492	-----	-----	-----	-----	-----	-----	-----	-----
2495	<1	<1	<1	<1	<1	<1	<1	<1
2497	0.016	0.031	0.112	0.003	0.413	0.151	0.0001	0.109
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	ND	ND	ND	ND	ND	-----	ND	ND
2516	-----	-----	-----	-----	-----	-----	-----	-----
2523	N.D.	N.D.	N.D.	N.D.	0.2650	0.2103	N.D.	0.0536
2550	-----	-----	-----	-----	-----	-----	-----	-----
2553	ND	ND	ND	ND	0.30 C	1.03 C	0.04 C	0.10 C
2560	<10	<0.1	<0.5	<0.5	<10	<10	<0.02	<0.5
2561	<2	<0.1	0.15	<0.1	0.42	0.37	<0.1	<0.1
2563	-----	-----	<0,1	n.d.	<0,5	0.2	-----	<0,1
2572	<1	<0.1	<0.5	<0.3	<1	<1	<0.01	<0.3
2582	ND	ND	ND	ND	0.95	NA	ND	0.10
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.
2606	-----	-----	-----	-----	-----	-----	-----	-----
2609	N.d./<0.1	N.d./<0.1	N.d./<0.1	N.d./<0.1	0.382	0.143	N.d./<0.1	0.138
2637	<0,05	<0,05	<0,2	<0,1	<0,2	0.18	<0,002	<0,2
2638	N/A	N/A	N/A	n.d	n.d	N/A	N/A	n.d

lab	Sb	As	Cr	Co	Cu	Mn	Hg	Ni
2644	-----	-----	-----	-----	-----	-----	-----	-----
2678	nd	nd	nd	nd	nd	-----	nd	nd
2741	<0.5	<0.03	<0.1	<0.1	<0.5	<0.25	<0.01	<0.1
2743	0.00876	0.00819	0.086696	0.08556	0.99441	0.29336	0.05644	0.13831
2789	0.126	0.086	0.408	0.068	0.630	0.346	0.024	0.174
2792	0.001	0.073	0.358	0.009	0.688	-----	0.000	0.104
2793	0.0	0	0.013677696	0	0.183385714	0.269329042	0	0.066512249
3117	-----	-----	-----	-----	-----	-----	-----	-----
3118	<0.25	<0.05	<0.05	<0.25	<0.5	-----	<0.02	<0.25
3146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3150	<0,1	<0,05	<0,1	<0,1	<0,2	-----	<0,01	<0,2
3153	<1	<0.3	<0.5	<1	<1	-----	<0.01	<1
3154	6.68	-----	0.33	-----	-----	0.20	-----	-----
3166	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 5	< 0.05	< 0.1	< 0.1	< 5	-----	< 0.01	< 0.2
3176	-----	-----	-----	-----	-----	-----	-----	-----
3179	0.001	0.024	0.012	0.004	0.236	0.331	<0,01	0.049
3197	<1	<0,1	<0,1	<0,1	<1	<1	<0,02	<0,1
3209	<1.00	<0.10	<1.00	<1.00	<1.00	<1.00	<0.02	<1.00
3210	<5	<0.1	2.6	7.8	<5	<5	<0.02	<0.5
3228	<0.5	<0.02	<0.5	<0.5	<0.5	-----	<0.02	<0.5
3237	-----	-----	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	0.246	n.d.	0.091

Lab 2553 first reported for Cu: 5.95, for Mn: 20.61, for Hg: 0.80 and for Ni: 1.68

Reported test results of Antimony (Sb), Arsenic (As), Cadmium (Cd), Copper (Cu), Lead (Pb), Manganese (Mn), Mercury (Hg), Nickel (Ni) and Zinc (Zn) on sample #17631; results in mg/kg

lab	Sb	As	Cd	Cu	Pb	Mn	Hg	Ni	Zn
110	0.122	<0.01	<0.01	<1	0.0478	0.318	0.0174	0.0384	<1
213	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<30	<0.2	<0.1	<25	<0.2	-----	<0.02	<1	-----
348	n.d.	n.d.	n.d.	n.d.	n.d.	-----	0.015	n.d.	-----
362	-----	0.174	-----	0.266	-----	-----	-----	-----	-----
551	ND	ND	ND	ND	ND	Not Appl.	ND	ND	Not Appl.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	<0.5	<0.02	<0.02	<5	<0.2	<1	<0.01	<0.5	<1
841	n.d	n.d	n.d	2.0	n.d	n.d	n.d	n.d	2.0
2108	<4	<0,1	<0,05	<4	<0,1	<4	0.0125	<0,1	<4
2115	-----	-----	-----	-----	0.104	0.623	-----	-----	-----
2129	<0,20	<0,10	<0,050	<5,0	<0,10	-----	0.025	<0,4	-----
2132	<2	<0.05	<0.02	<2	<0.1	-----	0.025	<0.3	-----
2135	<0,2	<0,2	<0,1	<2	<0,2	<1	<0,02	<0,2	<1
2165	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	-----
2172	<0.5	<0.02	<0.02	<5	<0.1	-----	-----	<0.1	-----
2184	< 0.5	< 0.02	< 0.02	< 0.5	0.110	-----	0.038	< 0.5	-----
2213	<5	<0.2	<0.1	<5	<0.2	<10	0.02	<0.5	<10
2230	<0.5	<0.02	<0.02	<5	<0.1	<0.25	<0.005	<0.1	<6
2241	0.000	0.003	0.006	0.197	0.057	-----	<0.02	0.056	-----
2255	ND	ND	ND	ND	ND	ND	ND	ND	ND
2265	< 2,0	< 0,1	< 0,03	< 5,0	< 0,1	< 5,0	0.033	< 0,2	< 5,0
2266	0.020	0	0	0.441	0	0.272	0	0.225	0.773
2271	N.D.	N.D.	N.D.	N.D.	N.D.	-----	N.D.	N.D.	-----
2289	-----	-----	-----	-----	-----	-----	-----	-----	-----
2290	<1	<0.1	<0.03	<1	<0.1	<1	0.0173	<0.3	<1
2295	<10	<0.1	<0.1	<10	<0.1	-----	<0.02	<0.5	-----
2301	<0.5	0.060	<0.02	<5.0	<0.1	0.300	<0.005	<0.1	<6.0
2303	0.0083	0.0030	0.0206	0.6331	0.0873	0.2521	0.0199	0.1592	0.8756
2310	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	1.21
2311	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	Not Detect.	<1
2330	ND	ND	ND	ND	ND	-----	ND	ND	-----
2347	<1	<0.1	<0.05	<5	<0.1	<0.5	<0.01	<0.5	<1
2350	<0.5	<0.02	<0.02	<5	<0.1	-----	<0.005	<0.1	-----
2352	nd	nd	nd	nd	nd	-----	nd	nd	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	<1.0	<0.1	<0.1	<5.0	<0.1	-----	<0.01	<0.5	-----
2363	nd	nd	nd	nd	nd	nd	0.037	nd	nd
2365	<1	<0.1	<0.05	<5	<0.1	<0.5	0.033	<0.5	<1
2369	<1	<0.2	<0.1	<5	<0.2	<1	<0.02	<0.5	<1
2370	<1	<0.2	<0.1	<5	<0.2	--	0.04	<0.5	--
2375	-----	-----	-----	-----	-----	-----	0.02	-----	-----
2379	<0.5	<0.05	<0.05	<0.5	0.086	-----	<0.02	<0.5	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----	-----
2385	<1	<0,1	<0,05	<0,5	0.135	<0,5	<0,01	<0,5	0.945

lab	Sb	As	Cd	Cu	Pb	Mn	Hg	Ni	Zn
2390	ND	ND	ND	ND	ND	ND	ND	ND	ND
2432	-----	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	1.48	-----	-----	-----	-----	-----
2452	0	0.06	0.0045	0.0665	0.0805	-----	0	0	-----
2459	<1.0	<0.1	<0.2	1.425	<0.2	<1.0	<0.02	0.88 C	4.47 C
2472	<0.35	-----	<0.06	<0.60	<0.35	-----	-----	<0.05	-----
2492	-----	-----	-----	-----	0.107	-----	0.0300	-----	-----
2495	<1	<1	<1	<1	<5	<1	<1	<1	<1
2497	0.018	0.021	0.006	0.708	0.087	0.242	0.021	0.099	1.90
2511	-----	-----	-----	-----	-----	-----	0.012	-----	-----
2514	ND	ND	ND	ND	ND	-----	ND	ND	-----
2516	-----	-----	-----	-----	-----	-----	-----	-----	1.61
2523	N.D.	N.D.	N.D.	0.2159	0.0865	0.2623	0.0590	0.0485	0.4038
2550	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	ND	ND	ND	0.34 C	0.22 C	2.00 C	0.18 C	0.15 C	0.44 C
2560	<10	<0.1	<0.1	<10	<0.1	<10	0.0247	<0.5	<10
2561	<2	<0.1	<0.1	0.32	0.34	0.39	<0.1	<0.1	0.38
2563	-----	-----	n.d.	<0,5	<0,1	0.26	-----	<0,1	4.1 C
2572	<1	<0.1	<0.03	<1	<0.1	<1	0.0146	<0.3	<1
2582	ND	ND	ND	1.89	ND	NA	ND	0.16	NA
2590	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	0.7012	<L.O.Q.	<L.O.Q.	<L.O.Q.
2606	-----	-----	-----	-----	-----	-----	-----	-----	-----
2609	N.d./<0.1	N.d./<0.1	N.d./<0.1	0.423	N.d./<0.1	0.194	N.d./<0.1	0.118	1.150
2637	<0,05	<0,05	<0,01	<0,2	0.05	0.24	0.01	<0,2	0.37
2638	N/A	N/A	n.d	n.d	n.d	N/A	N/A	n.d	N/A
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----
2678	nd	nd	nd	nd	nd	-----	0.02	nd	-----
2741	<0.5	<0.03	<0.01	<0.5	0.1	<0.25	0.02	<0.1	<6
2743	0.00686	0.00565	0.00762	1.24928	0.08369	0.28508	0.04621	0.13627	2.34126
2789	0.037	0.024	0.131	0.547	0.310	0.334	0.039	0.115	0.970
2792	0.001	0.056	0.000	0.702	0.056	-----	0.000	0.103	-----
2793	0	0	0	0.132498608	1.414982041	0.267866908	0.031732646	0.012588915	0
3117	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	<0.25	<0.05	<0.05	<0.5	<0.05	-----	<0.02	<0.25	-----
3146	n.d.	n.d.	n.d.	n.d.	0.102	n.d.	0.0215	n.d.	0.526
3150	<0,1	<0,05	<0,02	<0,2	<0,1	-----	0.03	<0,2	-----
3153	<1	<0.3	<0.03	<1	<0.3	-----	0.026	<1	-----
3154	2.90	-----	0.77	-----	-----	0.18	-----	-----	1.38
3166	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 5	< 0.05	< 0.02	< 5	< 0.1	-----	0.033	< 0.2	< 5
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----
3179	0.001	0.019	0.002	0.247	0.07	0.24	0.01817	0.057	0.504
3197	<1	<0,1	<0,1	<1	<0,1	<1	<0,02	<0,1	<1
3209	<1.00	<0.10	<0.50	<1.00	<1.00	<1.00	<0.02	<1.00	<1.00
3210	<5	<0.1	8.3	<5	2.2	<5	0.03	<0.5	7.7
3228	<0.5	<0.02	<0.02	<0.5	<0.1	-----	<0.02	<0.5	-----
3237	-----	-----	-----	-----	0.10	-----	0.020	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	0.060	0.271	0.016	0.112	n.d.

Lab 2459 first reported for Ni: 1.3 andfor Zn: 6.113
Lab 2553 first reported for Cu: 6.84, for Lead: 4.39, for Manganese: ND, for Mercury: 3.45, for Nickel: 2.89 and for Zn: 8.91
Lab 2563 first reported for Zn: 7.6

APPENDIX 3

Accreditation status and extraction ratio as reported by participants

labnrs	Is your laboratory accredited?	What ratio was used in gram textile per ml?	Remarks on Additional Questions:
110	Yes	1 gram textile per 50 ml perspiration liquid	
213	No	1 gram textile per 50 ml perspiration liquid	
339	No	1 gram textile per 50 ml perspiration liquid	
348	Yes	1 gram textile per 50 ml perspiration liquid	
362	Yes	1 gram textile per 50 ml perspiration liquid	
551	No	1 gram textile per 50 ml perspiration liquid	
623	Yes	1 gram textile per 50 ml perspiration liquid	
840	Yes	1 gram textile per 50 ml perspiration liquid	
841	---	---	
2108	Yes	1 gram textile per 20 ml perspiration liquid	
2115	Yes	1 gram textile per 50 ml perspiration liquid	
2129	Yes	1 gram textile per 50 ml perspiration liquid	
2132	Yes	1 gram textile per 50 ml perspiration liquid	
2135	Yes	1 gram textile per 50 ml perspiration liquid	
2165	Yes	1 gram textile per 50 ml perspiration liquid	
2172	Yes	1 gram textile per 50 ml perspiration liquid	
2184	No	1 gram textile per 50 ml perspiration liquid	
2213	Yes	1 gram textile per 50 ml perspiration liquid	
2230	Yes	1 gram textile per 50 ml perspiration liquid	
2241	Yes	1 gram textile per 50 ml perspiration liquid	
2255	Yes	1 gram textile per 50 ml perspiration liquid	
2265	No	1 gram per 40 ml	
2266	Yes	1 gram textile per 50 ml perspiration liquid	
2271	Yes	1 gram textile per 50 ml perspiration liquid	
2289	Yes	1 gram textile per 50 ml perspiration liquid	
2290	---	---	
2295	Yes	1 gram textile per 50 ml perspiration liquid	
2301	Yes	1 gram textile per 50 ml perspiration liquid	
2303	No	1 gram textile per 50 ml perspiration liquid	
2310	No	1 gram textile per 50 ml perspiration liquid	
2311	Yes	1 gram textile per 50 ml perspiration liquid	
2330	Yes	1 gram textile per 50 ml perspiration liquid	
2347	Yes	1 gram textile per 50 ml perspiration liquid	
2350	Yes	1 gram textile per 50 ml perspiration liquid	
2352	Yes	1 gram textile per 50 ml perspiration liquid	
2357	---	1 gram textile per 50 ml perspiration liquid	
2358	Yes	1 gram textile per 50 ml perspiration liquid	
2363	No	1 gram textile per 50 ml perspiration liquid	
2365	Yes	1 gram textile per 50 ml perspiration liquid	
2369	---	---	
2370	Yes	1 gram textile per 50 ml perspiration liquid	
2375	No	1 gram textile per 50 ml perspiration liquid	
2379	No	1 gram textile per 50 ml perspiration liquid	
2380	Yes	1 gram textile per 50 ml perspiration liquid	
2385	Yes	1 gram textile per 50 ml perspiration liquid	
2390	Yes	1 gram textile per 50 ml perspiration liquid	
2432	No	1 gram textile per 50 ml perspiration liquid	
2442	Yes	1 gram textile per 50 ml perspiration liquid	
2449	Yes	1 gram textile per 50 ml perspiration liquid	
2452	No	1 gram textile per 50 ml perspiration liquid	
2459	Yes	1 gram textile per 50 ml perspiration liquid	

labnrs	Is your laboratory accredited?	What ratio was used in gram textile per ml?	Remarks on Additional Questions:
2472	No	1 gram textile per 50 ml perspiration liquid	
2492	Yes	1 gram textile per 20 ml perspiration liquid	
2495	Yes	1 gram textile per 50 ml perspiration liquid	
2497	Yes	1 gram textile per 50 ml perspiration liquid	
2511	Yes	1 gram textile per 50 ml perspiration liquid	
2514	Yes	1.12 gram textile per 50 ml perspiration liquid	
2516	Yes	1 gram textile per 50 ml perspiration liquid	
2523	Yes	2 gram textile per 60 ml perspiration liquid	
2550	Yes	1 gram textile per 50 ml perspiration liquid	
2553	Yes	1 gram textile per 50 ml perspiration liquid	
2560	Yes	1 gram textile per 50 ml perspiration liquid	
2561	No	1 gram textile per 50 ml perspiration liquid	
2563	No	1 gram textile per 50 ml perspiration liquid	
2572	---	---	
2582	Yes	1 gram textile per 50 ml perspiration liquid	
2590	Yes	1 gram textile per 50 ml perspiration liquid	
2606	Yes	1 gram textile per 50 ml perspiration liquid	
2609	No	1 gram textile per 50 ml perspiration liquid	
2637	---	---	
2638	No	1 gram textile per 50 ml perspiration liquid	
2644	No	1 gram textile per 50 ml perspiration liquid	
2678	No	1 gram textile per 50 ml perspiration liquid	
2741	Yes	1 gram textile per 50 ml perspiration liquid	
2743	Yes	1 gram textile per 50 ml perspiration liquid	
2789	No	1 gram textile per 50 ml perspiration liquid	
2792	No	1 gram textile per 50 ml perspiration liquid	
2793	No	1 gram textile per 50 ml perspiration liquid	
3117	Yes	1 gram textile per 50 ml perspiration liquid	
3118	No	1 gram textile per 50 ml perspiration liquid	
3146	Yes	1 gram textile per 50 ml perspiration liquid	low sample amount
3150	Yes	1 gram textile per 50 ml perspiration liquid	
3153	Yes	1 gram textile per 50 ml perspiration liquid	
3154	Yes	1 gram textile per 50 ml perspiration liquid	
3172	Yes	1 gram textile per 50 ml perspiration liquid	
3176	Yes	1 gram textile per 50 ml perspiration liquid	
3179	Yes	1 gram textile per 50 ml perspiration liquid	
3197	Yes	1 gram textile per 50 ml perspiration liquid	
3209	Yes	1 gram textile per 50 ml perspiration liquid	
3210	Yes	1 gram textile per 50 ml perspiration liquid	
3228	---	---	
3237	---	---	
3246	Yes	1 gram textile per 50 ml perspiration liquid	
3246	---		

APPENDIX 4

Number of participants per country:

5 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA, Kingdom of
3 labs in FRANCE
11 labs in GERMANY
5 labs in HONG KONG
3 labs in INDIA
4 labs in INDONESIA
7 labs in ITALY
1 lab in JAPAN
1 lab in KOREA
1 lab in MOROCCO
18 labs in P.R. of CHINA
4 labs in PAKISTAN
2 labs in SPAIN
2 labs in SRI LANKA
3 labs in TAIWAN R.O.C.
1 lab in THAILAND
4 labs in TUNISIA
5 labs in TURKEY
2 labs in U.S.A.
1 lab in UNITED ARAB EMIRATES
2 labs in UNITED KINGDOM
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 calculations
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

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