Results of Proficiency Test Heavy Metals by Perspiration in textile November 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Corrector: ing. A.S. Noordman-de Neef

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CONTENTS

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 2019/2020, it was decided to continue the proficiency test for the analysis of perspirated metals in textile.
In this interlaboratory study 98 laboratories from 27 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each and respectively labelled #19630 and #19631. Each textile was artificially fortified with different metal dyes. The participants were 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/IEC17043:2010 (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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two different batches of textile were obtained from a third-party laboratory. The first batch was an orange colored cotton positive on Chromium and Nickel. The second batch was a pink colored cotton which was positive on Antimony and Arsenic.
Both batches were cut finely, well mixed and divided over 120 subsamples of 3 grams each and respectively labelled #19630 and #19631. The homogeneity was checked by the determination of perspirated Chromium and Nickel on sample #19630 and by the determination of perspirated Antimony and Arsenic on sample #19631 on 8 stratified randomly selected subsamples of each set.

	Sample #19630		Sample #19631	
	Perspirated Chromium in mg/kg	Perspirated Nickel in mg/kg	Perspirated Antimony in mg/kg	Perspirated Arsenic in mg/kg
Sample 1	3.9	15.6	28.7	8.6
Sample 2	3.7	15.5	28.6	8.5
Sample 3	3.4	15.5	28.5	7.7
Sample 4	3.9	15.5	30.3	8.7
Sample 5	3.5	15.5	29.7	8.3
Sample 6	3.4	15.6	30.6	8.5
Sample 7	3.4	15.2	30.3	8.8
Sample 8	3.6	15.5	30.6	8.8

Table 1: homogeneity test results of subsamples #19630 and #19631

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 ISO13528, Annex B2 in the next table.

	Sample #19630		Sample #19631	
	Perspirated Chromium in mg/kg	Perspirated Nickel in mg/kg	Perspirated Chromium in mg/kg	Perspirated Nickel in mg/kg
r (observed)	0.60	0.35	2.58	0.97
reference test method	EN16711-2:15	EN16711-2:15	EN16711-2:15	EN16711-2:15
0.3 x R (ref. test method)	0.45	1.30	4.98	1.43

Table 2: evaluation of the repeatabilities of subsamples #19630 and #19631

The calculated repeatabilities of each metal, except for Chromium, were in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19631 was assumed.
Sample #19630 was also used in a previous proficiency test iis13A03 as sample #13189 and had proven to be a good PT sample. Therefore, homogeneity of the subsamples #19630 was also assumed.

To each of the participating laboratories, one subsample of #19630 and one subsample of #19631 were sent on October 9, 2019.

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 appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, 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 two participants did not report any test results at all and one other laboratory reported the test results after the final reporting date. Not all laboratories were able to report all elements requested.
Finally, the 96 reporting laboratories reported 408 numerical test results. Observed were 13 outlying results, which is 3.2% 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, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER ELEMENT

In this section, the results are discussed per sample and per element.
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 element between laboratories (see table A.1 of DIN54233-3). 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 #19630

Chromium: The determination of Chromium was not problematic. 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.

Nickel: The determination of Nickel was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN16711-2:15.

Other Elements: The majority of the participants agreed on a content close to or below the quantification limits of Antimony, Arsenic, Cadmium, Cobalt, Copper, Lead, Manganese, Mercury and Zinc. Therefore, no z-scores were calculated. The test results are given in appendix 2.

Sample #19631

Antimony: The determination of Antimony was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN16711-2:15.

Arsenic: The determination of Arsenic was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN16711-2:15.

Other elements: The majority of the participants agreed on a content close to or below the quantification limits of Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel and Zinc. Therefore, no z-scores were calculated. The test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R (lit.)
Chromium as Cr	mg/kg	92	7.08	2.27	2.97
Nickel as Ni	mg/kg	91	25.9	7.8	7.3

Table 3: reproducibilities of perspirated metals in sample #19630

Component	unit	n	average	2.8 * sd	R (lit.)
Antimony as Sb	mg/kg	92	24.6	8.5	13.8
Arsenic as As	mg/kg	93	8.3	2.0	4.7

Table 4: reproducibilities of perspirated metals in sample #19631

From the tables above it can be concluded that, without statistical calculations, the group of participating laboratories do not have difficulties with the analyzes compared to the target reproducibility. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2019 WITH PREVIOUS PTS

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs, see below table.

Component	November 2019	November 2018	November 2017	October 2016	2010-2015	EN16711-2
Antimony as Sb	12%	8%	--	--	16-19%	20%
Arsenic as As	9%	--	--	--	--	20%
Cadmium as Cd	--	--	18%	(24%)	9-14%	10%
Chromium as Cr	11%	10%	13%	12%	15-19%	15%
Cobalt as Co	--	--	9%	13%	8-14%	13%
Copper as Cu	--	--	--	10%	9-22%	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 5: development of uncertainties over the last years

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

The observed relative standard deviations are in line with previous PTs and the target values.

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 74% 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 #19630 nor in sample #19631.

It should be noticed that differences in sample intake, perspiration time and/or 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 and a sample intake of at least 0.5 gram as per test method EN16711-2:15. Most of the participants reported to have used the 1:50 ration and a sample intake of 0.5-1.0 grams.

5 DISCUSSION

In this PT, the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant. Secondly, the homogeneity testing is done by one laboratory only. The test results of this ISO/IEC17025 accredited laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias. Also, each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the homogeneity test.

Sample #19630 was also used in a previous proficiency test iis13A03 as sample #13189. The obtained PT results are in line with the previous PT (see table 6). Therefore, it is concluded that the samples textile containing Chromium and Nickel are stable for 6 years at least.

Component	unit	Sample #19630			Sample #13189		
		n	average	2.8 * sd	n	average	2.8 * sd
Chromium as Cr	mg/kg	92	7.1	2.3	70	5.2	2.2
Nickel as Ni	mg/kg	91	25.9	7.8	68	25.5	7.6

Table 6: comparison sample #19630 vs #13189

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”.

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 6).
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 in mg/kg	Class 2: direct skin contact in mg/kg	Class 3: no direct skin contact in mg/kg	Class 4: decoration material in mg/kg
Antimony as Sb	30.0	30.0	30.0	--
Arsenic as As	0.2	1.0	1.0	1.0
Cadmium as Cd	0.1	0.1	0.1	0.1
Chromium as Cr	1.0	2.0	2.0	2.0
Cobalt as Co	1.0	4.0	4.0	4.0
Copper as Cu	25.0	50.0	50.0	50.0
Lead as Pb	0.2	1.0	1.0	1.0
Manganese as Mn	--	--	--	--
Mercury as Hg	0.02	0.02	0.02	0.02
Nickel as Ni	1.0	4.0	4.0	4.0
Zinc as Zn	--	--	--	--

Table 7: Ecolabelling Standards and Requirements for Textiles in EU

6 CONCLUSION

In this proficiency test the Heavy Metal content by perspiration on Textile 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

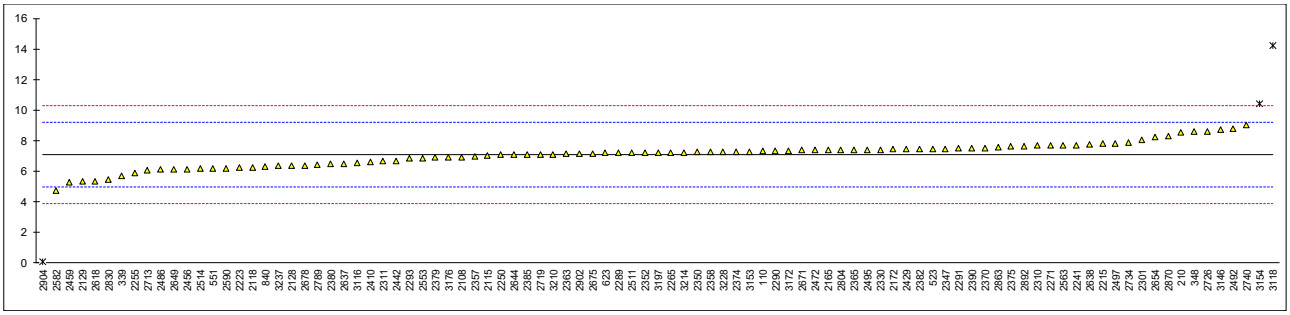
Determination of Chromium as Cr on sample #19630; results in mg/kg

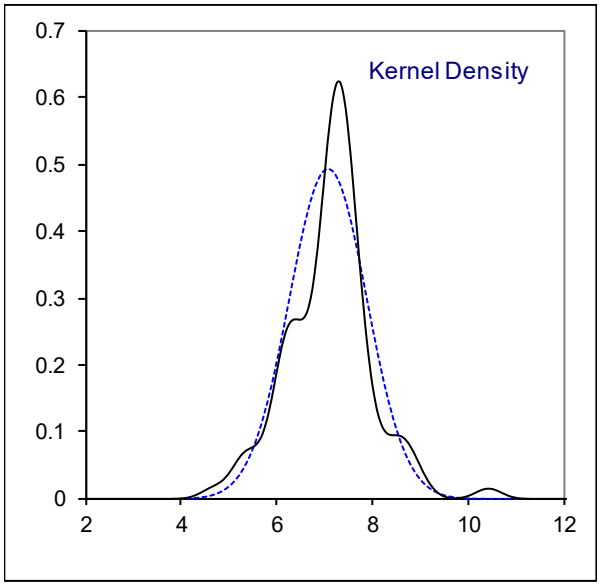
lab	method	value	mark	z(targ)	remarks
110	In house	7.33783		0.24	
210	EN16711-2	8.508		1.34	
339	ISO105E04Mod.	5.708		-1.29	
348	ISO105E04	8.583		1.42	
523	EN16711-2	7.443		0.34	
551	EN16711-2	6.1787		-0.85	
623	EN16711-2	7.18		0.09	
840	EN16711-2	6.30		-0.73	
2108	In house	6.92		-0.15	
2115	EN16711-2	7.0		-0.08	
2118	EN16711-2	6.2579		-0.77	
2128	ISO105E04	6.3782		-0.66	
2129		5.33		-1.65	
2165	EN16711-2	7.376		0.28	
2172	EN16711-2	7.417		0.32	
2190		-----		-----	
2215	EN16711-2	7.7847		0.66	
2223	EN16711-2	6.22		-0.81	
2241	EN16711-2	7.714		0.60	
2250	EN16711-2	7.057		-0.02	
2255	DIN54233-3	5.9		-1.11	
2265		7.213		0.13	
2271	EN16711-2	7.7		0.58	
2289	DIN54233-3	7.18		0.09	
2290	EN16711-2	7.349		0.25	
2291	EN16711-2	7.50		0.40	
2293	DIN54233-3	6.86		-0.21	
2301	EN16711-2	8.03		0.89	
2310	EN16711-2	7.7		0.58	
2311	EN16711-2	6.68		-0.38	
2330	ISO105E04	7.408		0.31	
2347	ISO105E04	7.47		0.37	
2350	EN16711-2	7.247		0.16	
2352	EN16711-2	7.2		0.11	
2357	EN16711-2	6.96		-0.11	
2358	EN16711-2	7.25		0.16	
2363	EN16711-2	7.15		0.07	
2365	EN16711-2	7.391		0.29	
2370	ISO105E04	7.51		0.41	
2374	EN16711-2	7.271		0.18	
2375	EN16711-2	7.6		0.49	
2379	EN16711-2	6.9091		-0.16	
2380	EN16711-2	6.474		-0.57	
2382	EN16711-2	7.44		0.34	
2385	EN16711-2	7.1		0.02	
2390	ISO105E04	7.501		0.40	
2410		6.6		-0.45	
2429	DIN54233-3	7.426		0.33	
2442	EN16711-2	6.68		-0.38	
2456	EN16711-2	6.107	C	-0.92	First reported 12.214
2459	EN16711-2	5.25		-1.72	
2472	GB/T17593	7.36		0.26	
2486	EN16711-2	6.097		-0.93	
2492	In house	8.78	C	1.60	First reported 9.960
2495	EN16711-2	7.4		0.30	
2497	EN16711-2	7.821		0.70	
2511	EN16711-2	7.19		0.10	
2514	EN16711-2	6.17		-0.86	
2553	EN16711-2	6.87		-0.20	
2563	EN16711-2	7.7		0.58	
2582	EN16711-2	4.70	C	-2.24	First reported 2.48
2590	EN16711-2	6.18	C	-0.85	First reported 4.66
2618	ISO105E04	5.34		-1.64	
2637	EN16711-2	6.49		-0.56	
2638	EN16711-2	7.7475		0.63	
2644	EN16711-2	7.06		-0.02	
2649	ISO105E04	6.10		-0.92	
2654	EN16711-2	8.208		1.06	
2671	EN16711-2	7.357		0.26	
2675	EN16711-2	7.158		0.07	
2678	EN16711-2	6.3812		-0.66	
2713	EN16711-2	6.056		-0.96	
2719	ISO105E04	7.1	C	0.02	First reported as sample #19631
2726	EN16711-2	8.601		1.43	

lab	method	value	mark	z(targ)	remarks
2734	EN16711-2	7.90		0.77	
2740	EN16711-2	9.00		1.81	
2789	EN16711-2	6.4		-0.64	
2804	EN16711-2	7.38		0.28	
2812		-----		-----	
2830	EN16711-2	5.45		-1.53	
2863	EN16711-2	7.5730		0.46	
2870	EN16711-2	8.3		1.15	
2877		-----		-----	
2892	EN16711-2	7.626		0.51	
2902	EN16711-2	7.15		0.07	
2904		0.096	C,R(0.01)	-6.58	First reported 0.573
3116	EN16711-2	6.519		-0.53	
3118	EN16711-2	14.2412	C,R(0.01)	6.74	First reported 18.3912
3146	EN16711-2	8.73		1.55	
3153	EN16711-2	7.275		0.18	
3154	EN16711-2	10.43	C,R(0.01)	3.15	First reported 11.304
3172	EN16711-2	7.35		0.25	
3176	EN16711-2	6.916		-0.15	
3197	EN16711-2	7.2		0.11	
3210	EN16711-2	7.10		0.02	
3214	EN16711-2	7.216		0.13	
3228	EN16711-2	7.27		0.18	
3237	EN16711-2	6.37		-0.67	

	normality	OK
	n	92
	outliers	3
	mean (n)	7.0798
	st.dev. (n)	0.80913
	R(calc.)	2.2656
	st.dev.(EN16711-2:15)	1.06197
	R(EN16711-2:15)	2.9735

RSD = 11%



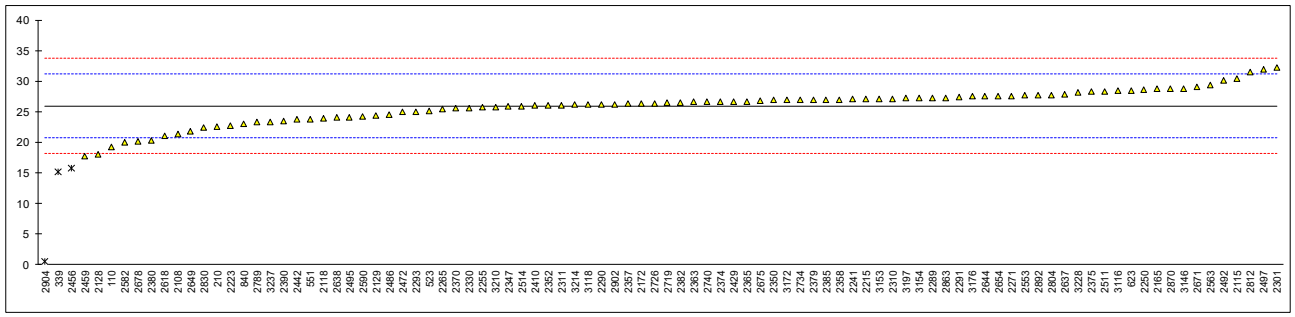


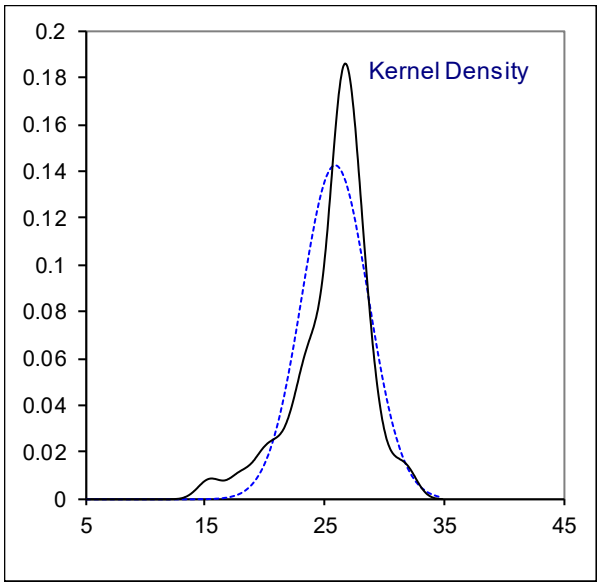
Determination of Nickel as Ni on sample #19630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	19.24830		-2.58	
210	EN16711-2	22.538		-1.31	
339	ISO105E04Mod.	15.077	R(0.05)	-4.19	
348		-----		-----	
523	EN16711-2	25.055		-0.34	
551	EN16711-2	23.8024		-0.82	
623	EN16711-2	28.49		0.98	
840	EN16711-2	22.95		-1.15	
2108	In house	21.3		-1.79	
2115	EN16711-2	30.4		1.72	
2118	EN16711-2	23.8346		-0.81	
2128	ISO105E04	17.9574		-3.08	
2129		24.35		-0.61	
2165	EN16711-2	28.70		1.07	
2172	EN16711-2	26.31		0.14	
2190		-----		-----	
2215	EN16711-2	27.077		0.44	
2223	EN16711-2	22.76		-1.22	
2241	EN16711-2	27.025		0.42	
2250	EN16711-2	28.597		1.03	
2255	DIN54233-3	25.7		-0.09	
2265		25.467		-0.18	
2271	EN16711-2	27.6		0.64	
2289	DIN54233-3	27.27		0.51	
2290	EN16711-2	26.226		0.11	
2291	EN16711-2	27.34		0.54	
2293	DIN54233-3	24.98		-0.37	
2301	EN16711-2	32.15		2.40	
2310	EN16711-2	27.12		0.46	
2311	EN16711-2	26.05		0.04	
2330	ISO105E04	25.555		-0.15	
2347	ISO105E04	25.84		-0.04	
2350	EN16711-2	26.890		0.37	
2352	EN16711-2	26.01		0.03	
2357	EN16711-2	26.27		0.13	
2358	EN16711-2	27.0		0.41	
2363	EN16711-2	26.56		0.24	
2365	EN16711-2	26.653		0.28	
2370	ISO105E04	25.5		-0.17	
2374	EN16711-2	26.573		0.25	
2375	EN16711-2	28.3		0.91	
2379	EN16711-2	26.9460		0.39	
2380	EN16711-2	20.262		-2.19	
2382	EN16711-2	26.45		0.20	
2385	EN16711-2	27		0.41	
2390	ISO105E04	23.502		-0.94	
2410		26		0.02	
2429	DIN54233-3	26.585		0.25	
2442	EN16711-2	23.70		-0.86	
2456	EN16711-2	15.767	C,R(0.05)	-3.92	First reported 31.533
2459	EN16711-2	17.75		-3.16	
2472	GB/T17593	24.95		-0.38	
2486	EN16711-2	24.478		-0.56	
2492	In house	30.066		1.59	
2495	EN16711-2	24.1		-0.71	
2497	EN16711-2	31.843		2.28	
2511	EN16711-2	28.31		0.91	
2514	EN16711-2	25.91		-0.01	
2553	EN16711-2	27.68		0.67	
2563	EN16711-2	29.3		1.30	
2582	EN16711-2	20.02	C	-2.28	First reported 18.07
2590	EN16711-2	24.19		-0.67	
2618	ISO105E04	21.08		-1.87	
2637	EN16711-2	27.82		0.73	
2638	EN16711-2	23.99		-0.75	
2644	EN16711-2	27.56		0.63	
2649	ISO105E04	21.75		-1.61	
2654	EN16711-2	27.56		0.63	
2671	EN16711-2	29.059		1.20	
2675	EN16711-2	26.784		0.33	
2678	EN16711-2	20.095		-2.25	
2713		-----		-----	
2719	ISO105E04	26.4	C	0.18	First reported as sample #19631
2726	EN16711-2	26.383		0.17	

lab	method	value	mark	z(targ)	remarks
2734	EN16711-2	26.91		0.38	
2740	EN16711-2	26.56	C	0.24	First reported 32.47
2789	EN16711-2	23.3		-1.02	
2804	EN16711-2	27.75		0.70	
2812	EN16711-2	31.48		2.14	
2830	EN16711-2	22.44		-1.35	
2863	EN16711-2	27.3032		0.53	
2870	EN16711-2	28.8		1.10	
2877		-----		-----	
2892	EN16711-2	27.730		0.69	
2902	EN16711-2	26.23		0.11	
2904		0.434	C,R(0.01)	-9.83	First reported 1.403
3116	EN16711-2	28.47		0.98	
3118	EN16711-2	26.2153		0.11	
3146	EN16711-2	28.8		1.10	
3153	EN16711-2	27.097		0.45	
3154	EN16711-2	27.24		0.50	
3172	EN16711-2	26.89		0.37	
3176	EN16711-2	27.503		0.60	
3197	EN16711-2	27.2		0.49	
3210	EN16711-2	25.7		-0.09	
3214	EN16711-2	26.149		0.08	
3228	EN16711-2	28.2		0.87	
3237	EN16711-2	23.31		-1.01	

normality	OK	
n	91	
outliers	3	
mean (n)	25.937	
st.dev. (n)	2.7980	RSD = 11%
R(calc.)	7.834	
st.dev.(EN16711-2:15)	2.5937	
R(EN16711-2:15)	7.262	

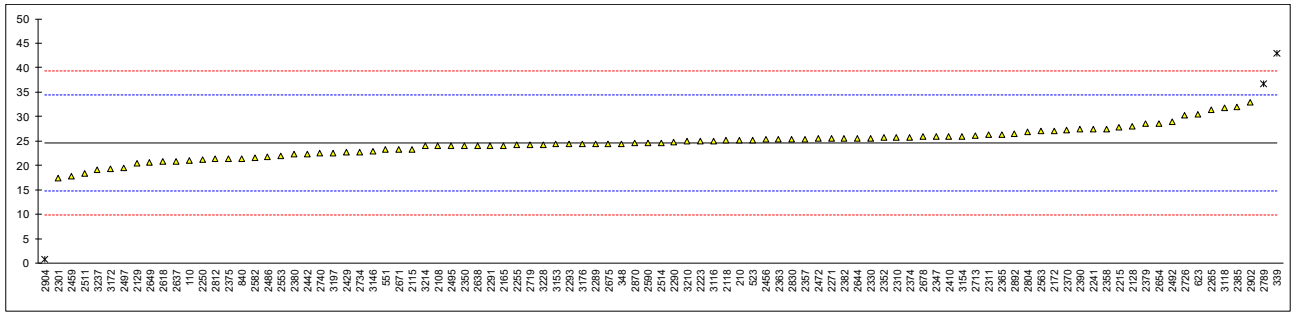


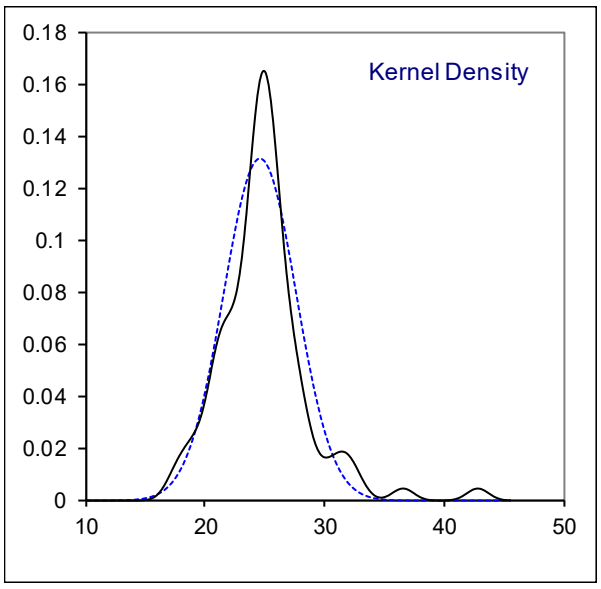


Determination of Antimony as Sb on sample #19631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	20.9734		-0.74	
210	EN16711-2	25.1213		0.10	
339	ISO105E04Mod.	42.86	R(0.01)	3.70	
348	ISO105E04	24.448		-0.04	
523	EN16711-2	25.233		0.12	
551	EN16711-2	23.2570		-0.28	
623	EN16711-2	30.5	C	1.19	First reported 38.77
840	EN16711-2	21.35		-0.67	
2108	In house	24.0		-0.13	
2115	EN16711-2	23.3		-0.27	
2118	EN16711-2	25.0992		0.10	
2128	ISO105E04	27.9051		0.67	
2129		20.4		-0.86	
2165	EN16711-2	24.12		-0.10	
2172	EN16711-2	27.12		0.51	
2190		-----		-----	
2215	EN16711-2	27.848		0.65	
2223	EN16711-2	24.99		0.07	
2241	EN16711-2	27.474		0.58	
2250	EN16711-2	21.164		-0.70	
2255	DIN54233-3	24.2		-0.09	
2265		31.4		1.38	
2271	EN16711-2	25.5		0.18	
2289	DIN54233-3	24.44		-0.04	
2290	EN16711-2	24.825		0.04	
2291	EN16711-2	24.10		-0.11	
2293	DIN54233-3	24.38		-0.05	
2301	EN16711-2	17.42	C	-1.46	First reported 15.56
2310	EN16711-2	25.73		0.22	
2311	EN16711-2	26.22		0.32	
2330	ISO105E04	25.550		0.19	
2347	ISO105E04	25.96		0.27	
2350	EN16711-2	24.048		-0.12	
2352	EN16711-2	25.64		0.21	
2357	EN16711-2	25.41		0.16	
2358	EN16711-2	27.5		0.58	
2363	EN16711-2	25.29		0.13	
2365	EN16711-2	26.330		0.35	
2370	ISO105E04	27.2		0.52	
2374	EN16711-2	25.810		0.24	
2375	EN16711-2	21.3		-0.68	
2379	EN16711-2	28.4869		0.78	
2380	EN16711-2	22.257		-0.48	
2382	EN16711-2	25.53		0.18	
2385	EN16711-2	32		1.50	
2390	ISO105E04	27.445		0.57	
2410		26		0.28	
2429	DIN54233-3	22.663		-0.40	
2442	EN16711-2	22.38		-0.46	
2456	EN16711-2	25.269	C	0.13	First reported 50.538
2459	EN16711-2	17.86		-1.37	
2472	GB/T17593	25.48		0.17	
2486	EN16711-2	21.708		-0.59	
2492	In house	28.916		0.87	
2495	EN16711-2	24.0		-0.13	
2497	EN16711-2	19.428		-1.06	
2511	EN16711-2	18.42		-1.26	
2514	EN16711-2	24.68		0.01	
2553	EN16711-2	21.97		-0.54	
2563	EN16711-2	27		0.48	
2582	EN16711-2	21.51		-0.63	
2590	EN16711-2	24.55	C	-0.02	First reported 237.06
2618	ISO105E04	20.88		-0.76	
2637	EN16711-2	20.9		-0.76	
2638	EN16711-2	24.09		-0.11	
2644	EN16711-2	25.54		0.19	
2649	ISO105E04	20.54		-0.83	
2654	EN16711-2	28.49		0.78	
2671	EN16711-2	23.259		-0.28	
2675	EN16711-2	24.445		-0.04	
2678	EN16711-2	25.8726		0.25	
2713	EN16711-2	26.027		0.28	
2719	ISO105E04	24.2	C	-0.09	First reported for #19630
2726	EN16711-2	30.169		1.13	

lab	method	value	mark	z(targ)	remarks
2734	EN16711-2	22.68		-0.39	
2740	EN16711-2	22.5		-0.43	
2789	EN16711-2	36.6	C,R(0.05)	2.43	First reported 2753.9
2804	EN16711-2	26.8		0.44	
2812	EN16711-2	21.29		-0.68	
2830	EN16711-2	25.34		0.15	
2863		-----		-----	
2870	EN16711-2	24.5		-0.03	
2877		-----		-----	
2892	EN16711-2	26.510		0.38	
2902	EN16711-2	32.82		1.66	
2904		0.764	C,R(0.01)	-4.84	First reported 1.618
3116	EN16711-2	25.02		0.08	
3118	EN16711-2	31.7712		1.45	
3146	EN16711-2	22.9		-0.35	
3153	EN16711-2	24.344		-0.06	
3154	EN16711-2	26.00	C	0.28	First reported 37.47
3172	EN16711-2	19.26		-1.09	
3176	EN16711-2	24.409		-0.04	
3197	EN16711-2	22.6		-0.41	
3210	EN16711-2	24.95		0.07	
3214	EN16711-2	23.987		-0.13	
3228	EN16711-2	24.2		-0.09	
3237	EN16711-2	19.12		-1.12	
	normality	OK			
	n	92			
	outliers	3			
	mean (n)	24.625			
	st.dev. (n)	3.0319	RSD = 12%		
	R(calc.)	8.489			
	st.dev.(EN16711-2:15)	4.9250			
	R(EN16711-2:15)	13.790			

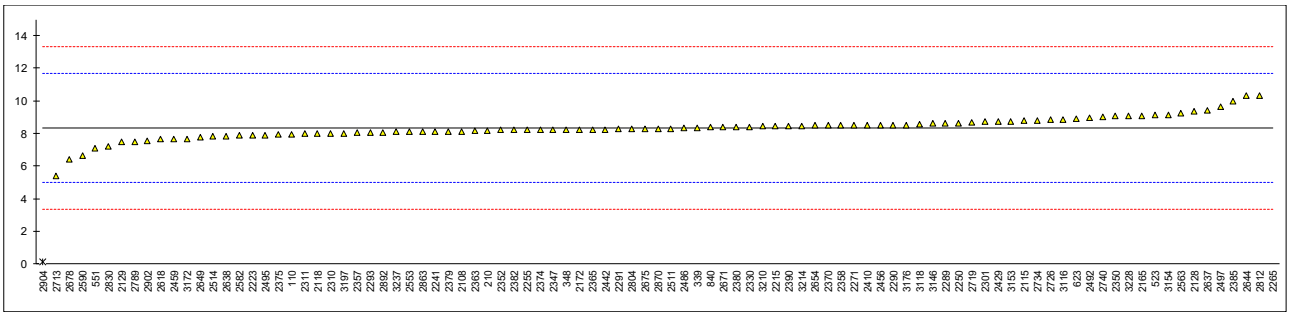


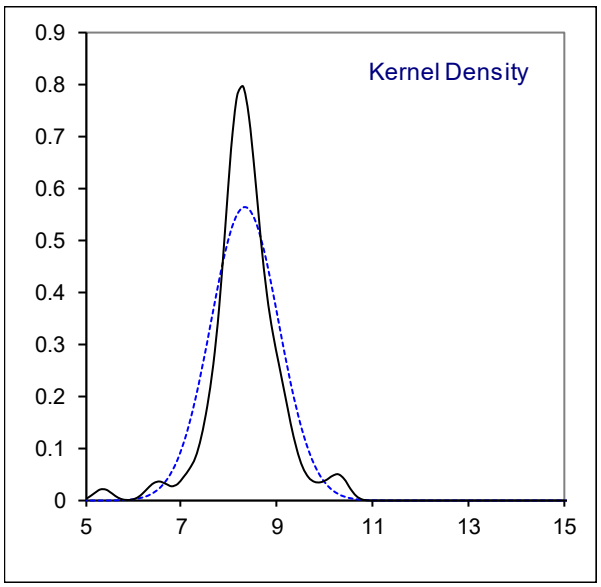


Determination of Arsenic as As on sample #19631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	7.97283		-0.22	
210	EN16711-2	8.1703		-0.10	
339	ISO105E04Mod.	8.348		0.01	
348	ISO105E04	8.232		-0.06	
523	EN16711-2	9.115		0.47	
551	EN16711-2	7.1066		-0.74	
623	EN16711-2	8.91		0.34	
840	EN16711-2	8.37		0.02	
2108	In house	8.14		-0.12	
2115	EN16711-2	8.8		0.28	
2118	EN16711-2	7.997		-0.20	
2128	ISO105E04	9.3579		0.61	
2129		7.50		-0.50	
2165	EN16711-2	9.106		0.46	
2172	EN16711-2	8.235		-0.06	
2190		-----		-----	
2215	EN16711-2	8.4515		0.07	
2223	EN16711-2	7.90		-0.26	
2241	EN16711-2	8.115		-0.13	
2250	EN16711-2	8.6405		0.18	
2255	DIN54233-3	8.2		-0.08	
2265		30.77	C,R(0.01)	13.45	First reported 13.73
2271	EN16711-2	8.5		0.10	
2289	DIN54233-3	8.61		0.16	
2290	EN16711-2	8.518		0.11	
2291	EN16711-2	8.26		-0.05	
2293	DIN54233-3	8.06		-0.17	
2301	EN16711-2	8.75	C	0.25	First reported ND
2310	EN16711-2	8		-0.20	
2311	EN16711-2	7.99		-0.21	
2330	ISO105E04	8.420		0.05	
2347	ISO105E04	8.22		-0.07	
2350	EN16711-2	9.060		0.43	
2352	EN16711-2	8.2		-0.08	
2357	EN16711-2	8.058		-0.17	
2358	EN16711-2	8.5		0.10	
2363	EN16711-2	8.17		-0.10	
2365	EN16711-2	8.239		-0.06	
2370	ISO105E04	8.50		0.10	
2374	EN16711-2	8.201		-0.08	
2375	EN16711-2	7.93		-0.24	
2379	EN16711-2	8.1199		-0.13	
2380	EN16711-2	8.403		0.04	
2382	EN16711-2	8.20		-0.08	
2385	EN16711-2	10		1.00	
2390	ISO105E04	8.483		0.09	
2410		8.5		0.10	
2429	DIN54233-3	8.751		0.25	
2442	EN16711-2	8.25		-0.05	
2456	EN16711-2	8.516	C	0.11	First reported 17.032
2459	EN16711-2	7.67		-0.40	
2472		-----		-----	
2486	EN16711-2	8.320		-0.01	
2492	In house	8.94	C	0.36	First reported 12.542
2495	EN16711-2	7.9		-0.26	
2497	EN16711-2	9.672		0.80	
2511	EN16711-2	8.31		-0.02	
2514	EN16711-2	7.82		-0.31	
2553	EN16711-2	8.10		-0.14	
2563	EN16711-2	9.27		0.56	
2582	EN16711-2	7.89		-0.27	
2590	EN16711-2	6.65		-1.01	
2618	ISO105E04	7.64		-0.42	
2637	EN16711-2	9.4		0.64	
2638	EN16711-2	7.8366		-0.30	
2644	EN16711-2	10.31		1.18	
2649	ISO105E04	7.75		-0.35	
2654	EN16711-2	8.484		0.09	
2671	EN16711-2	8.38		0.03	
2675	EN16711-2	8.300		-0.02	
2678	EN16711-2	6.4100		-1.16	
2713	EN16711-2	5.372	C	-1.78	First reported 4.721
2719	ISO105E04	8.7	C	0.22	First reported for #19630
2726	EN16711-2	8.835		0.30	

lab	method	value	mark	z(targ)	remarks
2734	EN16711-2	8.80		0.28	
2740	EN16711-2	9.00		0.40	
2789	EN16711-2	7.5		-0.50	
2804	EN16711-2	8.29		-0.03	
2812	EN16711-2	10.34		1.20	
2830	EN16711-2	7.19		-0.69	
2863	EN16711-2	8.1093		-0.14	
2870	EN16711-2	8.3		-0.02	
2877		-----		-----	
2892	EN16711-2	8.084		-0.15	
2902	EN16711-2	7.534		-0.48	
2904		0.152	C,R(0.01)	-4.91	First reported 0.462
3116	EN16711-2	8.862		0.31	
3118	EN16711-2	8.5850		0.15	
3146	EN16711-2	8.60		0.16	
3153	EN16711-2	8.755		0.25	
3154	EN16711-2	9.12		0.47	
3172	EN16711-2	7.68		-0.39	
3176	EN16711-2	8.537		0.12	
3197	EN16711-2	8.0		-0.20	
3210	EN16711-2	8.45		0.07	
3214	EN16711-2	8.483		0.09	
3228	EN16711-2	9.10		0.46	
3237	EN16711-2	8.09		-0.15	
	normality	not OK			
	n	93			
	outliers	2			
	mean (n)	8.3381			
	st.dev. (n)	0.70915	RSD = 9%		
	R(calc.)	1.9856			
	st.dev.(EN16711-2:15)	1.66762			
	R(EN16711-2:15)	4.6693			





APPENDIX 2

Reported test results of Antimony, Arsenic, Cadmium, Cobalt, Copper, Lead, Manganese, Mercury and Zinc on sample #19630; results in mg/kg

lab	Sb	As	Cd	Co	Cu	Pb	Mn	Hg	Zn
110	0.06633	0.00857	0.00717	0.01387	0.45983	0.02010	0.29303	0.00090	3.7418
210	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.5	<0.05	<0.05	<0.5	<0.5	<0.05	-----	<0.005	-----
348	< 1	< 0.1	< 0.05	< 0.5	< 5	< 0.1	-----	< 0.01	-----
523	<0.05	<0.05	<0.05	<0.05	<0.50	<0.05	<0.50	<0.05	2.255
551	ND	ND	ND	ND	4.5522	0.11901	0.27274	ND	2.4298
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	<0.3	<0.06	<0.03	<0.3	<2	<0.06	<1	<0.006	<2
2108	<4	<0,1	<0,05	<0,1	<4	<0,1	<4	<0,01	<4
2115	-----	-----	-----	-----	0.3	-----	1.1	-----	3.1
2118	0.06485	0	0	0	0	0.00305	0.2661	0.01675	2.3073
2128	<0,02	<0,02	<0,02	<0,02	0.1254	<0,02	0.2552	<0,002	2.6806
2129	<0,20	<0,10	<0,050	<0,20	<5,0	<0,10	-----	<0,010	-----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	-----
2172	<0.5	<0.1	<0.02	<0.1	<5	<0.1	<0.5	<0.02	<6
2190	-----	-----	-----	-----	-----	-----	-----	-----	-----
2215	<0.5	<0.02	<0.02	<0.1	<5	<0.1	<0.4	<0.005	<6
2223	<0.5	<0.05	<0.05	<0.05	<0.5	<0.05	<0.5	<0.05	2.15
2241	0.010	0.002	0.006	0.007	0.294	0.006	-----	0	-----
2250	<0,1	<0,02	<0,02	<0,05	<0,5	<0,1	0.1991	<0,005	2.443
2255	nd	nd	nd	nd	nd	nd	nd	nd	3.2
2265	-----	-----	-----	-----	-----	-----	-----	-----	4.62
2271	ND	ND	ND	ND	ND	ND	ND	ND	ND
2289	<1.0	<0.1	<0.03	<0.3	<1.0	<0.1	<1.0	<0.01	<1.0
2290	<1.0	<0.1	<0.03	<0.3	<1.0	<0.1	<5.0	<0.01	<5.0
2291	＜3.00	＜0.20	＜0.10	＜1.00	＜1.00	＜0.20	＜1.00	＜0.02	＜3.00
2293	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	ND	ND	ND	ND	ND	ND	-----	ND	-----
2310	not detected	not detected	not detected	not detected	not detected	not detected	<1.0	not detected	<5.0
2311	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2330	ND	ND	ND	ND	ND	ND	ND	ND	NA
2347	<1	<0.1	<0.05	<0.5	<5	<0.1	<10	<0.01	<10
2350	<0.5	<0.02	<0.02	<0.1	0.209	<0.1	-----	<0.005	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	<1	<0.1	<0.05	<0.5	<5	<0.1	N/A	<0.01	N/A
2363	ND	ND	ND	ND	ND	ND	-----	ND	-----
2365	<1.00	<0.10	<0.05	<0.50	<5.00	<0.10	-----	<0.01	-----
2370	<1	<0.2	<0.1	<0.1	<5	<0.2	<1	<0.02	<2
2374	-----	-----	-----	-----	-----	-----	-----	-----	-----
2375	<3	<0.06	<0.03	<0.3	<5	<0.06	<2.5	<0.006	<2.5
2379	ND	ND	ND	ND	ND	ND	-----	ND	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----	-----
2382	<1.0	<0.10	<0.050	<0.50	<5.0	<0.10	-----	<0.010	-----
2385	<0.1	<0.1	<0.01	<0.1	<0.5	<0.1	<0.5	<0.01	2.9
2390	nd	nd	nd	nd	nd	nd	nd	nd	nd
2410	-----	-----	-----	-----	-----	-----	-----	-----	-----
2429	<1.0	<0.1	<0.05	<0.5	<5.0	<0.1	<1.0	<0.02	<1.0
2442	ND	ND	ND	ND	ND	ND	-----	ND	-----
2456	-----	-----	-----	-----	-----	-----	-----	-----	1.956
2459	ND	ND	ND	ND	ND	ND	ND	ND	4.03
2472	<0.35	-----	<0.06	<0.10	<0.6	<0.35	-----	-----	-----
2486	ND	ND	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<0.1	<0.02	<0.02	<0.1	<1	<0.1	<1	<0.02	2.3
2497	-----	-----	-----	-----	3.362	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----	2.92
2553	ND	ND	ND	ND	ND	ND	ND	ND	ND
2563	-----	-----	-----	-----	-----	-----	0.24	-----	22.7
2582	0.03	ND	ND	ND	2.47	0.06	0.13	ND	7.47
2590	0.86	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	-----	<L.O.Q.	-----
2618	-----	-----	-----	-----	-----	-----	-----	-----	-----
2637	<0.02	<0.02	0.005	0.012	0.017	<0.05	0.32	<0.001	2.37
2638	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	1.95
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----
2649	ND	ND	ND	ND	ND	ND	ND	ND	4.0
2654	-----	-----	-----	-----	-----	-----	-----	-----	-----
2671	ND	ND	ND	ND	ND	ND	ND	ND	ND
2675	-----	-----	-----	-----	0.269	-----	0.202	-----	2.497
2678	nd	nd	nd	nd	nd	nd	-----	nd	-----
2713	< 7,5	< 0,05	< 0,025	-----	-----	< 0,05	-----	0.005	-----
2719	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	Sb	As	Cd	Co	Cu	Pb	Mn	Hg	Zn
2726	-----	-----	-----	-----	0.313	-----	-----	-----	-----
2734	nd	nd	nd	nd	nd	nd	nd	nd	nd
2740	-----	-----	-----	-----	-----	0.13	-----	-----	-----
2789	-----	-----	-----	-----	0.4	-----	0.2	-----	4.8
2804	<1.00	<0.05	<0.02	<0.30	<5.00	<0.10	-----	<0.005	-----
2812	-----	-----	-----	-----	-----	-----	-----	-----	-----
2830	ND	ND	ND	ND	< LQ = 12.5	< LQ = 0.5	-----	ND	-----
2863	< 0,05	< 0,05	< 0,05	< 0,05	-----	-----	0.318	< 0,05	-----
2870	ND	ND	ND	ND	2.8	ND	0.30	ND	3.2
2877	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	ND	ND	ND	ND	0.438	ND	0.250	ND	2.900
2902	<0.5	<0.1	<0.1	<0.5	<1	<0.5	-----	-----	-----
2904	0.021	-----	-----	-----	0.0875	0.0057	0.0313	0.0015	0.526
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	<0.25	<0.05	<0.05	<0.25	<0.25	<0.25	-----	<0.01	-----
3146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.91
3153	<1	<0.3	<0.03	<1	<1	<1	-----	<0.01	-----
3154	-----	-----	-----	-----	-----	-----	0.276	-----	0.841
3172	< 5	< 0.05	< 0.02	< 0.1	< 5	< 0.1	-----	< 0.01	< 5
3176	1.454	-----	-----	-----	-----	-----	-----	-----	2.136
3197	<1	<0,1	<0,1	<0,1	<1	<0,1	<1	<0,02	1.9
3210	<5.00	<0.20	<0.10	<1.00	<5.00	<0.20	<5.00	<0.02	<5.00
3214	<1	<0.1	<0.03	<0.3	<1	<0.1	<1	<0.02	<5
3228	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	-----	<0.02	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----

Reported test results of Cadmium, Chromium, Cobalt, Copper, Lead, Manganese, Mercury, Nickel and Zinc on sample #19631; results in mg/kg

lab	Cd	Cr	Co	Cu	Pb	Mn	Hg	Ni	Zn
110	0.00357	0.09453	0.00857	0.15390	0.03340	0.40673	0.00310	0.17753	2.5416
210	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.05	<0.25	<0.5	<0.5	<0.05	-----	<0.005	<0.5	-----
348	< 0.05	< 0.5	< 0.5	< 5	< 0.1	-----	< 0.01	< 0.5	-----
523	<0.05	0.060	<0.05	<0.50	<0.05	<0.50	<0.05	0.123	0.913
551	ND	0.14442	ND	3.8700	0.1245	0.3685	ND	0.11454	2.13147
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	<0.03	<0.3	<0.3	<2	<0.06	<1	<0.006	<0.15	<2
2108	<0,05	0.13	<0,1	<4	<0,1	<4	<0,01	0.16	<4
2115	-----	0.08	-----	-----	-----	1.3	-----	0.2	1.1
2118	0.09215	0.00135	0	0	0.03885	0.3922	0.0013	0.1184	0.6819
2128	<0,02	0.0657	<0,02	0.0443	0.0313	0.4392	<0,002	0.2009	1.0472
2129	<0,050	<0,20	<0,20	<5,0	<0,10	-----	<0,010	<0,40	-----
2165	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.	n.d.	-----
2172	<0.02	<0.1	<0.1	<5	<0.1	<0.5	<0.02	<0.1	<6
2190	-----	-----	-----	-----	-----	-----	-----	-----	-----
2215	<0.02	<0.1	<0.1	<5	<0.1	<0.4	<0.005	<0.1	<6
2223	<0.05	<0.05	<0.05	<0.5	<0.05	<0.5	<0.05	0.15	1.00
2241	0.001	0.060	0.007	0.100	0	-----	0.001	0.148	-----
2250	<0,02	<0,1	<0,05	<0,5	<0,1	0.3659	<0,005	<0,1	<1
2255	nd	nd	nd	nd	nd	nd	nd	nd	nd
2265	-----	0.091	-----	-----	-----	-----	0.0299	0.152	3.333
2271	ND	ND	ND	ND	ND	ND	ND	ND	ND
2289	<0.03	<0.5	<0.3	<1.0	<0.1	<1.0	<0.01	<0.3	<1.0
2290	<0.03	<0.5	<0.3	<1.0	<0.1	<5.0	<0.01	<0.3	<5.0
2291	＜0.10	＜0.50	＜1.00	＜1.00	＜0.20	＜1.00	＜0.02	＜1.00	＜3.00
2293	-----	-----	-----	-----	-----	-----	-----	-----	-----
2301	ND	ND	ND	ND	ND	-----	ND	ND	-----
2310	not detected	not detected	not detected	not detected	not detected	<1.0	not detected	not detected	<5.0
2311	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2330	ND	ND	ND	ND	ND	ND	ND	ND	NA
2347	<0.05	<1	<0.5	<5	<0.1	<0.5	<0.01	<0.5	<1
2350	<0.02	<0.1	<0.1	<0.05	<0.1	-----	0.0182	<0.1	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	<0.05	<0.5	<0.5	<5	<0.1	N/A	<0.01	<0.5	N/A
2363	ND	ND	ND	ND	ND	-----	ND	ND	-----
2365	<0.05	<0.50	<0.50	<5.00	<0.10	-----	<0.01	<0.50	-----
2370	<0.1	<0.5	<0.1	<5	<0.2	<1	<0.02	<0.1	<1
2374	-----	-----	-----	-----	-----	-----	-----	-----	-----
2375	<0.03	<10	<0.3	<5	<0.06	<2.5	<0.006	0.17	<2.5
2379	ND	ND	ND	ND	ND	-----	ND	ND	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.050	<0.50	<0.50	<5.0	<0.10	-----	<0.010	<0.50	-----
2385	<0.01	<0.5	<0.1	<0.5	<0.1	<0.5	<0.01	<0.5	0.88
2390	nd	nd	nd	nd	nd	nd	nd	nd	nd
2410	-----	-----	-----	-----	-----	-----	-----	-----	-----
2429	<0.05	<0.5	<0.5	<5.0	<0.1	<1.0	<0.02	<0.1	<1.0
2442	ND	ND	ND	ND	ND	-----	ND	ND	-----
2456	0.560	-----	-----	-----	-----	0.360	-----	-----	1.0395
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND
2472	<0.06	<0.06	<0.10	<0.6	<0.35	-----	-----	<0.05	-----
2486	ND	ND	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<0.02	<0.1	<0.1	<1	<0.1	<1	<0.02	<0.1	<1
2497	-----	-----	-----	3.768	0.128	-----	-----	0.948	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	ND	ND	ND	ND	ND	ND	ND	ND	ND
2563	-----	-----	-----	-----	-----	0.42	-----	-----	23.1
2582	ND	ND	ND	2.63	ND	0.33	ND	ND	1.92
2590	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	<L.O.Q.	-----	<L.O.Q.	<L.O.Q.	-----
2618	-----	-----	-----	-----	-----	-----	-----	-----	-----
2637	<0.005	0.16	0.009	<0.1	0.05	0.49	<0.001	0.24	0.8
2638	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	0.5116
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----
2649	ND	ND	ND	ND	ND	ND	ND	ND	ND
2654	-----	-----	-----	-----	-----	-----	-----	-----	-----
2671	ND	ND	ND	ND	ND	ND	ND	ND	ND
2675	-----	-----	-----	0.099	-----	0.364	-----	0.188	1.739
2678	ND	ND	ND	ND	ND	-----	ND	ND	-----
2713	< 0,025	< 0,5	-----	-----	< 0,05	-----	0.045	-----	-----
2719	-----	-----	-----	-----	-----	-----	-----	-----	-----
2726	-----	-----	-----	-----	-----	-----	-----	-----	-----
2734	ND	ND	ND	ND	ND	ND	ND	ND	ND

lab	Cd	Cr	Co	Cu	Pb	Mn	Hg	Ni	Zn
2740	-----	-----	-----	-----	0.11	-----	0.002	-----	-----
2789	-----	0.3	-----	-----	-----	0.4	-----	0.2	5.2
2804	<0.02	<0.10	<0.30	<5.00	<0.10	-----	0.0079	<0.30	-----
2812	-----	-----	-----	-----	-----	-----	-----	-----	-----
2830	ND	< LQ = 1.0	ND	< LQ = 12.5	< LQ = 0.5	-----	ND	ND	-----
2863	< 0,05	-----	< 0,05	-----	-----	0.5050	< 0,05	-----	2.5795
2870	ND	ND	ND	2.7	ND	0.35	ND	0.19	0.63
2877	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	ND	ND	ND	ND	ND	0.509	ND	ND	0.935
2902	<0.1	0.57	<0.5	<1	<0.5	-----	-----	<0.5	-----
2904	-----	0.0689	-----	0.108	0.0087	0.0397	0.0041	0.137	0.401
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	<0.05	<0.5	<0.25	<0.25	<0.05	-----	0.1825	<0.25	-----
3146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3153	<0.03	<0.5	<1	<1	<1	-----	<0.01	<1	-----
3154	-----	-----	-----	-----	-----	0.431	-----	-----	-----
3172	< 0.02	< 0.1	< 0.1	< 5	< 0.1	-----	< 0.01	< 0.2	< 5
3176	-----	0.115	-----	-----	-----	-----	-----	0.233	0.988
3197	<0,1	<0,1	<0,1	<1	<0,1	<1	<0,02	0.17	<1
3210	<0.10	<1.00	<1.00	<5.00	<0.20	<5.00	<0.02	<1.00	<5.00
3214	<0.03	<0.5	<0.3	<1	<0.1	<1	<0.02	<1	<5
3228	<0.02	<0.5	<0.5	<0.5	<0.02	-----	<0.02	<0.5	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3

Analytical Details

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
110	---	---		---
210	---	---		1 gram textile per 50 mL perspiration liquid
339	---	---		---
348	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
523	No	Used as received	1g	1 gram textile per 50 mL perspiration liquid
551	Yes	Used as received	1.004g / 1.0083g	1 gram textile per 50 mL perspiration liquid
623	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
840	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
2108	Yes	Further Cut	1 g	1 gram textile per 20 mL perspiration liquid
2115	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
2118	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2128	No	Used as received	1g	1 gram textile per 20 mL perspiration liquid
2129	---	---		---
2165	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2172	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2190	---	---		---
2215	Yes	Used as received	1.0106g / 1.0038g	1 gram textile per 50 mL perspiration liquid
2223	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
2241	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2250	Yes	Used as received	0,5	0.5 gram textile per 25 mL perspiration liquid
2255	Yes	Used as received	1.0	1 gram textile per 50 mL perspiration liquid
2265	Yes	Used as received	1,0	1 gram textile per 30 mL perspiration liquid
2271	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
2289	Yes	Further Cut	1.0	1 gram textile per 50 mL perspiration liquid
2290	---	---		---
2291	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
2293	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2301	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
2310	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
2311	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2330	Yes	Further Cut	1 gram	1 gram textile per 50 mL perspiration liquid
2347	Yes	---		---
2350	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
2352	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2357	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
2358	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2363	Yes	---		---
2365	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2370	Yes	Further Cut	1 g	1 gram textile per 50 mL perspiration liquid
2374	No	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2375	Yes	Further Cut	-	1 gram textile per 50 mL perspiration liquid
2379	Yes	Used as received	1 grams	1 gram textile per 50 mL perspiration liquid
2380	Yes	Further Cut	0.50	1 gram textile per 50 mL perspiration liquid
2382	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
2385	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2390	Yes	Further Cut	1.0 g	1 gram textile per 50 mL perspiration liquid
2410	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2429	Yes	Further Cut	1.0052	1 gram textile per 50 mL perspiration liquid
2442	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
2456	Yes	Used as received	2.5 gram	2.5 gram textile per 50 mL perspiration liquid
2459	---	---		---
2472	Yes	Further Cut	1gram	1 gram textile per 50 mL perspiration liquid
2486	Yes	Further Cut	1.0081g / 1.0029g	1 gram textile per 50 mL perspiration liquid
2492	Yes	Further Cut	0.5 gram	1 gram textile per 50 mL perspiration liquid
2495	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2497	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
2511	Yes	Used as received	1 g	1 gram textile per 20 mL perspiration liquid
2514	Yes	Further Cut	0.5001	1 gram textile per 50 mL perspiration liquid
2553	Yes	Used as received	01	1 gram textile per 50 mL perspiration liquid
2563	No	Used as received		1 gram textile per 50 mL perspiration liquid
2582	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2590	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2618	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2637	---	---		---
2638	No	Used as received	1 gm	1 gram textile per 50 mL perspiration liquid
2644	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2649	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
2654	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2671	Yes	Used as received	0.5	1 gram textile per 50 mL perspiration liquid
2675	No	Used as received		1 gram textile per 50 mL perspiration liquid
2678	---	---		---
2713	No	Used as received	1 gr / 50 ml	1 gram textile per 50 mL perspiration liquid
2719	Yes	Further Cut	0.5	1 gram textile per 50 mL perspiration liquid

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
2726	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2734	---	---		---
2740	Yes	Used as received	about 0,75 g	1 gram textile per 50 mL perspiration liquid
2789	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
2804	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
2812	Yes	Further Cut	0.15	1 gram textile per 50 mL perspiration liquid
2830	---	---		---
2863	Yes	---		1 gram textile per 50 mL perspiration liquid
2870	Yes	Further Cut	0.5034g / 0.5002g	1 gram textile per 50 mL perspiration liquid
2877	---	---		---
2892	Yes	Further Cut	0.5g	1 gram textile per 50 mL perspiration liquid
2902	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
2904	No	Used as received	2 g	1 gram textile per 50 mL perspiration liquid
3116	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
3118	Yes	Further Cut	0.5	1 gram textile per 50 mL perspiration liquid
3146	Yes	Used as received	0.6 gram	1 gram textile per 50 mL perspiration liquid
3153	Yes	Further Cut	1.0g	1 gram textile per 50 mL perspiration liquid
3154	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
3172	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
3176	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
3197	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
3210	Yes	Further Cut	1g	1 gram textile per 50 mL perspiration liquid
3214	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
3228	Yes	Further Cut	1	1 gram textile per 50 mL perspiration liquid
3237	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid

APPENDIX 4

Number of participants per country

7 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in CAMBODIA
3 labs in FRANCE
12 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
5 labs in INDIA
3 labs in INDONESIA
9 labs in ITALY
1 lab in MEXICO
3 labs in MOROCCO
17 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in POLAND
1 lab in SERBIA
2 lab in SOUTH KOREA
2 labs in SPAIN
2 labs in SRI LANKA
1 lab in SWITZERLAND
2 labs in TAIWAN R.O.C.
1 lab in THAILAND
2 labs in TUNISIA
7 labs in TURKEY
1 lab in U.S.A.
4 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
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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