Institute for
Interlaboratory Studies

Results of Proficiency Test
Heavy Metals by Perspiration
in Textile
November 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 some Ecolabelling schemes are imposing environmental requirements for textile products on a voluntary basis e.g. EU Ecolabel regulation 2014/350/EU, Oeko-Tex® Standard (Switzerland), BlueSign® (Switzerland) and American Apparel and Footwear Association (United States).

Since 2002 the Institute of Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Heavy Metals by Perspiration in Textile every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Heavy Metals by Perspiration in Textile.

In this interlaboratory study 84 laboratories in 24 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of Heavy Metals by Perspiration in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different textile samples of approximately 3 grams each labelled #22750 and #22751 respectively.
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

For the first sample a batch of pink cotton pieces was selected which was artificially fortified with Antimony and Arsenic by a third party. After homogenization 105 small plastic bags were filled with approximately 3 grams each and labelled #22750.
The batch for sample #22750 was used in a previous proficiency test on Heavy Metals by Perspiration in Textile as sample #19631 in iis19A02. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of red cotton pieces was selected which was artificially fortified with Copper and Nickel by a third party. After homogenization 115 small plastic bags were filled with approximately 3 grams each and labelled #22751.
The batch for sample #22751 was used in a previous proficiency test on Heavy Metals by Perspiration in Textile as sample #20700 in iis20A12. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two textile samples labelled #22750 and #22751 respectively were sent on October 5, 2022.

2.5 ANALYZES

The participants were requested to determine on both samples: Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Cobalt as Co, Copper as Cu, Lead as Pb, Mercury as Hg and Nickel as Ni 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.
It was requested not to use less than 0.5 gram per determination to ensure homogeneity.
It was also requested to report if the laboratory was accredited for the requested components and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.

The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

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

3.1 STATISTICS

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or

DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Nine participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all elements requested. In total 83 participants reported 324 numerical test results. Observed were 6 outlying test results, which is 1.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER ELEMENT

In this section the reported test 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 test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of Heavy Metals by Perspiration in Textile the EN16711-2 is considered to be the official test method. This method mentions the standard deviation and variation coefficient per element between laboratories. The reproducibility of each metal was calculated by multiplying the variation coefficient of the metal with 2.8.

sample #22750

Antimony as Sb: The determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN16711-2:15.

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

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

sample #22751

Copper as Cu: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the outliers is in agreement with the requirements of EN16711-2:15.

Nickel as Ni: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN16711-2:15.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported 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 and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next two tables.

Element	unit	n	average	2.8 * sd	R(lit)
Antimony as Sb	mg/kg	79	23.2	6.5	13.0
Arsenic as As	mg/kg	80	8.26	1.87	4.62

Table 1: reproducibilities of test on sample #22750

Element	unit	n	average	2.8 * sd	R(lit)
Copper as Cu	mg/kg	78	28.9	7.1	12.9
Nickel as Ni	mg/kg	81	2.81	0.54	0.79

Table 2: reproducibilities of tests on sample #22751

Without further statistical calculations it can be concluded that for all tests there is a good compliance of the group of participants with the reference test method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	November 2021	November 2020	November 2019	November 2018
Number of reporting laboratories	83	86	79	96	91
Number of test results	324	418	314	408	254
Number of statistical outliers	6	12	11	13	8
Percentage of statistical outliers	1.9%	2.9%	3.5%	3.2%	3.1%

Table 3: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

Element	November 2022	November 2021	November 2020	November 2019	2010-2018	EN16711-2
Antimony as Sb	10%	--	--	12%	8-19%	20%
Arsenic as As	8%	--	--	9%	--	20%
Cadmium as Cd	--	9-12%	--	--	9-18%	10%
Chromium as Cr	--	--	--	11%	10-19%	15%
Cobalt as Co	--	12%	--	--	8-14%	13%
Copper as Cu	9%	10%	8-13%	--	9-22%	16%
Lead as Pb	--	--	--	--	35-40%	40%
Mercury as Hg	--	--	--	--	34-41%	31%
Nickel as Ni	7%	11%	10%	11%	7-14%	10%

Table 4: development of the uncertainties over the years

The uncertainties observed in this PT are comparable or better than the uncertainties observed in previous PTs.

Sample #22750 was used in a previous PT as sample #19631 in iis19A12. The averages found in both PTs for this sample are similar. The calculated reproducibility for Antimony improved in the 2022 PT compared to the 2019 PT.

Element	unit	sample #22750			sample #19631		
		n	average	R(calc)	n	average	R(calc)
Antimony as Sb	mg/kg	79	23.2	6.5	92	24.6	8.5
Arsenic as As	mg/kg	80	8.26	1.87	93	8.33	1.99

Table 5: comparison of sample #22750 with #19631

Sample #22751 was used in a previous PT as sample #20700 in iis20A12. The averages found in both PTs for this sample are similar. The calculated reproducibility for the Copper and Nickel improved in the 2022 PT compared to the 2020 PT.

Element	unit	sample #22751			sample #20700		
		n	average	R(calc)	n	average	R(calc)
Copper as Cu	mg/kg	78	28.9	7.1	79	29.2	10.6
Nickel as Ni	mg/kg	81	2.81	0.54	74	2.76	0.77

Table 6: comparison of sample #22751 with #20700

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested and are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 85% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- About 75% mentioned to use the sample as received and about 25% did further cut or further grind the sample prior to analysis.
- About 75% used a sample intake of 1 gram and about 20% used 0.5 grams.
- The vast majority of the participants used a ratio of 1 g to 50 mL. Remarkably, three participants reported a ratio of 1g to 20-30 mL. Please note that in the method it is described that there can be a risk that not all the fabric is wetted sufficiently when a smaller amount of simulant is used.

For the elements present in the samples the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

Almost all participants identified all added metals in both textile samples correctly: sample #22750 contained Antimony and Arsenic and sample #22751 contained Copper and Nickel

When the results of this interlaboratory study were compared to the OEKO-TEX® 100 (see table below), it was noticed that all participants would have made identical decisions about the acceptability of sample #22750 but not for sample #22751.
All reporting participants would have rejected sample #22750 for all categories.
All reporting participants would have rejected sample #22751 for Class 1 and almost all reporting participants, except one, would have accepted sample #22751 for Class 2, 3 and 4.

	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
Mercury as Hg	0.02	0.02	0.02	0.02
Nickel as Ni	1.0	4.0	4.0	4.0

Table 7: Overview from OEKO-TEX® 100

6 CONCLUSION

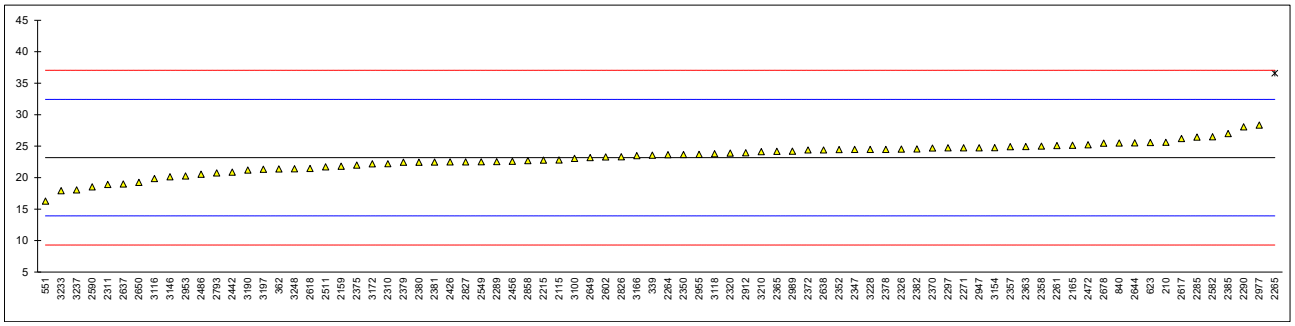
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

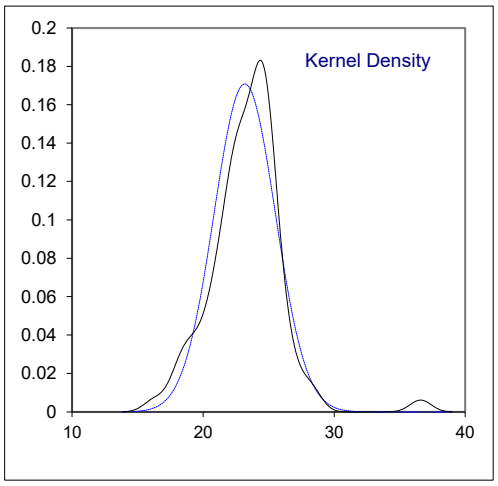
APPENDIX 1

Determination of Antimony as Sb on sample #22750; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	EN16711-2	25.619		0.53	
339	ISO105E04	23.584		0.09	
362	In house	21.39		-0.38	
551	EN16711-2	16.27		-1.49	
623	EN16711-2	25.58		0.52	
840	EN16711-2	25.5		0.50	
2115	EN16711-2	22.80		-0.08	
2159	In house	21.804		-0.30	
2165	EN16711-2	25.154		0.43	
2215	EN16711-2	22.768		-0.09	
2261	GB/T17593.2	25.11		0.42	
2264	EN16711-2	23.639		0.10	
2265		36.60	R(0.01)	2.90	
2271	EN16711-2	24.74		0.34	
2285	EN16711-2	26.434		0.70	
2289	DIN54233-3	22.54		-0.14	
2290	EN16711-2	28.061		1.05	
2297	EN16711-2	24.74		0.34	
2310	EN16711-2	22.2		-0.21	
2311	EN16711-2	18.92		-0.92	
2320	EN16711-2	23.9	C	0.16	first reported 32.08
2326	EN16711-2	24.532		0.29	
2347	EN16711-2	24.48		0.28	
2350	EN16711-2	23.678		0.11	
2352	EN16711-2	24.460		0.28	
2357	EN16711-2	24.92		0.38	
2358	EN16711-2	25		0.39	
2363	EN16711-2	24.94		0.38	
2365	EN16711-2	24.184		0.22	
2370	ISO105E04	24.7		0.33	
2372	EN16711-2	24.4		0.26	
2375	EN16711-2	22		-0.25	
2378	EN16711-2	24.5		0.29	
2379	EN16711-2	22.4422		-0.16	
2380	EN16711-2	22.450		-0.16	
2381	EN16711-2	22.48		-0.15	
2382	EN16711-2	24.55		0.30	
2385	EN16711-2	27		0.83	
2423	EN16711-2	not analyzed		-----	
2426	EN16711-2	22.50		-0.14	
2442	EN16711-2	20.878		-0.49	
2456	EN16711-2/EN11885	22.614		-0.12	
2472	GB/T17593	25.21		0.44	
2486	EN16711-2	20.5470		-0.57	
2511	EN16711-2	21.72		-0.31	
2549	EN16711-2	22.510		-0.14	
2582	EN16711-2	26.50		0.72	
2590	EN16711-2	18.54		-1.00	
2602	EN16711-2	23.3		0.03	
2617	EN16711-2	26.218		0.66	
2618	EN16711-2	21.45	C	-0.37	first reported 10.4
2637	EN16711-2	19		-0.90	
2638	EN16711-2	24.4		0.26	
2644	EN16711-2	25.53		0.51	
2649	EN16711-2	23.2		0.01	
2650	EN16711-2	19.25		-0.85	
2678	EN16711-2	25.470		0.50	
2793	EN16711-2	20.7607		-0.52	
2826	EN16711-2	23.31		0.03	
2827	EN16711-2	22.501		-0.14	
2858	EN16711-2	22.695		-0.10	
2912	EN16711-2	23.955		0.17	
2947	OEKO-TEX St201 M-10&ML-10	24.743		0.34	
2953	EN16711-2	20.266		-0.63	
2955	EN16711-2	23.7		0.11	
2977	EN16711-2	28.337		1.11	
2984		-----		-----	
2989	ISO105E04	24.2021		0.22	
3010		-----		-----	
3100	EN16711-2	23.046		-0.03	
3116		19.848		-0.72	
3118	EN16711-2	23.8110		0.14	
3146	EN16711-2	20.14		-0.65	
3154	EN16711-2	24.77		0.34	
3166	In house	23.5		0.07	

lab	method	value	mark	z(targ)	remarks
3172	EN16711-2	22.175		-0.22	
3190	EN16711-2	21.2		-0.43	
3197	EN16711-2	21.34		-0.40	
3210	EN16711-2	24.139		0.21	
3228	EN16711-2	24.48		0.28	
3233	EN16711-2	17.92		-1.13	
3237	EN16711-2	18.04		-1.11	
3248	EN16711-2	21.4		-0.38	
6191		-----		-----	
	normality	OK			
	n	79			
	outliers	1			
	mean (n)	23.1720			
	st.dev. (n)	2.33600	RSD = 10%		
	R(calc.)	6.5408			
	st.dev.(EN16711-2:15)	4.63439			
	R(EN16711-2:15)	12.9763			

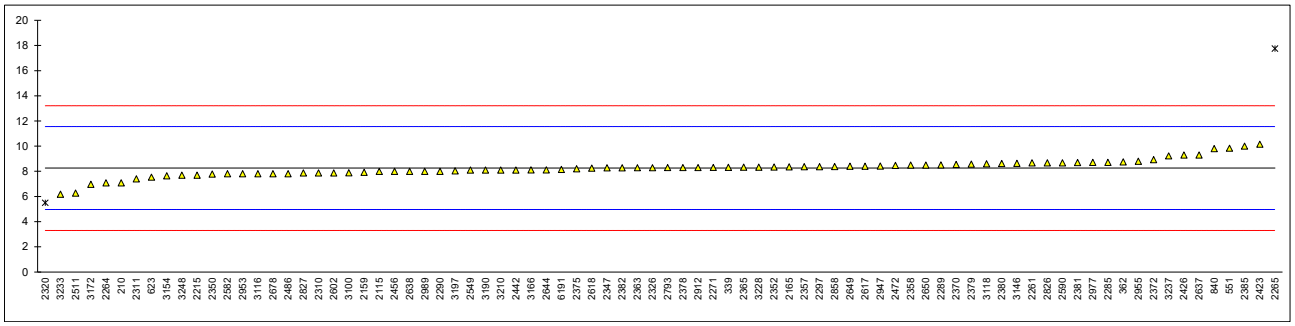


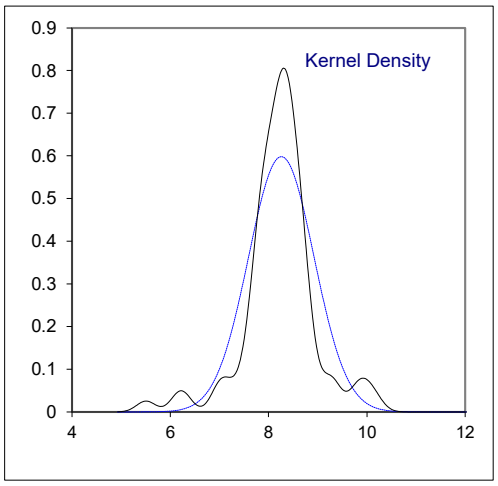


Determination of Arsenic as As on sample #22750; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	EN16711-2	7.092		-0.71	
339	ISO105E04	8.322		0.04	
362	In house	8.75		0.30	
551	EN16711-2	9.838		0.96	
623	EN16711-2	7.53		-0.44	
840	EN16711-2	9.8		0.93	
2115	EN16711-2	7.99		-0.16	
2159	In house	7.928		-0.20	
2165	EN16711-2	8.353		0.06	
2215	EN16711-2	7.701		-0.34	
2261	GB/T17593.2	8.67		0.25	
2264	EN16711-2	7.087		-0.71	
2265		17.75	R(0.01)	5.75	
2271	EN16711-2	8.31		0.03	
2285	EN16711-2	8.714		0.28	
2289	DIN54233-3	8.51		0.15	
2290	EN16711-2	8.006		-0.15	
2297	EN16711-2	8.37		0.07	
2310	EN16711-2	7.88		-0.23	
2311	EN16711-2	7.40		-0.52	
2320	EN16711-2	5.505	R(0.01)	-1.67	
2326	EN16711-2	8.292		0.02	
2347	EN16711-2	8.27		0.01	
2350	EN16711-2	7.7796		-0.29	
2352	EN16711-2	8.350		0.06	
2357	EN16711-2	8.37		0.07	
2358	EN16711-2	8.5		0.15	
2363	EN16711-2	8.29		0.02	
2365	EN16711-2	8.338		0.05	
2370	ISO105E04	8.55		0.18	
2372	EN16711-2	8.93		0.41	
2375	EN16711-2	8.2		-0.04	
2378	EN16711-2	8.30		0.03	
2379	EN16711-2	8.5710		0.19	
2380	EN16711-2	8.630		0.22	
2381	EN16711-2	8.70		0.27	
2382	EN16711-2	8.28		0.01	
2385	EN16711-2	10		1.05	
2423	EN16711-2	10.159		1.15	
2426	EN16711-2	9.30		0.63	
2442	EN16711-2	8.105		-0.09	
2456	EN16711-2/EN11885	7.9996		-0.16	
2472	GB/T17593	8.48		0.13	
2486	EN16711-2	7.8100		-0.27	
2511	EN16711-2	6.26		-1.21	
2549	EN16711-2	8.100		-0.10	
2582	EN16711-2	7.80		-0.28	
2590	EN16711-2	8.68		0.26	
2602	EN16711-2	7.88		-0.23	
2617	EN16711-2	8.408		0.09	
2618	EN16711-2	8.25	C	-0.01	first reported 2.8
2637	EN16711-2	9.3		0.63	
2638	EN16711-2	8.0		-0.16	
2644	EN16711-2	8.117		-0.09	
2649	EN16711-2	8.4		0.09	
2650	EN16711-2	8.5		0.15	
2678	EN16711-2	7.807		-0.27	
2793	EN16711-2	8.2950		0.02	
2826	EN16711-2	8.67		0.25	
2827	EN16711-2	7.871		-0.23	
2858	EN16711-2	8.377		0.07	
2912	EN16711-2	8.301		0.03	
2947	OEKO-TEX St201 M-10&ML-10	8.4132		0.09	
2953	EN16711-2	7.8		-0.28	
2955	EN16711-2	8.8		0.33	
2977	EN16711-2	8.707		0.27	
2984		-----		-----	
2989	ISO105E04	8.0010		-0.16	
3010		-----		-----	
3100	EN16711-2	7.887		-0.22	
3116		7.805		-0.27	
3118	EN16711-2	8.5907		0.20	
3146	EN16711-2	8.635		0.23	
3154	EN16711-2	7.64		-0.37	
3166	In house	8.11		-0.09	

lab	method	value	mark	z(targ)	remarks
3172	EN16711-2	6.975		-0.78	
3190	EN16711-2	8.1		-0.10	
3197	EN16711-2	8.04		-0.13	
3210	EN16711-2	8.10		-0.10	
3228	EN16711-2	8.34		0.05	
3233	EN16711-2	6.18		-1.26	
3237	EN16711-2	9.23		0.59	
3248	EN16711-2	7.7		-0.34	
6191	EN16711-2	8.154		-0.06	
	normality	not OK			
	n	80			
	outliers	2			
	mean (n)	8.2585			
	st.dev. (n)	0.66692	RSD = 8%		
	R(calc.)	1.8674			
	st.dev.(EN16711-2:15)	1.65170			
	R(EN16711-2:15)	4.6248			

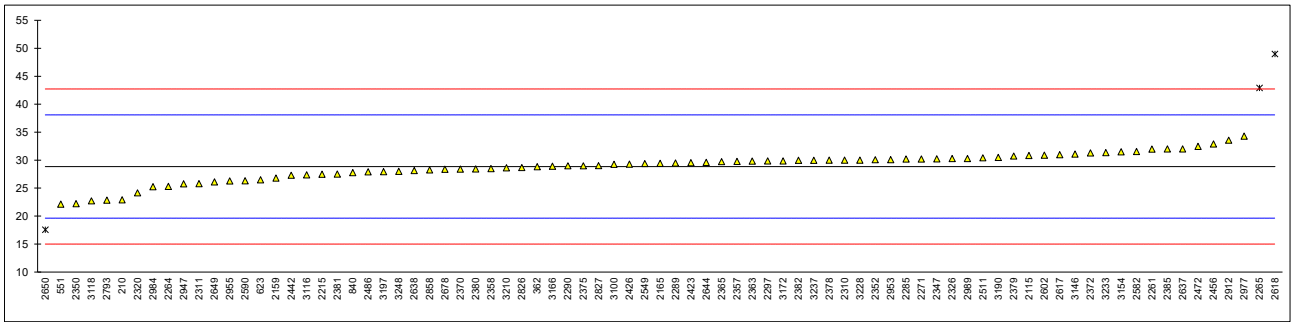


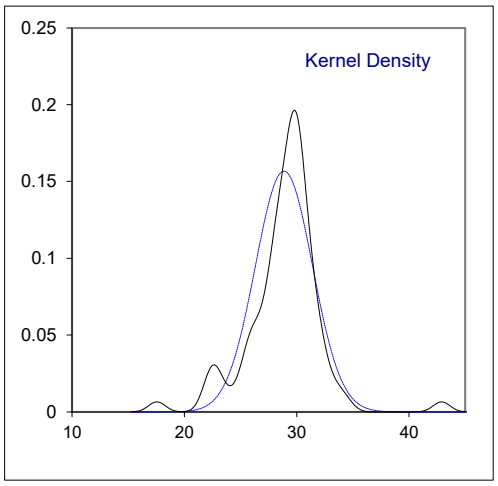


Determination of Copper as Cu on sample #22751; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	EN16711-2	22.915		-1.29	
339	ISO105E04	not analyzed		-----	
362	In house	28.84		-0.01	
551	EN16711-2	22.138		-1.46	
623	EN16711-2	26.47		-0.52	
840	EN16711-2	27.78		-0.23	
2115	EN16711-2	30.85		0.43	
2159	In house	26.802		-0.45	
2165	EN16711-2	29.446		0.13	
2215	EN16711-2	27.487		-0.30	
2261	GB/T17593.2	31.97		0.67	
2264	EN16711-2	25.318		-0.77	
2265		42.90	R(0.01)	3.04	
2271	EN16711-2	30.22		0.29	
2285	EN16711-2	30.213		0.29	
2289	DIN54233-3	29.49		0.14	
2290	EN16711-2	28.991		0.03	
2297	EN16711-2	29.86		0.22	
2310	EN16711-2	30		0.25	
2311	EN16711-2	25.79		-0.67	
2320	EN16711-2	24.149		-1.02	
2326	EN16711-2	30.274		0.31	
2347	EN16711-2	30.23		0.30	
2350	EN16711-2	22.218		-1.44	
2352	EN16711-2	30.070		0.26	
2357		29.78		0.20	
2358	EN16711-2	28.5		-0.08	
2363	EN16711-2	29.84		0.21	
2365	EN16711-2	29.738		0.19	
2370	ISO105E04	28.4		-0.10	
2372	EN16711-2	31.3		0.53	
2375	EN16711-2	29		0.03	
2378	EN16711-2	30.0		0.25	
2379	EN16711-2	30.7351		0.41	
2380	EN16711-2	28.428		-0.09	
2381	EN16711-2	27.50		-0.30	
2382	EN16711-2	29.96		0.24	
2385	EN16711-2	32		0.68	
2423	EN16711-2	29.547		0.15	
2426	EN16711-2	29.28		0.09	
2442	EN16711-2	27.296		-0.34	
2456	EN16711-2/EN11885	32.910		0.88	
2472	GB/T17593	32.46		0.78	
2486	EN16711-2	27.9110		-0.21	
2511	EN16711-2	30.41		0.33	
2549	EN16711-2	29.400		0.12	
2582	EN16711-2	31.55		0.58	
2590	EN16711-2	26.33		-0.55	
2602	EN16711-2	30.9		0.44	
2617	EN16711-2	30.966		0.46	
2618	EN16711-2	49.000	R(0.01)	4.36	
2637	EN16711-2	32.0		0.68	
2638	EN16711-2	28.155		-0.15	
2644	EN16711-2	29.59		0.16	
2649	EN16711-2	26.1		-0.60	
2650	EN16711-2	17.55	R(0.01)	-2.45	
2678	EN16711-2	28.386		-0.10	
2793	EN16711-2	22.8510		-1.30	
2826	EN16711-2	28.67		-0.04	
2827	EN16711-2	29.012		0.03	
2858	EN16711-2	28.263		-0.13	
2912	EN16711-2	33.573		1.02	
2947	OEKO-TEX St201 M-10&ML-10	25.7512		-0.67	
2953	EN16711-2	30.102		0.27	
2955	EN16711-2	26.3		-0.56	
2977	EN16711-2	34.32		1.18	
2984		25.2696		-0.78	
2989	ISO105E04	30.2750		0.31	
3010		-----		-----	
3100	EN16711-2	29.258		0.09	
3116		27.385		-0.32	
3118	EN16711-2	22.7112		-1.33	
3146		31.09		0.48	
3154	EN16711-2	31.51		0.57	
3166	In house	28.9		0.01	

lab	method	value	mark	z(targ)	remarks
3172	EN16711-2	29.875		0.22	
3190	EN16711-2	30.5		0.35	
3197	EN16711-2	27.93		-0.20	
3210	EN16711-2	28.61		-0.05	
3228	EN16711-2	30.00		0.25	
3233	EN16711-2	31.34	C	0.54	first reported 41.69
3237	EN16711-2	29.96		0.24	
3248	EN16711-2	28		-0.19	
6191		-----		-----	
	normality	OK			
	n	78			
	outliers	3			
	mean (n)	28.8635			
	st.dev. (n)	2.54501	RSD = 9%		
	R(calc.)	7.1260			
	st.dev.(EN16711-2:15)	4.61815			
	R(EN16711-2:15)	12.9308			

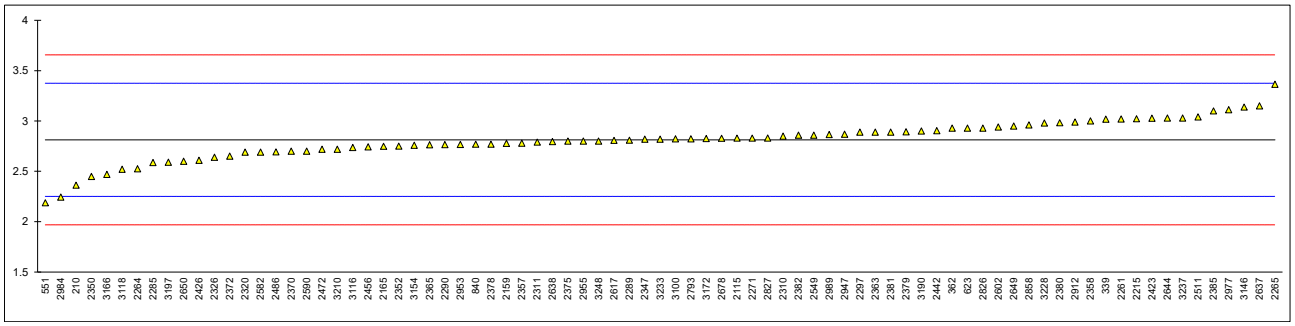


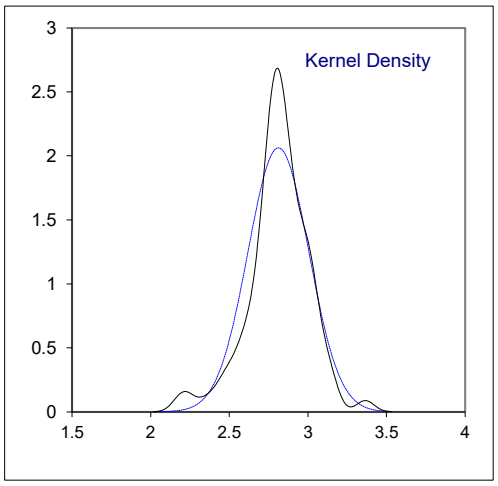


Determination of Nickel as Ni on sample #22751; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	EN16711-2	2.362	C	-1.60	first reported 2.147
339	ISO105E04	3.018		0.73	
362	In house	2.93		0.42	
551	EN16711-2	2.188		-2.22	
623	EN16711-2	2.93		0.42	
840	EN16711-2	2.77		-0.15	
2115	EN16711-2	2.83		0.06	
2159	In house	2.778		-0.12	
2165	EN16711-2	2.749		-0.23	
2215	EN16711-2	3.023		0.75	
2261	GB/T17593.2	3.02		0.74	
2264	EN16711-2	2.526		-1.02	
2265		3.365		1.96	
2271	EN16711-2	2.83		0.06	
2285	EN16711-2	2.587		-0.80	
2289	DIN54233-3	2.81		-0.01	
2290	EN16711-2	2.765		-0.17	
2297	EN16711-2	2.89		0.27	
2310	EN16711-2	2.85		0.13	
2311	EN16711-2	2.79		-0.08	
2320	EN16711-2	2.69		-0.44	
2326	EN16711-2	2.640		-0.61	
2347	EN16711-2	2.82		0.03	
2350	EN16711-2	2.4492		-1.29	
2352	EN16711-2	2.750		-0.22	
2357		2.78		-0.12	
2358	EN16711-2	3.0		0.67	
2363	EN16711-2	2.89		0.27	
2365	EN16711-2	2.764		-0.17	
2370	ISO105E04	2.70		-0.40	
2372	EN16711-2	2.65		-0.58	
2375	EN16711-2	2.8		-0.05	
2378	EN16711-2	2.77		-0.15	
2379	EN16711-2	2.8929		0.28	
2380	EN16711-2	2.982		0.60	
2381	EN16711-2	2.89		0.27	
2382	EN16711-2	2.86		0.17	
2385	EN16711-2	3.1		1.02	
2423	EN16711-2	3.028		0.77	
2426	EN16711-2	2.61		-0.72	
2442	EN16711-2	2.904		0.32	
2456	EN16711-2/EN11885	2.743		-0.25	
2472	GB/T17593	2.72		-0.33	
2486	EN16711-2	2.6933		-0.42	
2511	EN16711-2	3.04		0.81	
2549	EN16711-2	2.860		0.17	
2582	EN16711-2	2.69		-0.44	
2590	EN16711-2	2.70		-0.40	
2602	EN16711-2	2.94		0.45	
2617	EN16711-2	2.808		-0.02	
2618	EN16711-2	Not Detected		-----	possibly a false negative test result?
2637	EN16711-2	3.15		1.20	
2638	EN16711-2	2.796		-0.06	
2644	EN16711-2	3.03		0.77	
2649	EN16711-2	2.95		0.49	
2650	EN16711-2	2.6		-0.76	
2678	EN16711-2	2.828		0.05	
2793	EN16711-2	2.8232	C	0.04	first reported 2.1837
2826	EN16711-2	2.93		0.42	
2827	EN16711-2	2.831		0.06	
2858	EN16711-2	2.961		0.53	
2912	EN16711-2	2.990		0.63	
2947	OEKO-TEX St201 M-10&ML-10	2.869		0.20	
2953	EN16711-2	2.766		-0.17	
2955	EN16711-2	2.8		-0.05	
2977	EN16711-2	3.112		1.06	
2984		2.2433	C	-2.02	first reported 2.0253
2989	ISO105E04	2.8651		0.19	
3010		-----		-----	
3100	EN16711-2	2.823		0.04	
3116		2.737		-0.27	
3118	EN16711-2	2.5213		-1.04	
3146		3.139		1.16	
3154	EN16711-2	2.76		-0.19	
3166	In house	2.47		-1.22	

lab	method	value	mark	z(targ)	remarks
3172	EN16711-2	2.825		0.04	
3190	EN16711-2	2.9		0.31	
3197	EN16711-2	2.59		-0.79	
3210	EN16711-2	2.72		-0.33	
3228	EN16711-2	2.98		0.59	
3233	EN16711-2	2.82		0.03	
3237	EN16711-2	3.03		0.77	
3248	EN16711-2	2.8		-0.05	
6191		-----		-----	
	normality	suspect			
	n	81			
	outliers	0			
	mean (n)	2.8128			
	st.dev. (n)	0.19346	RSD = 7%		
	R(calc.)	0.5417			
	st.dev.(EN16711-2:15)	0.28128			
	R(EN16711-2:15)	0.7876			





APPENDIX 2 Other reported elements

Other reported elements on sample #22750; results in mg/kg

lab	Cd	Cr	Co	Cu	Pb	Hg	Ni
210	not detected	not detected	not detected	not detected	not detected	not detected	not detected
339	not detected	not detected	not detected	not detected	not detected	not detected	0.164
362	<0.05	<0.05	<0.50	<5.00	<0.10	<0.01	<0.50
551	-----	0.071	0.005	0.048	0.135	0.011	-----
623	Not Detected	Not Detected	0.01	Not Detected	Not Detected	Not Detected	0.15
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	0.08	-----	-----	-----	-----	0.10
2159	not determined	not determined	not determined	not determined	not determined	not determined	<0.25
2165	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2215	<0.02	<0.1	<0.1	<5	<0.1	<0.005	<0.1
2261	-----	-----	-----	-----	-----	-----	-----
2264	not detected	< 0.04 mg/kg	not detected	< 0.04 mg/kg	not detected	not detected	< 0.04 mg/kg
2265	-----	0.160	-----	-----	-----	-----	0.246
2271	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2285	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2289	<0.03	<0.5	<0.3	<1.0	<0.1	<0.01	<0.3
2290	<0.05	<0.5	<0.5	<5	<0.1	<0.02	0.148
2297	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2310	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
2320	<0.05	<0.5	<0.5	<5.0	<0.1	<0.01	<0.5
2326	ND	ND	ND	ND	ND	ND	ND
2347	<0.05	<1	<0.5	<5	<0.1	<0.01	<0.5
2350	<0.02	0.1059	<0.1	<5	<0.06	<0.005	<0.1
2352	-----	-----	-----	-----	-----	-----	-----
2357	<0.1	<1	<1	<5	<0.2	<0.02	<0.5
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<0.05	<0.5	<0.5	<5	<0.1	<0.01	<0.5
2365	＜0.1	＜0.5	＜0.5	＜5	＜0.1	＜0.02	＜0.5
2370	<0.1	<0.5	<0.1	<5.0	<0.2	<0.02	<0.5
2372	< 0.1	< 0.5	< 0.1	< 5	< 0.2	< 0.02	< 0.5
2375	<0.03	<0.3	<0.3	<5.0	<0.06	0.015	0.18
2378	＜0.03	＜0.3	＜0.1	＜5.0	＜0.06	＜0.006	＜0.1
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.1596
2380	<0.03	<0.3	<0.3	<5	<0.06	<0.006	<0.1
2381	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2382	＜0.050	＜0.30	＜0.50	＜5.0	＜0.10	＜0.010	＜0.50
2385	<0.05	0.1	<0.1	<0.5	<0.1	<0.01	<0.5
2423	not detected	not detected	not detected	1.548	not detected	not detected	not detected
2426	ND	ND	ND	ND	ND	ND	ND
2442	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2456	-----	-----	-----	-----	-----	-----	-----
2472	<0.06	<0.06	<0.10	<0.6	<0,35	-----	<0.05
2486	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----	-----	-----	-----	-----
2549	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2582	Not detected	Not detected	Not detected	1.92	Not detected	Not detected	Not detected
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2602	not determined	not determined	not determined	not determined	not determined	-----	-----
2617	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2618	Not Detected	Not Detected	Not Detected	29.100	Not Detected	Not Detected	Not Detected
2637	not detected	not detected	not detected	not detected	not detected	not detected	0.15
2638	not detected	not detected	not detected	not detected	not detected	not available	not detected
2644	not detected	0.115	not detected	not detected	not detected	not detected	not detected
2649	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2650	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2678	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2793	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2912	-----	-----	-----	-----	-----	-----	-----
2947	not detected	0.0808	not detected	not detected	0.0482	not detected	0.1675
2953	-----	-----	-----	-----	-----	-----	-----
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	< 0.05	< 0.10	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
2984	-----	-----	-----	0.7396	-----	-----	0.3708
2989	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3010	-----	-----	-----	-----	-----	-----	-----
3100	＜0.03	＜0.5	＜0.3	＜1	＜0.1	＜0.01	＜0.3
3116	<0.02	<0.1	<0.1	<5	<0.1	<0.005	0.132
3118	<0.05	<0.50	<0.25	<0.25	<0.05	<0.01	<0.25
3146	not detected	not detected	not detected	not detected	not detected	not detected	not detected

lab	Cd	Cr	Co	Cu	Pb	Hg	Ni
3154	-----	-----	-----	-----	-----	-----	-----
3166	ND	0.10	0.010	0.056	0.024	0.010	0.142
3172	< 0.02	< 0.1	< 0.1	< 1	< 0.1	0.01	< 0.1
3190	<0.03	<0.5	<0.3	<1.0	<0.1	<0.01	<0.3
3197	<0.1	<0.1	<0.1	<1	<0.1	<0.01	<0.1
3210	<0.1	<1	<1	<5	<0.2	<0.02	<1
3228	<0.02	<0.5	<0.5	<0.5	<0.02	<0.02	<0.5
3233	< 0.05	0.07	< 0.05	0.13	0.06	< 0.01	< 0.05
3237	-----	-----	-----	-----	-----	-----	-----
3248	<0.02	<0.1	<0.1	<5	<0.1	<0.005	0.14
6191	not detected	0.291	-----	-----	not detected	-----	-----

Other reported metals on sample #22751; results in mg/kg

lab	Sb	As	Cd	Cr	Co	Pb	Hg
210	not detected	not detected	not detected	not detected	not detected	not detected	not detected
339	not detected	not detected	not detected	not detected	not detected	not detected	not detected
362	<1.0	<0.10	<0.05	<0.05	<0.50	<0.10	<0.01
551	0.115	-----	-----	0.03	0.02	-----	0.03
623	0.02	0.01	Not Detected	Not Detected	0.01	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	-----	-----	-----	-----	-----
2159	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2165	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2215	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	<0.005
2261	-----	-----	-----	-----	-----	-----	-----
2264	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	0.114	-----	-----	0.018
2271	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2285	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2289	<1.0	<0.1	<0.03	<0.5	<0.3	<0.1	<0.01
2290	<3	<0.1	<0.05	<0.5	<0.5	<0.1	<0.02
2297	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2310	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
2320	<1.0	<0.1	<0.05	<0.5	<0.5	<0.1	<0.01
2326	ND	ND	ND	ND	ND	ND	ND
2347	<1	<0.01	<0.05	<1	<0.5	<0.1	<0.01
2350	<0.5	<0.02	<0.02	<0.1	<0.1	<0.06	<0.005
2352	-----	-----	-----	-----	-----	-----	-----
2357	<1	<0.2	<0.1	<1	<1	<0.2	<0.02
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<1	<0.1	<0.05	<0.5	<0.5	<0.1	<0.01
2365	＜1	＜0.1	＜0.1	＜0.5	＜0.5	＜0.1	＜0.02
2370	<1.0	<0.2	<0.1	<0.5	<0.1	<0.2	<0.02
2372	< 1	< 0.2	< 0.1	< 0.5	< 0.1	< 0.2	< 0.02
2375	<3	<0.06	<0.03	<0.3	<0.3	<0.06	<0.006
2378	＜1.0	＜0.06	＜0.03	＜0.3	＜0.1	＜0.06	＜0.006
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<3	<0.06	<0.03	<0.3	<0.3	<0.06	<0.006
2381	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2382	＜1.00	＜0.10	＜0.050	＜0.30	＜0.50	＜0.10	＜0.010
2385	<0.1	<0.1	<0.05	<0.1	<0.1	<0.1	<0.01
2423	not analyzed	not detected	not detected	not detected	not detected	not detected	not detected
2426	ND	ND	ND	ND	ND	ND	ND
2442	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2456	-----	-----	-----	-----	-----	-----	-----
2472	<0.35	<0.20	<0.06	<0.06	<0.10	<0.35	-----
2486	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----	-----	-----	-----	-----
2549	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2582	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2602	not determined	not determined	not determined	not determined	not determined	not determined	-----
2617	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2618	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2637	not detected	not detected	not detected	not detected	not detected	0.04	not detected
2638	not detected	not detected	not detected	not detected	not detected	not detected	not available
2644	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2649	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2650	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2678	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2793	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2912	-----	-----	-----	-----	-----	-----	-----

lab	Sb	As	Cd	Cr	Co	Pb	Hg
2947	not detected	not detected	not detected	0.0295	not detected	0.0228	not detected
2953	-----	-----	-----	-----	-----	-----	-----
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	0.131	< 0.05	< 0.05	< 0.10	< 0.05	< 0.05	< 0.10
2984	-----	-----	-----	-----	-----	-----	-----
2989	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3010	-----	-----	-----	-----	-----	-----	-----
3100	＜1	＜0.1	＜0.03	＜0.5	＜0.3	＜0.1	＜0.01
3116	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	<0.005
3118	<0.25	<0.05	<0.05	<0.50	<0.25	<0.05	<0.01
3146	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3154	1.25	-----	0.10	-----	-----	-----	-----
3166	0.08	ND	ND	0.04	0.007	0.028	ND
3172	< 1	< 0.02	< 0.02	< 0.1	< 0.1	< 0.1	< 0.005
3190	<1.0	<0.1	<0.03	<0.5	<0.3	<0.1	<0.01
3197	<1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.01
3210	<5	<0.2	<0.1	<1	<1	<0.2	<0.02
3228	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.02
3233	< 0.05	< 0.05	< 0.05	0.06	< 0.05	0.08	< 0.01
3237	-----	-----	-----	-----	-----	-----	-----
3248	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	<0.005
6191	-----	not detected	not detected	not detected	-----	not detected	-----

APPENDIX 3 Analytical Details

lab	ISO/IEC1702 accr.	sample preparation	Sample intake (g)	Ratio gram textile per ml
210	Yes	Further cut		---
339	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
362	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
551	No	Further cut	1g	1 gram textile per 50 mL perspiration liquid
623	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
840	Yes	Used as received	2g	1 gram textile per 50 mL perspiration liquid
2115	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2159	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2165	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2215	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2261	Yes	Used as received	2	1 gram textile per 50 mL perspiration liquid
2264	No	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2265	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2271	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2285	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2289	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2290	Yes	---		---
2297	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2310	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
2311	Yes	Further grinded	1	1 gram textile per 50 mL perspiration liquid
2320	Yes	Used as received	0.5 grams	1 gram textile per 50 mL perspiration liquid
2326	Yes	Further cut	1 GRAM	1 gram textile per 50 mL perspiration liquid
2347	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2350	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2352	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2357	---	---		---
2358	Yes	Used as received	1.0	1 gram textile per 50 mL perspiration liquid
2363	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2365	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2370	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2372	No	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2375	Yes	Further cut	0.6074g, 30mL	1 gram textile per 50 mL perspiration liquid
2378	No	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2379	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
2380	Yes	Further cut	0.5 g	1 gram textile per 50 mL perspiration liquid
2381	Yes	Used as received	1 gm	1 gram textile per 50 mL perspiration liquid
2382	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2385	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2423	No	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2426	Yes	Used as received		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		1 gram textile per 50 mL perspiration liquid
2472	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2486	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2511	---	---		---
2549	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2582	Yes	Used as received	#22750=1.0025, #22751=1.0010	1 gram textile per 50 mL perspiration liquid
2590	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2602	Yes	Used as received	1,0 g	1 gram textile per 50 mL perspiration liquid
2617	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2618	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2637	Yes	Further cut		1 gram textile per 50 mL perspiration liquid
2638	No	Further cut	1 gm	1 gram textile per 50 mL perspiration liquid
2644	Yes	Used as received	0.5 g	1 gram textile per 20 mL perspiration liquid
2649	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2650	Yes	Used as received	2g for each sample	1 gram textile per 50 mL perspiration liquid
2678	Yes	Used as received	1gram	1 gram textile per 50 mL perspiration liquid
2793	No	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2826	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2827	Yes	Further cut	0.5g	1 gram textile per 50 mL perspiration liquid
2858	Yes	Used as received	1.0 gm	1 gm textile per 20 mL perspiration liquid
2912	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2947	No	Used as received	1	1 gram textile per 20 mL perspiration liquid
2953	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
2955	Yes	Used as received	1gm	1 gram textile per 50 mL perspiration liquid
2977	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
2984	Yes	Used as received	#22750=1.0033, #22751=1.0016	1 gram textile per 50 mL perspiration liquid
2989	---	---		---
3010	---	---		---
3100	Yes	Further cut	1g	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 gram C	1 gram textile per 50 mL perspiration liquid
3146	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
3154	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
3166	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid

lab	ISO/IEC1702 accr.	sample preparation	Sample intake (g)	Ratio gram textile per ml
3172	Yes	---		---
3190	Yes	Used as received	0.5g	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	1	1 gram textile per 50 mL perspiration liquid
3228	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
3233	No	Further cut	#22751=0.5158, #22750=0.5265	1 gram textile per 50 mL perspiration liquid
3237	Yes	Used as received	1 gr	1 gram textile per 50 mL perspiration liquid
3248	Yes	Used as received	1.0000	1 gram textile per 50 mL perspiration liquid
6191	No	Used as received	1 g	1 gram textile per 50 mL perspiration liquid

Lab 3118 first reported a sample intake of 5 gram

APPENDIX 4

Number of participants per country

1 lab in	AUSTRIA
8 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
4 labs in	FRANCE
6 labs in	GERMANY
4 labs in	HONG KONG
4 labs in	INDIA
4 labs in	INDONESIA
8 labs in	ITALY
1 lab in	KOREA, Republic of
1 lab in	MOROCCO
19 labs in	P.R. of CHINA
4 labs in	PAKISTAN
1 lab in	PERU
1 lab in	SERBIA
1 lab in	SPAIN
2 labs in	SRI LANKA
2 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TUNISIA
4 labs in	TURKEY
1 lab in	U.S.A.
2 labs in	VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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