Results of Proficiency Test Heavy Metals by Perspiration in textile November 2020

Organized 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 Heavy Metals by Perspiration in textile every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of perspirated heavy metals in textile.
In this interlaboratory study 86 laboratories from 26 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report the results of this 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 respectively labelled #20700 and #20701 of 3 grams each. The samples were artificially fortified with different Elements.
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 dark brownish red cotton positive on some metals was selected. After cutting and homogenization 100 plastic bags were filled with approximately 3 grams each and labelled #20700.
The homogeneity of the subsamples was checked by the determination of perspirated Copper according to an in house method on 8 stratified randomly selected subsamples.

	Perspirated Copper in mg/kg
Sample #20700-1	20.2
Sample #20700-2	18.8
Sample #20700-3	20.9
Sample #20700-4	20.6
Sample #20700-5	21.3
Sample #20700-6	20.2
Sample #20700-7	21.3
Sample #20700-8	21.0

Table 1: homogeneity test results of subsamples #20700

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Perspirated Copper in mg/kg
r (observed)	2.3
reference test method	EN16711-2:15
0.3 x R (reference test method)	2.8

Table 2: evaluation of the repeatability of subsamples #20700

The calculated repeatability was in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of light blue cotton positive on some metals was selected. After cutting and homogenization 100 plastic bags were filled with approximately 3 grams each and labelled #20701.
The homogeneity of the subsamples was checked by the determination of perspirated Copper and Nickel according to an in house method on 10 stratified randomly selected subsamples.

	Perspirated Copper in mg/kg	Perspirated Nickel in mg/kg
Sample #20701-1	85.5	20.8
Sample #20701-2	83.1	21.1
Sample #20701-3	80.2	20.4
Sample #20701-4	84.9	21.5
Sample #20701-5	83.0	21.2
Sample #20701-6	84.8	22.2
Sample #20701-7	80.4	20.7
Sample #20701-8	79.9	20.7
Sample #20701-9	85.0	21.5
Sample #20701-10	85.9	21.8

Table 3 homogeneity test results of subsamples #20701

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Perspirated Copper in mg/kg	Perspirated Nickel in mg/kg
r (observed)	6.6	1.6
reference test method	EN16711-2:15	EN16711-2:15
0.3 x R (reference test method)	11.2	1.8

Table 4: evaluation of the repeatabilities of subsamples #20701

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one subsample of #20700 and one subsample of #20701 were sent on October 7, 2020.

2.5 ANALYZES

The participants were requested to determine on samples #20700 and #20701 the 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.

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

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

3.1 STATISTICS

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers,

this check was repeated. If a data set 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. 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. 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.
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 interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. One participant reported the test results after this period and seven participants did not report any test results. Not all participants were able to report all elements requested.
Finally, the 79 reporting laboratories reported 314 numerical test results. Observed were 11 outlying results, which is 3.5% 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 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 perspirated from 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 standard deviation (or variation coefficient) of the metal with 2.8.

Sample #20700

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

Nickel: The determination of Nickel was not problematic. Three statistical outliers were observed. 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 for the other elements a content close to or below the quantification limits. Therefore, no z-scores were calculated. The test results are given in appendix 2.

Sample #20701

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

Nickel: The determination of Nickel was not problematic. Three statistical outliers were observed. 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 for the other elements a content close to or below the quantification limits. 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 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 test method (in casu EN16711-2) are presented in the next two tables.

Component	unit	n	average	2.8 * sd	R(lit)
Copper as Cu	mg/kg	79	29.2	10.6	13.1
Nickel as Ni	mg/kg	74	2.8	0.8	0.8

Table 5: reproducibilities of perspirated metals in sample #20700

Component	unit	n	average	2.8 * sd	R(lit)
Copper as Cu	mg/kg	74	74.0	17.3	33.1
Nickel as Ni	mg/kg	76	17.7	4.8	4.9

Table 6: reproducibilities of perspirated metals in sample #20701

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 2020 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 2020	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%	--	9-14%	10%
Chromium as Cr	--	11%	10%	13%	12%	15-19%	15%
Cobalt as Co	--	--	--	9%	13%	8-14%	13%
Copper as Cu	8-13%	--	--	--	10%	9-22%	16%
Lead as Pb	--	--	--	40%	35%	--	40%
Mercury as Hg	--	--	34%	--	--	41%	31%
Nickel as Ni	10%	11%	--	--	--	7-14%	10%

Table 7: development of uncertainties over the last years

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 85% of the reporting laboratories mentioned to be accredited for the determination of Heavy Metals by Perspiration in textile.
It was requested to report if the sample was further cut/grinded and the amount of sample intake. It appeared that no effect was observed on the reported test results for the determined metals in sample #20700 nor in sample #20701 because the observed reproducibilities are in line with the reference test method EN16711-2:15.

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 averages of the homogeneity test results are not complete in line with the averages (consensus values) 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 #20700 was also used in a previous proficiency test iis15A04 as sample #15206. The obtained PT results are in line with the previous PT (see table 8). Therefore, it is concluded that the samples textile containing Copper and Nickel are stable for 5 years at least.

Component	unit	Sample #20700			Sample #15206		
		n	average	2.8 * sd	n	average	2.8 * sd
Copper as Cu	mg/kg	79	29.2	10.6	82	30.0	7.7
Nickel as Ni	mg/kg	74	2.8	0.8	80	2.8	0.9

Table 8: comparison sample #20700 vs #15206

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 9).

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

Table 9: Ecolabelling Standards and Requirements for Textiles in EU

6 CONCLUSION

In this proficiency test the Heavy Metals 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.

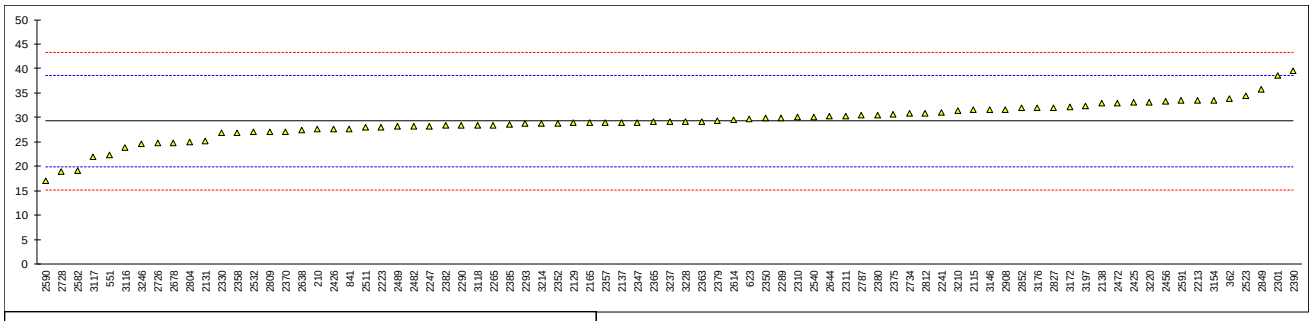
A possible explanation for the variation could be the preparation or the conditioning of the sample and/or by the performance of the analysis by the laboratory. Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

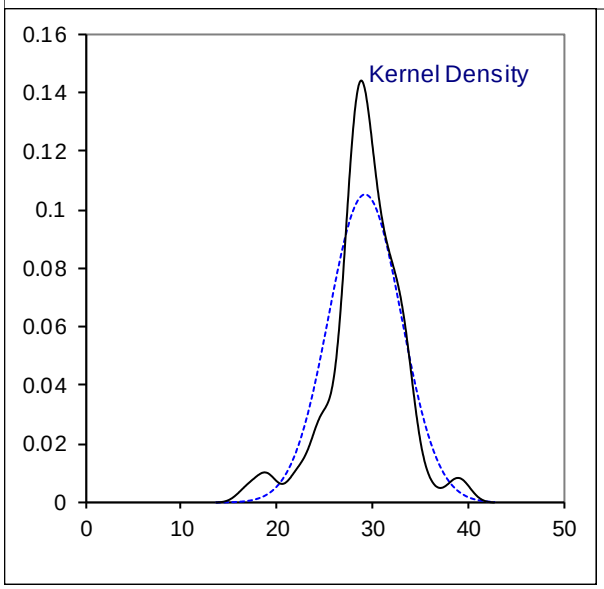
APPENDIX 1

Determination of Copper as Cu on sample #20700; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO105E04	27.55		-0.36	
339		-----		-----	
348		-----		-----	
362	In house	33.8		0.98	
551	EN16711-2	22.3999	C	-1.46	First reported 46.9852
623	EN16711-2	29.63		0.08	
841	EN16711-2	27.7		-0.33	
2115	EN16711-2	31.60		0.51	
2129	EN16711-2	28.86		-0.08	
2131	In house	25.1455		-0.87	
2137	KS K0731	28.942		-0.06	
2138	EN16711-2	32.9		0.78	
2165	EN16711-2	28.88		-0.08	
2213		33.49		0.91	
2223	EN16711-2	28.03		-0.26	
2241	EN16711-2	31.095		0.40	
2247	EN16711-2	28.26		-0.21	
2265	EN16711-2	28.40		-0.18	
2289	DIN54233-3	29.81		0.12	
2290	EN16711-2	28.36		-0.19	
2293	EN16711-2	28.66		-0.12	
2301	EN16711-2	38.48		1.98	
2310	EN16711-2	30		0.16	
2311	EN16711-2	30.284		0.22	
2330	ISO105E04	26.9230		-0.49	
2347	EN16711-2	29		-0.05	
2350	EN16711-2	29.8025		0.12	
2352	EN16711-2	28.703		-0.11	
2357	EN16711-2	28.94		-0.06	
2358	EN16711-2	26.95		-0.49	
2363	EN16711-2	29.2		-0.01	
2365	EN16711-2	29.041		-0.04	
2370	ISO105E04	27.1		-0.46	
2375	EN16711-2	30.62		0.30	
2379	EN16711-2	29.3279		0.02	
2380	ISO105E04	30.447		0.26	
2382	EN16711-2	28.35		-0.19	
2385	EN16711-2	28.5		-0.16	
2390	ISO105E04	39.44		2.18	
2425	EN16711-2	33.17		0.84	
2426	EN16711-2	27.607		-0.35	
2429		-----		-----	
2442		-----		-----	
2456	EN16711-2	33.29		0.87	
2472	GB/T17593	32.90		0.78	
2482	EN16711-2	28.2		-0.22	
2489	EN16711-2	28.1		-0.24	
2511	EN16711-2	27.97		-0.27	
2523	ISO105-E04/EPA200.7	34.3400		1.09	
2532	EN16711-2	27.0		-0.48	
2540	EN16711-2	30.0426		0.17	
2582	EN16711-2	19.10		-2.17	
2590	EN16711-2	17.009	C	-2.61	First reported 14.7051
2591	EN16711-2	33.431		0.90	
2614	EN16711-2	29.51		0.06	
2638	EN16711-2	27.42		-0.39	
2644	EN16711-2	30.20		0.21	
2678		24.787		-0.95	
2726	EN16711-2	24.708		-0.97	
2728	In house	18.89		-2.21	
2734	EN16711-2	30.783		0.33	
2787	EN16711-2	30.3731		0.24	
2804	EN16711-2	24.9294		-0.92	
2809	ISO105E04	27.0333		-0.47	
2812	EN16711-2	30.92		0.36	
2827	EN16711-2	32.02		0.60	
2849	EN16711-2	35.744		1.39	
2852	EN16711-2	31.89		0.57	
2908	GB/T17593	31.63		0.51	
2946		-----		-----	
3116	EN16711-2	23.9		-1.14	
3117	EN16711-2	22.025		-1.54	
3118	EN16711-2	28.37		-0.18	

lab	method	value	mark	z(targ)	remarks
3146	EN16711-2	31.6		0.51	
3154	EN16711-2	33.50		0.91	
3172	EN16711-2	32.09		0.61	
3176	DIN54233-3	31.98		0.59	
3190		-----		-----	
3197	EN16711-2	32.31		0.66	
3210	EN16711-2	31.418		0.47	
3214	EN16711-2	28.68		-0.12	
3220	EN16711-2	33.183		0.84	
3228	EN16711-2	29.1		-0.03	
3237	EN16711-2	29.09		-0.03	
3246	EN16711-2	24.623		-0.99	
	normality	suspect			
	n	79			
	outliers	0			
	mean (n)	29.234			
	st.dev. (n)	3.7843	RSD = 13%		
	R(calc.)	10.596			
	st.dev.(EN16711-2:15)	4.6774			
	R(EN16711-2:15)	13.097			

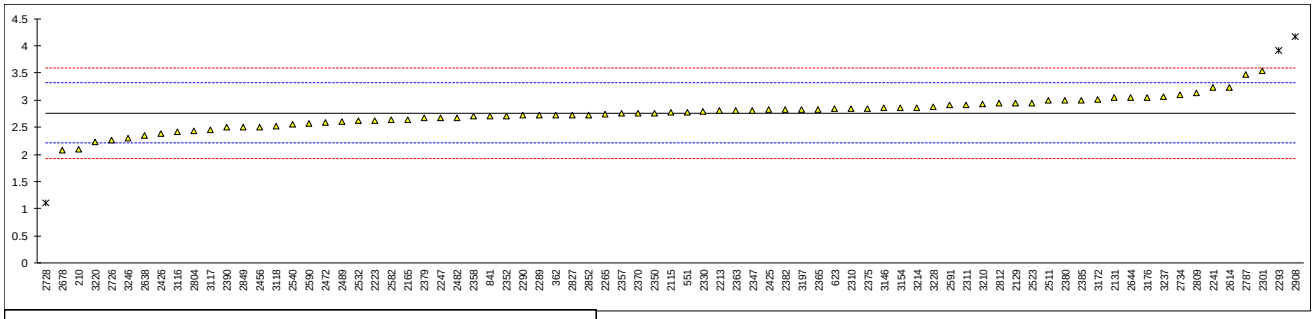


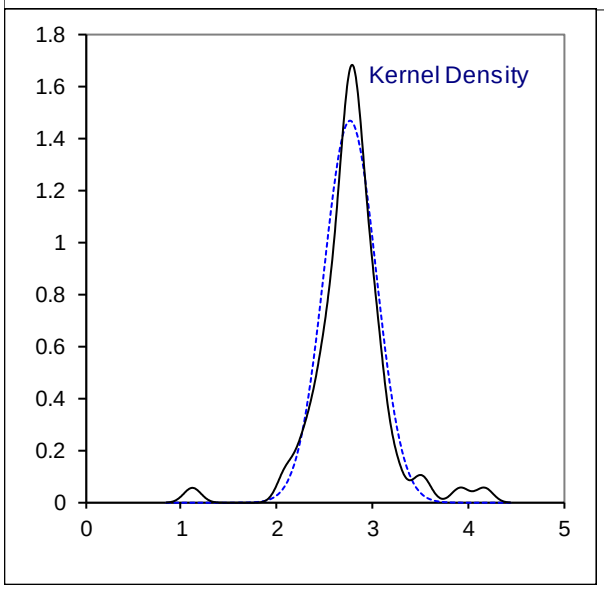


Determination of Nickel as Ni on sample #20700; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO105E04	2.10		-2.39	
339		-----		-----	
348		-----		-----	
362	In house	2.73		-0.11	
551	EN16711-2	2.7814	C	0.08	First reported 3.774
623	EN16711-2	2.84		0.29	
841	EN16711-2	2.7		-0.21	
2115	EN16711-2	2.78		0.08	
2129	EN16711-2	2.945		0.67	
2131	In house	3.046		1.04	
2137		-----		-----	
2138		-----	W	-----	Test result with drawn, reported Not Detected
2165	EN16711-2	2.631		-0.46	
2213		2.80		0.15	
2223	EN16711-2	2.628		-0.48	
2241	EN16711-2	3.240		1.74	
2247	EN16711-2	2.68		-0.29	
2265	EN16711-2	2.74		-0.07	
2289	DIN54233-3	2.73		-0.11	
2290	EN16711-2	2.72		-0.14	
2293	EN16711-2	3.913	R(0.01)	4.18	
2301	EN16711-2	3.54		2.83	
2310	EN16711-2	2.84		0.29	
2311	EN16711-2	2.908		0.54	
2330	ISO105E04	2.7972		0.14	
2347	EN16711-2	2.8		0.15	
2350	EN16711-2	2.763		0.01	
2352	EN16711-2	2.712		-0.17	
2357	EN16711-2	2.75		-0.03	
2358	EN16711-2	2.7		-0.21	
2363	EN16711-2	2.8		0.15	
2365	EN16711-2	2.824		0.23	
2370	ISO105E04	2.76		0.00	
2375	EN16711-2	2.85		0.33	
2379	EN16711-2	2.6680		-0.33	
2380	ISO105E04	3.00		0.87	
2382	EN16711-2	2.82		0.22	
2385	EN16711-2	3		0.87	
2390	ISO105E04	2.50		-0.94	
2425	EN16711-2	2.82		0.22	
2426	EN16711-2	2.387		-1.35	
2429		-----		-----	
2442		-----		-----	
2456	EN16711-2	2.51		-0.90	
2472	GB/T17593	2.59		-0.61	
2482	EN16711-2	2.68		-0.29	
2489	EN16711-2	2.6		-0.58	
2511	EN16711-2	2.99		0.84	
2523	ISO105-E04/EPA200.7	2.9450		0.67	
2532	EN16711-2	2.62		-0.50	
2540	EN16711-2	2.5485		-0.76	
2582	EN16711-2	2.63		-0.47	
2590	EN16711-2	2.5762		-0.66	
2591	EN16711-2	2.903		0.52	
2614	EN16711-2	3.24		1.74	
2638	EN16711-2	2.35		-1.48	
2644	EN16711-2	3.05		1.05	
2678		2.075		-2.48	
2726	EN16711-2	2.2598		-1.81	
2728	In house	1.11	R(0.01)	-5.98	
2734	EN16711-2	3.098		1.23	
2787	EN16711-2	3.4647		2.56	
2804	EN16711-2	2.4423		-1.15	
2809	ISO105E04	3.1367		1.37	
2812	EN16711-2	2.94		0.65	
2827	EN16711-2	2.73		-0.11	
2849	EN16711-2	2.502		-0.93	
2852	EN16711-2	2.73		-0.11	
2908	GB/T17593	4.1636	R(0.01)	5.09	
2946		-----		-----	
3116	EN16711-2	2.41		-1.27	
3117	EN16711-2	2.455		-1.10	
3118	EN16711-2	2.52		-0.87	
3146	EN16711-2	2.86		0.37	

lab	method	value	mark	z(targ)	remarks
3154	EN16711-2	2.86	C	0.37	First reported 5.20
3172	EN16711-2	3.01		0.91	
3176	DIN54233-3	3.05		1.05	
3190		-----		-----	
3197	EN16711-2	2.82		0.22	
3210	EN16711-2	2.926		0.60	
3214	EN16711-2	2.86		0.37	
3220	EN16711-2	2.235		-1.90	
3228	EN16711-2	2.87		0.40	
3237	EN16711-2	3.07		1.13	
3246	EN16711-2	2.299		-1.67	
	normality	OK			
	n	74			
	outliers	3			
	mean (n)	2.759			
	st.dev. (n)	0.2724	RSD = 10%		
	R(calc.)	0.763			
	st.dev.(EN16711-2:15)	0.2759			
	R(EN16711-2:15)	0.773			

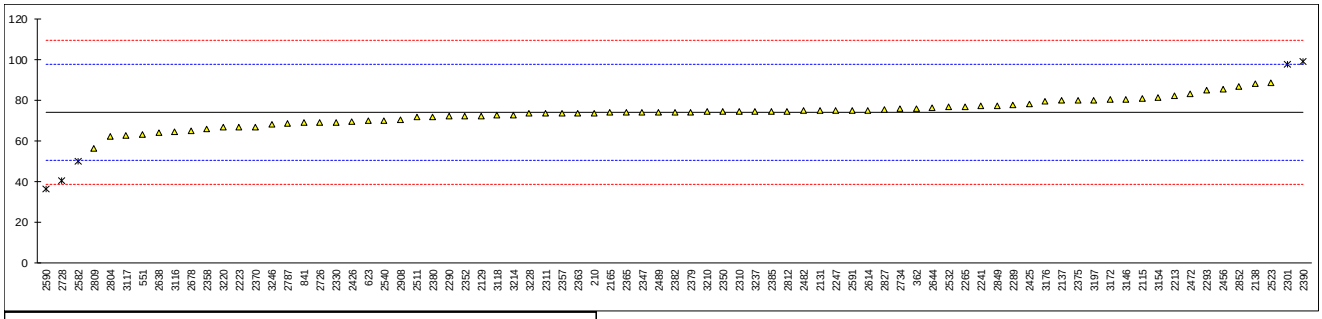


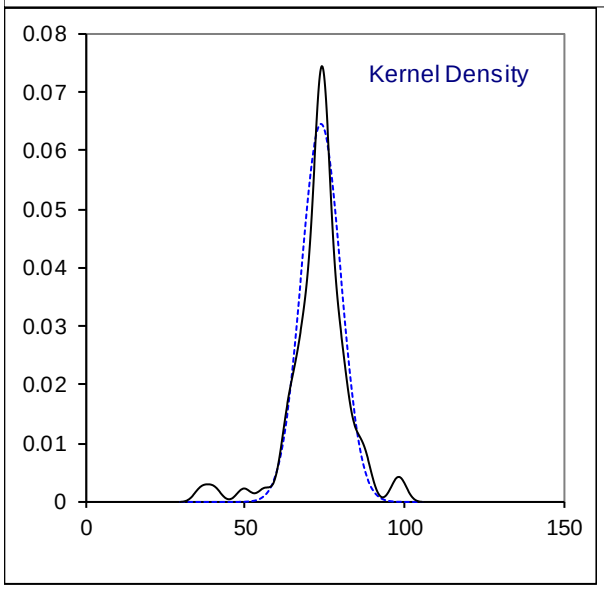


Determination of Copper as Cu on sample #20701; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO105E04	73.75		-0.02	
339		-----		-----	
348		-----		-----	
362	In house	75.8		0.16	
551	EN16711-2	63.2735	C	-0.90	First reported 168.209
623	EN16711-2	69.75		-0.36	
841	EN16711-2	68.9		-0.43	
2115	EN16711-2	80.97		0.59	
2129	EN16711-2	72.12		-0.16	
2131	In house	74.9715		0.09	
2137	KS K0731	79.669		0.48	
2138	EN16711-2	88.1		1.19	
2165	EN16711-2	73.85		-0.01	
2213		82.22		0.70	
2223	EN16711-2	66.89		-0.60	
2241	EN16711-2	77.233		0.28	
2247	EN16711-2	74.98		0.09	
2265	EN16711-2	76.80		0.24	
2289	DIN54233-3	77.61		0.31	
2290	EN16711-2	71.98		-0.17	
2293	EN16711-2	84.85		0.92	
2301	EN16711-2	97.52	R(0.05)	1.99	
2310	EN16711-2	74.4		0.04	
2311	EN16711-2	73.661		-0.03	
2330	ISO105E04	69.1058		-0.41	
2347	EN16711-2	74		0.00	
2350	EN16711-2	74.304		0.03	
2352	EN16711-2	72.113		-0.16	
2357	EN16711-2	73.69		-0.02	
2358	EN16711-2	66.0		-0.67	
2363	EN16711-2	73.7		-0.02	
2365	EN16711-2	73.996		0.00	
2370	ISO105E04	66.9		-0.60	
2375	EN16711-2	79.7		0.48	
2379	EN16711-2	74.1553		0.02	
2380	ISO105E04	71.795		-0.18	
2382	EN16711-2	74.11		0.01	
2385	EN16711-2	74.5		0.05	
2390	ISO105E04	99.10	R(0.05)	2.12	
2425	EN16711-2	78.10		0.35	
2426	EN16711-2	69.395		-0.39	
2429		-----		-----	
2442		-----		-----	
2456	EN16711-2	85.48		0.97	
2472	GB/T17593	83.09		0.77	
2482	EN16711-2	74.9	C	0.08	First reported 17.6
2489	EN16711-2	74		0.00	
2511	EN16711-2	71.78		-0.18	
2523	ISO105-E04/EPA200.7	88.5100		1.23	
2532	EN16711-2	76.6		0.22	
2540	EN16711-2	69.9070		-0.34	
2582	EN16711-2	49.84	R(0.05)	-2.04	
2590	EN16711-2	36.5308	R(0.01)	-3.16	
2591	EN16711-2	74.994		0.09	
2614	EN16711-2	75.08		0.09	
2638	EN16711-2	63.94		-0.85	
2644	EN16711-2	76.30		0.20	
2678		64.946		-0.76	
2726	EN16711-2	69.0382		-0.42	
2728	In house	40.52	R(0.01)	-2.83	
2734	EN16711-2	75.778		0.15	
2787	EN16711-2	68.6736		-0.45	
2804	EN16711-2	62.2014		-0.99	
2809	ISO105E04	56.4000		-1.48	
2812	EN16711-2	74.50		0.05	
2827	EN16711-2	75.44		0.12	
2849	EN16711-2	77.242		0.28	
2852	EN16711-2	86.51		1.06	
2908	GB/T17593	70.2442		-0.31	
2946		-----		-----	
3116	EN16711-2	64.3		-0.82	
3117	EN16711-2	62.862		-0.94	
3118	EN16711-2	72.65		-0.11	
3146	EN16711-2	80.4		0.54	

lab	method	value	mark	z(targ)	remarks
3154	EN16711-2	81.16		0.61	
3172	EN16711-2	80.35		0.54	
3176	DIN54233-3	79.48		0.47	
3190		-----		-----	
3197	EN16711-2	79.83		0.50	
3210	EN16711-2	74.262		0.03	
3214	EN16711-2	72.77		-0.10	
3220	EN16711-2	66.620		-0.62	
3228	EN16711-2	73.4		-0.05	
3237	EN16711-2	74.40		0.04	
3246	EN16711-2	68.064		-0.50	
	normality	OK			
	n	74			
	outliers	5			
	mean (n)	73.965			
	st.dev. (n)	6.1693	RSD = 8%		
	R(calc.)	17.274			
	st.dev.(EN16711-2:15)	11.8345			
	R(EN16711-2:15)	33.137			

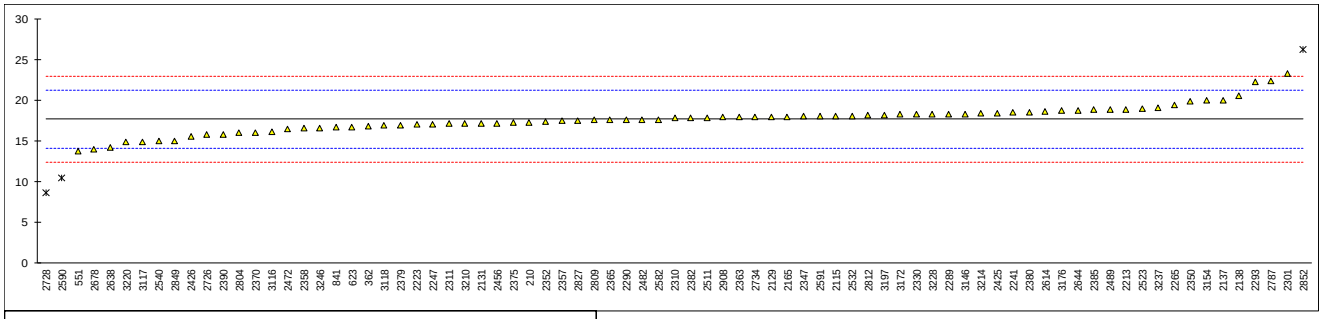


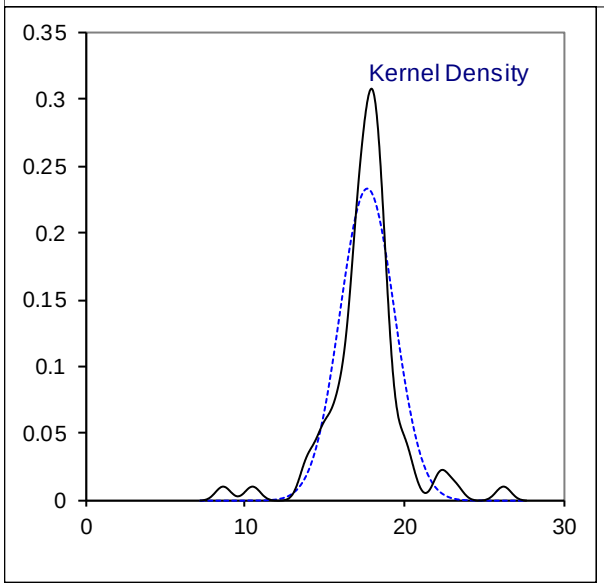


Determination of Nickel as Ni on sample #20701; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	ISO105E04	17.23		-0.25	
339		-----		-----	
348		-----		-----	
362	In house	16.8		-0.49	
551	EN16711-2	13.7226	C	-2.23	First reported 28.2423
623	EN16711-2	16.72		-0.53	
841	EN16711-2	16.7		-0.55	
2115	EN16711-2	18.07		0.23	
2129	EN16711-2	17.94		0.16	
2131	In house	17.1825		-0.27	
2137	KS K0731	20.013		1.33	
2138	EN16711-2	20.5		1.61	
2165	EN16711-2	17.98		0.18	
2213		18.89		0.69	
2223	EN16711-2	16.98		-0.39	
2241	EN16711-2	18.459		0.45	
2247	EN16711-2	17.02		-0.36	
2265	EN16711-2	19.45		1.01	
2289	DIN54233-3	18.29		0.35	
2290	EN16711-2	17.58		-0.05	
2293	EN16711-2	22.24		2.59	
2301	EN16711-2	23.2		3.13	
2310	EN16711-2	17.81		0.08	
2311	EN16711-2	17.158		-0.29	
2330	ISO105E04	18.2317		0.32	
2347	EN16711-2	18		0.19	
2350	EN16711-2	19.8972		1.26	
2352	EN16711-2	17.332		-0.19	
2357	EN16711-2	17.43		-0.13	
2358	EN16711-2	16.6		-0.60	
2363	EN16711-2	17.9		0.13	
2365	EN16711-2	17.536		-0.07	
2370	ISO105E04	16.0		-0.94	
2375	EN16711-2	17.2		-0.26	
2379	EN16711-2	16.9429		-0.41	
2380	ISO105E04	18.518		0.48	
2382	EN16711-2	17.81		0.08	
2385	EN16711-2	18.8		0.64	
2390	ISO105E04	15.78		-1.07	
2425	EN16711-2	18.43		0.43	
2426	EN16711-2	15.493		-1.23	
2429		-----		-----	
2442		-----		-----	
2456	EN16711-2	17.19		-0.27	
2472	GB/T17593	16.42		-0.70	
2482	EN16711-2	17.6		-0.04	
2489	EN16711-2	18.8		0.64	
2511	EN16711-2	17.83		0.09	
2523	ISO105-E04/EPA200.7	18.9400		0.72	
2532	EN16711-2	18.08		0.24	
2540	EN16711-2	14.9414		-1.54	
2582	EN16711-2	17.62		-0.02	
2590	EN16711-2	10.448	C,R(0.01)	-4.09	First reported 8.6856
2591	EN16711-2	18.007		0.19	
2614	EN16711-2	18.62		0.54	
2638	EN16711-2	14.16		-1.98	
2644	EN16711-2	18.76		0.62	
2678		13.946		-2.10	
2726	EN16711-2	15.7541		-1.08	
2728	In house	8.60	R(0.01)	-5.13	
2734	EN16711-2	17.922		0.15	
2787	EN16711-2	22.340		2.65	
2804	EN16711-2	15.955		-0.97	
2809	ISO105E04	17.5333		-0.07	
2812	EN16711-2	18.20		0.30	
2827	EN16711-2	17.51		-0.09	
2849	EN16711-2	15.004		-1.51	
2852	EN16711-2	26.23	R(0.01)	4.85	
2908	GB/T17593	17.8741		0.12	
2946		-----		-----	
3116	EN16711-2	16.1		-0.89	
3117	EN16711-2	14.882		-1.57	
3118	EN16711-2	16.93		-0.42	
3146	EN16711-2	18.3		0.36	

lab	method	value	mark	z(targ)	remarks
3154	EN16711-2	19.95		1.29	
3172	EN16711-2	18.22		0.31	
3176	DIN54233-3	18.71		0.59	
3190		-----		-----	
3197	EN16711-2	18.20		0.30	
3210	EN16711-2	17.17		-0.28	
3214	EN16711-2	18.41		0.42	
3220	EN16711-2	14.818		-1.61	
3228	EN16711-2	18.26		0.34	
3237	EN16711-2	19.04		0.78	
3246	EN16711-2	16.611		-0.60	
	normality	suspect			
	n	76			
	outliers	3			
	mean (n)	17.664			
	st.dev. (n)	1.7087	RSD = 10%		
	R(calc.)	4.784			
	st.dev.(EN16711-2:15)	1.7664			
	R(EN16711-2:15)	4.946			





APPENDIX 2

Reported test results of Antimony, Arsenic, Cadmium, Chromium, Cobalt, Lead, Manganese, Mercury and Zinc on sample #20700; results in mg/kg

lab	Sb	As	Cd	Cr	Co	Pb	Mn	Hg	Zn
110	-----	-----	-----	-----	-----	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	-----	-----	-----	-----	-----	-----	-----	-----
348	-----	-----	-----	-----	-----	-----	-----	-----	-----
362	-----	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	2.1161	-----	0.1443	0.1643	-----	0.4581	0.2141	1.2249
623	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	1.38
841	<0.2	<0.025	<0.025	<0.1	<0.1	<0.1	<5	<0.005	<5
2115	-----	-----	-----	-----	-----	-----	1.12	-----	1.07
2129	-----	-----	-----	-----	-----	-----	N.A.	-----	N.A.
2131	<0.1	<0.02	<0.02	<0.02	<0.02	<0.02	<0.40	<0.01	9.4705
2137	-----	-----	-----	-----	-----	-----	-----	-----	-----
2138	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2165	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2213	<5	<0.05	<0.02	<0.1	<0.1	<0.1	<10	<0.01	<10
2223	<0.5	<0.05	<0.05	<0.05	<0.05	<0.05	<0.5	<0.05	0.9687
2241	0.016	0.004	0.002	0.038	0.005	0.036	-----	0.000	-----
2247	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2265	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2289	<1.0	<0.1	<0.03	<0.5	<0.3	<0.1	<1.0	<0.01	<1.0
2290	<1	<0.1	<0.03	<0.5	<0.3	<0.1	<5	<0.01	<5
2293	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	3.441
2301	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2310	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	<5
2311	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2330	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	N.A.
2347	<1	<0.1	<0.05	<1	<0.5	<0.1	<0.5	<0.010	<1
2350	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	-----	<0.005	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	<1	<0.2	<0.1	<1	<1	<0.2	-----	<0.02	-----
2358	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	n.d.	N.A.
2363	<1.0	<0.1	<0.05	<0.5	<0.5	<0.1	N.A.	<0.01	N.A.
2365	<1.0	<0.1	<0.05	<1.0	<0.5	<0.1	<0.5	<0.01	<1.0
2370	<1	<0.2	<0.1	<0.5	<0.1	<0.2	<1	<0.02	<1
2375	<0.05	<0.002	<0.002	<0.05	<0.05	<0.05	<0.5	<0.005	1.18
2379	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2380	<3	<0.06	<0.03	<0.3	<0.3	<0.06	-----	<0.006	-----
2382	<1.0	<0.10	<0.050	<0.50	<0.50	<0.10	<10.0	<0.010	<10.0
2385	<0,1	<0,1	<0,05	<0,1	<0,1	<0,1	0.23	<0,01	0.87
2390	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2425	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
2426	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	N.A.
2429	-----	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----	-----
2456	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	<0.35	-----	<0.06	<0.06	<0.10	<0.35	-----	-----	-----
2482	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	0.184	< 0,025	-----
2489	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2523	not det.	not det.	not det.	not det.	not det.	not det.	0.1753	not det.	0.7447
2532	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2540	-----	-----	-----	-----	-----	-----	-----	-----	-----
2582	not det.	not det.	not det.	not det.	not det.	0.18	0.18	not det.	1.68
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	-----	< L.O.Q.
2591	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2614	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2638	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----
2678	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
2726	-----	-----	-----	-----	-----	-----	-----	-----	-----
2728	-----	-----	0.00	-----	-----	0.00	-----	-----	-----
2734	not det.	not det.	not det.	not det.	not det.	not det.	2.396	not det.	not det.
2787	<LOD	<LOD	8.7594	0.05526	0.00324	<LOD	-----	0.09014	-----
2804	<1	<0.05	<0.02	<0.1	<0.3	<0.1	-----	<0.005	-----
2809	-----	-----	-----	-----	-----	-----	-----	-----	-----
2812	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	N.A.
2827	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2849	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	8.004
2852	<0.1	<0.1	<0.1	<0.1	<0.5	<0.1	<0.1	<0.02	<0.1
2908	0.0514	not det.	0.0173	not det.	not det.	0.3756	not det.	not det.	not det.
2946	-----	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3117	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	Sb	As	Cd	Cr	Co	Pb	Mn	Hg	Zn
3118	<0.25	<0.05	<0.05	<0.5	<0.25	<0.25	-----	<0.01	-----
3146	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	0.831
3154	0.68	0.022	-----	-----	-----	0.058	0.21	0.015	1.14
3172	< 5.0	< 0.05	< 0.02	< 0.1	< 0.1	< 0.1	--	< 0.01	--
3176	-----	-----	-----	-----	-----	-----	-----	-----	1.42
3190	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<1	<0,1	<0,1	<0,1	<0,1	<0,1	<1	<0,01	<2
3210	<5	<0.2	<0.1	<1	<1	<0.2	<5	<0.02	<5
3214	<1	<0.1	<0.03	<0.5	<0.3	<0.1	<0.3	<0.01	<5
3220	not det.	not det.	not det.	not det.	not det.	not det.	0.189	not det.	0.634
3228	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.5	<0.02	<1
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----
3246	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	0.501

Reported test results of Antimony, Arsenic, Cadmium, Chromium, Cobalt, Lead, Manganese, Mercury and Zinc on sample #20701; results in mg/kg

lab	Sb	As	Cd	Cr	Co	Pb	Mn	Hg	Zn
110	-----	-----	-----	-----	-----	-----	-----	-----	-----
210	-----	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	-----	-----	-----	-----	-----	-----	-----	-----
348	-----	-----	-----	-----	-----	-----	-----	-----	-----
362	-----	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	2.9142	-----	0.14471	-----	-----	0.96307	0.09953	1.712
623	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	1.50
841	<0.2	<0.025	<0.025	<0.1	<0.1	<0.1	<5	<0.005	<5
2115	-----	-----	-----	-----	-----	-----	1.06	-----	1.10
2129	-----	-----	-----	-----	-----	-----	N.A.	-----	N.A.
2131	<0.1	<0.02	<0.02	<0.02	<0.02	<0.02	<0.40	<0.01	10.1445
2137	-----	-----	-----	-----	-----	-----	-----	-----	-----
2138	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2165	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2213	<5	<0.05	<0.02	<0.1	<0.1	<0.1	<10	<0.01	<10
2223	<0.5	<0.05	<0.05	<0.05	<0.05	<0.05	<0.5	<0.05	1.113
2241	0.017	0.002	0.004	0.009	0.004	0.011	-----	0.001	-----
2247	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2265	-----	-----	-----	-----	-----	-----	-----	-----	-----
2289	<1.0	<0.1	<0.03	<0.5	<0.3	<0.1	<1.0	<0.01	<1.0
2290	<1	<0.1	<0.03	<0.5	<0.3	<0.1	<5	<0.01	<5
2293	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	1.718
2301	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2310	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	<5
2311	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2330	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	N.A.
2347	<1	<0.1	<0.05	<1	<0.5	<0.1	<0.5	<0.01	<1
2350	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	-----	<0.005	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	<1	<0.2	<0.1	<1	<1	<0.2	-----	<0.02	-----
2358	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	N.A.
2363	<1	<0.1	<0.05	<0.5	<0.5	<0.1	N.A.	<0.01	N.A.
2365	<1.0	<0.1	<0.05	<1.0	<0.5	<0.1	<0.5	<0.01	<1.0
2370	<1	<0.2	<0.1	<0.5	<0.1	<0.2	<1	<0.02	<1
2375	<0.05	<0.002	<0.002	<0.05	<0.05	<0.05	<0.5	<0.005	1.12
2379	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2380	<3	<0.06	<0.03	<0.3	<0.3	<0.06	-----	<0.006	-----
2382	<1.0	<0.10	<0.050	<0.50	<0.50	<0.10	<10.0	<0.010	<10.0
2385	<0,1	<0,1	<0,05	<0,1	<0,1	<0,1	0.44	<0,01	1.04
2390	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2425	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
2426	not det.	not det.	not det.	not det.	not det.	not det.	N.A.	not det.	N.A.
2429	-----	-----	-----	-----	-----	-----	-----	-----	-----
2442	-----	-----	-----	-----	-----	-----	-----	-----	-----
2456	-----	-----	-----	-----	-----	-----	-----	-----	-----
2472	<0.35	-----	<0.06	<0.06	<0.10	<0.35	-----	-----	-----
2482	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	0.348	< 0,025	-----
2489	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2523	not det.	not det.	not det.	not det.	not det.	not det.	0.3228	not det.	1.0430
2532	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2540	-----	-----	-----	-----	-----	-----	-----	-----	-----
2582	not det.	not det.	not det.	not det.	not det.	0.13	0.28	not det.	2.12
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	-----	< L.O.Q.
2591	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2614	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.	-----
2638	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----	not det.
2644	-----	-----	-----	-----	-----	-----	-----	-----	-----
2678	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
2726	-----	-----	-----	-----	-----	-----	-----	-----	-----
2728	-----	-----	0.00	-----	-----	0.00	-----	-----	-----
2734	not det.	not det.	not det.	not det.	not det.	not det.	2.282	not det.	not det.
2787	<LOD	<LOD	0.0076	0.0803	0.03797	-----	-----	<LOD	-----
2804	<1	<0.05	<0.02	<0.1	<0.3	<0.1	-----	<0.005	-----
2809	-----	-----	-----	-----	-----	-----	-----	-----	-----
2812	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	N.A.
2827	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2849	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	9.003
2852	<0.1	<0.1	<0.1	<0.1	<0.5	<0.1	<0.1	<0.02	<0.1
2908	not det.	not det.	not det.	not det.	not det.	0.4018	not det.	not det.	not det.
2946	-----	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3117	-----	-----	-----	-----	-----	-----	-----	-----	-----
3118	<0.25	<0.05	<0.05	<0.5	<0.25	<0.25	-----	<0.01	-----
3146	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	1.01

lab	Sb	As	Cd	Cr	Co	Pb	Mn	Hg	Zn
3154	0.44	0.041	-----	-----	0.012	0.042	0.415	0.010	1.58
3172	< 5.0	< 0.05	< 0.02	< 0.1	< 0.1	< 0.1	--	< 0.01	--
3176	-----	-----	-----	-----	-----	-----	-----	-----	0.99
3190	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	<1	<0,1	<0,1	<0,1	<0,1	<0,1	<1	<0,01	<2
3210	<5	<0.2	<0.1	<1	<1	<0.2	<5	<0.02	<5
3214	<1	<0.1	<0.03	<0.5	<0.3	<0.1	<0.3	<0.01	<5
3220	not det.	not det.	not det.	not det.	not det.	not det.	0.242	not det.	0.625
3228	<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.5	<0.02	<1.0
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----
3246	not det.	not det.	not det.	not det.	not det.	not det.	0.236	not det.	0.629

APPENDIX 3

Analytical Details

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
110	---	---		---
210	No	---		---
339	---	---		---
348	---	---		---
362	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
551	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
623	Yes	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
841	Yes	Further cut	0.5016	1 gram textile per 50 mL perspiration liquid
2115	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
2129	Yes	Further cut	1,0	1 gram textile per 50 mL perspiration liquid
2131	Yes	Further cut	1.5 g	**1 gram textile per 20 mL perspiration liquid**
2137	Yes	Used as received	0.5	1 gram textile per 50 mL perspiration liquid
2138	Yes	Used as received	1.0 g	1 gram textile per 50 mL perspiration liquid
2165	Yes	---	1	1 gram textile per 50 mL perspiration liquid
2213	Yes	Used as received	0.5 gm	1 gram textile per 50 mL perspiration liquid
2223	No	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2241	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2247	Yes	Used as received	1.0	1 gram textile per 50 mL perspiration liquid
2265	Yes	Used as received	3	**1.5 gram textile per 30 mL perspiration liquid**
2289	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2290	Yes	---		---
2293	Yes	Used as received	1.0 gm	1 gram textile per 50 mL perspiration liquid
2301	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2310	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
2311	Yes	Further cut	0.5	1 gram textile per 50 mL perspiration liquid
2330	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2347	---	---		1 gram textile per 50 mL perspiration liquid
2350	Yes	Further cut	1 g	1 gram textile per 50 mL perspiration liquid
2352	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2357	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2358	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2363	No	Further cut	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	1g	1 gram textile per 50 mL perspiration liquid
2375	---	---		---
2379	Yes	Further cut	0.5 g/25 mL	1 gram textile per 50 mL perspiration liquid
2380	---	---		---
2382	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2385	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2390	---	---		---
2425	Yes	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
2426	Yes	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
2429	---	---		---
2442	---	---		---
2456	Yes	Used as received	2.5	1 gram textile per 50 mL perspiration liquid
2472	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2482	Yes	Used as received	0,5	1 gram textile per 50 mL perspiration liquid
2489	No	Further cut	0.5 g	1 gram textile per 50 mL perspiration liquid
2511	---	---		---
2523	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2532	Yes	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
2540	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2582	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2590	Yes	Used as received	1 gr	1 gram textile per 50 mL perspiration liquid
2591	No	Further cut	1.0 gram	1 gram textile per 50 mL perspiration liquid
2614	Yes	Further cut	1g	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	2 g	1 gram textile per 50 mL perspiration liquid
2678	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2726	Yes	Used as received	1gram	1 gram textile per 50 mL perspiration liquid
2728	No	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2734	Yes	Used as received	3	1 gram textile per 50 mL perspiration liquid
2787	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
2804	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2809	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
2812	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
2827	Yes	Further cut	1.0	1 gram textile per 50 mL perspiration liquid
2849	No	Used as received	t 1 gram	1 gram textile per 50 mL perspiration liquid
2852	Yes	Used as received	2 g	1 gram textile per 50 mL perspiration liquid
2908	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2946	---	---		---
3116	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
3117	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

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
3146	Yes	Used as received	0.7 gram	1 gram textile per 50 mL perspiration liquid
3154	Yes	---	1	1 gram textile per 50 mL perspiration liquid
3172	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
3176	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
3190	---	---		---
3197	Yes	Used as received	0,5	1 gram textile per 50 mL perspiration liquid
3210	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
3214	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
3220	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
3228	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
3237	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
3246	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid

APPENDIX 4

Number of participants per country

3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
2 labs in	FRANCE
6 labs in	GERMANY
1 lab in	GUATEMALA
3 labs in	HONG KONG
9 labs in	INDIA
4 labs in	INDONESIA
6 labs in	ITALY
4 labs in	MOROCCO
14 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	POLAND
1 lab in	SERBIA
4 labs in	SOUTH KOREA
2 labs in	SPAIN
1 lab in	SRI LANKA
2 labs in	SWITZERLAND
3 labs in	TAIWAN
1 lab in	THAILAND
2 labs in	TUNISIA
6 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

Literature

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