Institute for
Interlaboratory Studies

Results of Proficiency Test
Heavy Metals by Perspiration
in Leather/Footwear
November 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A. Ouwerkerk & ing. G.A. Oosterlaken-Buijs
Approved by: ing. A.S. Noordman-de Neef

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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 leather consumer products. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather some Ecolabelling schemes are imposing environmental requirements for leather 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 2020 the Institute for Interlaboratory Studies organizes a proficiency scheme for the determination of Heavy Metals by Perspiration in Leather/Footwear 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 Leather/Footwear.

In this interlaboratory study 51 laboratories in 18 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Heavy Metals by Perspiration in Leather/Footwear 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 one leather sample of approximately 6 grams labelled #22760.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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

A batch of grey leather pieces was selected which was artificially fortified to contain one or more heavy metals. After homogenization 75 small plastic bags were filled with approximately 6 grams each and labelled #22760.
The homogeneity of the subsamples was checked by the determination of Copper in accordance with ISO17072-1 on 8 stratified randomly selected subsamples.

	Copper as Cu in mg/kg
sample #22760-1	94.9
sample #22760-2	89.2
sample #22760-3	88.6
sample #22760-4	88.1
sample #22760-5	94.8
sample #22760-6	88.7
sample #22760-7	89.9
sample #22760-8	92.7

Table 1: homogeneity test results of subsamples #22760

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.

	Copper as Cu in mg/kg
r (observed)	7.9
reference test method	EN16711-2:15
0.3 x R (reference test method)	12.2

Table 2: evaluation of the repeatability of subsamples #22760

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

To each of the participating laboratories one leather sample labelled #22760 was sent on October 19, 2022.

2.5 ANALYZES

The participants were requested to determine: 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. 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 sample as if it was a routine sample, but not to age nor dry the sample 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 no problems were encountered with the dispatch of the samples. Four 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 50 participants reported 145 numerical test results. Observed were 6 outlying test results, which is 4.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER ELEMENT

In this section the reported test results are discussed 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 Leather/Footwear the ISO17072-1 is considered to be the official test method. Regretfully only precision data for Lead are mentioned at a very low value of 0.6 mg/kg. Also, the use of the Horwitz equation is very strict. Therefore, it was decided to use for the target reproducibilities the precision data from test method EN16117-2:15. Test method EN16117-2 is a test method for the determination of heavy metals extracted by acidic artificial perspiration solution from textile. 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.

Cadmium: 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.

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

Copper: 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.

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 table.

Element	unit	n	average	2.8 * sd	R(lit)
Cadmium as Cd	mg/kg	48	43.2	10.1	12.1
Chromium as Cr	mg/kg	46	43.2	18.0	18.1
Copper as Cu	mg/kg	45	117	50	52

Table 3: reproducibilities of tests in sample #22760

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
Number of reporting laboratories	50	50	55
Number of test results	145	144	55
Number of statistical outliers	6	7	4
Percentage of statistical outliers	4.1%	4.9%	7.3%

Table 4: 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	EN16711-2
Antimony as Sb	--	--	--	20%
Arsenic as As	--	--	--	20%
Cadmium as Cd	8%	--	--	10%
Chromium as Cr	15%	22%	15%	15%
Cobalt as Co	--	9%	--	13%
Copper as Cu	15%	--	--	16%
Lead as Pb	--	--	--	40%
Mercury as Hg	--	--	--	31%
Nickel as Ni	--	10%	--	10%

Table 5: development of the uncertainties over the years

The uncertainties observed in this PT are comparable with the uncertainties observed in previous PTs and to the test method EN16711-2.

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 50% mentioned to use the sample as received and about 50% did further cut or further grind the sample prior to analysis.
- About 70% used a sample intake of 1 gram, about 10% used 0.5 grams and about 20% used 2 grams or more.
- All the reporting participants used a ratio of 1 g to 50 mL.

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

5 DISCUSSION

Limits for Heavy Metals via Perspiration are specified in the Leather Standard by Oeko-Tex®. When the test results of this interlaboratory study are compared to the Oeko-Tex® (see table 6), it was noticed that all participants would have made identical decisions about the acceptability of the leather for the determined parameters.

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	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 6: Leather Standard by Oeko-Tex®

All reporting laboratories would have rejected the sample based on the test results for one or more elements.

6 CONCLUSION

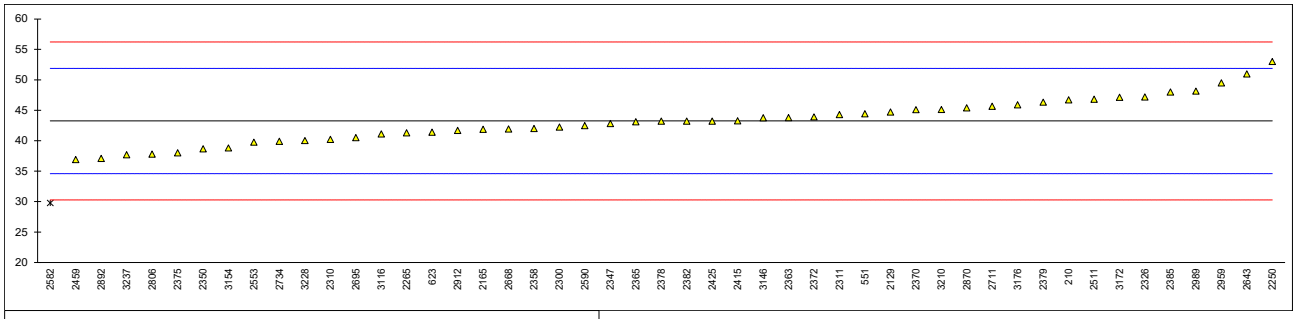
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Cadmium as Cd on sample #22760; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17072-1	46.715		0.80	
551	ISO17072-1	44.411		0.27	
623	EN16711-2	41.40		-0.43	
2129	ISO17072-1	44.715		0.34	
2165	ISO17072-1	41.87		-0.32	
2250	ISO17072-1	53.01		2.26	
2265	ISO17072-1	41.30		-0.45	
2300	ISO17072-1	42.23		-0.24	
2310	ISO17072-1	40.2		-0.70	
2311	ISO17072-1	44.30		0.24	
2326	EN16711-2	47.176		0.91	
2347	ISO17072-1	42.83		-0.10	
2350	EN16711-2	38.657		-1.06	
2358	ISO17072-1	42.0		-0.29	
2363	ISO17072-1	43.8		0.13	
2365	ISO17072-1	43.115		-0.03	
2370	ISO105E04	45.1		0.43	
2372	ISO17072-1	43.9		0.15	
2375	ISO17072-1	38		-1.21	
2378	ISO17072-1	43.2		-0.01	
2379	ISO17072-1	46.3305		0.71	
2382	ISO17072-1	43.2		-0.01	
2385	EN16711-2	48		1.10	
2415	ISO17072-1	43.26		0.00	
2425	ISO17072-1	43.20		-0.01	
2459	ISO17072-1	36.89		-1.47	
2511	ISO17072-1	46.8		0.82	
2553	ISO17072-1	39.758		-0.81	
2582	EN16711-2	29.76	R(0.05)	-3.12	
2590	ISO17072-1	42.49		-0.18	
2643	ISO17072-1	50.967		1.78	
2668	EN16711-2	41.93		-0.30	
2695	ISO17072-1/17294-2	40.50		-0.64	
2711	ISO17072-1	45.65		0.56	
2734	EN16711-2	39.90		-0.77	
2806	ISO17072-1	37.8		-1.26	
2870	ISO17072-1	45.38		0.49	
2882		-----		-----	
2892	ISO17072-1	37.082		-1.43	
2912	ISO17072-1	41.673		-0.36	
2959	ISO17072-1	49.5		1.45	
2977		-----		-----	
2989	ISO105E04	48.1310		1.13	
3116	ISO17072-1	41.1		-0.50	
3146	ISO17072-1	43.77		0.12	
3154	EN16711-2	38.81		-1.03	
3172	ISO17072-1	47.13		0.90	
3176	ISO17072-1	45.90		0.61	
3210	ISO17072-1	45.11	C	0.43	First reported 4.511
3228	ISO17072-1	40.02		-0.75	
3237	ISO17072-1	37.69		-1.29	

normality	OK	
n	48	
outliers	1	
mean (n)	43.2479	
st.dev. (n)	3.61492	RSD = 8%
R(calc.)	10.1218	
st.dev.(EN16711-2:15)	4.32479	
R(EN16711-2:15)	12.1094	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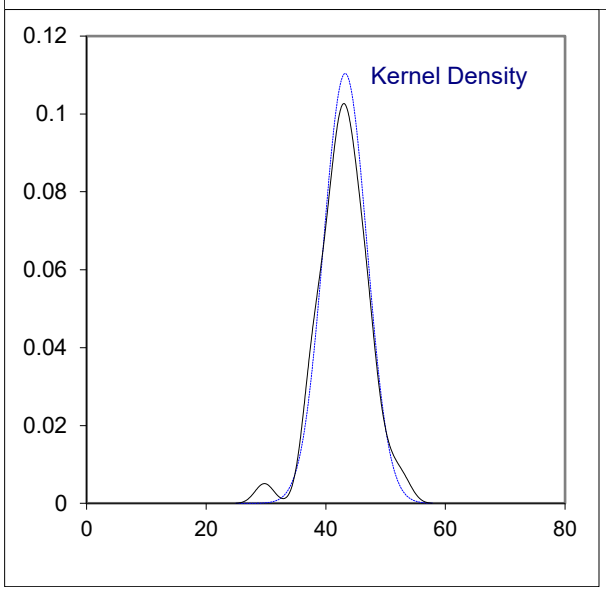
Kernel Density
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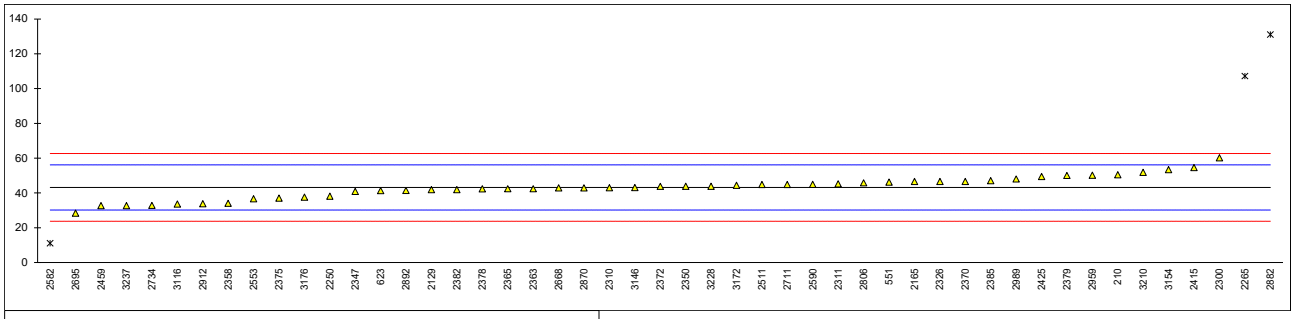
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60
80

Determination of Chromium as Cr on sample #22760; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17072-1	50.428		1.12	
551	ISO17072-1	46.2305		0.48	
623	EN16711-2	41.31		-0.28	
2129	ISO17072-1	41.852		-0.20	
2165	ISO17072-1	46.49		0.52	
2250	ISO17072-1	38.11		-0.78	
2265	ISO17072-1	107.15	R(0.01)	9.89	
2300	ISO17072-1	60.12	C	2.62	First reported 56.68
2310	ISO17072-1	43		-0.02	
2311	ISO17072-1	45.19		0.31	
2326	EN16711-2	46.500		0.52	
2347	ISO17072-1	40.87		-0.35	
2350	EN16711-2	43.834		0.11	
2358	ISO17072-1	34.0	C	-1.41	First reported 27.9
2363	ISO17072-1	42.4		-0.12	
2365	ISO17072-1	42.371		-0.12	
2370	ISO105E04	46.5		0.52	
2372	ISO17072-1	43.7		0.08	
2375	ISO17072-1	37		-0.95	
2378	ISO17072-1	42.3		-0.13	
2379	ISO17072-1	49.9960		1.06	
2382	ISO17072-1	41.9		-0.19	
2385	EN16711-2	47		0.59	
2415	ISO17072-1	54.56		1.76	
2425	ISO17072-1	49.37		0.96	
2459	ISO17072-1	32.65		-1.62	
2511	ISO17072-1	44.8		0.25	
2553	ISO17072-1	36.642		-1.01	
2582	EN16711-2	11.09	R(0.01)	-4.95	
2590	ISO17072-1	44.95		0.28	
2643		-----		-----	
2668	EN16711-2	42.87		-0.04	
2695	ISO17072-1/17294-2	28.32		-2.29	
2711	ISO17072-1	44.80		0.25	
2734	EN16711-2	32.78		-1.60	
2806	ISO17072-1	45.8		0.41	
2870	ISO17072-1	42.87		-0.04	
2882	ISO17072-1	130.9488	R(0.01)	13.56	
2892	ISO17072-1	41.4	C	-0.27	First reported 61.406
2912	ISO17072-1	33.742	C	-1.45	First reported 15.817
2959	ISO17072-1	50.2		1.09	
2977		-----		-----	
2989	ISO105E04	48.0110		0.75	
3116	ISO17072-1	33.6	C	-1.48	First reported 27.4
3146	ISO17072-1	43.03		-0.02	
3154	EN16711-2	53.40		1.58	
3172	ISO17072-1	44.335		0.18	
3176	ISO17072-1	37.57		-0.86	
3210	ISO17072-1	51.77	C	1.33	First reported 5.177
3228	ISO17072-1	43.84		0.11	
3237	ISO17072-1	32.66		-1.62	

normality	OK	
n	46	
outliers	3	
mean (n)	43.1537	
st.dev. (n)	6.41995	RSD = 15%
R(calc.)	17.9759	
st.dev.(EN16711-2:15)	6.47306	
R(EN16711-2:15)	18.1246	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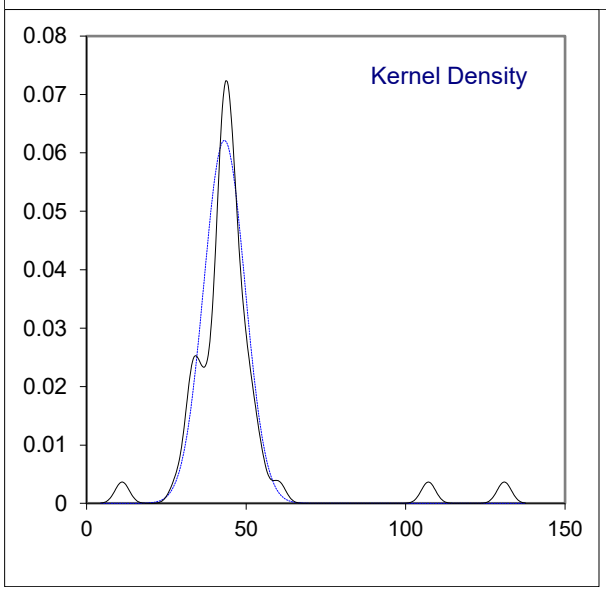
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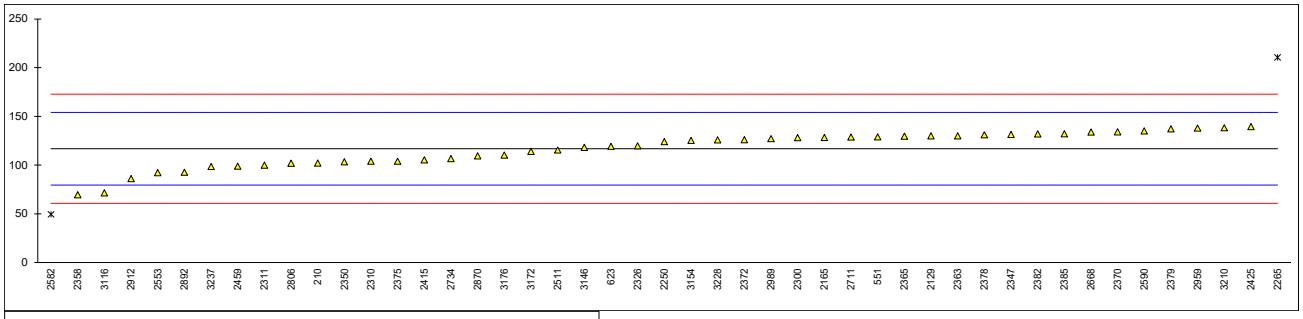
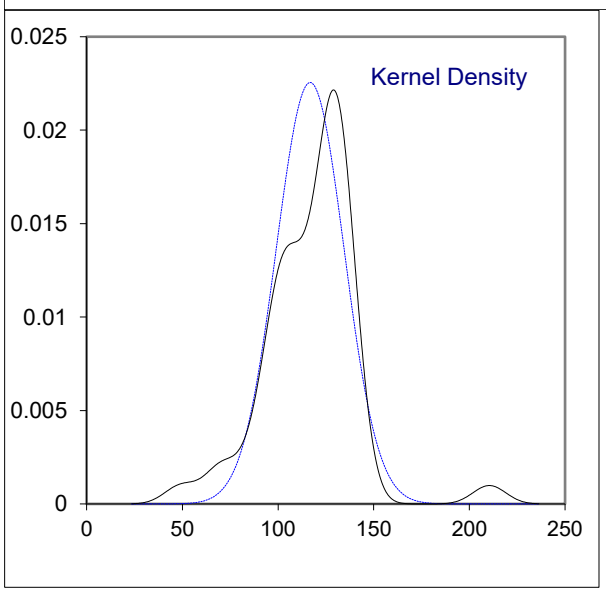
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
50
100
150

Determination of Copper as Cu on sample #22760; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17072-1	101.97		-0.79	
551	ISO17072-1	128.8765		0.65	
623	EN16711-2	119.19		0.13	
2129	ISO17072-1	129.865		0.70	
2165	ISO17072-1	128.27		0.61	
2250	ISO17072-1	123.98		0.38	
2265	ISO17072-1	210.50	R(0.01)	5.01	
2300	ISO17072-1	128.19		0.61	
2310	ISO17072-1	104		-0.69	
2311	ISO17072-1	99.81		-0.91	
2326	EN16711-2	119.719		0.16	
2347	ISO17072-1	131.4		0.78	
2350	EN16711-2	103.3		-0.72	
2358	ISO17072-1	69.5		-2.53	
2363	ISO17072-1	130.0		0.71	
2365	ISO17072-1	129.653		0.69	
2370	ISO105E04	134		0.92	
2372	ISO17072-1	126		0.49	
2375	ISO17072-1	104		-0.69	
2378	ISO17072-1	131.0		0.76	
2379	ISO17072-1	137.1353		1.09	
2382	ISO17072-1	131.9		0.81	
2385	EN16711-2	132		0.81	
2415	ISO17072-1	105.26		-0.62	
2425	ISO17072-1	139.45		1.21	
2459	ISO17072-1	98.78		-0.96	
2511	ISO17072-1	115.417		-0.07	
2553	ISO17072-1	92.149		-1.32	
2582	EN16711-2	49.41	R(0.05)	-3.61	
2590	ISO17072-1	135.02		0.97	
2643		-----		-----	
2668	EN16711-2	133.9		0.91	
2695	ISO17072-1/17294-2	Not detected		-----	Possibly a false negative test result?
2711	ISO17072-1	128.70		0.64	
2734	EN16711-2	106.70		-0.54	
2806	ISO17072-1	101.8		-0.80	
2870	ISO17072-1	109.45		-0.39	
2882		-----		-----	
2892	ISO17072-1	92.615		-1.29	
2912	ISO17072-1	86.127		-1.64	
2959	ISO17072-1	138		1.13	
2977		-----		-----	
2989	ISO105E04	127.1010		0.55	
3116	ISO17072-1	71.4		-2.43	
3146	ISO17072-1	118.2		0.07	
3154	EN16711-2	125.3		0.45	
3172	ISO17072-1	114.0		-0.15	
3176	ISO17072-1	110.27		-0.35	
3210	ISO17072-1	138.3	C	1.15	First reported 13.83
3228	ISO17072-1	125.92		0.49	
3237	ISO17072-1	98.52		-0.98	
	normality	OK			
	n	45			
	outliers	2			
	mean (n)	116.8030			
	st.dev. (n)	17.69757	RSD = 15%		
	R(calc.)	49.5532			
	st.dev.(EN16711-2:15)	18.68849			
	R(EN16711-2:15)	52.3278			


250
200
150
100
50
0
2582
2358
3116
2912
2553
2892
3237
2459
2311
2806
210
2350
2310
2375
2415
2734
2870
3176
3172
2511
3146
623
2326
2250
3154
3228
2372
2989
2300
2165
2711
551
2365
2129
2363
2378
2347
2382
2385
2668
2370
2590
2379
2959
3210
2425
2265


Kernel Density
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150
200
250

APPENDIX 2

Reported test results of other requested elements; results in mg/kg

lab	Sb	As	Co	Pb	Hg	Ni
210	-----	-----	-----	-----	-----	-----
551	-----	-----	0.002	-----	-----	0.0475
623	0.015	Not Detected	Not Detected	Not Detected	Not Detected	0.194
2129	-----	-----	-----	-----	-----	-----
2165	not detected	not detected	not detected	not detected	not detected	not detected
2250	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	-----	-----	-----
2300	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
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND	ND	ND	ND
2347	<1	<0.1	<0.5	<0.1	<0.01	<0.5
2350	<0.5	<0.02	<0.1	<0.1	<0.005	0.1408
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	<1	<0.1	<0.5	<0.1	<0.01	<0.5
2365	＜1.0	＜0.1	＜0.5	＜0.1	＜0.01	＜0.5
2370	<1	<0.2	<0.1	<0.2	<0.02	<0.5
2372	< 1	< 0.2	< 0.1	< 0.2	< 0.02	< 0.5
2375	<3	<0.06	<0.3	<0.06	<0.006	<0.1
2378	≤1.0	≤0.06	≤0.1	≤0.06	≤0.006	≤0.1
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2382	＜1.00	＜0.10	＜0.50	＜0.10	＜0.010	＜0.50
2385	<0.1	<0.1	<0.1	<0.1	<0.01	<0.5
2415	-----	-----	-----	-----	-----	-----
2425	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2459	ND	ND	ND	ND	ND	ND
2511	-----	-----	-----	-----	-----	-----
2553	NOT DETECTED	<0.05	<0.1	<0.1	NOT DETECTED	<0.1
2582	Not detected	Not detected	Not detected	Nor detected	Not detected	Not detected
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2643	-----	-----	-----	-----	-----	-----
2668	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2695	Not detected	Not Detected	Not Detected	Not detected	Not detected	Not detected
2711	<2	<2	<2	<2	-----	3.30
2734	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2806	< 0,1	< 0,1	< 0,1	< 0,1	< 0,02	< 0,1
2870	-----	-----	-----	-----	-----	-----
2882	-----	-----	-----	-----	-----	-----
2892	not detected	not detected	not detected	not detected	not detected	0.241
2912	-----	-----	-----	-----	-----	-----
2959	-----	-----	-----	-----	-----	-----
2977	-----	-----	-----	-----	-----	-----
2989	-----	-----	-----	-----	-----	-----
3116	<0.5	<0.02	<0.1	<0.1	<0.005	<0.1
3146	< 2.0	< 0.10	< 0.50	< 0.10	< 0.01	< 0.50
3154	-----	-----	-----	-----	-----	-----
3172	< 0.5	< 0.02	< 0.1	< 0.1	< 0.01	< 0.1
3176	-----	-----	-----	-----	-----	-----
3210	<5	<0,2	<1	<0,2	<0,02	<1
3228	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
3237	-----	-----	-----	-----	-----	-----

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample preparation	Sample intake (grams)	Ratio gram textile per ml
210	Yes	---		---
551	Yes	Further cut	1	1 gram per 50 mL
623	Yes	Further cut	1	1 gram per 50 mL
2129	Yes	Further cut	1	1 gram per 50 mL
2165	Yes	Further cut	1.0	1 gram per 50 mL
2250	Yes	Further cut	0,5	0.5 gram per 25 mL
2265	No	Further cut	1	1 gram per 50 mL
2300	Yes	Further cut	1	1 gram per 50 mL
2310	Yes	Further cut	1	1 gram per 50 mL
2311	Yes	Further cut	1	1 gram per 50 mL
2326	Yes	Further cut	1.0042	1 gram per 50 mL
2347	Yes	Used as received	1	2 gram per 100 mL
2350	Yes	Further cut	1	1 gram per 50 mL
2358	Yes	Used as received	2	2 gram per 100 mL
2363	Yes	Used as received	2	2 gram per 100 mL
2365	Yes	Used as received	1.0	1 gram per 50 mL
2370	Yes	Further cut	1	1 gram per 50 mL
2372	Yes	Further cut	1	1 gram per 50 mL
2375	Yes	Used as received	1.0032	1 gram per 50 mL
2378	No	Further cut	1	1 gram per 50 mL
2379	Yes	Further cut	1	1 gram per 50 mL
2382	Yes	Used as received	2	2 gram per 100 mL
2385	Yes	---		---
2415	Yes	Further cut	1	1 gram per 50 mL
2425	Yes	Further cut	1.0	1 gram per 50 mL
2459	Yes	Used as received	2	2 gram per 100 mL
2511	Yes	Further grinded		2 gram per 100 mL
2553	Yes	Used as received	0.5	---
2582	Yes	Used as received	1.0005	1 gram per 50 mL
2590	Yes	Used as received	1	1 gram per 50 mL
2643	Yes	Used as received	1.0	1 gram per 50 mL
2668	Yes	Further cut	1.0	1 gram per 50 mL
2695	Yes	Used as received	2	2 gram per 100 mL
2711	No	Further cut	1,0	1 gram per 50 mL
2734	Yes	Used as received	4,5	1 gram per 50 mL
2806	Yes	Used as received		---
2870	Yes	Used as received	2	2 gram per 100 mL
2882	No	Used as received	2.0	2 gram per 100 mL
2892	Yes	Further cut	1.0051	1 gram per 50 mL
2912	No	Used as received		0.5 gram per 25 mL---
2959	No	Used as received		2 gram per 100 mL
2977	---	---		---
2989	No	Further cut	1	1 gram per 50 mL
3116	Yes	Used as received	1	1 gram per 50 mL
3146	Yes	Used as received	0.6	0.6 gram per 30 mL
3154	Yes	Used as received	1	1 gram per 50 mL
3172	Yes	---		---
3176	Yes	Used as received	1	1 gram per 50 mL
3210	Yes	Further cut	1	1 gram per 50 mL
3228	No	Further cut	0.5	0.5 gram per 25 mL
3237	Yes	Used as received	2	2 gram per 100 mL

APPENDIX 4

Number of participants per country

1 lab in	BANGLADESH
1 lab in	BRAZIL
1 lab in	FRANCE
6 labs in	GERMANY
2 labs in	HONG KONG
5 labs in	INDIA
1 lab in	INDONESIA
8 labs in	ITALY
2 labs in	KOREA, Republic of
1 lab in	MOROCCO
8 labs in	P.R. of CHINA
4 labs in	PAKISTAN
2 labs in	SRI LANKA
2 labs in	TAIWAN
1 lab in	THAILAND
1 lab in	TUNISIA
3 labs in	TURKEY
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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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