Results of Proficiency Test Heavy Metals by Perspiration in Textile November 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. E.R. Montenij-Bos
Correctors: ing. R.J. Starink & ing. C.M. Nijssen-Wester
Report: iis21T06

January 2022

CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 4

2.4 SAMPLES 4

2.5 ANALYZES 5

3 RESULTS 5

3.1 STATISTICS 6

3.2 GRAPHICS 6

3.3 Z-SCORES 7

4 EVALUATION 8

4.1 EVALUATION PER SAMPLE AND PER ELEMENT 8

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 9

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2021 WITH PREVIOUS PTS 9

4.4 EVALUATION OF ANALYTICAL DETAILS 10

5 DISCUSSION 11

6 CONCLUSION 11

Appendices:

1. Data, statistical and graphic results 12
2. Other reported metals 22
3. Analytical Details 26
4. Number of participants per country 28
5. Abbreviations and literature 29

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 2021/2022 it was decided to continue with the proficiency test for the analysis of Heavy Metals by Perspiration in Textile.

In this interlaboratory study 93 laboratories in 28 different 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 3 grams each and labelled #21750 and #21751. The samples were artificially fortified with different heavy metals.
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 white cotton was selected which was obtained from a third party. After cutting and homogenization 111 small plastic bags were filled with approximately 3 grams each and labelled #21750.
The batch was used in previous proficiency test on Heavy Metals by Perspiration in Textile as sample #15205 in iis15A04. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of yellow cotton was prepared. After cutting and homogenization 120 small plastic bags were filled with approximately 3 grams each and labelled #21751.
The homogeneity of the subsamples was checked by the determination of Cadmium and Cobalt according to test method DIN 54233-3 on 10 stratified randomly selected subsamples.

	Cadmium in mg/kg	Cobalt in mg/kg
sample #21751-1	14.72	25.52
sample #21751-2	14.12	24.41
sample #21751-3	14.63	24.90
sample #21751-4	14.04	24.12
sample #21751-5	14.82	25.32
sample #21751-6	14.72	26.59
sample #21751-7	15.37	26.21
sample #21751-8	14.09	26.26
sample #21751-9	14.19	24.34
sample #21751-10	14.65	26.68

Table 1 homogeneity test results of subsamples #21751

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.

	Cadmium in mg/kg	Cobalt in mg/kg
r (observed)	1.18	2.71
Reference test method	EN16711-2:15	EN16711-2:15
0.3 x R (reference test method)	1.22	2.78

Table 2: evaluation of the repeatabilities of subsamples #21751

The calculated repeatabilities are 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 sample labelled #21750 and one sample labelled #21751 were sent on October 6, 2021.

2.5 ANALYZES

The participants were requested to determine on samples #21750 and #21751 the Heavy Metals by Perspiration: 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 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 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.

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, e.g. EN reproducibilities, 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. Seven participants reported test results after the final reporting date and seven other participants did not report any test results. Not all participants were able to report all elements requested.
In total 86 participants reported 418 numerical test results. Observed were 12 outlying test results, which is 2.9%. 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 standard deviation (or variation coefficient) of the metal with 2.8.

sample #21750

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

Copper as Cu: 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.

Nickel as Ni: 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.

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

sample #21751

Cadmium as Cd: This determination was problematic for a number of participants. Six statistical outliers were observed. The calculated reproducibility after rejection of the outliers is in agreement with the requirements of EN16711-2:15.

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

The majority of participants agreed on a concentration near or below the limit of detection for all other requested elements. 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 literature reference test method (in casu EN16711-2) are presented in the next two tables.

Element	unit	n	average	2.8 * sd	R(lit)
Cadmium as Cd	mg/kg	79	2.83	0.93	0.79
Copper as Cu	mg/kg	84	33.7	8.9	15.1
Nickel as Ni	mg/kg	81	3.04	0.94	2.64

Table 3: reproducibilities of Heavy Metals by Perspiration in sample #21750

Element	unit	n	average	2.8 * sd	R(lit)
Cadmium as Cd	mg/kg	79	11.9	3.1	3.3
Cobalt as Co	mg/kg	83	25.1	8.1	9.2

Table 4: reproducibilities of Heavy Metals by Perspiration in sample #21751

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 2021 WITH PREVIOUS PTS

	November 2021	November 2020	November 2019	November 2018	November 2017
Number of reporting laboratories	86	79	96	91	93
Number of test results	418	314	408	254	674
Number of statistical outliers	12	11	13	8	15
Percentage of statistical outliers	2.9%	3.5%	3.2%	3.1%	2.2%

Table 5: 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 expressed as relative standard deviation (uncertainties). The conclusions are given in the following table.

Element	November 2021	November 2020	November 2019	November 2018	2010-2017	EN16711-2
Antimony as Sb	--	--	12%	8%	16-19%	20%
Arsenic as As	--	--	9%	--	--	20%
Cadmium as Cd	9-12%	--	--	--	9-18%	10%
Chromium as Cr	--	--	11%	10%	12-19%	15%
Cobalt as Co	12%	--	--	--	8-14%	13%
Copper as Cu	10%	8-13%	--	--	9-22%	16%
Lead as Pb	--	--	--	--	35-40%	40%
Mercury as Hg	--	--	--	34%	41%	31%
Nickel as Ni	11%	10%	11%	--	7-14%	10%

Table 6: 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 participants were asked to provide some analytical details which are given in appendix 3. Based on the reported answers the following can be summarized:

- A vast majority (90%) mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- For the samples about 30% further cut the sample prior to analysis and 70% used the sample as received.
- Almost 75% used 1.0 grams of sample intake.
- The vast majority of the participants used the ratio of 1 g to 50 mL. Remarkably, two participants chose 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.

It appeared that no effect was observed on the reported test results for the determined elements in sample #21750 nor in sample #21751 because the observed reproducibilities are in line with the reference test method EN16711-2:15.

5 DISCUSSION

Sample #21750 was also used in a previous proficiency test iis15A04 as sample #15205. A comparison is given in table below.

Element	unit	Sample #21750			Sample #15205		
		n	average	2.8 * sd	n	average	2.8 * sd
Cadmium as Cd	mg/kg	79	2.83	0.93	79	2.86	0.95
Copper as Cu	mg/kg	84	33.7	8.9	83	33.6	10.2
Nickel as Ni	mg/kg	81	3.04	0.94	80	3.00	0.94

Table 7: comparison sample #21750 with #15205

The current PT results are in line with the previous PT. Therefore, it is concluded that the samples textile containing Cadmium, Copper and Nickel is stable for six years at least.

When the results of this interlaboratory study were compared to the Oekotex 100 (see table below), it was noticed that all participants would have made identical decisions about the acceptability of the textiles for the determined components.

	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 8: Overview from Oekotex 100

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

6 CONCLUSION

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

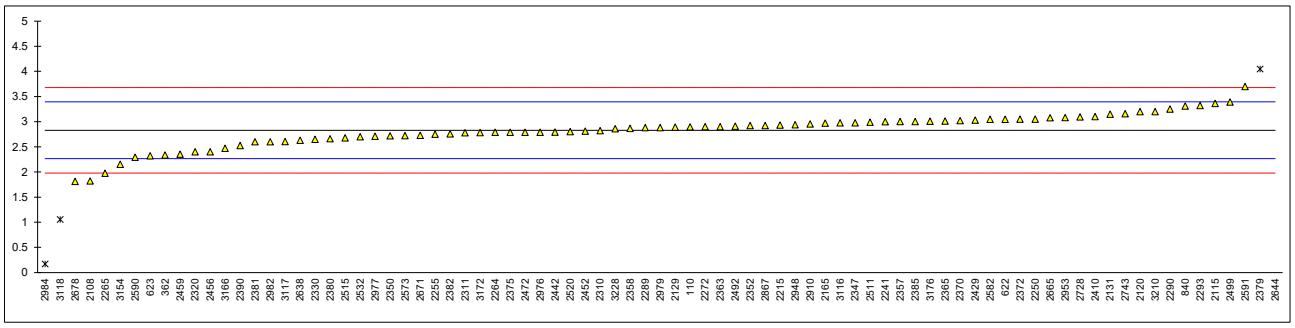
Determination of Cadmium as Cd on sample #21750; results in mg/kg

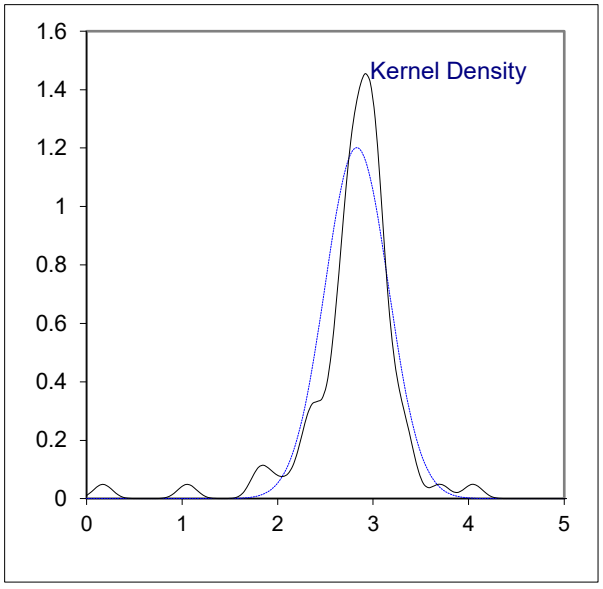
lab	method	value	mark	z(targ)	remarks
110	ISO105E04	2.896		0.24	
210		-----		-----	
339		-----	W	-----	test result withdrawn, reported 0.95
362	ISO105E04	2.335		-1.75	
551		-----		-----	
622		3.047		0.77	
623	EN16711-2	2.32		-1.80	
840	EN16711-2	3.31		1.70	
2108	In house	1.818	C	-3.57	first reported 1.604
2115	EN1611-2	3.36		1.88	
2120	EN16711-2	3.2		1.31	
2129	EN16711-2	2.89		0.22	
2131	In house	3.15		1.13	
2165	EN16711-2	2.970		0.50	
2215	EN16711-2	2.932		0.36	
2241	EN16711-2	2.998		0.60	
2250	EN16711-2	3.05		0.78	
2255	EN16711-2	2.75		-0.28	
2264	ISO17294/105E04	2.788		-0.15	
2265		1.973		-3.03	
2272	EN16711-2	2.9		0.25	
2287	EN16711-2	<5		-----	
2289	DIN54233-3	2.88		0.18	
2290	EN16711-2	3.25		1.49	
2293	EN16711-2	3.32		1.74	
2295		-----		-----	
2310	EN16711-2	2.82		-0.03	
2311	EN16711-2	2.78		-0.17	
2320	In house	2.4	C	-1.52	first reported 1.446
2330	ISO105E04	2.647		-0.64	
2347	EN16711-2	2.978		0.53	
2350	EN16711-2	2.718		-0.39	
2352	EN16711-2	2.923		0.33	
2357	EN16711-2	3.0		0.60	
2358	EN16711-2	2.866		0.13	
2363	EN16711-2	2.9		0.25	
2365	EN16711-2	3.011		0.64	
2370	ISO105E04	3.02		0.67	
2372	EN16711-2	3.05		0.78	
2375	EN16711-2	2.79		-0.14	
2379	EN16711-2	4.0459	R(0.05)	4.30	
2380	EN16711-2	2.66		-0.60	
2381	EN16711-2	2.60		-0.81	
2382	EN16711-2	2.7570		-0.25	
2385	EN16711-2	3.0		0.60	
2390	ISO105E04	2.522		-1.09	
2410	EN16711-2	3.1		0.96	
2426		-----		-----	
2429	EN16711-2	3.03		0.71	
2442	EN16711-2	2.7932		-0.13	
2452	EN16711-2	2.81		-0.07	
2456	EN16711-2	2.4		-1.52	
2459	EN16711-2	2.351		-1.69	
2472	GB/T17593	2.79		-0.14	
2492	EN16711-2	2.911		0.29	
2499	EN16711-2	3.3911		1.99	
2511	EN16711-2	2.99		0.57	
2515	ISO105E04	2.676		-0.54	
2520	ISO105E04	2.8		-0.10	
2532	EN16711-2	2.7		-0.46	
2573	EN16711-2	2.723		-0.38	
2582	EN16711-2	3.045		0.76	
2590	EN16711-2	2.29		-1.91	
2591	EN16711-2	3.70		3.08	
2618		-----		-----	
2638	EN16711-2	2.63		-0.70	
2644	EN16711-2	12.52	R(0.01)	34.25	
2665	EN16711-2	3.077		0.88	
2671	EN16711-2	2.73		-0.35	
2678	EN16711-2	1.81		-3.60	
2728	SNI7334	3.093	C	0.93	first reported 0.685
2743	EN16711-2	3.158		1.16	
2773		-----		-----	
2859		-----		-----	
2867	EN16711-2	2.923		0.33	

lab	method	value	mark	z(targ)	remarks
2910	EN16711-2	2.952		0.43	
2912		-----		-----	
2948	EN16711-2	2.936		0.38	
2953	EN16711-2	3.08		0.89	
2976	EN16711-2	2.79		-0.14	
2977	EN16711-2	2.708		-0.43	
2979	EN16711-2	2.881		0.18	
2982	EN16711-2	2.60		-0.81	
2984	SNI7334	0.1702	R(0.01)	-9.40	
3116		2.976		0.52	
3117	EN16711-2	2.604		-0.80	
3118	EN16711-2	1.0539	C,R(0.01)	-6.27	first reported 1.5141
3154	EN16711-2	2.15		-2.40	
3166	In house	2.47		-1.27	
3172	EN16711-2	2.7825		-0.16	
3176	EN16711-2	3.008		0.63	
3210	EN16711-2	3.201		1.31	
3228	EN16711-2	2.86		0.11	

	normality	suspect
	n	79
	outliers	4
	mean (n)	2.8291
	st.dev. (n)	0.33198
	R(calc.)	0.9295
	st.dev.(EN16711-2:15)	0.28291
	R(EN16711-2:15)	0.7921

RSD = 12%





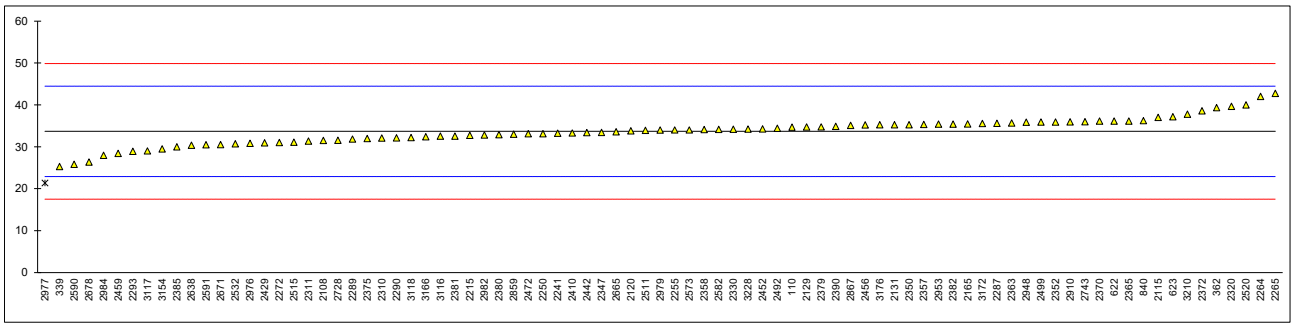
Determination of Copper as Cu on sample #21750; results in mg/kg

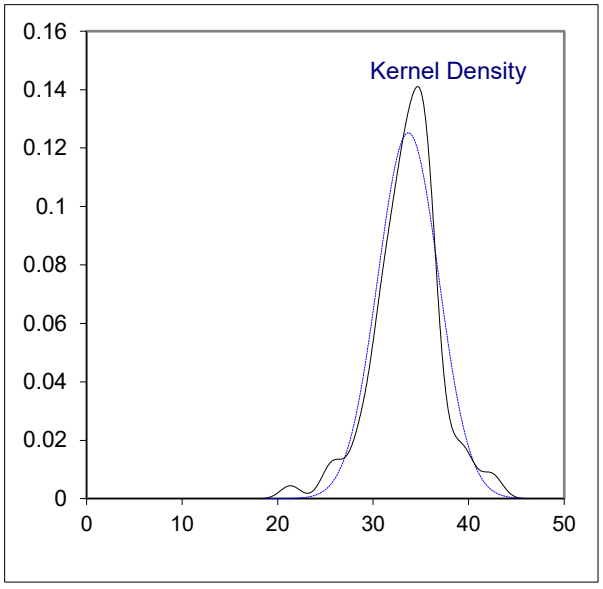
lab	method	value	mark	z(targ)	remarks
110	ISO105E04	34.65		0.18	
210		-----		-----	
339	EN16711-2	25.28		-1.56	
362	ISO105E04	39.35		1.05	
551		-----		-----	
622		36.124		0.45	
623	EN16711-2	37.16		0.64	
840	EN16711-2	36.21		0.47	
2108	In house	31.497		-0.41	
2115	EN1611-2	37.04		0.62	
2120	EN16711-2	33.8		0.02	
2129	EN16711-2	34.72		0.19	
2131	In house	35.24		0.29	
2165	EN16711-2	35.429		0.32	
2215	EN16711-2	32.7406		-0.18	
2241	EN16711-2	33.230		-0.09	
2250	EN16711-2	33.1		-0.11	
2255	EN16711-2	34.0		0.06	
2264	ISO17294/105E04	42.028		1.54	
2265		42.767		1.68	
2272	EN16711-2	31		-0.50	
2287	EN16711-2	35.61		0.35	
2289	DIN54233-3	31.82		-0.35	
2290	EN16711-2	32.13		-0.29	
2293	EN16711-2	28.89		-0.89	
2295		-----		-----	
2310	EN16711-2	32.1		-0.30	
2311	EN16711-2	31.38		-0.43	
2320	In house	39.632		1.10	
2330	ISO105E04	34.187		0.09	
2347	EN16711-2	33.428		-0.05	
2350	EN16711-2	35.26		0.29	
2352	EN16711-2	35.913		0.41	
2357	EN16711-2	35.32		0.30	
2358	EN16711-2	34.120		0.08	
2363	EN16711-2	35.7		0.37	
2365	EN16711-2	36.130		0.45	
2370	ISO105E04	36.1		0.44	
2372	EN16711-2	38.6		0.91	
2375	EN16711-2	32		-0.32	
2379	EN16711-2	34.7500		0.19	
2380	EN16711-2	32.9		-0.15	
2381	EN16711-2	32.54		-0.22	
2382	EN16711-2	35.3970		0.31	
2385	EN16711-2	30		-0.69	
2390	ISO105E04	34.885		0.22	
2410	EN16711-2	33.3		-0.07	
2426		-----		-----	
2429	EN16711-2	30.92		-0.52	
2442	EN16711-2	33.4024		-0.06	
2452	EN16711-2	34.27		0.11	
2456	EN16711-2	35.2		0.28	
2459	EN16711-2	28.441		-0.98	
2472	GB/T17593	33.09		-0.11	
2492	EN16711-2	34.426		0.13	
2499	EN16711-2	35.9030		0.41	
2511	EN16711-2	33.89		0.03	
2515	ISO105E04	31.069		-0.49	
2520	ISO105E04	40		1.17	
2532	EN16711-2	30.73		-0.55	
2573	EN16711-2	34.00		0.06	
2582	EN16711-2	34.159		0.08	
2590	EN16711-2	25.80		-1.47	
2591	EN16711-2	30.49		-0.60	
2618		-----		-----	
2638	EN16711-2	30.34		-0.62	
2644		-----		-----	
2665	EN16711-2	33.593		-0.02	
2671	EN16711-2	30.55		-0.58	
2678	EN16711-2	26.36		-1.36	
2728	SNI7334	31.539		-0.40	
2743	EN16711-2	35.961		0.42	
2773		-----		-----	
2859	EN16711-2	33.001		-0.13	
2867	EN16711-2	35.11		0.26	

lab	method	value	mark	z(targ)	remarks
2910	EN16711-2	35.953		0.42	
2912		-----		-----	
2948	EN16711-2	35.87		0.40	
2953	EN16711-2	35.38		0.31	
2976	EN16711-2	30.81		-0.54	
2977	EN16711-2	21.354	R(0.05)	-2.29	
2979	EN16711-2	33.992		0.05	
2982	EN16711-2	32.83		-0.16	
2984	SNI7334	27.9553		-1.07	
3116		32.52		-0.22	
3117	EN16711-2	29.006		-0.87	
3118	EN16711-2	32.1962		-0.28	
3154	EN16711-2	29.47		-0.78	
3166	In house	32.4		-0.24	
3172	EN16711-2	35.575		0.35	
3176	EN16711-2	35.239		0.29	
3210	EN16711-2	37.78		0.76	
3228	EN16711-2	34.24		0.10	

normality	suspect
n	84
outliers	1
mean (n)	33.701
st.dev. (n)	3.1877
R(calc.)	8.926
st.dev.(EN16711-2:15)	5.3922
R(EN16711-2:15)	15.098

RSD = 10%



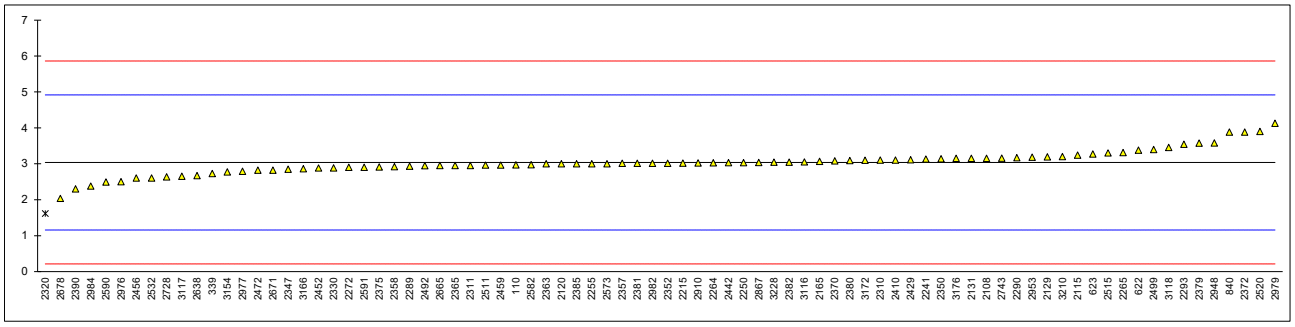


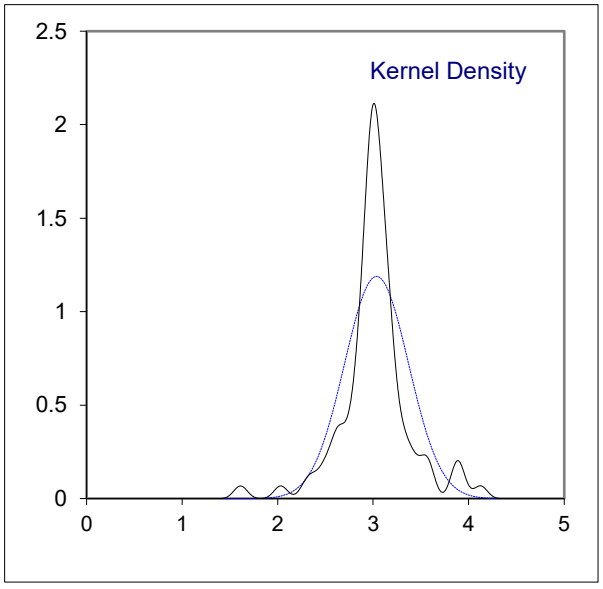
Determination of Nickel as Ni on sample #21750; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	2.97		-0.07	
210		-----		-----	
339	EN16711-2	2.73		-0.32	
362	ISO105E04	<0.5		-----	
551		-----		-----	
622		3.378		0.36	
623	EN16711-2	3.27		0.25	
840	EN16711-2	3.88		0.90	
2108	In house	3.153		0.12	
2115	EN1611-2	3.24		0.22	
2120	EN16711-2	3.0		-0.04	
2129	EN16711-2	3.19		0.16	
2131	In house	3.152		0.12	
2165	EN16711-2	3.066		0.03	
2215	EN16711-2	3.015		-0.02	
2241	EN16711-2	3.129		0.10	
2250	EN16711-2	3.03		-0.01	
2255	EN16711-2	3.0		-0.04	
2264	ISO17294/105E04	3.026		-0.01	
2265		3.313		0.29	
2272	EN16711-2	2.9		-0.14	
2287	EN16711-2	<5		-----	
2289	DIN54233-3	2.93		-0.11	
2290	EN16711-2	3.17		0.14	
2293	EN16711-2	3.54		0.54	
2295		-----		-----	
2310	EN16711-2	3.1		0.07	
2311	EN16711-2	2.95		-0.09	
2320	In house	1.612	R(0.01)	-1.51	
2330	ISO105E04	2.887		-0.16	
2347	EN16711-2	2.849		-0.20	
2350	EN16711-2	3.139		0.11	
2352	EN16711-2	3.013		-0.02	
2357	EN16711-2	3.01		-0.03	
2358	EN16711-2	2.921		-0.12	
2363	EN16711-2	3.0		-0.04	
2365	EN16711-2	2.949		-0.09	
2370	ISO105E04	3.08		0.05	
2372	EN16711-2	3.88		0.90	
2375	EN16711-2	2.91		-0.13	
2379	EN16711-2	3.5756		0.57	
2380	EN16711-2	3.09		0.06	
2381	EN16711-2	3.01		-0.03	
2382	EN16711-2	3.0410		0.01	
2385	EN16711-2	3.0		-0.04	
2390	ISO105E04	2.302		-0.78	
2410	EN16711-2	3.1		0.07	
2426		-----		-----	
2429	EN16711-2	3.11		0.08	
2442	EN16711-2	3.0272		-0.01	
2452	EN16711-2	2.88		-0.17	
2456	EN16711-2	2.6		-0.46	
2459	EN16711-2	2.962		-0.08	
2472	GB/T17593	2.82		-0.23	
2492	EN16711-2	2.945		-0.10	
2499	EN16711-2	3.3965		0.38	
2511	EN16711-2	2.96		-0.08	
2515	ISO105E04	3.300		0.28	
2520	ISO105E04	3.9		0.92	
2532	EN16711-2	2.60		-0.46	
2573	EN16711-2	3.00		-0.04	
2582	EN16711-2	2.972		-0.07	
2590	EN16711-2	2.49		-0.58	
2591	EN16711-2	2.90		-0.14	
2618		-----		-----	
2638	EN16711-2	2.67		-0.39	
2644		-----		-----	
2665	EN16711-2	2.947		-0.09	
2671	EN16711-2	2.82		-0.23	
2678	EN16711-2	2.033		-1.07	
2728	SNI7334	2.635		-0.43	
2743	EN16711-2	3.155		0.13	
2773		-----		-----	
2859		-----		-----	
2867	EN16711-2	3.034		0.00	

lab	method	value	mark	z(targ)	remarks
2910	EN16711-2	3.019		-0.02	
2912		-----		-----	
2948	EN16711-2	3.58		0.58	
2953	EN16711-2	3.18		0.15	
2976	EN16711-2	2.50		-0.57	
2977	EN16711-2	2.786		-0.27	
2979	EN16711-2	4.124		1.16	
2982	EN16711-2	3.01		-0.03	
2984	SNI7334	2.3762		-0.70	
3116		3.052		0.02	
3117	EN16711-2	2.653		-0.41	
3118	EN16711-2	3.4535		0.44	
3154	EN16711-2	2.77		-0.28	
3166	In house	2.86		-0.19	
3172	EN16711-2	3.0975		0.07	
3176	EN16711-2	3.151		0.12	
3210	EN16711-2	3.200		0.17	
3228	EN16711-2	3.04		0.00	

	normality	not OK
	n	81
	outliers	1
	mean (n)	3.0358
	st.dev. (n)	0.33550 RSD = 11%
	R(calc.)	0.9394
	st.dev.(EN16711-2:15)	0.94109
	R(EN16711-2:15)	2.6351



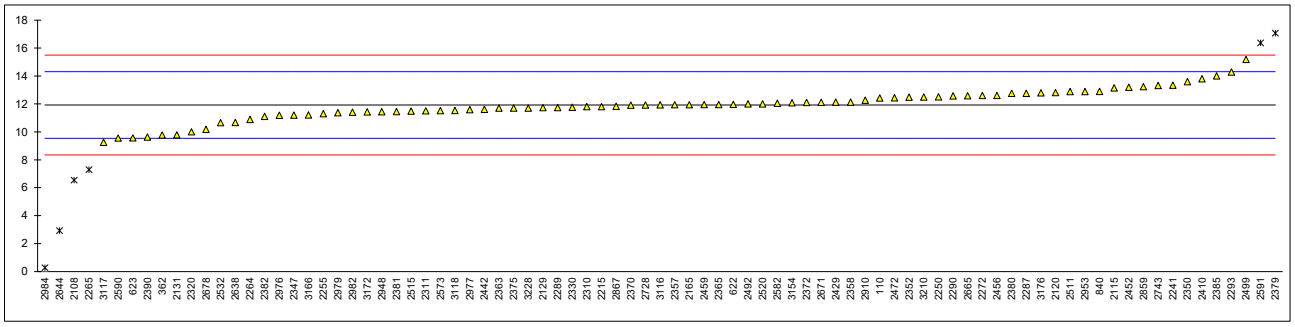


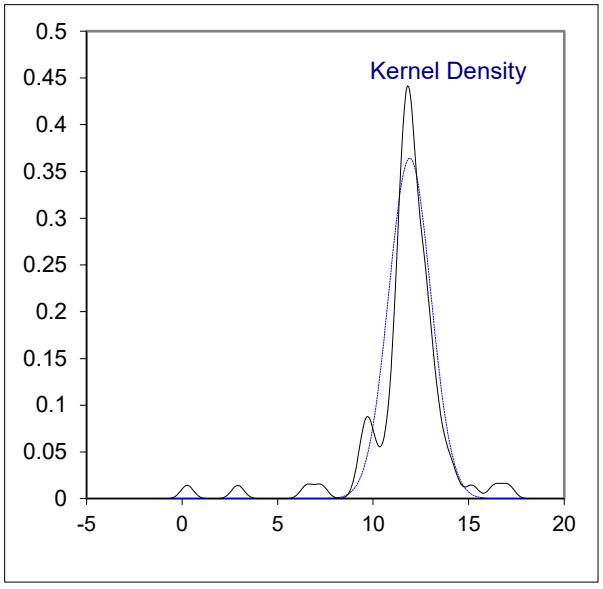
Determination of Cadmium as Cd on sample #21751; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	12.42		0.42	
210		-----		-----	
339		-----	W	-----	test result withdrawn, reported 4.08
362	ISO105E04	9.765		-1.81	
551		-----		-----	
622	EN16711-2	11.964		0.04	
623		9.56		-1.98	
840	EN16711-2	12.89		0.81	
2108	In house	6.540	C,R(0.01)	-4.51	first reported 5.912
2115	EN1611-2	13.14		1.02	
2120	EN16711-2	12.8		0.74	
2129	EN16711-2	11.73		-0.16	
2131	In house	9.78		-1.80	
2165	EN16711-2	11.931		0.01	
2215	EN16711-2	11.807		-0.09	
2241	EN16711-2	13.341		1.19	
2250	EN16711-2	12.5		0.49	
2255	EN16711-2	11.3		-0.52	
2264	ISO105E04	10.889		-0.86	
2265		7.283	R(0.01)	-3.89	
2272		12.6		0.57	
2287	EN16711-2	12.75		0.70	
2289	DIN54233-3	11.74		-0.15	
2290	EN16711-2	12.56		0.54	
2293	EN16711-2	14.27		1.97	
2295		-----		-----	
2310	EN16711-2	11.8		-0.10	
2311	EN16711-2	11.50		-0.35	
2320	In house	10.0	C	-1.61	first reported 5.158
2330	ISO105E04	11.763		-0.13	
2347	EN16711-2	11.193		-0.61	
2350	EN16711-2	13.59		1.40	
2352	EN16711-2	12.474		0.47	
2357	EN16711-2	11.93		0.01	
2358	EN16711-2	12.124		0.17	
2363	EN16711-2	11.7		-0.18	
2365	EN16711-2	11.944		0.02	
2370	ISO105E04	11.9		-0.02	
2372	EN16711-2	12.1		0.15	
2375	EN16711-2	11.7		-0.18	
2379	EN16711-2	17.0584	R(0.01)	4.31	
2380	EN16711-2	12.749		0.70	
2381	EN16711-2	11.44		-0.40	
2382	EN16711-2	11.1020		-0.69	
2385	EN16711-2	14		1.75	
2390	ISO105E04	9.632		-1.92	
2410	EN16711-2	13.8		1.58	
2426		-----		-----	
2429	EN16711-2	12.12		0.17	
2442	EN16711-2	11.6075		-0.26	
2452	EN16711-2	13.19		1.07	
2456	EN16711-2	12.6		0.57	
2459	EN16711-2	11.941		0.02	
2472	GB/T17593	12.43		0.43	
2492	EN16711-2	11.997		0.06	
2499	EN16711-2	15.1752		2.73	
2511	EN16711-2	12.88		0.81	
2515	ISO105E04	11.479		-0.37	
2520	ISO105E04	12		0.07	
2532	EN16711-2	10.65		-1.07	
2573	EN16711-2	11.52		-0.34	
2582	EN16711-2	12.04	C	0.10	first reported 4.014
2590	EN16711-2	9.55		-1.99	
2591	EN16711-2	16.36	R(0.01)	3.73	
2618		-----		-----	
2638	EN16711-2	10.67		-1.05	
2644	EN16711-2	2.92	R(0.01)	-7.55	
2665	EN16711-2	12.574		0.55	
2671	EN16711-2	12.11		0.16	
2678	EN16711-2	10.17		-1.47	
2728	SNI7334	11.915	C	0.00	first reported 0.457
2743	EN16711-2	13.318		1.17	
2773		-----		-----	
2859	EN16711-2	13.226		1.10	
2867	EN16711-2	11.82		-0.08	

lab	method	value	mark	z(targ)	remarks
2910	EN16711-2	12.263		0.29	
2912		-----		-----	
2948	EN16711-2	11.438		-0.40	
2953	EN16711-2	12.88		0.81	
2976	EN16711-2	11.18		-0.62	
2977	EN16711-2	11.590		-0.28	
2979	EN16711-2	11.363		-0.47	
2982	EN16711-2	11.40		-0.44	
2984	SNI7334	0.2718	R(0.01)	-9.77	
3116		11.92		0.00	
3117	EN16711-2	9.250		-2.24	
3118	EN16711-2	11.5364		-0.32	
3154	EN16711-2	12.08		0.13	
3166	In house	11.2		-0.60	
3172	EN16711-2	11.425		-0.42	
3176	EN16711-2	12.778		0.72	
3210	EN16711-2	12.493		0.48	
3228	EN16711-2	11.70		-0.18	

	normality	OK
	n	79
	outliers	6
	mean (n)	11.9197
	st.dev. (n)	1.09571 RSD = 9%
	R(calc.)	3.0680
	st.dev.(EN16711-2:15)	1.19197
	R(EN16711-2:15)	3.3375



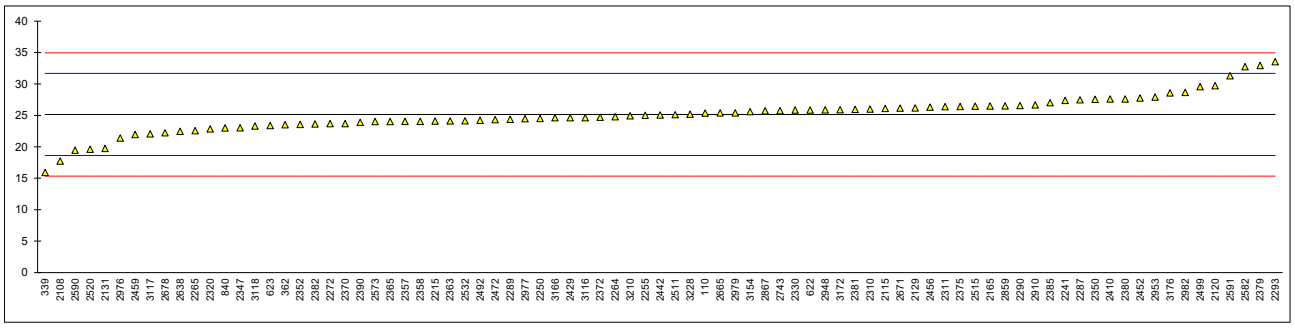


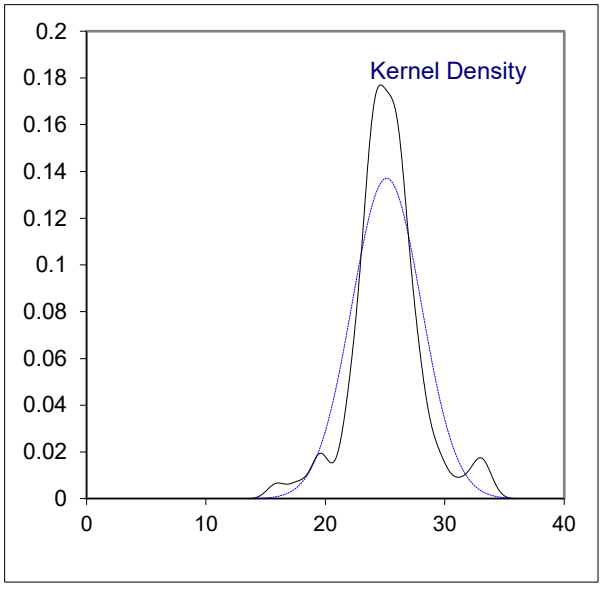
Determination of Cobalt as Co on sample #21751; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	25.35		0.06	
210		-----		-----	
339	EN16711-2	15.91		-2.83	
362	ISO105E04	23.5	C	-0.50	first reported <1.0
551		-----		-----	
622	EN16711-2	25.854		0.22	
623		23.38		-0.54	
840	EN16711-2	23.00		-0.66	
2108	In house	17.721		-2.27	
2115	EN1611-2	26.11		0.29	
2120	EN16711-2	29.7		1.39	
2129	EN16711-2	26.17		0.31	
2131	In house	19.72		-1.66	
2165	EN16711-2	26.480		0.41	
2215	EN16711-2	24.084		-0.33	
2241	EN16711-2	27.376		0.68	
2250	EN16711-2	24.5		-0.20	
2255	EN16711-2	25.0		-0.05	
2264	ISO105E04	24.777		-0.11	
2265		22.567		-0.79	
2272		23.7		-0.44	
2287	EN16711-2	27.46		0.71	
2289	DIN54233-3	24.36		-0.24	
2290	EN16711-2	26.55		0.43	
2293	EN16711-2	33.54		2.57	
2295		-----		-----	
2310	EN16711-2	26		0.26	
2311	EN16711-2	26.38		0.38	
2320	In house	22.857		-0.70	
2330	ISO105E04	25.837		0.21	
2347	EN16711-2	23.007		-0.66	
2350	EN16711-2	27.55		0.73	
2352	EN16711-2	23.553		-0.49	
2357	EN16711-2	24.02		-0.35	
2358	EN16711-2	24.033		-0.34	
2363	EN16711-2	24.1		-0.32	
2365	EN16711-2	24.011		-0.35	
2370	ISO105E04	23.7		-0.44	
2372	EN16711-2	24.7		-0.14	
2375	EN16711-2	26.4		0.38	
2379	EN16711-2	32.9364		2.38	
2380	EN16711-2	27.603		0.75	
2381	EN16711-2	25.95		0.25	
2382	EN16711-2	23.6220		-0.47	
2385	EN16711-2	27		0.57	
2390	ISO105E04	23.877		-0.39	
2410	EN16711-2	27.6		0.75	
2426		-----		-----	
2429	EN16711-2	24.62		-0.16	
2442	EN16711-2	25.0517		-0.03	
2452	EN16711-2	27.75		0.80	
2456	EN16711-2	26.3		0.35	
2459	EN16711-2	21.962		-0.97	
2472	GB/T17593	24.31		-0.26	
2492	EN16711-2	24.213		-0.29	
2499	EN16711-2	29.5577		1.35	
2511	EN16711-2	25.1		-0.01	
2515	ISO105E04	26.456		0.40	
2520	ISO105E04	19.6		-1.70	
2532	EN16711-2	24.12		-0.31	
2573	EN16711-2	24.00		-0.35	
2582	EN16711-2	32.73	C	2.32	first reported 16.365
2590	EN16711-2	19.46		-1.74	
2591	EN16711-2	31.28		1.88	
2618		-----		-----	
2638	EN16711-2	22.45		-0.83	
2644		-----		-----	
2665	EN16711-2	25.388		0.07	
2671	EN16711-2	26.16		0.31	
2678	EN16711-2	22.22		-0.90	
2728		-----		-----	
2743	EN16711-2	25.745		0.18	
2773		-----		-----	
2859	EN16711-2	26.506		0.42	
2867	EN16711-2	25.72		0.17	

lab	method	value	mark	z(targ)	remarks
2910	EN16711-2	26.666		0.46	
2912		-----		-----	
2948	EN16711-2	25.87		0.22	
2953	EN16711-2	27.91		0.84	
2976	EN16711-2	21.40		-1.15	
2977	EN16711-2	24.474		-0.21	
2979	EN16711-2	25.39		0.07	
2982	EN16711-2	28.64		1.07	
2984		-----		-----	
3116		24.62		-0.16	
3117	EN16711-2	22.057		-0.95	
3118	EN16711-2	23.3058		-0.56	
3154	EN16711-2	25.57		0.13	
3166	In house	24.6		-0.17	
3172	EN16711-2	25.885		0.23	
3176	EN16711-2	28.581		1.05	
3210	EN16711-2	24.935		-0.07	
3228	EN16711-2	25.20		0.02	

	normality	not OK
	n	83
	outliers	0
	mean (n)	25.1488
	st.dev. (n)	2.90959 RSD = 12%
	R(calc.)	8.1468
	st.dev.(EN16711-2:15)	3.26934
	R(EN16711-2:15)	9.1542





APPENDIX 2 Other reported metals on sample #21750; results in mg/kg

lab	Sb	As	Cr	Co	Pb	Hg
110	less than 1	less than 0.05	less than 0.5	less than 0.5	less than 0.05	less than 0.02
210	-----	-----	-----	-----	-----	-----
339	not detected	not detected	not detected	not detected	not detected	not detected
362	<1.0	<0.2	<1.0	<1.0	<0.1	<0.02
551	-----	-----	-----	-----	-----	-----
622	0.004	0	0.154	0	0	0.015
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected
2108	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	-----	-----	-----	-----
2120	<2.5	<0.10	<0.5	<0.5	<0.10	<0.013
2129	not detected	not detected	not detected	not detected	not detected	not detected
2131	not detected	not detected	not detected	not detected	not detected	not detected
2165	not detected	not detected	not detected	not detected	not detected	not detected
2215	<0.5	<0.02	<0.1	<0.1	<0.1	<0.005
2241	0.004	0.003	0.008	0.002	0.003	0
2250	not detected	not detected	not detected	not detected	not detected	not detected
2255	not detected	not detected	not detected	not detected	not detected	not detected
2264	<0.033	0.009	<0.030	<0.012	<0.044	<0.005
2265	not detected	not detected	not detected	not detected	not detected	not detected
2272	-----	-----	-----	-----	-----	-----
2287	<5	<5	<5	<5	<5	<5
2289	<1.0	<0.1	<0.5	<0.3	<0.1	<0.01
2290	< 3	< 0.1	< 0.5	< 0.5	< 0.1	< 0.02
2293	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2295	-----	-----	-----	-----	-----	-----
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
2320	<1.0	<0.1	<0.5	<0.5	<0.1	<0.01
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<1	<0.1	<1	<0.5	<0.1	<0.01
2350	<0.5	<0.02	<0.1	<0.1	<0.1	<0.005
2352	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	not detected	not detected	not detected	not detected	not detected	not detected
2365	<1.0	<0.1	<0.5	<0.5	<0.1	<0.01
2370	<1	<0.2	<0.5	<0.1	<0.2	<0.02
2372	< 1	< 0.2	< 0.5	< 0.1	< 0.2	< 0.02
2375	<0.05	<0.02	<0.05	<0.05	<0.05	<0.005
2379	Not detected	Not detected	Not detected	Not detected	0.0517	Not detected
2380	-----	-----	-----	-----	-----	-----
2381	not detected	not detected	not detected	not detected	not detected	not detected
2382	<1.0	<0.10	<0.50	<0.50	<0.10	<0.010
2385	<0,1	<0,1	<0,1	<0,1	<0,1	<0,01
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2410	< 5	< 0.2	< 1	< 1	< 0.2	< 0.02
2426	-----	-----	-----	-----	-----	-----
2429	<1.0	<0.1	<0.5	<0.3	<0.1	<0.01
2442	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2452	0.06	not detected	0.07	0.06	0.06	not detected
2456	not detected	not detected	not detected	not detected	not detected	-----
2459	ND	ND	ND	ND	ND	ND
2472	<0.35	-----	<0.06	<0.10	<0.35	-----
2492	-----	-----	-----	-----	-----	-----
2499	not detected	not detected	not detected	not detected	not detected	not detected
2511	nd	nd	nd	nd	nd	nd
2515	not detected	not detected	not detected	not detected	not detected	not detected
2520	< 2.5	< 0.01	< 0.25	< 0.5	0.17	0.5
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2573	-----	-----	-----	-----	-----	-----
2582	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2591	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2618	-----	-----	-----	-----	-----	-----
2638	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2644	-----	-----	-----	27.20	-----	-----
2665	not detected	not detected	not detected	not detected	not detected	not detected
2671	ND	ND	ND	ND	ND	ND
2678	nd	nd	0.5208	nd	nd	nd
2728	-----	-----	-----	-----	0.108	-----
2743	-----	-----	-----	-----	-----	-----
2773	-----	-----	-----	-----	-----	-----
2859	-----	-----	-----	-----	-----	-----
2867	not detected	not detected	not detected	not detected	not detected	not detected

lab	Sb	As	Cr	Co	Pb	Hg
2910	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----
2948	Not detected	Not detected	Not Ddetected	Not Ddetected	Not detected	Not detected
2953	-----	-----	-----	-----	-----	-----
2976	not detected	not detected	not detected	not detected	not detected	not detected
2977	<0.05 (LOQ)	<0.05 (LOQ)	<0.05 (LOQ)	<0.05 (LOQ)	<0.05 (LOQ)	<0.05 (LOQ)
2979	N.D	N.D	ND	ND	ND	0.293
2982	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2984	-----	-----	-----	-----	not detected	-----
3116	<0.5	<0.02	<0.1	<0.1	<0.1	<0.005
3117	-----	-----	-----	-----	-----	-----
3118	<0.25	<0.05	<0.5	<0.25	<0.25	<0.01
3154	0.71	-----	-----	-----	-----	-----
3166	<0.03	<0.02	<0.02	<0.002	<0.006	<0.003
3172	< 1	< 0.02	< 0.1	< 0.1	< 0.1	< 0.005
3176	-----	-----	-----	-----	-----	-----
3210	<5	<0.2	<1	<1	<0.2	<0.02
3228	<0.5	<0.02	<0.5	<0.5	<0.02	<0.02

Other reported metals on sample #21751; results in mg/kg

lab	Sb	As	Cr	Cu	Pb	Hg	Ni
110	less than 1	less than 0.05	less than 0.5	less than 1	less than 0.05	less than 0.02	less than 0.5
210	-----	-----	-----	-----	-----	-----	-----
339	not detected	not detected	not detected	not detected	not detected	not detected	not detected
362	<1.0	<0.2	<1.0	1.310	<0.1	<0.02	<0.5
551	-----	-----	-----	-----	-----	-----	-----
622	0	0	0	0.568	0	0.010	0
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected	0.08
2108	not detected	not detected	not detected	not detected	not detected	not detected	0.180
2115	-----	-----	-----	-----	-----	-----	-----
2120	<2.5	<0.10	<0.5	<2.5	<0.10	<0.013	<0.5
2129	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2131	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2165	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2215	<0.5	<0.02	<0.1	<5	<0.1	<0.005	<0.1
2241	0.005	0.005	0.008	0.301	0	0	0.060
2250	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2264	<0.033	<0.009	<0.030	8.739	<0.044	<0.002	0.076
2265	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2272	-----	-----	-----	-----	-----	-----	-----
2287	<5	<5	<5	<5	<5	<5	<5
2289	<1.0	<0.1	<0.5	<1.0	<0.1	<0.01	<0.3
2290	< 3	< 0.1	< 0.5	< 5	< 0.1	< 0.02	< 0.1
2293	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2295	-----	-----	-----	-----	-----	-----	-----
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	<0.5	<1.0	<0.1	<0.01	<0.5
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<1	<0.1	<1	<5	<0.1	<0.01	<0.5
2350	<0.5	<0.02	<0.1	<5	<0.1	<0.005	<0.1
2352	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	<1.0	<0.1	<0.5	<5	<0.1	<0.01	<0.5
2370	<1	<0.2	<0.5	<5	<0.2	<0.02	<0.5
2372	< 1	< 0.2	< 0.5	< 5	< 0.2	< 0.02	< 0.5
2375	<0.05	<0.02	<0.05	<5.0	<0.05	<0.005	<0.1
2379	Not detected	Not detected	Not detected	Not detected	0.0354	Not detected	0.1539
2380	-----	-----	-----	-----	-----	-----	-----
2381	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2382	<1.0	<0.10	<0.50	<5.0	<0.10	<0.010	<0.50
2385	<0,1	<0,1	<0,1	<0,5	<0,1	<0,01	<0,5
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2410	< 5	< 0.2	< 1	< 5	< 0.2	< 0.02	< 1
2426	-----	-----	-----	-----	-----	-----	-----
2429	<1.0	<0.1	<0.5	<1.0	<0.1	<0.01	<0.3
2442	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2452	0.06	not detected	0.07	0.5	0.06	not detected	0.08
2456	not detected	not detected	not detected	not detected	not detected	-----	not detected
2459	ND	ND	ND	ND	ND	ND	ND
2472	<0.35	-----	<0.06	<0.6	<0.35	-----	<0.05
2492	-----	-----	-----	-----	-----	-----	-----
2499	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2511	nd	nd	nd	nd	nd	nd	nd
2515	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2520	< 2.5	< 0.01	< 0.25	1	0.15	0.2	< 0.04
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2573	-----	-----	-----	-----	-----	-----	-----
2582	Not Detected	Not Detected	Not Detected	Not detected	Not Detected	Not Detected	Not Detected
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
2591	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2618	-----	-----	-----	-----	-----	-----	-----
2638	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2644	-----	-----	-----	37.62	-----	-----	2.95
2665	not detected	not detected	not detected	0.2894	not detected	not detected	not determined
2671	ND	ND	ND	ND	ND	ND	ND
2678	nd	nd	nd	nd	0.2	nd	0.27
2728	-----	-----	-----	0.955	0.064	-----	0.169
2743	-----	-----	-----	2.315	-----	-----	-----
2773	-----	-----	-----	-----	-----	-----	-----
2859	-----	-----	-----	-----	-----	-----	-----
2867	not detected	not detected	not detected	not detected	not detected	not detected	not detected

lab	Sb	As	Cr	Cu	Pb	Hg	Ni
2910	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----	-----
2948	Not detected	Not detected	Not detected	2.27	Not detected	Not detected	1.25
2953	-----	-----	-----	-----	-----	-----	-----
2976	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	<0.05 (LOQ)	<0.05 (LOQ)	0.209	0.722	<0.05 (LOQ)	<0.05 (LOQ)	0.760
2979	N.D	N.D	N.D	0.922	N.D	0.244	N.D
2982	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2984	-----	-----	-----	1.7771	not detected	-----	0.4646
3116	<0.5	<0.02	<0.1	<5	<0.1	<0.005	<0.1
3117	-----	-----	-----	0.576	-----	-----	-----
3118	<0.25	<0.05	<0.5	<0.25	<0.25	<0.01	<0.25
3154	-----	-----	-----	1.25	-----	-----	-----
3166	<0.03	<0.02	<0.02	0.33	<0.002	<0.003	0.07
3172	< 1	< 0.02	< 0.1	< 1	< 0.1	< 0.005	< 0.1
3176	-----	-----	-----	0.374	-----	-----	0.074
3210	<5	<0.2	<1	<5	<0.2	<0.02	<1
3228	<0.5	<0.02	<0.5	<0.5	<0.02	<0.02	<0.5

APPENDIX 3 Analytical Details

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
110	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
210	---	---		---
339	---	---		---
362	No	Used as received	1	1 gram textile per 50 mL perspiration liquid
551	---	---		---
622	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
623	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
840	Yes	Further cut	2g	0.5g textile per 25 mL perspiration liquid
2108	Yes	Further cut	1 g	1 gram textile per 20 mL perspiration liquid
2115	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
2120	Yes	Used as received		1 gram textile per 50 mL perspiration liquid
2129	Yes	Further cut	1 g	1 gram textile per 50 mL perspiration liquid
2131	Yes	Further cut	1.5 gram in duplicate	2.5 gram textile per 50mL
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
2241	Yes	Used as received	21750:1g / 21751:1g	1 gram textile per 50 mL perspiration liquid
2250	Yes	Used as received	0,5	1 gram textile per 50 mL perspiration liquid
2255	Yes	Used as received	1.0	1 gram textile per 50 mL perspiration liquid
2264	Yes	Used as received	2.0	1 gram textile per 50 mL perspiration liquid
2265	Yes	Used as received		1g / 30 mL
2272	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2287	No	Further cut	0.5g	1 gram textile per 50 mL perspiration liquid
2289	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2290	Yes	---		---
2293	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2295	---	---		---
2310	Yes	Further cut	1g	1 gram textile per 50 mL perspiration liquid
2311	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
2320	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2330	Yes	Further cut	0.5 gram	1 gram textile per 50 mL perspiration liquid
2347	---	---		---
2350	Yes	Further cut	1 g	1 gram textile per 50 mL perspiration liquid
2352	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2357	---	---		---
2358	No	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2363	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2365	Yes	Used as received	1.0g	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		1 gram textile per 50 mL perspiration liquid
2375	---	---		---
2379	Yes	Used as received	1 gram	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 gram	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	1 gram textile per 50 mL perspiration liquid
2390	Yes	Further cut	21750=1.0033 g 21751=1.0039 g	1 gram textile per 50 mL perspiration liquid
2410	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2426	---	---		---
2429	Yes	Further cut	#21750:1.0040 #21751:0.9880	1 gram textile per 50 mL perspiration liquid
2442	Yes	Further cut	1.0093g	1 gram textile per 50 mL perspiration liquid
2452	Yes	Other	1 g	1 gram textile per 50 mL perspiration liquid
2456	Yes	Used as received	All received sample	1 gram textile per 50 mL perspiration liquid
2459	Yes	Used as received	0.50g	1 gram textile per 50 mL perspiration liquid
2472	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2492	Yes	Used as received	0.5g	1 gram textile per 50 mL perspiration liquid
2499	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2511	---	---		---
2515	Yes	Used as received	0.5 g	1 gram textile per 50 mL perspiration liquid
2520	Yes	Used as received	1g /test	1 gram textile per 50 mL perspiration liquid
2532	Yes	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
2573	Yes	Used as received	1g	1 gram textile per 50 mL perspiration liquid
2582	No	Used as received	21750- 1.0009g 21751-1.0002g	1 gram textile per 50 mL perspiration liquid
2590	Yes	Further cut	0.5	1 gram textile per 50 mL perspiration liquid
2591	No	Further cut	1 gram	1 gram textile per 50 mL perspiration liquid
2618	---	---		---
2638	No	Used as received	1 gm	1 gram textile per 50 mL perspiration liquid
2644	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2665	Yes	Used as received	3x 1 gram	1 gram textile per 50 mL perspiration liquid

lab	laboratory accredited	sample further grinded/cut	Sample intake (in grams)	Ratio gram textile per ml
2671	Yes	Used as received	0.3 grams	1 gram textile per 50 mL perspiration liquid
2678	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2728	Yes	Further cut	1	1 gram textile per 50 mL perspiration liquid
2743	Yes	Used as received	1 g	1 gram textile per 50 mL perspiration liquid
2773	---	---		---
2859	Yes	Used as received	1.0094g,1.0040g	1 gram textile per 50 mL perspiration liquid
2867	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2910	Yes	Used as received	1.0g	1 gram textile per 50 mL perspiration liquid
2912	---	---		---
2948	---	---		---
2953	No	Further cut	1	1 gram textile per 50 mL perspiration liquid
2976	No	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
2977	Yes	Further cut	all	1 gram textile per 50 mL perspiration liquid
2979	Yes	Used as received	2gr	2 gram textile per 100ml perspiration liquid
2982	Yes	Used as received	1 gm	1 gram textile per 50 mL perspiration liquid
2984	Yes	Used as received	#21750: 1.0016 g #21751: 1.0008 g	1 gram textile per 50 mL perspiration liquid
3116	Yes	Used as received	1 gram	1 gram textile per 50 mL perspiration liquid
3117	Yes	Used as received	1gram	---
3118	Yes	Further cut	0.5 gram	---
3154	Yes	---	1	1 gram textile per 50 mL perspiration liquid
3166	Yes	Used as received	0.5	1 gram textile per 50 mL perspiration liquid
3172	Yes	---		---
3176	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
3210	Yes	Used as received	1	1 gram textile per 50 mL perspiration liquid
3228	Yes	Used as received	1.0	1 gram textile per 50 mL perspiration liquid

APPENDIX 4

Number of participants per country

6 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
2 labs in	EGYPT
2 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
3 labs in	HONG KONG
5 labs in	INDIA
5 labs in	INDONESIA
10 labs in	ITALY
1 lab in	JAPAN
1 lab in	MOROCCO
19 labs in	P.R. of CHINA
5 labs in	PAKISTAN
1 lab in	PERU
1 lab in	PORTUGAL
2 labs in	SOUTH KOREA
1 lab in	SPAIN
2 labs in	SRI LANKA
1 lab in	SWITZERLAND
2 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TUNISIA
3 labs in	TURKEY
2 labs 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
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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)