Results of Proficiency Test
Heavy Metals by perspiration
in textile
October 2016

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's, many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, 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 test scheme for perspirated metals in textile. During the annual proficiency testing program 2016/2017, it was decided to continue the proficiency test for the determination of perspirated metals in textile.
In the interlaboratory study of October 2016, 84 laboratories from 27 different countries did register for participation. See appendix for the number of participants per country. In this report, the results of the 2016 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different textile samples, which were artificially fortified with different metals and to use a solid/liquid ratio of 1/50 by preference. Participants were also 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/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls in 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 organisation 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 April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two different textile samples were selected. The first batch (#16625) was a cotton, fortified with Chromium and Cobalt. The second batch (#16626) also a cotton was fortified with Copper and Lead.
Samples #16625 and #16626 were finely cut, well mixed and divided over 100 subsamples of approx. 3 grams each. The homogeneity of subsamples #16625 and #16626 was checked by the determination of Chromium and Cobalt content on sample #16625 and by the determination of Copper content on sample #16626 on respectively 7 and 5 stratified randomly selected subsamples.

	Perspirated Chromium in mg/kg	*Perspirated Cobalt in mg/kg*
Sample #16625-1	2.77	8.42
Sample #16625-2	2.83	8.44
Sample #16625-3	2.92	8.92
Sample #16625-4	3.01	8.87
Sample #16625-5	2.71	8.65
Sample #16625-6	2.76	9.22
Sample #16625-7	2.82	8.54

Table 1: homogeneity test results of subsamples #16625

	Perspirated Copper in mg/kg
Sample #16626-1	105.5
Sample #16626-2	106.1
Sample #16626-3	107.1
Sample #16626-4	105.5
Sample #16626-5	107.7

Table 2: homogeneity test results of subsamples #16626

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibilities of the reference test method, in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	Perspirated Chromium in mg/kg	*Perspirated Cobalt in mg/kg*	*Perspirated Copper in mg/kg*
r (observed) #16625	0.28	0.82	--
r (observed) #16626	--	--	2.8
reference test method	EN16711-2:15	EN16711-2:15	EN16711-2:15
0.3 x R (ref. test method)	0.33	0.85	13.4

Table 3: repeatabilities of subsamples #16625 and #16626

The calculated repeatabilities of each metal is in good agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneities of the subsamples #16625 and #16626 were assumed.

To each of the participating laboratories, one of sample #16625 and one of sample #16626 were sent on October 12, 2016.

2.5 ANALYSES

The participants were requested to determine on both samples: perspirated heavy metals: Antimony, Arsenic, Cadmium, Chromium, Cobalt, Copper, Lead, Mercury and Nickel, applying the analysis procedure that is routinely used in the laboratory, but also to use preferably a solid/liquid ratio of 1/50 g/ml as prescribed in EN16711-2:15 (DIN 54233-3:10).
It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report ‘less than’ results, which are above the detection limit, because such results can’t be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.
The laboratories were also requested to confirm the sample receipt on the same data entry portal together with some details of the test methods used.

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 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 reanalysis).

Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results should be used with due care.

According to ISO 5725 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise 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 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. ASTM 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.

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

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Only two participants did not report any test result at all. Not all laboratories were able to report all metals requested.
Finally, the 82 reporting laboratories reported 489 numerical test results. Observed were 14 outlying results, which is 2.9% 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.

In 2010 the draft method DIN 54233-3 was issued. This method mentions the standard deviation and variation coefficient per metal between laboratories (see table A.1). The reproducibility of each metal was calculated by multiplying the standard deviation (or variation coefficient) of the metal with 2.8. In 2015 this test method was finalized and published as EN16711-2.

4.1 EVALUATION PER SAMPLE AND PER METAL

In this section, the results are discussed per sample and per metal.
The participants were requested to report 9 different metals. The majority of participants did only detect Chromium and Cobalt for sample #16625 and Copper and Lead for sample #16626.
The participants were also requested to report the ratio used (see appendix 2) and whether or not they are accredited to perform these tests. Of all reporting laboratories, 73% is ISO/IEC 17025 accredited.

Sample #16625:

Chromium: The determination of this metal was problematic for a number of laboratories at a perspiration level of 2.9 mg/kg. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the reproducibility of EN16711-2:15 (table A.1).

Cobalt: The determination of this metal was not problematic at a perspiration level of 7.7 mg/kg. No statistical outliers were observed. The calculated reproducibility is in full agreement with the reproducibility of EN16711-2:15 (table A.1).

Mercury: Although this metal was not added to the blank textile, it is remarkable that twenty participants reported a test result larger than the allowable limit of the textile standards of 0.02 mg/kg and thus would reject this sample.
No statistical outliers were observed. After conversion of less than test results, thus including the information from 21 negative test results, the consensus value is reduced from 0.024 mg/kg to 0.018 mg/kg.
Therefore no z-scores were calculated.

Other metals: Two participants did report a positive test result for Arsenic and one participant reported a positive test result for Copper. None of the participants did report a positive test result for any of other metals on sample #16625.

Sample #16626

Cadmium: Although this metal was not added to the blank textile, it is remarkable that thirty-eight participants reported a test result above 0.025 mg/kg. Two statistical outliers were observed. The consensus value may be below or near the detection limit of the test method. Therefore no z-scores were calculated.

Copper: The determination of this metal was not problematic at a perspiration level of 114 mg/kg. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the reproducibility of EN16711-2:15 (table A.1).

Lead: The determination of this metal was problematic for a number of participants a perspiration level of 1.2 mg/kg. Four statistical outliers were observed and three participants reported a false negative test result. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the reproducibility of EN16711-2:15 (table A.1).

Other metals: One participant reported a positive test result for Cobalt. None of the participants did report a positive test result for any of other metals on sample #16626.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities from the reference test method EN16711-2:15 and the reproducibilities as found for the group of participating laboratories. The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities from the reference test method EN16711-2:15 are compared in the next two tables.

Parameter	*Unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Chromium as Cr	mg/kg	76	2.87	0.95	1.20
Cobalt as Co	mg/kg	82	7.67	2.82	2.79
Mercury as Hg	mg/kg	32	0.024	0.030	(0.021)

Table 4: reproducibilities of perspirated metals in sample #16625

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Cadmium as Cd	mg/kg	37	0.059	0.040	(0.017)
Copper as Cu	mg/kg	77	114.1	31.1	51.1
Lead as Pb	mg/kg	66	1.18	1.16	1.33

Table 5: reproducibilities of perspirated metals in sample #16626

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories do not have difficulties with the analysis when compared with the target reproducibility of the reference test method EN16711-2:15. See also the discussions in paragraphs 4.1 and 6.

5 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2016 WITH THE PREVIOUS PTS

The uncertainties that were found in the results during the present PT are in line with the uncertainties as observed in previous rounds and with the target requirements (see below table).

Parameter	*Oct. 2016*	*Oct. 2015*	*Oct. 2014*	*Oct. 2013*	*Oct. 2012*	*Oct. 2011*	*Oct. 2010*	*Oct. 2009*	*EN16711 -2:2015*
Arsenic	--	--	--	--	--	--	--	--	20%
Antimony	--	--	16%	--	--	19%	--	15%	20%
Cadmium	(24%)	12%	--	9%	14%	--	14%	--	10%
Chromium	12%	--	--	15%	--	19%	--	--	15%
Cobalt	13%	--	14%	--	11%	8%	11%	--	13%
Copper	10%	9-11%	10%	10%	--	22%	--	16-17%	16%
Lead	35%	--	--	--	--	--	--	--	40%
Mercury	(45%)	--	--	--	41%	--	--	--	31%
Nickel	--	11%	--	11-13%	--	10-14%	7%	--	10%

Table 6: development of uncertainties over the last years

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

6 DISCUSSION

When the results of this interlaboratory study are compared to the Ecolabelling Standards and Requirements for Textiles in EU (table 7), it is noticed that some participants would make different decisions about the acceptability of the textiles for the determined parameters, to the majority of the group. The detection limit reported by some laboratories does not meet the requirements of the Standards (reported detection limit is larger than the maximum required concentration by the Ecolabelling standard).

Ecolabel	Class 1: baby clothes	Class 2: in direct skin contact	Class 3: with no direct skin contact	Class 4: Decoration material
Arsenic (As) mg/kg	0.2	1.0	1.0	1.0
Antimony (Sb) mg/kg	30.0	30.0	30.0	--
Cadmium (Cd) mg/kg	0.1	0.1	0.1	0.1
Chromium (Cr) mg/kg	1.0	2.0	2.0	2.0
Cobalt (Co) mg/kg	1.0	4.0	4.0	4.0
Copper (Cu) mg/kg	25.0	50.0	50.0	50.0
Lead (Pb) mg/kg	0.2	1.0	1.0	1.0
Mercury (Hg) mg/kg	0.02	0.02	0.02	0.02
Nickel (Ni) mg/kg	1.0	4.0	4.0	4.0

Table 7: Ecolabelling Standards and Requirements for Textiles in EU

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”.
It should be noticed that for the results reported in this proficiency test, all participants have probably performed the acid perspiration step according to the same conditions. Differences in sample intake and perspiration time and temperature may be parameters of importance. In the past, the solid/liquid ratio (grams of textile per ml perspiration liquid) appeared to be a parameter of utmost importance (see reports iis07A05 and iis08A05 on “Perspirated Metals in Textile”). Therefore in this proficiency test the laboratories were advised to use preferably a ratio of 1:50 as in the test method EN16711-2:15.

Each laboratory has to 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 the quality of the analytical results.

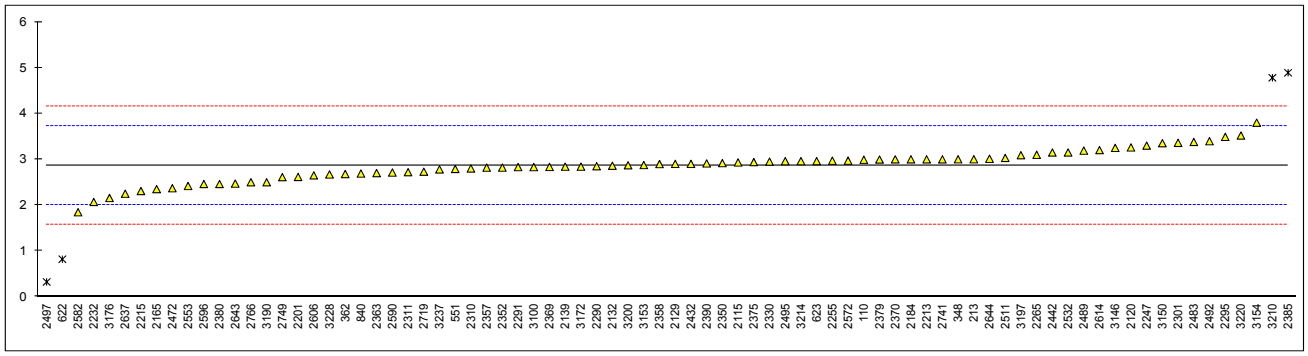
APPENDIX 1

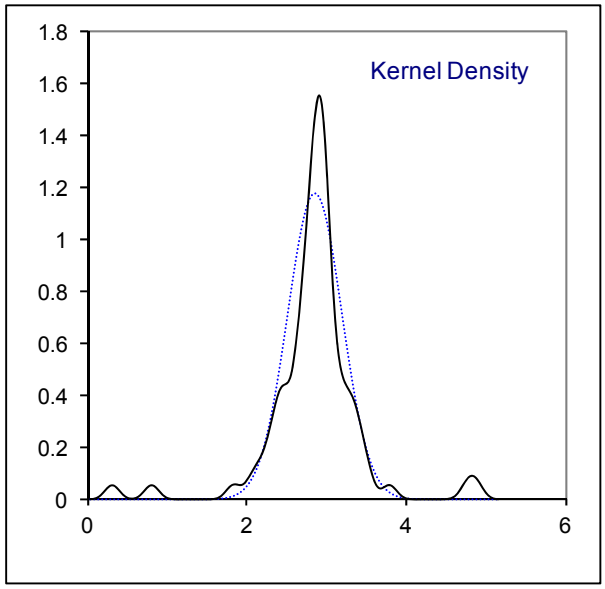
Determination of Chromium as Cr on sample #16625; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	2.991		0.29	
213	ISO105E04	3.002		0.32	
348	ISO105E04	3.001		0.32	
362		2.679		-0.43	
551	ISO105E04	2.79		-0.18	
622	ISO105E04	0.820	R(0.01)	-4.76	
623	ISO105E04	2.961		0.22	
840	ISO105E04	2.69		-0.41	
2115		2.932		0.15	
2120	EN16711-2	3.26		0.92	
2129	DIN54233-3	2.90		0.08	
2132	DIN54233-3	2.86		-0.01	
2139	ISO105E04	2.84	C	-0.06	First reported 4.46
2165	DIN54233-3	2.35		-1.20	
2184	DIN54233-3	3.00		0.31	
2201	DIN54233-3	2.616		-0.58	
2213	ISO105E04	3.0		0.31	
2215	DIN54233-3	2.308		-1.30	
2232	DIN54233-3	2.07		-1.85	
2247	ISO105E04	3.3		1.01	
2255	DIN54233-3	2.97		0.24	
2265	DIN54233-3	3.099		0.54	
2290	DIN54233-3	2.85		-0.04	
2291	GB/T17593	2.83		-0.08	
2295	EN16711-2	3.49		1.45	
2301	DIN54233-3	3.360		1.15	
2310	ISO105E04	2.801		-0.15	
2311	ISO105E04	2.72		-0.34	
2330	ISO105E04	2.9500		0.20	
2350	ISO105E04	2.917		0.12	
2352	DIN54233-3	2.820		-0.11	
2357		2.819		-0.11	
2358	DIN54233-3	2.8960		0.07	
2363	DIN54233-3	2.7		-0.38	
2369	DIN54233-3	2.834		-0.07	
2370	DIN54233-3	2.999		0.31	
2375	DIN54233-3	2.94		0.17	
2379	DIN54233-3	2.996		0.30	
2380	DIN54233-3	2.462		-0.94	
2385	DIN54233-3	4.89	R(0.01)	4.71	
2390	ISO105E04	2.91		0.10	
2432	ISO105E04	2.9007		0.08	
2442	In house	3.15		0.66	
2472	GB/T17593	2.37		-1.15	
2483	ISO105E04	3.38		1.20	
2489	ISO105E04	3.19		0.76	
2492	In house	3.396		1.23	
2495	ISO105E04	2.96		0.22	
2497	DIN54233-3	0.325	C,R(0.01)	-5.91	First reported 4.392
2511	EN16711-2	3.031		0.39	
2520		-----		-----	
2532	DIN54233-3	3.15		0.66	
2553	DIN54233-3	2.419		-1.04	
2572	ISO105E04	2.97		0.24	
2582	DIN54233-3	1.845		-2.37	
2590	GB/T17593	2.712		-0.36	
2596	GB/T17593	2.461	C	-0.94	First reported --
2606	In house	2.65		-0.50	
2614	ISO105E04	3.202		0.78	
2637		2.25		-1.43	
2638	ISO105E04	n.d		-----	
2643	DIN54233-3	2.47		-0.92	
2644	ISO16711-2	3.0125		0.34	
2719	DIN54233-3	2.73		-0.31	
2723	DIN54233-3	n.d.	C	-----	First reported 5
2741	DIN54233-3	3.0		0.31	
2749	EN16711-2	2.612		-0.59	
2766	ISO105E04	2.5	C	-0.85	First reported 1.437
3100	DIN54233-3	2.8307		-0.08	
3146	DIN54233-3	3.250		0.89	
3150	EN16711-2	3.352		1.13	
3153	ISO105E04	2.875		0.02	
3154	DIN54233-3	3.80	C	2.17	First reported 4.64
3172	EN16711	2.84		-0.06	
3176	DIN54233-3	2.157		-1.65	

3190	DIN54233-3	2.5		-0.85
3197	ISO105E04	3.09		0.52
3200	EN16711-2	2.87		0.01
3209		-----		-----
3210	ISO105E04	4.78	R(0.01)	4.45
3214	DIN54233-3	2.96		0.22
3220	EN16711-2	3.52		1.52
3228	DIN54233-3	2.67		-0.45
3237	ISO105E04	2.78		-0.20

normality	OK
n	76
outliers	4
mean (n)	2.865
st.dev. (n)	0.3402
R(calc.)	0.953
R(EN16711-2:15)	1.203



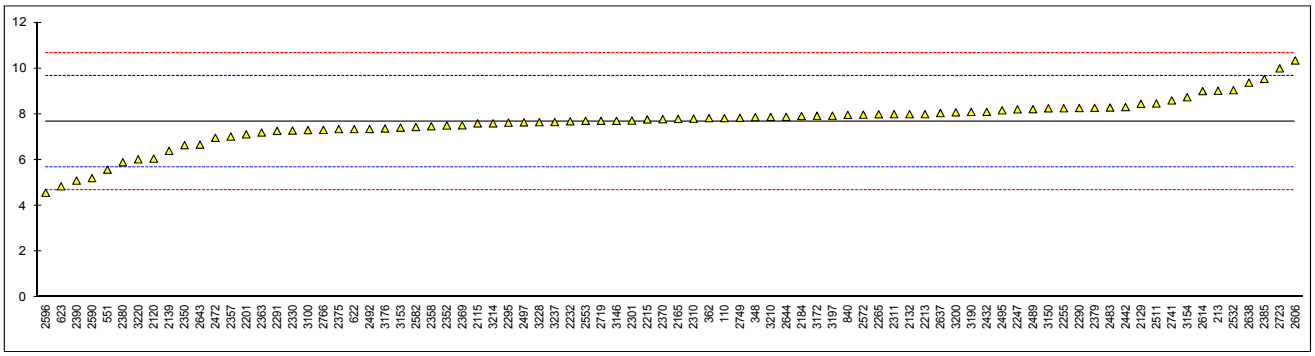


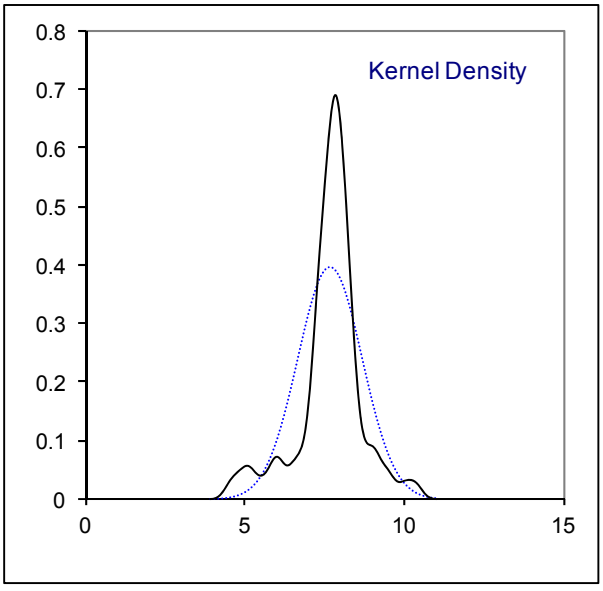
Determination of Cobalt as Co on sample #16625; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	7.829		0.16	
213	ISO105E04	9.02		1.36	
348	ISO105E04	7.871		0.20	
362		7.828		0.16	
551	ISO105E04	5.58		-2.09	
622	ISO105E04	7.350		-0.32	
623	ISO105E04	4.85		-2.83	
840	ISO105E04	7.97		0.30	
2115		7.60		-0.07	
2120	EN16711-2	6.06		-1.61	
2129	DIN54233-3	8.45		0.78	
2132	DIN54233-3	8.0		0.33	
2139	ISO105E04	6.40		-1.27	
2165	DIN54233-3	7.79		0.12	
2184	DIN54233-3	7.92		0.25	
2201	DIN54233-3	7.120		-0.55	
2213	ISO105E04	8.0		0.33	
2215	DIN54233-3	7.774		0.11	
2232	DIN54233-3	7.69		0.02	
2247	ISO105E04	8.21		0.54	
2255	DIN54233-3	8.26		0.59	
2265	DIN54233-3	7.997		0.33	
2290	DIN54233-3	8.27		0.60	
2291	GB/T17593	7.27		-0.40	
2295	EN16711-2	7.63		-0.04	
2301	DIN54233-3	7.720		0.05	
2310	ISO105E04	7.804		0.14	
2311	ISO105E04	8.0		0.33	
2330	ISO105E04	7.2850		-0.38	
2350	ISO105E04	6.648		-1.02	
2352	DIN54233-3	7.506		-0.16	
2357		7.024		-0.65	
2358	DIN54233-3	7.4720		-0.20	
2363	DIN54233-3	7.2		-0.47	
2369	DIN54233-3	7.516		-0.15	
2370	DIN54233-3	7.782		0.11	
2375	DIN54233-3	7.35		-0.32	
2379	DIN54233-3	8.275		0.61	
2380	DIN54233-3	5.900		-1.77	
2385	DIN54233-3	9.54		1.88	
2390	ISO105E04	5.10	C	-2.58	First reported 3.32
2432	ISO105E04	8.1017		0.43	
2442	In house	8.31		0.64	
2472	GB/T17593	6.97		-0.70	
2483	ISO105E04	8.29		0.62	
2489	ISO105E04	8.22		0.55	
2492	In house	7.354		-0.32	
2495	ISO105E04	8.17		0.50	
2497	DIN54233-3	7.642		-0.03	
2511	EN16711-2	8.468		0.80	
2520		-----		-----	
2532	DIN54233-3	9.05		1.39	
2553	DIN54233-3	7.710		0.04	
2572	ISO105E04	7.98		0.31	
2582	DIN54233-3	7.438		-0.23	
2590	GB/T17593	5.210		-2.47	
2596	GB/T17593	4.572		-3.11	
2606	In house	10.346		2.69	
2614	ISO105E04	9.010		1.35	
2637		8.05		0.38	
2638	ISO105E04	9.377		1.71	
2643	DIN54233-3	6.67		-1.00	
2644	ISO16711-2	7.8815		0.21	
2719	DIN54233-3	7.71		0.04	
2723	DIN54233-3	10		2.34	
2741	DIN54233-3	8.6		0.93	
2749	EN16711-2	7.843		0.18	
2766	ISO105E04	7.317		-0.35	
3100	DIN54233-3	7.3059		-0.36	
3146	DIN54233-3	7.710		0.04	
3150	EN16711-2	8.257		0.59	
3153	ISO105E04	7.412		-0.26	
3154	DIN54233-3	8.74		1.07	
3172	EN16711	7.93		0.26	
3176	DIN54233-3	7.374		-0.30	
3190	DIN54233-3	8.1		0.43	

3197	ISO105E04	7.93	0.26
3200	EN16711-2	8.08	0.41
3209		-----	-----
3210	ISO105E04	7.88	0.21
3214	DIN54233-3	7.60	-0.07
3220	EN16711-2	6.03	-1.64
3228	DIN54233-3	7.65	-0.02
3237	ISO105E04	7.66	-0.01

normality	not OK
n	82
outliers	0
mean (n)	7.668
st.dev. (n)	1.0053
R(calc.)	2.815
R(EN16711-2:15)	2.791





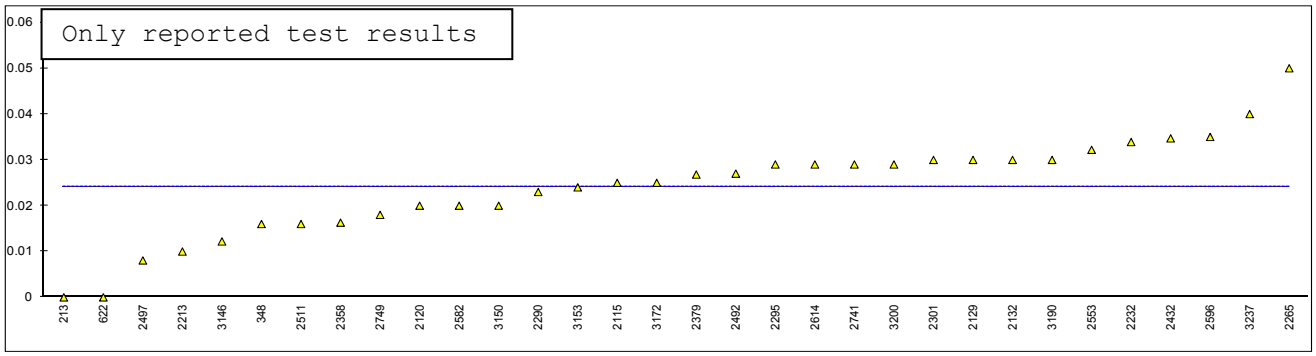
Determination of Mercury as Hg on sample #16625; results in mg/kg

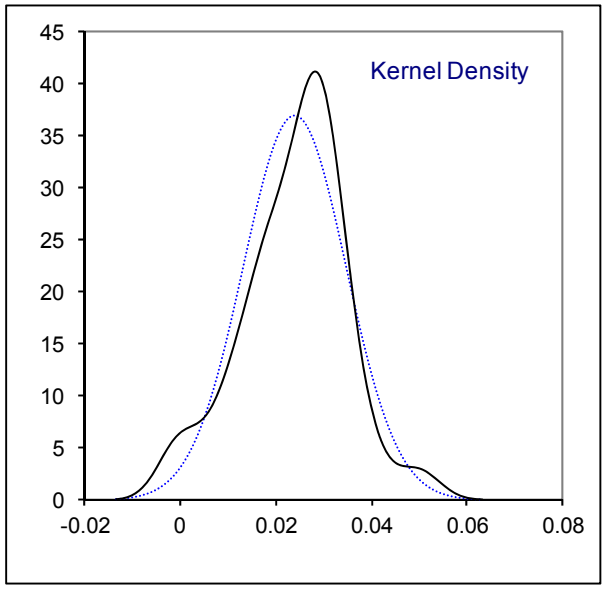
lab	method	value	mark	z(targ)	remarks
110	ISO105E04	<0.05		-----	
213	ISO105E04	0.0		-----	
348	ISO105E04	0.016		-----	
362		-----		-----	
551	ISO105E04	n.d.		-----	
622	ISO105E04	0.000		-----	
623	ISO105E04	n.d.		-----	
840	ISO105E04	ND		-----	
2115		0.025		-----	
2120	EN16711-2	0.020		-----	
2129	DIN54233-3	0.030		-----	
2132	DIN54233-3	0.030		-----	
2139	ISO105E04	< 0.01		-----	
2165	DIN54233-3	ND		-----	
2184	DIN54233-3	<0.02		-----	
2201	DIN54233-3	<0.01		-----	
2213	ISO105E04	0.01		-----	
2215	DIN54233-3	<0.005		-----	
2232	DIN54233-3	0.0339		-----	
2247	ISO105E04	ND		-----	
2255	DIN54233-3	nd		-----	
2265	DIN54233-3	0.050		-----	
2290	DIN54233-3	0.023		-----	
2291	GB/T17593	< 0.01		-----	
2295	EN16711-2	0.029		-----	
2301	DIN54233-3	0.030		-----	
2310	ISO105E04	Not Det.		-----	
2311	ISO105E04	Not Det.		-----	
2330	ISO105E04	ND		-----	
2350	ISO105E04	<0.005		-----	
2352	DIN54233-3	nd		-----	
2357		<0.02		-----	
2358	DIN54233-3	0.0163		-----	
2363	DIN54233-3	<0.02		-----	
2369	DIN54233-3	<0.01		-----	
2370	DIN54233-3	<0.02		-----	
2375	DIN54233-3	-----		-----	
2379	DIN54233-3	0.0268		-----	
2380	DIN54233-3	-----		-----	
2385	DIN54233-3	<0,01		-----	
2390	ISO105E04	ND		-----	
2432	ISO105E04	0.0347		-----	
2442	In house	-----		-----	
2472	GB/T17593	-----		-----	
2483	ISO105E04	ND		-----	
2489	ISO105E04	ND		-----	
2492	In house	0.027		-----	
2495	ISO105E04	<1		-----	
2497	DIN54233-3	0.00806	C	-----	First reported 0.0806
2511	EN16711-2	0.016		-----	
2520		-----		-----	
2532	DIN54233-3	Not Det.		-----	
2553	DIN54233-3	0.0322		-----	
2572	ISO105E04	<0.01		-----	
2582	DIN54233-3	0.02		-----	
2590	GB/T17593	< L.O.Q.		-----	
2596	GB/T17593	0.035		-----	
2606	In house	n.d.	C	-----	First reported 0.078
2614	ISO105E04	0.029		-----	
2637		<0,05		-----	
2638	ISO105E04	N/A		-----	
2643	DIN54233-3	< 0.01		-----	
2644	ISO16711-2	-----		-----	
2719	DIN54233-3	<0.02		-----	
2723	DIN54233-3	< 0.5		-----	
2741	DIN54233-3	0.029		-----	
2749	EN16711-2	0.018		-----	
2766	ISO105E04	Not Det.		-----	
3100	DIN54233-3	<0.01		-----	
3146	DIN54233-3	0.0122		-----	
3150	EN16711-2	0.02		-----	
3153	ISO105E04	0.024		-----	
3154	DIN54233-3	-----		-----	
3172	EN16711	0.025		-----	
3176	DIN54233-3	-----		-----	
3190	DIN54233-3	0.03		-----	

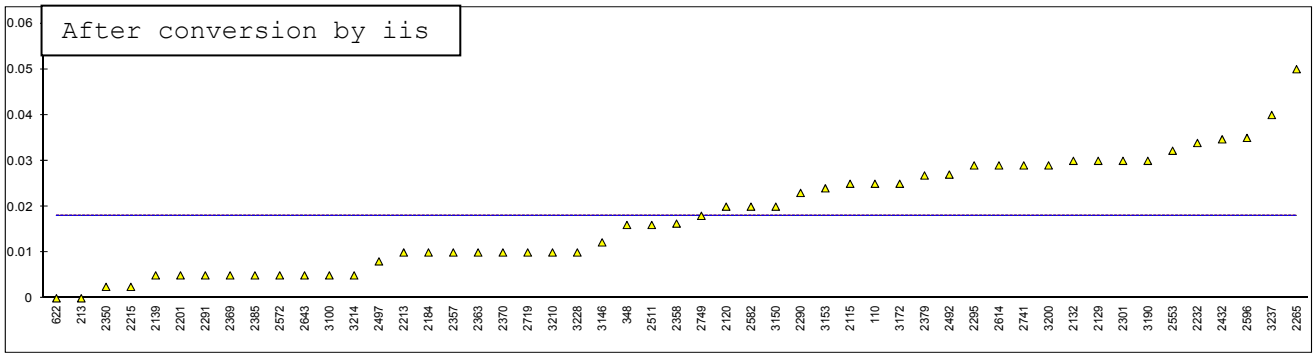
3197	ISO105E04	ND	-----
3200	EN16711-2	0.029	-----
3209		-----	-----
3210	ISO105E04	<0.020	-----
3214	DIN54233-3	<0.01	-----
3220	EN16711-2	ND	-----
3228	DIN54233-3	<0.02	-----
3237	ISO105E04	0.04	-----

		After conversion by iis *):
	normality	OK	OK
	n	32	51
	outliers	0	0
	mean (n)	0.0240	0.0179
	st.dev. (n)	0.01079	0.01206
	R(calc.)	0.0302	0.0338
	R(EN16711-2:15)	(0.0209)	(0.0156)

*) In the calculation of the mean, standard deviation, the reproducibility and in below graph, a reported value of '<x' is changed into x/2 (for example 0.02- into 0.01). This was done only for reported results <0.1 or smaller.







Determination of Antimony (Sb), Arsenic (As), Cadmium (Cd), Copper (Cu), Lead (Pb) and Nickel (Ni) on sample #16625; results in mg/kg

lab	method	Sb	As	Cd	Cu	Pb	Ni
110	ISO105E04	<0.05	<0.05	<0.05	<5.00	<0.05	<0.05
213	ISO105E04	0.0	0.0	0.0	0.0	0.0	0.0
348	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362		-----	-----	-----	-----	-----	-----
551	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
622	ISO105E04	0.002	0.000	0.000	0.096	0.000	0.000
623	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	ISO105E04	ND	ND	ND	ND	ND	ND
2115		-----	-----	-----	0.642	0.041	0.062
2120	EN16711-2	< 2,5	< 0,10	< 0,05	< 5,0	< 0,10	< 1,2
2129	DIN54233-3	<0,2	<0,1	<0,05	<5	<0,1	<0,4
2132	DIN54233-3	<5	<0.03	<0.03	<5	0.099	<0.5
2139	ISO105E04	< 5	< 0.1	< 0.05	< 5	< 0.1	< 0.5
2165	DIN54233-3	ND	ND	ND	ND	ND	ND
2184	DIN54233-3	<0.5	<0.02	<0.02	<0.5	<0.02	<0.5
2201	DIN54233-3	<1	<0.1	<0.03	<1	<0.1	<0.3
2213	ISO105E04	Not Detected	Not Detected	Not Detected	Not Detected	0.073	Not Detected
2215	DIN54233-3	<0.5	<0.02	<0.02	<5	<0.1	<0.1
2232	DIN54233-3	ND	0.0399	ND	ND	ND	ND
2247	ISO105E04	ND	ND	ND	ND	ND	ND
2255	DIN54233-3	nd	nd	nd	nd	nd	nd
2265	DIN54233-3	n. d.	n.d.	n.d.	n. d.	n.d.	0.277
2290	DIN54233-3	< 1.0	< 0.1	< 0.03	< 1.0	< 0.1	< 0.3
2291	GB/T17593	< 3.00	< 0.20	< 0.10	< 1.00	< 0.20	< 1.00
2295	EN16711-2	-----	-----	-----	-----	-----	-----
2301	DIN54233-3	ND	ND	ND	ND	ND	0.220
2310	ISO105E04	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	ISO105E04	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ISO105E04	ND	ND	ND	ND	ND	ND
2350	ISO105E04	<0.5	<0.02	<0.05	<5.0	<0.1	<0.5
2352	DIN54233-3	nd	nd	nd	nd	nd	nd
2357		<1	<0.2	<0.1	<5	<0.2	<0.5
2358	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	DIN54233-3	<1.0	<0.2	<0.1	<5.0	<0.2	<1.0
2369	DIN54233-3	<1.0	<0.1	<0.05	<5.0	<0.1	<0.5
2370	DIN54233-3	<1	<0.2	<0.1	<5	<0.2	<1
2375	DIN54233-3	-----	-----	-----	-----	-----	-----
2379	DIN54233-3	< 0.5	< 0.05	< 0.05	< 0.5	< 0.05	< 0.5
2380	DIN54233-3	-----	-----	-----	-----	-----	-----
2385	DIN54233-3	<0,1	<0,05	<0,01	<0,5	<0,1	<0,4
2390	ISO105E04	ND	ND	ND	ND	ND	ND
2432	ISO105E04	-----	-----	-----	-----	-----	-----
2442	In house	-----	-----	-----	-----	n.d.	-----
2472	GB/T17593	<0.35	-----	<0.06	<0.6	<0.35	<0.05
2483	ISO105E04	ND	ND	ND	0.33	ND	ND
2489	ISO105E04	ND	ND	ND	ND	ND	ND
2492	In house	-----	-----	-----	-----	-----	-----
2495	ISO105E04	<1	<1	<1	<1	<5	<1
2497	DIN54233-3	0.0237	0.0188	0.0067	1.594	0.132	0.154
2511	EN16711-2	-----	-----	-----	-----	0.044	-----
2520		-----	-----	-----	-----	-----	-----
2532	DIN54233-3	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2553	DIN54233-3	ND	0.0312	ND	ND	0.043	0.1233
2572	ISO105E04	<1.0	<0.1	< 0.03	< 1.0	< 0.1	< 0.3
2582	DIN54233-3	ND	0.015	0.05	***204.9***	ND	ND
2590	GB/T17593	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2596	GB/T17593	--	--	--	0.181	--	--
2606	In house	-----	-----	-----	-----	-----	-----
2614	ISO105E04	ND	ND	ND	ND	0.151	0.174
2637		<0,05	<0,1	<0,02	<0,2	<0,05	<0,2
2638	ISO105E04	N/A	N/A	n.d	n.d	n.d	n.d
2643	DIN54233-3	< 0.1	< 0.1	< 0.1	< 2.0	< 0.1	< 0.1
2644	ISO16711-2	-----	-----	-----	-----	-----	-----
2719	DIN54233-3	<0.5	<0.5	<0.1	<2	<0.5	<0.1
2723	DIN54233-3	< 1	< 1	< 1	n.d.	< 0.5	< 1
2741	DIN54233-3	< 2.0	< 0.05	< 0.02	< 2.0	0.084	< 0.3
2749	EN16711-2	0.005	0.001	0.008	0.427	0.115	0.103
2766	ISO105E04	Not detected	**0.49**	Not detected	0.48	Not detected	0.48
3100	DIN54233-3	<1	<0.1	<0.03	<1	<0.1	<0.3
3146	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3150	EN16711-2	<1,00	<0,05	<0,05	<0,35	0.126	<0,15
3153	ISO105E04	<1	<0.3	<0.03	2.119	<0.3	<1
3154	DIN54233-3	-----	-----	-----	-----	-----	-----
3172	EN16711	< 1	< 0.05	< 0.02	< 5	0.09	< 0.2
3176	DIN54233-3	-----	-----	-----	0.396	0.081	0.035
3190	DIN54233-3	<1.0	<1.0	<0.03	<1.0	<0.1	<0.3

3197	ISO105E04	ND	ND	ND	ND	ND	ND
3200	EN16711-2	<0.50	<0.10	<0.050	<5.0	<0.10	<0.40
3209		-----	-----	-----	-----	-----	-----
3210	ISO105E04	<30	<0.20	<0.10	<25	<0.20	<1.0
3214	DIN54233-3	<1	<0.1	<0.03	<1	<0.1	<0.3
3220	EN16711-2	ND	**0.21**	ND	0.31	ND	0.10
3228	DIN54233-3	<0.5	<0.02	<0.02	<0.5	<0.02	<0.5
3237	ISO105E04	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown
	n	65	58	64	69	69	68
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<5	<0.2	<0.1	<25	<0.2	<1
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(lit)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

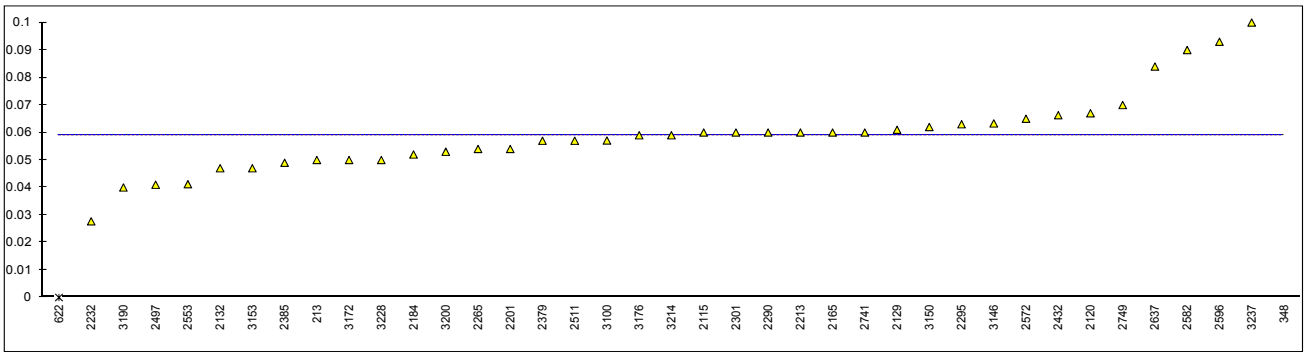
*) bold and underlined are marked as false positive test result

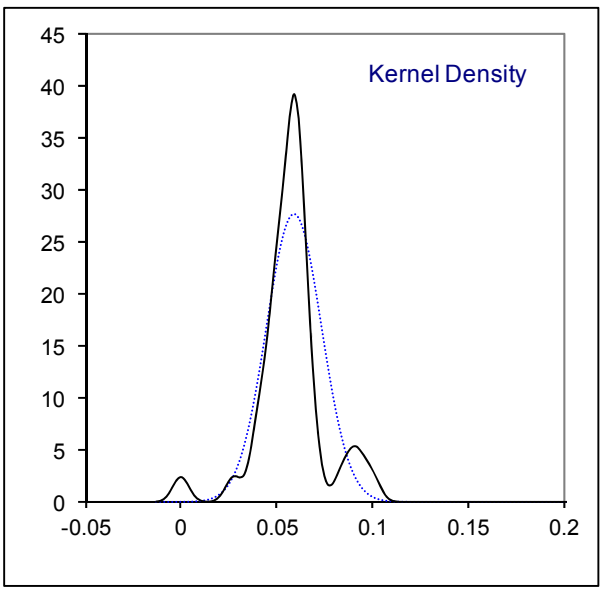
Determination of Cadmium as Cd on sample #16626; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	<0.05		-----	
213	ISO105E04	0.05		-----	
348	ISO105E04	0.532	R(0.01)	-----	
362		-----		-----	
551	ISO105E04	n.d.		-----	
622	ISO105E04	0.000	R(0.05)	-----	
623	ISO105E04	n.d.		-----	
840	ISO105E04	ND		-----	
2115		0.06		-----	
2120	EN16711-2	0.067		-----	
2129	DIN54233-3	0.061		-----	
2132	DIN54233-3	0.047		-----	
2139	ISO105E04	< 0.05		-----	
2165	DIN54233-3	0.06		-----	
2184	DIN54233-3	0.052		-----	
2201	DIN54233-3	0.054		-----	
2213	ISO105E04	0.06		-----	
2215	DIN54233-3	<0.02		-----	
2232	DIN54233-3	0.0277		-----	
2247	ISO105E04	ND		-----	
2255	DIN54233-3	nd		-----	
2265	DIN54233-3	0.054		-----	
2290	DIN54233-3	0.060		-----	
2291	GB/T17593	< 0.10		-----	
2295	EN16711-2	0.063		-----	
2301	DIN54233-3	0.060		-----	
2310	ISO105E04	Not Detected		-----	
2311	ISO105E04	Not Detected		-----	
2330	ISO105E04	ND		-----	
2350	ISO105E04	<0.05		-----	
2352	DIN54233-3	nd		-----	
2357		<0.1		-----	
2358	DIN54233-3	n.d.		-----	
2363	DIN54233-3	<0.1		-----	
2369	DIN54233-3	<0.05		-----	
2370	DIN54233-3	<0.1		-----	
2375	DIN54233-3	-----		-----	
2379	DIN54233-3	0.057		-----	
2380	DIN54233-3	-----		-----	
2385	DIN54233-3	0.049		-----	
2390	ISO105E04	ND		-----	
2432	ISO105E04	0.0663		-----	
2442	In house	-----		-----	
2472	GB/T17593	<0.06		-----	
2483	ISO105E04	ND		-----	
2489	ISO105E04	ND		-----	
2492	In house	-----		-----	
2495	ISO105E04	<1		-----	
2497	DIN54233-3	0.041		-----	
2511	EN16711-2	0.057		-----	
2520		-----		-----	
2532	DIN54233-3	Not Detected		-----	
2553	DIN54233-3	0.0412		-----	
2572	ISO105E04	0.065		-----	
2582	DIN54233-3	0.09		-----	
2590	GB/T17593	< L.O.Q.		-----	
2596	GB/T17593	0.093		-----	
2606	In house	-----		-----	
2614	ISO105E04	n.d		-----	
2637		0.084		-----	
2638	ISO105E04	n.d		-----	
2643	DIN54233-3	< 0.1		-----	
2644	ISO16711-2	-----		-----	
2719	DIN54233-3	<0.1		-----	
2723	DIN54233-3	< 1		-----	
2741	DIN54233-3	0.06		-----	
2749	EN16711-2	0.070		-----	
2766	ISO105E04	Not detected		-----	
3100	DIN54233-3	0.0571		-----	
3146	DIN54233-3	0.0633		-----	
3150	EN16711-2	0.062		-----	
3153	ISO105E04	0.047		-----	
3154	DIN54233-3	-----		-----	
3172	EN16711	0.05		-----	
3176	DIN54233-3	0.059		-----	
3190	DIN54233-3	0.04		-----	

3197	ISO105E04	ND	-----
3200	EN16711-2	0.053	-----
3209		-----	-----
3210	ISO105E04	<0.10	-----
3214	DIN54233-3	0.059	-----
3220	EN16711-2	ND	-----
3228	DIN54233-3	0.05	-----
3237	ISO105E04	0.10	-----

normality	suspect
n	37
outliers	2
mean (n)	0.0592
st.dev. (n)	0.01444
R(calc.)	0.0404
R(EN16711-2:15)	(0.0166)



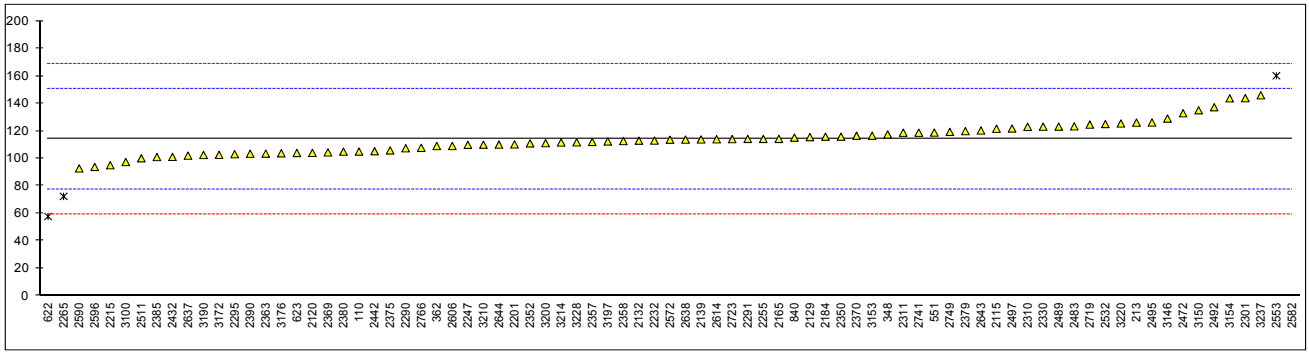


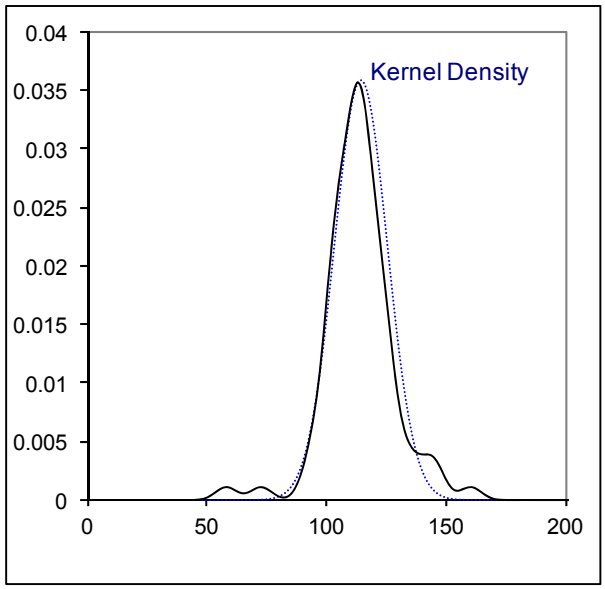
Determination of Copper as Cu on sample #16626; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	105		-0.50	
213	ISO105E04	126.03		0.65	
348	ISO105E04	117.417		0.18	
362		109		-0.28	
551	ISO105E04	118.8		0.26	
622	ISO105E04	57.754	R(0.01)	-3.09	
623	ISO105E04	103.96		-0.56	
840	ISO105E04	115.1		0.05	
2115		121.6		0.41	
2120	EN16711-2	104.0		-0.55	
2129	DIN54233-3	115.5		0.08	
2132	DIN54233-3	112.9		-0.07	
2139	ISO105E04	113.80		-0.02	
2165	DIN54233-3	114.21		0.01	
2184	DIN54233-3	115.8		0.09	
2201	DIN54233-3	110.20		-0.21	
2213	ISO105E04	Not Det.		-----	
2215	DIN54233-3	95.018		-1.05	
2232	DIN54233-3	113		-0.06	
2247	ISO105E04	109.87		-0.23	
2255	DIN54233-3	114.2		0.00	
2265	DIN54233-3	72.386	R(0.01)	-2.29	
2290	DIN54233-3	107.4		-0.37	
2291	GB/T17593	114.20		0.00	
2295	EN16711-2	103.1		-0.60	
2301	DIN54233-3	143.850		1.63	
2310	ISO105E04	123.01		0.49	
2311	ISO105E04	118.7		0.25	
2330	ISO105E04	123.1125		0.49	
2350	ISO105E04	115.819		0.09	
2352	DIN54233-3	110.927		-0.17	
2357		111.981		-0.12	
2358	DIN54233-3	112.6500		-0.08	
2363	DIN54233-3	103.5		-0.58	
2369	DIN54233-3	104.361		-0.53	
2370	DIN54233-3	116.50		0.13	
2375	DIN54233-3	105.79		-0.46	
2379	GB/T17593	119.840		0.31	
2380	DIN54233-3	104.910		-0.50	
2385	DIN54233-3	101		-0.72	
2390	ISO105E04	103.4		-0.59	
2432	ISO105E04	101.0839		-0.71	
2442	In house	105.20		-0.49	
2472	GB/T17593	132.85		1.03	
2483	ISO105E04	123.33		0.50	
2489	ISO105E04	123.13		0.49	
2492	In house	137.249		1.27	
2495	ISO105E04	126.10		0.66	
2497	DIN54233-3	121.74		0.42	
2511	EN16711-2	100.1		-0.77	
2520		-----		-----	
2532	DIN54233-3	125		0.60	
2553	DIN54233-3	160.1033	R(0.01)	2.52	
2572	ISO105E04	113.6		-0.03	
2582	DIN54233-3	427.04	C,R(0.01)	17.14	First reported 286.72
2590	GB/T17593	92.704		-1.17	
2596	GB/T17593	93.792		-1.11	
2606	In house	109.0		-0.28	
2614	ISO105E04	113.903		-0.01	
2637		102		-0.66	
2638	ISO105E04	113.694		-0.02	
2643	DIN54233-3	120.3		0.34	
2644	ISO16711-2	110.05		-0.22	
2719		124.6		0.57	
2723	DIN54233-3	114		-0.01	
2741	DIN54233-3	118.7		0.25	
2749	EN16711-2	119.3		0.28	
2766	ISO105E04	107.7		-0.35	
3100	DIN54233-3	97.3915		-0.92	
3146	DIN54233-3	129.0		0.82	
3150	EN16711-2	135.03		1.15	
3153	ISO105E04	116.527		0.13	
3154	DIN54233-3	143.7		1.62	
3172	EN16711	102.6		-0.63	
3176	DIN54233-3	103.742		-0.57	
3190	DIN54233-3	102.5		-0.64	

3197	ISO105E04	112.35	-0.10
3200	EN16711-2	111.1	-0.17
3209		-----	-----
3210	ISO105E04	110	-0.23
3214	DIN54233-3	111.6	-0.14
3220	EN16711-2	125.40	0.62
3228	DIN54233-3	111.70	-0.13
3237	ISO105E04	145.89	1.74

normality	OK
n	77
outliers	4
mean (n)	114.118
st.dev. (n)	11.1100
R(calc.)	31.108
R(EN16711-2:15)	51.125



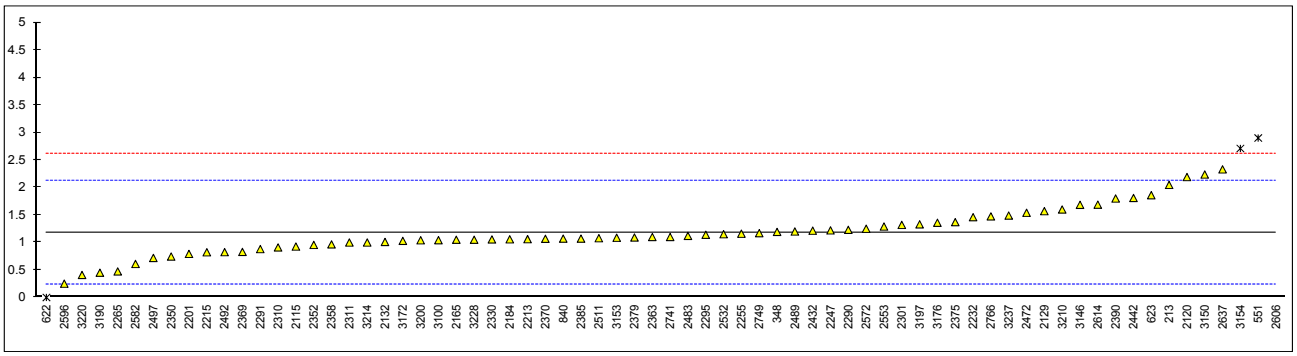


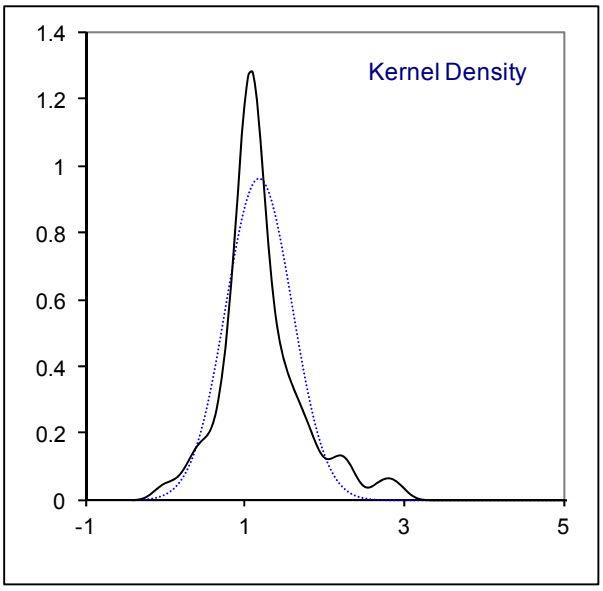
Determination of Lead as Pb on sample #16626; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO105E04	<0.05		<-2.39	False negative test result?
213	ISO105E04	2.0465		1.82	
348	ISO105E04	1.193		0.02	
362		-----		-----	
551	ISO105E04	2.9	R(0.05)	3.63	
622	ISO105E04	0.000	R(0.05)	-2.50	
623	ISO105E04	1.86		1.43	
840	ISO105E04	1.07		-0.24	
2115		0.926		-0.54	
2120	EN16711-2	2.19		2.13	
2129	DIN54233-3	1.57		0.82	
2132	DIN54233-3	1.01		-0.37	
2139	ISO105E04	< 0.1		<-2.29	False negative test result?
2165	DIN54233-3	1.05		-0.28	
2184	DIN54233-3	1.058		-0.27	
2201	DIN54233-3	0.792		-0.83	
2213	ISO105E04	1.06		-0.26	
2215	DIN54233-3	0.822		-0.76	
2232	DIN54233-3	1.46		0.58	
2247	ISO105E04	1.22		0.08	
2255	DIN54233-3	1.16		-0.05	
2265	DIN54233-3	0.474	C	-1.50	First reported 31.165
2290	DIN54233-3	1.23		0.10	
2291	GB/T17593	0.88		-0.64	
2295	EN16711-2	1.14		-0.09	
2301	DIN54233-3	1.320		0.29	
2310	ISO105E04	0.910		-0.58	
2311	ISO105E04	1.0		-0.39	
2330	ISO105E04	1.0562		-0.27	
2350	ISO105E04	0.745		-0.93	
2352	DIN54233-3	0.956		-0.48	
2357		-----		-----	
2358	DIN54233-3	0.9653		-0.46	
2363	DIN54233-3	1.1		-0.18	
2369	DIN54233-3	0.829		-0.75	
2370	DIN54233-3	1.067		-0.25	
2375	DIN54233-3	1.37		0.39	
2379	DIN54233-3	1.091		-0.20	
2380	DIN54233-3	-----		-----	
2385	DIN54233-3	1.07		-0.24	
2390	ISO105E04	1.80		1.30	
2432	ISO105E04	1.2147		0.07	
2442	In house	1.81		1.32	
2472	GB/T17593	1.54		0.75	
2483	ISO105E04	1.12		-0.13	
2489	ISO105E04	1.2		0.03	
2492	In house	0.825		-0.76	
2495	ISO105E04	<5		-----	
2497	DIN54233-3	0.719		-0.98	
2511	EN16711-2	1.078		-0.22	
2520		-----		-----	
2532	DIN54233-3	1.15		-0.07	
2553	DIN54233-3	1.2906		0.23	
2572	ISO105E04	1.25		0.14	
2582	DIN54233-3	0.61		-1.21	
2590	GB/T17593	< L.O.Q.		-----	
2596	GB/T17593	0.251		-1.97	
2606	In house	12.01	C,R(0.01)	22.87	First reported 20.9
2614	ISO105E04	1.688		1.07	
2637		2.33		2.42	
2638	ISO105E04	n.d		-----	
2643	DIN54233-3	< 0.1		<-2.29	False negative test result?
2644	ISO16711-2	-----		-----	
2719	DIN54233-3	-----		-----	
2723	DIN54233-3	< 0.5		-----	
2741	DIN54233-3	1.1		-0.18	
2749	EN16711-2	1.170		-0.03	
2766	ISO105E04	1.475		0.62	
3100	DIN54233-3	1.0421		-0.30	
3146	DIN54233-3	1.685		1.06	
3150	EN16711-2	2.236		2.22	
3153	ISO105E04	1.086		-0.21	
3154	DIN54233-3	2.71	R(0.05)	3.22	
3172	EN16711	1.03		-0.32	
3176	DIN54233-3	1.359		0.37	
3190	DIN54233-3	0.45		-1.55	

3197	ISO105E04	1.33	0.31
3200	EN16711-2	1.04	-0.30
3209		-----	-----
3210	ISO105E04	1.60	0.88
3214	DIN54233-3	1.00	-0.39
3220	EN16711-2	0.41	-1.63
3228	DIN54233-3	1.05	-0.28
3237	ISO105E04	1.49	0.65

normality	OK
n	66
outliers	4
mean (n)	1.184
st.dev. (n)	0.4151
R(calc.)	1.162
R(EN16711-2:15)	1.326





Determination of Antimony (Sb), Arsenic (As), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Mercury (Hg) and Nickel (Ni) on sample #16626; results in mg/kg

lab	method	Sb	As	Cr	Co	Hg	Ni
110	ISO105E04	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
213	ISO105E04	0.0	0.0	0.0	0.0	0.0	0.0
348	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
362		-----	-----	-----	-----	-----	-----
551	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
622	ISO105E04	0.060	0.000	0.000	0.000	0.000	0.000
623	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	ISO105E04	ND	ND	ND	ND	ND	ND
2115		-----	-----	0.012	0.048	0.008	0.115
2120	EN16711-2	< 2,5	< 0,10	< 0,50	< 1,2	< 0,013	< 1,2
2129	DIN54233-3	<0,2	<0,1	<0,2	<0,2	<0,02	<0,4
2132	DIN54233-3	<5	<0.03	<0.5	<0.5	<0.005	<0.5
2139	ISO105E04	< 5	< 0.1	< 0.5	< 0.5	< 0.01	< 0.5
2165	DIN54233-3	ND	ND	ND	ND	ND	ND
2184	DIN54233-3	<0.5	<0.02	<0.5	<0.5	<0.02	<0.5
2201	DIN54233-3	<1	<0.1	<0.5	<0.3	<0.01	<0.3
2213	ISO105E04	Not Detected	Not Detected	Not Detected	**100.5**	Not Detected	0.18
2215	DIN54233-3	<0.5	<0.02	<0.1	<0.1	<0.005	<0.1
2232	DIN54233-3	ND	0.0287	ND	ND	0.00685	ND
2247	ISO105E04	ND	ND	ND	ND	ND	ND
2255	DIN54233-3	nd	nd	nd	nd	nd	nd
2265	DIN54233-3	n.d.	n.d.	n.d.	n.d.	0.013	0.202
2290	DIN54233-3	< 1.0	< 0.1	< 0.5	< 0.3	< 0.01	< 0.3
2291	GB/T17593	< 3.00	< 0.20	< 0.50	< 1.00	< 0.01	< 1.00
2295	EN16711-2	-----	-----	-----	-----	-----	0.118
2301	DIN54233-3	ND	ND	ND	ND	ND	0.260
2310	ISO105E04	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2311	ISO105E04	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	ISO105E04	ND	ND	ND	ND	ND	ND
2350	ISO105E04	<0.5	<0.02	<0.1	<0.1	<0.005	<0.5
2352	DIN54233-3	nd	nd	nd	nd	nd	nd
2357		<1	<0.2	<0.5	<1	<0.02	<0.5
2358	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	DIN54233-3	<1.0	<0.2	<1.0	<1.0	<0.02	<1.0
2369	DIN54233-3	<1.0	<0.1	<0.5	<0.5	<0.01	<0.5
2370	DIN54233-3	<1	<0.2	<0.5	<0.5	<0.02	<1
2375	DIN54233-3	-----	-----	-----	-----	-----	-----
2379	DIN54233-3	< 0.5	< 0.05	< 0.5	< 0.5	< 0.02	< 0.5
2380	DIN54233-3	-----	-----	-----	-----	-----	-----
2385	DIN54233-3	<0,1	<0,05	<0,1	<0,1	<0,01	<0,4
2390	ISO105E04	ND	ND	ND	ND	ND	ND
2432	ISO105E04	-----	-----	-----	-----	-----	-----
2442	In house	-----	-----	-----	-----	-----	-----
2472	GB/T17593	<0.35	-----	<0.06	<0.1	-----	<0.05
2483	ISO105E04	ND	ND	0.55	ND	ND	0.61
2489	ISO105E04	ND	ND	ND	ND	ND	ND
2492	In house	-----	-----	-----	-----	-----	-----
2495	ISO105E04	<1	<1	<1	<1	<1	<1
2497	DIN54233-3	0.037	0.018	0.251	0.042	0.0001	0.152
2511	EN16711-2	-----	-----	-----	-----	-----	0.146
2520		-----	-----	-----	-----	-----	-----
2532	DIN54233-3	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2553	DIN54233-3	ND	0.0317	ND	ND	ND	0.116
2572	ISO105E04	< 1.0	< 0.1	< 0.5	< 0.3	< 0.01	< 0.3
2582	DIN54233-3	ND	ND	ND	ND	ND	ND
2590	GB/T17593	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2596	GB/T17593	--	--	--	--	0.007	0.081
2606	In house	-----	-----	-----	-----	n.d.	-----
2614	ISO105E04	ND	ND	ND	ND	ND	0.129
2637		<0,05	<0,1	<0,5	<0,1	<0,05	<0,2
2638	ISO105E04	N/A	N/A	n.d	n.d	N/A	n.d
2643	DIN54233-3	< 0.1	< 0.1	< 0.1	< 0.1	< 0.01	< 0.1
2644	ISO16711-2	-----	-----	-----	-----	-----	-----
2719	DIN54233-3	<0.5	<0.5	<0.1	<0.1	<0.02	<0.5
2723	DIN54233-3	< 1	< 1	< 0.5	< 1	< 0.5	< 1
2741	DIN54233-3	< 2.0	< 0.05	< 0.1	< 0.3	< 0.005	< 0.3
2749	EN16711-2	0.008	0.004	0.048	0.030	0.005	0.117
2766	ISO105E04	Not detected	Not detected	0.49	Not detected	Not detected	Not detected
3100	DIN54233-3	<1	<0.1	<0.5	<0.3	<0.01	<0.3
3146	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3150	EN16711-2	<1,00	<0,05	<0,05	<0,05	<0,02	0.156
3153	ISO105E04	<1	<0.3	<0.5	<1	<0.01	<1
3154	DIN54233-3	-----	-----	-----	-----	-----	-----
3172	EN16711	< 1	< 0.05	< 0.1	< 0.1	< 0.005	0.22
3176	DIN54233-3	-----	-----	-----	-----	-----	0.068
3190	DIN54233-3	<1.0	<0.1	<0.5	<0.3	<0.01	<0.3

3197	ISO105E04	ND	ND	ND	ND	ND	ND
3200	EN16711-2	<0.50	<0.10	<0.40	<0.40	<0.020	<0.40
3209		-----	-----	-----	-----	-----	-----
3210	ISO105E04	<30	<0.20	<1.0	<1.0	<0.02	<1.0
3214	DIN54233-3	<1	<0.1	<0.5	<0.3	<0.01	<0.3
3220	EN16711-2	ND	0.2	ND	ND	ND	ND
3228	DIN54233-3	<0.5	<0.02	<0.5	<0.5	<0.02	<0.5
3237	ISO105E04	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown
	n	65	61	69	66	65	71
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<5	<0.2	<1	<1	<0.02	<1
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(lit)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

*) bold and underlined are marked as false positive test result

APPENDIX 2

Accreditation status and dilution ratio as reported by participants

labnrs	Is your laboratory accredited?	What ratio was used in gram textile per ml?	Remarks on Additional Questions:
110	No	1 gram textile per 50 ml perspiration liquid	
213	No	1 gram textile per 50 ml perspiration liquid	
348	Yes	1 gram textile per 50 ml perspiration liquid	
362	No	1 gram textile per 50 ml perspiration liquid	
551	No	1 gram textile per 50 ml perspiration liquid	
622	Yes	1 gram textile per 50 ml perspiration liquid	
623	Yes	1 gram textile per 50 ml perspiration liquid	
840	Yes	1 gram textile per 50 ml perspiration liquid	
2115	No	1 gram textile per 50 ml perspiration liquid	
2120	Yes	1 gram textile per 50 ml perspiration liquid	Oeko-tex used 1g/ 20 ml. Not accredited for mercury
2129	Yes	1 gram textile per 50 ml perspiration liquid	
2132	No	1 gram textile per 50 ml perspiration liquid	
2139	Yes	1 gram textile per 50 ml perspiration liquid	
2165	No	1 gram textile per 50 ml perspiration liquid	
2184	No	1 gram textile per 50 ml perspiration liquid	
2201	Yes	1 gram textile per 50 ml perspiration liquid	
2213	Yes	1 gram textile per 50 ml perspiration liquid	
2215	Yes	1 gram textile per 50 ml perspiration liquid	
2232	Yes	1 gram textile per 50 ml perspiration liquid	
2247	No	1 gram textile per 50 ml perspiration liquid	
2255	Yes	1 gram textile per 50 ml perspiration liquid	
2265	No	1 gram textile per 50 ml perspiration liquid	
2290	---	---	
2291	Yes	1 gram textile per 20 ml perspiration liquid	
2295	Yes	1 gram textile per 50 ml perspiration liquid	
2301	Yes	1 gram textile per 50 ml perspiration liquid	
2310	Yes	1 gram textile per 50 ml perspiration liquid	
2311	Yes	1 gram textile per 50 ml perspiration liquid	
2330	No	1 gram textile per 50 ml perspiration liquid	
2350	---	1 gram textile per 50 ml perspiration liquid	
2352	Yes	1 gram textile per 50 ml perspiration liquid	
2357	Yes	---	
2358	Yes	1 gram textile per 50 ml perspiration liquid	
2363	No	1 gram textile per 50 ml perspiration liquid	
2369	Yes	1 gram textile per 50 ml perspiration liquid	
2370	Yes	1 gram textile per 50 ml perspiration liquid	
2375	Yes	1 gram textile per 50 ml perspiration liquid	-
2379	No	1 gram textile per 50 ml perspiration liquid	
2380	Yes	1 gram textile per 50 ml perspiration liquid	
2385	Yes	1 gram textile per 50 ml perspiration liquid	
2390	Yes	1 gram textile per 50 ml perspiration liquid	
2432	No	1 gram textile per 50 ml perspiration liquid	
2442	Yes	1 gram textile per 50 ml perspiration liquid	
2472	Yes	1 gram textile per 50 ml perspiration liquid	
2483	Yes	1 gram textile per 50 ml perspiration liquid	
2489	No	0.5 gram textile per 15 ml perspiration liquid	
2492	Yes	1 gram textile per 20 ml perspiration liquid	

labnrs	Is your laboratory accredited?	What ratio was used in gram textile per ml?	Remarks on Additional Questions:
2495	Yes	1 gram textile per 50 ml perspiration liquid	
2497	Yes	1 gram textile per 50 ml perspiration liquid	
2511	Yes	1 gram textile per 50 ml perspiration liquid	
2520	--	--	
2532	Yes	0.5 gram textile per 25 ml perspiration liquid	
2553	Yes	1 gram textile per 50 ml perspiration liquid	
2572	Yes	1 gram textile per 50 ml perspiration liquid	
2582	Yes	1 gram textile per 50 ml perspiration liquid	
2590	Yes	1 gram textile per 20 ml perspiration liquid	Method used for all metals is GB/T 17593
2596	No	1 gram textile per 20 ml perspiration liquid	
2606	Yes	1 gram textile per 50 ml perspiration liquid	
2614	Yes	1 gram textile per 50 ml perspiration liquid	
2637	Yes	1 gram textile per 50 ml perspiration liquid	
2638	No	1 gram textile per 50 ml perspiration liquid	
2643	---	---	
2644	No	1 gram textile per 50 ml perspiration liquid	
2719	Yes	1 gram textile per 50 ml perspiration liquid	
2723	Yes	1 gram textile per 50 ml perspiration liquid	
2741	Yes	1 gram textile per 50 ml perspiration liquid	
2749	Yes	1 gram textile per 50 ml perspiration liquid	
2766	No	1 gram textile per 50 ml perspiration liquid	
3100	Yes	0.5 gram textile per 25 ml perspiration liquid	
3146	Yes	1 gram textile per 50 ml perspiration liquid	low sample amount
3150	No	1 gram textile per 50 ml perspiration liquid	
3153	Yes	1 gram textile per 50 ml perspiration liquid	
3154	Yes	1 gram textile per 50 ml perspiration liquid	
3172	Yes	1 gram textile per 50 ml perspiration liquid	
3176	Yes	1 gram textile per 50 ml perspiration liquid	
3190	Yes	1 gram textile per 50 ml perspiration liquid	
3197	Yes	1 gram textile per 50 ml perspiration liquid	
3200	No	1 gram textile per 50 ml perspiration liquid	
3209	--	--	
3210	Yes	1 gram textile per 50 ml perspiration liquid	
3214	Yes	1 gram textile per 50 ml perspiration liquid	
3220	Yes	1 gram textile per 50 ml perspiration liquid	
3228	Yes	1 gram textile per 50 ml perspiration liquid	
3237	Yes	1 gram textile per 50 ml perspiration liquid	

APPENDIX 3

Number of participants per country:

3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	EGYPT
1 lab in	FRANCE
7 labs in	GERMANY
6 labs in	HONG KONG
9 labs in	INDIA
3 labs in	INDONESIA
6 labs in	ITALY
1 lab in	MOROCCO
14 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	PORTUGAL
1 lab in	SINGAPORE
3 labs in	SOUTH KOREA
1 lab in	SPAIN
2 labs in	SRI /LANKA
2 labs in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
1 lab in	TUNISIA
5 labs in	TURKEY
1 lab in	U.A.E.
1 lab in	U.S.A.
3 labs in	VIETNAM

APPENDIX 4

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 calculations
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

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