Results of Proficiency Test Heavy Metals by perspiration in textile October 2015

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's, many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, 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) and Thai Green Label (Thailand).

Since 2002 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for perspirated metals in textile. It was decided to continue this scheme as part of the proficiency testing program 2015/2016.

In this 2015 interlaboratory study 88 laboratories in 23 different countries participated. See appendix 3 for the number of participants per country. In this report the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory.

It was decided to use 2 different textile samples, both positive on metals, and to request the participants to use a solid/liquid ratio of 1/50 by preference (see paragraph 6 and the PT report iis07A05). Participants were also requested to report test results with at least one extra figure. These unrounded results were preferably used for the statistical evaluations.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 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 internet site 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

The materials used in this proficiency test were prepared by subcontractors. The two different coloured finely cut textile samples, #15205 and #15206 were each well mixed and divided over 100 subsamples of approx. 3 grams. The samples were labelled and tested for homogeneity on 8 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted, ISO17025 accredited laboratory. See the following tables for the homogeneity test results.

	Perspirated Copper in mg/kg
Sample #15205-1	29.6
Sample #15205-2	30.6
Sample #15205-3	29.1
Sample #15205-4	31.3
Sample #15205-5	32.7
Sample #15205-6	33.0
Sample #15205-7	29.4
Sample #15205-8	30.7

Table 1: homogeneity test results of subsamples #15205

	Perspirated Copper in mg/kg
Sample #15206-1	20.2
Sample #15206-2	18.8
Sample #15206-3	20.9
Sample #15206-4	20.6
Sample #15206-5	21.3
Sample #15206-6	20.2
Sample #15206-7	21.3
Sample #15206-8	21.0

Table 2: homogeneity test results of subsamples #15206

From the above results of the homogeneity tests, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Perspirated Copper in mg/kg	Perspirated Copper in mg/kg		
r (observed) #15205	4.1			
r (observed) #15206		2.3		
reference method	DIN54233-3:2010	DIN54233-3:2010		
0.3 x R (reference method)	4.1	2.8		

Table 3: repeatability of subsamples #15205 and #15206

The calculated repeatability of each subsample is in good agreement with 0.3 times the estimated target reproducibility of the reference test method. Therefore, homogeneity of both subsamples was assumed.

To each of the participating laboratories, two samples, one of sample #15205 and one of sample #15206 were sent on October 14, 2015.

2.5 ANALYSES

The participants were requested to determine on both samples: perspirated heavy metals: Arsenic, Antimony, 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 E-DIN54233-3:2010. It was explicitly requested to treat the samples as if they were routine samples and to report the analytical 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 cannot be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.

A form to confirm receipt of the samples and a letter of instructions were added to the samples.

3 RESULTS

During four weeks after sample dispatch, the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk./sgs-iis-cts/. The original data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder was sent to those laboratories that had not yet reported.

Shortly after the deadline, the available results were screened for suspect data. A 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 (raw data of the) reported results.

Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 4.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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 results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 4, no.18). Stragglers are marked by D(0.05) for the Dixon 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 the averages and the 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 each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 4; nos.14 and 15). Also a normal Gauss curve was projected over the Kernel Density Graph.

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 spread 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 in accordance with:

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

The $z_{(target)}$ scores are listed in the 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 maybe as follows:

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

4 EVALUATION

During the execution of this proficiency test, no problems occurred with sample dispatch. Four laboratories reported test results after the final reporting date and three other participating laboratories did not report any test results.

The 85 reporting laboratories did report in total 514 numerical results.

Observed were 15 statistical outlying results, which is 2.9% of the numerical 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.

4.1 EVALUATION PER SAMPLE AND PER METAL

In this section, the determinations per sample and perspirated metal are discussed. All statistical results reported on the samples are summarised in appendix 1.

For sample #15205

Cadmium:

The determination of this metal may be problematic at a perspiration level of 2.86 mg/kg. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of draft-DIN54233-3:10 (table A.1).

Copper:

The determination of this metal was not problematic at a perspiration level of 33.6 mg/kg. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility of draft-DIN54233-3:10 (table A.1).

Nickel:

The determination of this metal may be problematic at a perspiration level of 3.00 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of draft-DIN54233-3:10 (table A.1).

Other metals:

None of the participants did report positive test results for any of the other metals on sample #15205.

For sample #15206

Copper:

The determination of this metal was not problematic at a perspiration level of 30.0 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility of draft-DIN54233-3:10 (table A.1).

Nickel:

The determination of this metal was not problematic at a perspiration level of 2.82 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility of draft-DIN54233-3:10 (table A.1).

Other metals:

None of the participants, except four, did report positive test results for any of the other metals on sample #15206. Four participants reported test results between 0.34 and 0.751 mg/kg for Cadmium.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities from the reference test method draft-DIN54233-3:10 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 draft-DIN54233-3:10 are compared in the next two tables.

Parameter	Unit	n	average	2.8 * sd	R (target)
Perspirated Cadmium	mg/kg	79	2.86	0.95	0.80
Perspirated Copper	mg/kg	83	33.6	10.2	15.1
Perspirated Nickel	mg/kg	80	3.00	0.94	0.84

Table 4: reproducibilities of perspirated metals in sample #15205

Parameter	unit	n	average	2.8 * sd	R (target)
Perspirated Copper	mg/kg	82	30.0	7.7	13.4
Perspirated Nickel	mg/kg	80	2.82	0.87	0.79

Table 5: reproducibilities of perspirated metals in sample #15206

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories have hardly any difficulties with the analysis when compared with the target reproducibility of the reference test method draft-DIN54233-3:10. See also the discussions in paragraphs 4.1 and 6.

5 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2015 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.	Oct.	Oct.	Oct.	Oct.	Oct.	Oct.	Nov.	draft-DIN
	2015	2014	2013	2012	2011	2010	2009	2008	<i>54</i> 233-3
Arsenic									20%
Antimony		16%			19%		15%	16%	20%
Cadmium	12%		9%	14%		14%			10%
Chromium			15%		19%				15%
Cobalt		14%		11%	8%	11%		10%	13%
Copper	9-11%	10%	10%		22%		16-17%	9%	16%
Lead									40%
Mercury				41%					31%
Nickel	11%		11-13%		10-14%	7%		8%	10%

Table 6: development of uncertainties over the last years

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	Class 2: in	Class 3: with no	Class 4:
	clothes	direct skin	direct skin	Decoration
		contact	contact	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".

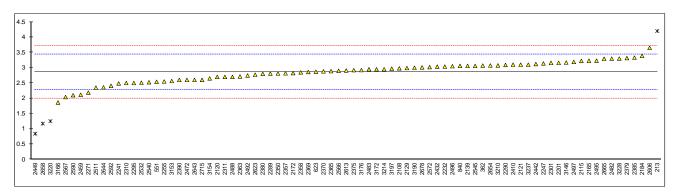
It should be noticed that for the results reported in this proficiency test, all participants have probably performed the acid perspiration step according to almost the same conditions. Differences in sample intake and perspiration time and temperature may be parameters of importance. In the past, the 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 draft test method DIN 54233-3:2010.

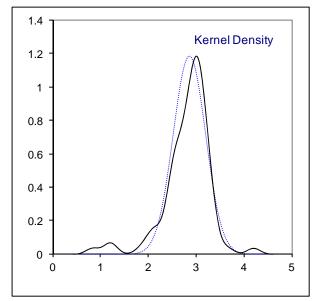
APPENDIX 1

Determination of Cadmium as Cd on sample #15205; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110					
213	ISO105E04	4.201	R(0.05)	4.69	
362 551	In house ISO105E04	3.0695 2.54		0.73 -1.12	
623	DIN54233-3	2.87		0.03	
840	ISO105E04	3.06		0.70	
2108	In house	2.98		0.42	
2115	DIN54233-3	3.222		1.26	
2120 2121	DIN54233-3 In house	2.700 3.10	С	-0.56 0.84	First reported 6.14
2121	DIN EN16711-2	2.99	C	0.45	riist reported 0.14
2139	ISO105E04	3.06		0.70	
2165	DIN54233-3	3.23		1.29	
2172	ISO105E04	2.824		-0.13	
2184 2190	DIN54233-3	3.388		1.84	
2201	DIN54233-3	3.160		1.05	
2232	DIN54233-3	3.0366		0.62	
2241	ISO105E04	2.485		-1.31	
2247	ISO105E04	3.14		0.98	
2255 2271	DIN54233-3 GB/T17593	2.55 2.184		-1.08 -2.36	
2289	DIN54233-3	2.104		-2.36 -0.18	
2290	DIN54233-3	3.09		0.80	
2295	DIN54233-3	2.5		-1.26	
2301	DIN54233-3	3.16		1.05	
2310	ISO105E04	2.5		-1.26 -0.56	
2311 2350	ISO105E04 ISO105E04	2.7 2.811	С	-0.56 -0.17	First reported 1.514
2357	DIN54233-3	2.817	Ü	-0.15	Thotropolica north
2358	ISO105E04	2.85		-0.04	
2363	DIN54233-3	2.714		-0.51	
2365	DIN54233-3	2.888		0.10	
2369 2370	ISO105E04 DIN54233-3	2.86 2.88		0.00 0.07	
2375	In house	2.92		0.21	
2379	DIN54233-3	3.316		1.59	
2380	ISO105E04	2.802		-0.20	
2385 2390	DIN54233-3	3.33 2.60		1.64 -0.91	
2390	ISO105E04 DIN54233-3	3.1		0.84	
2432	ISO105E04	3.036		0.61	
2442	In house	3.12		0.91	
2449	DIN54233-3	0.84	R(0.01)	-7.06	5 1
2459	DIN54233-3	2.116	С	-2.60	First reported 0.948
2472 2482	GB/T17593 prEN16711	2.6 3.298		-0.91 1.53	
2483	ISO105E04	2.95		0.31	
2488	ISO105E04	2.70		-0.56	
2492	In house	2.745		-0.40	
2495	DIN54233-3	3.23		1.29	
2496 2497	DIN54233-3 DIN54233-3	3.050 3.19	С	0.66 1.15	First reported 1.726
2511	DIN54233-3	2.348	J	-1.79	apolica in Eo
2532	DIN54233-3	2.51		-1.22	
2540	DIN54233-3	2.525		-1.17	
2545 2566	GB/T17593	3.06		0.70	
2566 2567	ISO105E04 DIN54233-3	2.90 2.04	С	0.14 -2.87	First reported 2.61
2572	DIN54233-3	3.02	J	0.56	aponou z.o.
2590	ISO105E04	2.10		-2.66	
2592	ISO105E04	2.4092		-1.58	
2606 2613	In house ISO105E04	3.65 2.91		2.76 0.17	
2623	GB/T17593	2.772		-0.31	
2638	ISO105E04	n.d			
2643	DIN54233-3	2.6		-0.91	
2644	DIN54233-3	2.36		-1.75	
2654 2658	ISO105E04	3.073	P(0.04)	0.74 -5.91	
2658 2665	ISO105E04 prEN16711	1.17 3.293	R(0.01)	-5.91 1.51	
2678	DIN54233-3	3.01		0.52	
2715	GB/T17593	2.603		-0.90	

3118				
3146	DIN54233-3	3.17		1.08
3153	ISO105E04	2.565		-1.03
3154	DIN54233-3	2.65		-0.74
3166	In house	1.86		-3.50
3172	DIN54233-3	2.95		0.31
3176	DIN54233-3	2.922		0.22
3190	DIN54233-3	3.00		0.49
3197	DIN54233-3	2.97		0.38
3210	ISO105E04	3.075		0.75
3214	DIN54233-3	2.95		0.31
3220	ISO105E04	1.25	R(0.01)	-5.63
3228	DIN54233-3	3.3		1.54
3232	ISO105E04	n.d		
3237	In house	3.10		0.84
	normality	OK		
	n	79		
	outliers	4		
	mean (n)	2.860		
	st.dev. (n)	0.3379		
	R(calc.)	0.946		
	R(DIN54233-3:2010)	0.801		
	11(2111012000.2010)	0.001		

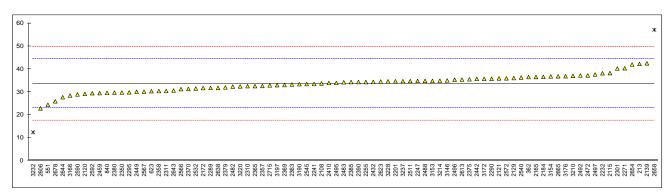


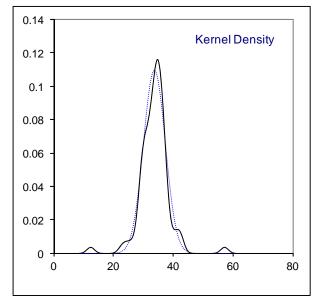


Determination of Copper as Cu on sample #15205; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110					
213	ISO105E04	42.223		1.59	
362	In house	36.47		0.52	
551	ISO105E04	24.39		-1.72	
623 840	DIN54233-3 ISO105E04	30.31 29.6		-0.62 -0.75	
2108	In house	33.7		0.73	
2115	DIN54233-3	38.213		0.85	
2120	DIN54233-3	29.10		-0.84	
2121	In house	35.9	С	0.42	First reported 7.19
2129 2139	DIN EN16711-2	36.03		0.44 1.64	
2165	ISO105E04 DIN54233-3	42.46 36.50		0.53	
2172	ISO105E04	31.66		-0.37	
2184	DIN54233-3	36.54		0.54	
2190					
2201	DIN54233-3	34.650		0.19	
2232 2241	DIN54233-3 ISO105E04	38.142 33.445		0.84 -0.04	
2247	ISO105E04	34.76		0.21	
2255	DIN54233-3	34.3		0.12	
2271	GB/T17593	40.334		1.24	
2289	DIN54233-3	31.72		-0.36	
2290	DIN54233-3	35.73		0.39	
2295 2301	DIN54233-3 DIN54233-3	29.8 40.22		-0.71 1.22	
2310	ISO105E04	32.5		-0.21	
2311	ISO105E04	30.4		-0.60	
2350	ISO105E04	29.73		-0.73	
2357	DIN54233-3	32.585		-0.20	
2358 2363	ISO105E04	30.37 33.192		-0.61 -0.08	
2365	DIN54233-3 DIN54233-3	32.518		-0.08	
2369	ISO105E04	32.99		-0.12	
2370	DIN54233-3	31.3		-0.44	
2375	In house	35.49		0.34	
2379	DIN54233-3	31.934		-0.32	
2380 2385	ISO105E04 DIN54233-3	29.709 34.3		-0.73 0.12	
2390	ISO105E04	34.30		0.12	
2410	DIN54233-3	33.9		0.05	
2432	ISO105E04	34.374		0.14	
2442	In house	35.70		0.38	
2449 2459	DIN54233-3 DIN54233-3	30.05 29.463		-0.67 -0.78	
2472	GB/T17593	37.2		0.66	
2482	prEN16711	32.332		-0.24	
2483	ISO105E04	34.2		0.10	
2488	ISO105E04	34.79		0.21	
2492	In house DIN54233-3	37.174		0.66	
2495 2496	DIN54233-3 DIN54233-3	33.90 35.339		0.05 0.31	
2497	DIN54233-3	37.59		0.73	
2511	DIN54233-3	34.705		0.20	
2532	DIN54233-3	31.5		-0.40	
2540 2545	DIN54233-3 GB/T17503	36.279 33.40		0.49 -0.05	
2545 2566	GB/T17593 ISO105E04	33.40 31.20		-0.05 -0.45	
2567	DIN54233-3	30.1		-0.43	
2572	DIN54233-3	35.94		0.43	
2590	ISO105E04	28.90		-0.88	
2592	ISO105E04	29.4392	C	-0.78	First reported 10.0
2606 2613	In house ISO105E04	22.8 35.35	С	-2.01 0.32	First reported 19.9
2623	GB/T17593	34.534		0.32	
2638	ISO105E04	31.726		-0.36	
2643	DIN54233-3	30.6		-0.57	
2644	DIN54233-3	27.67		-1.11	
2654 2658	ISO105E04	41.93 57.1	D(0.04)	1.54 4.36	
2658 2665	ISO105E04 prEN16711	57.1 36.810	R(0.01)	4.36 0.59	
2678	DIN54233-3	25.90		-1.44	
2715	GB/T17593	32.845		-0.15	
3118	B.N. II. 46				
3146	DIN54233-3	35.0		0.25	
3153	ISO105E04	34.796		0.21	

3154 3166 3172 3176 3190 3197 3210 3214 3220 3228 3232 3237	DIN54233-3 In house DIN54233-3 DIN54233-3 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 In house	36.75 28.4 35.70 36.837 33.37 32.93 37.01 34.90 32.4 34.6 12.35 34.68	R(0.01)	0.58 -0.97 0.38 0.59 -0.05 -0.13 0.63 0.23 -0.23 0.18 -3.96 0.19
	normality n outliers mean (n) st.dev. (n) R(calc.) R(DIN54233-3:2010)	OK 83 2 33.645 3.6425 10.199 15.073		

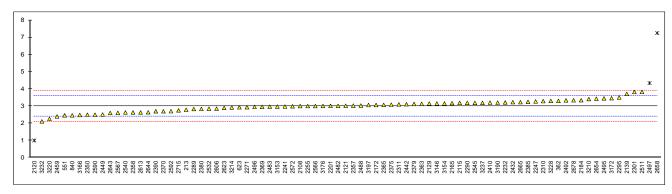


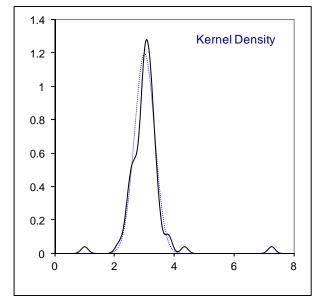


Determination of Nickel as Ni on sample #15205; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	metriou		man	<u> </u>	Tomarko
213	ISO105E04	2.787		-0.72	
362	In house	3.3055		1.01	
551	ISO105E04	2.46		-1.81	
623	DIN54233-3	2.93		-0.24	
840	ISO105E04	2.46		-1.81	
2108	In house	3.00		-0.01	
2115	DIN54233-3	3.188	D(0.04)	0.62	
2120 2121	DIN54233-3	1.000 3.02	R(0.01) C	-6.67 0.06	First reported 6.43
2121	In house DIN EN16711-2	3.15	C	0.06	Filst reported 6.45
2139	ISO105E04	3.71		2.35	
2165	DIN54233-3	3.17		0.56	
2172	ISO105E04	3.063		0.20	
2184	DIN54233-3	3.343		1.13	
2190					
2201	DIN54233-3	3.010		0.02	
2232	DIN54233-3	3.2128		0.70	
2241 2247	ISO105E04	2.972		-0.10	
2255	ISO105E04 DIN54233-3	3.27 3.0		0.89 -0.01	
2271	GB/T17593	2.934		-0.23	
2289	DIN54233-3	2.83		-0.58	
2290	DIN54233-3	3.19		0.62	
2295	DIN54233-3	3.5		1.66	
2301	DIN54233-3	3.82		2.72	
2310	ISO105E04	3.29		0.96	
2311	ISO105E04	3.1		0.32	
2350	ISO105E04	2.497		-1.68	
2357 2358	DIN54233-3 ISO105E04	3.027 2.63		0.08 -1.24	
2363	DIN54233-3	3.132		0.43	
2365	DIN54233-3	3.068		0.43	
2369	ISO105E04	2.96		-0.14	
2370	DIN54233-3	2.70		-1.01	
2375	In house	3.08		0.26	
2379	DIN54233-3	3.121		0.39	
2380	ISO105E04	2.832		-0.57	
2385	DIN54233-3	3.25		0.82	
2390 2410	ISO105E04 DIN54233-3	2.70		-1.01 0.66	
2432	ISO105E04	3.2 3.228		0.75	
2442	In house	3.11		0.75	
2449	DIN54233-3	2.51		-1.64	
2459	DIN54233-3	2.395		-2.02	
2472	GB/T17593	< 0.05		<-3.51	False negative result?
2482	prEN16711	3.010		0.02	
2483	ISO105E04	2.97		-0.11	
2488 2492	ISO105E04	3.03 3.332		0.09 1.10	
2492	In house DIN54233-3	3.45		1.10	
2496	DIN54233-3	2.949		-0.18	
2497	DIN54233-3	4.34	R(0.05)	4.45	
2511	DIN54233-3	3.829	/	2.75	
2532	DIN54233-3	2.85		-0.51	
2540	DIN54233-3	2.629		-1.24	
2545	GB/T17593	3.19		0.62	
2566 2567	ISO105E04	3.0	C	-0.01	First reported 1 04
2567 2572	DIN54233-3 DIN54233-3	2.61 2.99	С	-1.31 -0.04	First reported 1.04
2590	ISO105E04	2.50		-0.04	
2592	ISO105E04	2.7027		-1.00	
2606	In house	2.85	С	-0.51	First reported 60.4
2613	ISO105E04	2.63		-1.24	·
2623	GB/T17593	2.895		-0.36	
2638	ISO105E04	n.d			
2643	DIN54233-3	2.6		-1.34	
2644	DIN54233-3	2.64		-1.21	
2654 2658	ISO105E04	3.432	D(0.04)	1.43 14.14	
2658 2665	ISO105E04 prEN16711	7.25 3.228	R(0.01)	0.75	
2678	DIN54233-3	3.34		1.12	
2715	GB/T17593	2.751		-0.84	
3118					
3146	DIN54233-3	3.15		0.49	
3153	ISO105E04	2.971		-0.11	

3154 3166 3172 3176 3190 3197 3214 3220 3228 3232 3237	DIN54233-3 In house DIN54233-3 DIN54233-3 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 In house normality n outliers mean (n) st.dev. (n)	3.15 2.48 3.46 3.005 3.20 3.06 3.409 2.92 2.26 3.3 2.11 3.19 OK 80 3 3.003 0.3345	0.49 -1.74 1.52 0.01 0.66 0.19 1.35 -0.28 -2.47 0.99 -2.97 0.62
	st.dev. (n)	0.3345	
	R(calc.) R(DIN54233-3:2010)	0.937 0.841	

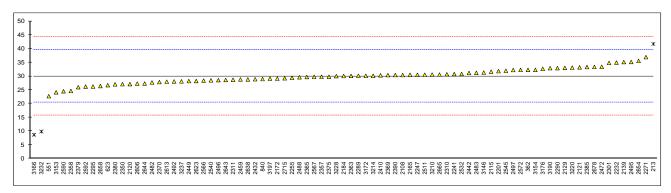


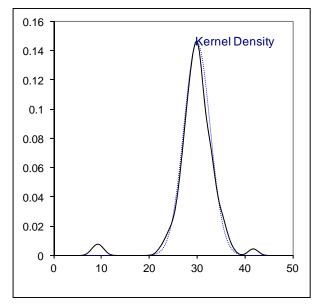


Determination of Copper as Cu on sample #15206; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110					
213	ISO105E04	41.72	R(0.01)	2.45	
362	In house	32.275		0.48	
551	ISO105E04	22.71		-1.52	
623 840	DIN54233-3	26.72		-0.68 -0.22	
2108	ISO105E04 In house	28.94 30.4		0.09	
2115	DIN54233-3	31.637		0.03	
2120	DIN54233-3	27.10		-0.60	
2121	In house	33.2	С	0.67	First reported 6.24
2129	DIN EN16711-2	33.03		0.64	
2139	ISO105E04	35.12		1.07	
2165	DIN54233-3	30.50		0.11	
2172 2184	ISO105E04	29.11 30.05		-0.18 0.01	
2190	DIN54233-3				
2201	DIN54233-3	31.830		0.39	
2232	DIN54233-3	34.928		1.03	
2241	ISO105E04	30.764		0.16	
2247	ISO105E04	30.50		0.11	
2255	DIN54233-3	29.4		-0.12	
2271	GB/T17593	36.921		1.45	
2289 2290	DIN54233-3 DIN54233-3	30.13 32.92		0.03 0.61	
2295	DIN54233-3	26.2		-0.79	
2301	DIN54233-3	34.82		1.01	
2310	ISO105E04	30.7		0.15	
2311	ISO105E04	28.7		-0.27	
2350	ISO105E04	27.08		-0.60	
2357	DIN54233-3	29.780		-0.04	
2358 2363	ISO105E04 DIN54233-3	24.61 30.086		-1.12 0.02	
2365	DIN54233-3	29.688		-0.02	
2369	ISO105E04	30.36		0.08	
2370	DIN54233-3	27.84		-0.45	
2375	In house	29.8	С	-0.04	First reported 2.94
2379	DIN54233-3	25.988		-0.83	
2380	ISO105E04	27.017		-0.62	
2385 2390	DIN54233-3 ISO105E04	33.3 30.40		0.69 0.09	
2410	DIN54233-3	30.3		0.07	
2432	ISO105E04	28.882		-0.23	
2442	In house	31.15		0.24	
2449	DIN54233-3	28.21		-0.37	
2459	DIN54233-3	28.813		-0.24	
2472 2482	GB/T17593 prEN16711	33.4 27.691		0.71 -0.48	
2483	ISO105E04	31.225		0.26	
2488	ISO105E04	29.60		-0.08	
2492	In house	28.087		-0.39	
2495	DIN54233-3	35.15		1.08	
2496	DIN54233-3	28.544		-0.30	
2497 2511	DIN54233-3 DIN54233-3	32.23 30.509		0.47 0.11	
2532	DIN54233-3	30.8		0.17	
2540	DIN54233-3	28.482		-0.31	
2545	GB/T17593	31.97		0.41	
2566	ISO105E04	28.40		-0.33	
2567	DIN54233-3	29.75		-0.05	
2572 2590	DIN54233-3 ISO105E04	32.26 24.50		0.47 -1.14	
2590 2592	ISO105E04	24.50 26.1977		-0.79	
2606	In house	27.3		-0.56	
2613	ISO105E04	27.98		-0.42	
2623	GB/T17593	28.234		-0.36	
2638	ISO105E04	28.817		-0.24	
2643	DIN54233-3	28.6		-0.29	
2644 2654	DIN54233-3 ISO105E04	27.32 35.55		-0.55 1.16	
2658	ISO105E04	26.4		-0.75	
2665	prEN16711	30.567		0.12	
2678	DIN54233-3	33.36		0.70	
2715	GB/T17593	29.230		-0.16	
3118 3146	DINEADOD O	31.3		0.27	
3146	DIN54233-3 ISO105E04	24.129		-1.22	
3.30		= .= 5			

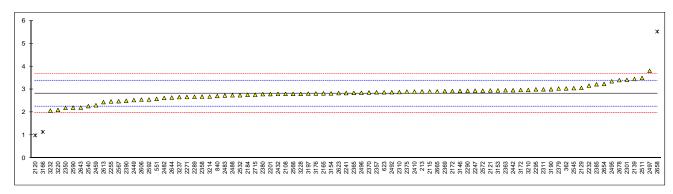
3154 3166 3172 3176 3190 3197 3210 3214 3220 3228 3232 3237	DIN54233-3 In house DIN54233-3 DIN54233-3 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 DIN54233-3 ISO105E04 In house	32.28 8.68 30.13 32.674 32.90 29.09 30.56 30.15 33.1 30.0 9.85 28.10	R(0.01)	0.48 -4.44 0.03 0.56 0.61 -0.19 0.12 0.04 0.65 0.00 -4.20 -0.39
3231	normality n outliers mean (n) st.dev. (n) R(calc.) R(DIN54233-3:2010)	OK 82 3 29.981 2.7370 7.664 13.432		-0.39

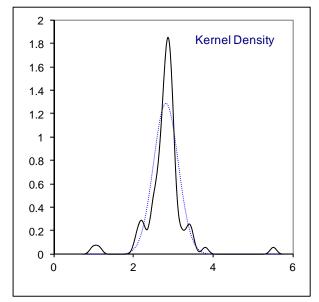




Determination of Nickel as Ni on sample #15206; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110					
213	ISO105E04	2.906		0.29	
362 551	In house	3.038		0.76	
623	ISO105E04 DIN54233-3	2.58 2.87		-0.86 0.17	
840	ISO105E04	2.71		-0.40	
2108	In house	2.80		-0.08	
2115	DIN54233-3	2.907		0.30	
2120	DIN54233-3	0.990	R(0.01)	-6.49	First are arts d. 5.00
2121 2129	In house DIN EN16711-2	2.95 3.06	С	0.45 0.84	First reported 5.96
2139	ISO105E04	3.45		2.22	
2165	DIN54233-3	2.82		-0.01	
2172	ISO105E04	2.920		0.34	
2184	DIN54233-3	2.759		-0.23	
2190 2201	DIN54233-3	2.790		-0.12	
2232	DIN54233-3	3.1582		1.19	
2241	ISO105E04	2.834		0.04	
2247	ISO105E04	2.94		0.42	
2255	DIN54233-3	2.47		-1.25	
2271 2289	GB/T17593 DIN54233-3	2.663 2.67		-0.57 -0.54	
2290	DIN54233-3	2.94		0.42	
2295	DIN54233-3	3		0.63	
2301	DIN54233-3	3.41		2.08	
2310	ISO105E04	2.88		0.20	
2311 2350	ISO105E04 ISO105E04	3.0 2.193		0.63 -2.23	
2357	DIN54233-3	2.870		0.17	
2358	ISO105E04	2.68		-0.51	
2363	DIN54233-3	2.959		0.48	
2365	DIN54233-3	2.842		0.07	
2369 2370	ISO105E04 DIN54233-3	2.92 2.86		0.34 0.13	
2375	In house	2.9	С	0.10	First reported 29.8
2379	DIN54233-3	3.033		0.74	•
2380	ISO105E04	2.788		-0.12	
2385 2390	DIN54233-3	3.22 2.50		1.41 -1.14	
2390 2410	ISO105E04 DIN54233-3	2.50 2.9		0.27	
2432	ISO105E04	2.798		-0.09	
2442	In house	2.96		0.49	
2449	DIN54233-3	2.53		-1.04	
2459 2472	DIN54233-3 GB/T17593	2.303 <0.05		-1.84 <-3.51	False negative result?
2472	prEN16711	2.622		-0.71	raise negative result?
2483	ISO105E04	2.73		-0.33	
2488	ISO105E04	2.74		-0.29	
2492	In house	2.874		0.18	
2495 2496	DIN54233-3 DIN54233-3	3.35 2.849		1.87 0.09	
2490	DIN54233-3 DIN54233-3	3.81		3.50	
2511	DIN54233-3	3.494		2.38	
2532	DIN54233-3	2.74		-0.29	
2540 2545	DIN54233-3 GR/T17503	2.266		-1.97 0.80	
2545 2566	GB/T17593 ISO105E04	3.05 2.8		-0.80	
2567	DIN54233-3	2.48	С	-1.21	First reported 0.74
2572	DIN54233-3	2.94		0.42	•
2590	ISO105E04	2.20		-2.21	
2592 2606	ISO105E04	2.5509		-0.96 -0.97	
2606 2613	In house ISO105E04	2.55 2.44		-0.97 -1.36	
2623	GB/T17593	2.833		0.04	
2638	ISO105E04	n.d			
2643	DIN54233-3	2.2		-2.21	
2644 2654	DIN54233-3	2.64		-0.65 1.45	
2654 2658	ISO105E04 ISO105E04	3.233 5.52	R(0.01)	9.56	
2665	prEN16711	2.907	(0.01)	0.30	
2678	DIN54233-3	3.40	С	2.04	First reported 3.77
2715	GB/T17593	2.760		-0.22	
3118 3146	DIN54233-3	2.93		0.38	
3146	ISO105E04	2.93 2.952		0.36	
2.50	· - · - ·				





Determination of Antimony (Sb), Arsenic (As), Chromium (Cr), Cobalt (Co), Lead (Pb) and Mercury (Hg) on sample #15205; results in mg/kg

lab	method	Sb	As	Cr	Со	Pb	Hg
110							
213 362	ISO105E04 In house	0	0	0	0	0	0
551	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
623	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	ISO105E04	<0.5	<0.02	<0.1	<0.1	<0.1	<0.005
2108 2115	In house DIN54233-3	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
2120	DIN54233-3	<2.5	<0.02	<0.50	<0.50	<0.02	< 0.02
2121	In house	0	0	0	0	0	0
2129	DIN EN16711-2						
2139 2165	ISO105E04 DIN54233-3	<5 n.d.	<0.1 n.d.	<0.5 n.d.	<0.5 n.d.	<0.1 n.d.	<0.01 n.d.
2172	ISO105E04	<0.5	<0.1	<0.1	<0.1	<0.1	<0.005
2184	DIN54233-3	<0.5	< 0.02	<0.5	<0.5	< 0.02	< 0.02
2190	DINIE 4000 0						
2201 2232	DIN54233-3 DIN54233-3	<1.0 n.d.	<0.10 n.d.	<0.50 n.d.	<0.30 n.d.	<0.10 n.d.	<0.010 n.d.
2241	ISO105E04	<5.0	<0.1	<3.0	<1.0	<0.1	<0.01
2247	ISO105E04	n.d.	N.D	N.D	N.D	N.D	N.D
2255	DIN54233-3						
2271 2289	GB/T17593 DIN54233-3	<1.0 n.d.	<0.2 n.d.	<0.2 n.d.	<0.2 n.d.	<0.2 n.d.	<0.02 n.d.
2290	DIN54233-3 DIN54233-3	11.u. <1	<0.1	<0.5	<0.3	<0.1	<0.01
2295	DIN54233-3						
2301	DIN54233-3						
2310 2311	ISO105E04 ISO105E04	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.	n.d. n.d.
2350	ISO105E04	<0.5	<0.02	<0.1	<0.1	<0.1	<0.005
2357	DIN54233-3	<1	<0.2	<0.5	<1	<0.2	<0.02
2358	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363 2365	DIN54233-3	<1 <1.00	<0.1 <0.10	< 0.5	<0.5 <0.50	<0.1	<0.01
2369	DIN54233-3 ISO105E04	<1.00	<0.10	<1.00 <0.5	<0.50	<0.10 <0.1	<0.01 <0.01
2370	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	In house						
2379 2380	DIN54233-3 ISO105E04	<1.0 n.d.	<0.2 n.d.	<1.0 n.d.	<1.0 n.d.	<0.2 n.d.	<0.02 n.d.
2385	DIN54233-3	<0.05	<0.05	<0.1	<0.1	<0.1	<0.01
2390	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2410	DIN54233-3	<5.0	<0.2	<1.0	<1.0	<0.2	< 0.02
2432 2442	ISO105E04 In house		0.22	0.75			
2449	DIN54233-3						
2459	DIN54233-3	N. D	N. D	N. D	N. D	N. D	N. D
2472	GB/T17593	<0.35		<0.06	<0.10	< 0.30	
2482	prEN16711 ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2488	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d	n.d.
2492	In house						
2495	DIN54233-3	<2.5	<2.5	<2.5	<2.5	<5	<2.5
2496 2497	DIN54233-3 DIN54233-3	<3 0.008	<0.2 0.007	<0.2 0.052	<0.2 0.0008	<0.2 0.0006	0.008
2511	DIN54233-3						
2532	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2540 2545	DIN54233-3 GB/T17593	N.D	N.D	N.D	 N.D	N.D	N.D
2566	ISO105E04	ND	ND ND	ND	ND	ND	ND
2567	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2572	DIN54233-3	<1	<0.1	<0.5	<0.3	<0.1	<0.01
2590 2592	ISO105E04 ISO105E04	<l.o.q. 0.074267</l.o.q. 	<l.o.q. 0.03022</l.o.q. 	0.40 0.5104	<l.o.q. 0.01385</l.o.q. 	<l.o.q. 0.01487</l.o.q. 	<l.o.q.< td=""></l.o.q.<>
2606	In house	0.074267	0.03022	0.5104	0.01365	0.01467	
2613	ISO105E04						
2623	GB/T17593		 N1/A				 N1/A
2638 2643	ISO105E04 DIN54233-3	n.d 	N/A 	n.d 	n.d 	n.d 	N/A
2644	DIN54233-3 DIN54233-3						
2654	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2658	ISO105E04			0.91	0.46		
2665 2678	prEN16711 DIN54233-3	<0.20 n.d.	<0.20 n.d.	<0.20 n.d.	<0.20 n.d.	<0.10 n.d.	<0.02 n.d.
2715	GB/T17593	0.07	0.024	n.a. 0.143	0.013	0.103	0.000
3118							

3146 3153 3154	DIN54233-3 ISO105E04 DIN54233-3	n.d. <1	n.d. <0.3	n.d. <0.5	n.d. <1	n.d. <0.3	n.d. <0.01
3166	In house	<0.08	<0.01	0.03	< 0.004	<0.004	<0.003
3172	DIN54233-3	<5	<0.05	<0.1	<0.1	<0.1	<0.01
3176	DIN54233-3						
3190	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3197	DIN54233-3						
3210	ISO105E04	0.0287	<0.03	0.165	<0.02	<0.20	<0.02
3214	DIN54233-3	<1.0	<0.1	<0.5	<0.1	<0.1	<0.01
3220	ISO105E04	n.d.	0.21	0.1	n.d.	0.1	n.d.
3228	DIN54233-3	<0.5	<0.02	<0.5	<0.5	<0.02	< 0.02
3232	ISO105E04	n.d	n.d	n.d	n.d	n.d	n.d
3237	In house	0.03	n.d.	0.02	n.d.	n.d.	n.d.
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	35	34	37	35	34	30
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<5	<1	<2	<4	<1	<0.02
	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(DIN54233-3:2010)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Determination of Antimony (Sb), Arsenic (As), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Lead (Pb) and Mercury (Hg) on sample #15206; results in mg/kg

loh	mathad	Ch	۸۵	C4	C.	Co	Dh	Ца
110	method	Sb	As	Cd 	Cr 	Co	Pb	Hg
213	ISO105E04	0	0	0	0	0	0	0
362	In house							
551	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
623	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	ISO105E04	<0.5	< 0.02	< 0.02	<0.1	<0.1	<0.1	< 0.005
2108	In house							
2115		<0.02	<0.02	<0.02	0.032	<0.02	0.030	<0.02
2120		<2.5	<0.10	<0.05	<0.50	<0.50	<0.10	<0.013
2121	In house DIN EN16711-2	0	0	0	0	0	0	0
	ISO105E04	<5	<0.1	< 0.05	<0.5	<0.5	<0.1	<0.01
	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	ISO105E04	<0.5	<0.1	<0.1	<0.1	<0.1	<0.1	<0.005
2184		<0.5	<0.02	<0.02	<0.5	<0.5	<0.02	<0.02
2190								
2201	DIN54233-3	<1.0	<0.10	< 0.030	< 0.50	< 0.30	<0.10	<0.010
	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2241	ISO105E04	<5.0	<0.1	<0.5	<3.0	<1.0	<0.1	<0.01
2247		N.D	N.D	N.D	N.D	N.D	N.D	N.D
2255 2271	DIN54233-3 GB/T17593	<1.0	<0.2	0.340	<0.2	<0.2	<0.2	
2289		< 1.0 n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<0.02 n.d.
	DIN54233-3	<1.u.	<0.1	<0.03	<0.5	<0.3	<0.1	<0.01
	DIN54233-3							
2301	DIN54233-3							
	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2350	ISO105E04	<0.5	<0.02	<0.02	<0.1	<0.1	<0.1	<0.005
2357		<1	<0.2	<0.1	<0.5	<1	<0.2	<0.02
	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	DIN54233-3	<1	<0.1	< 0.05	<0.5	<0.5	<0.1	<0.01
	DIN54233-3 ISO105E04	<1.00 <1.0	<0.10 <0.1	<0.05 <0.05	<1.00 <0.5	<0.50 <0.5	<0.10 <0.1	<0.01 <0.01
	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	In house							
2379	DIN54233-3	<1.0	<0.2	<0.1	<1.0	<1.0	< 0.2	< 0.02
	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2385	DIN54233-3	< 0.05	< 0.05	<0.01	<0.1	<0.1	<0.1	<0.01
	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	DIN54233-3	<5.0	<0.2	<0.1	<1.0	<1.0	<0.2	<0.02
	ISO105E04							
	In house		0.45	 0 75	0.72			
	DIN54233-3 DIN54233-3	N. D	 N. D	<u>0.75</u> 0.751	 N. D	N. D	N. D	N. D
	GB/T17593	<0.35	IN. D	< 0.06	<0.06	<0.10	<0.30	IN. D
	prEN16711							
2483	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2488	ISO105E04	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2492	In house							
2495	DIN54233-3	<2.5	<2.5	<2.5	<2.5	<2.5	<5	<2.5
2496	DIN54233-3	<3	<0.2	<0.05	<0.2	<0.2	<0.2	
2497	DIN54233-3	0.007	0.021	0.008	0.058	0.007	0.008	0.009
2511 2532	DIN54233-3 DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2540	DIN54233-3 DIN54233-3	11.u. 	11.u. 	11.u. 	11.u.	11.u.	11.u.	11.u.
2545	GB/T17593	N.D	N.D	N.D	N.D	N.D	N.D	N.D
2566	ISO105E04	ND	ND	ND	ND	ND	ND	ND
2567	DIN54233-3	n.d.	n.d.		n.d.	n.d.	n.d.	n.d.
2572	DIN54233-3	<1	<0.1	< 0.03	<0.5	<0.3	<0.1	<0.01
2590	ISO105E04	<l.o.q.< td=""><td><l.o.q.< td=""><td><l.o.q.< td=""><td>0.40</td><td><l.o.q.< td=""><td><l.o.q.< td=""><td><l.o.q.< td=""></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<>	<l.o.q.< td=""><td><l.o.q.< td=""><td>0.40</td><td><l.o.q.< td=""><td><l.o.q.< td=""><td><l.o.q.< td=""></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<>	<l.o.q.< td=""><td>0.40</td><td><l.o.q.< td=""><td><l.o.q.< td=""><td><l.o.q.< td=""></l.o.q.<></td></l.o.q.<></td></l.o.q.<></td></l.o.q.<>	0.40	<l.o.q.< td=""><td><l.o.q.< td=""><td><l.o.q.< td=""></l.o.q.<></td></l.o.q.<></td></l.o.q.<>	<l.o.q.< td=""><td><l.o.q.< td=""></l.o.q.<></td></l.o.q.<>	<l.o.q.< td=""></l.o.q.<>
2592	ISO105E04	0.05299	0.01345	0.008443	0.51799	0.01499	0.01452	
2606	In house							
2613	ISO105E04			n d				
2623 2638	GB/T17593 ISO105E04	n.d	N/A	n.d. n.d	n.d	n.d	n.d	N/A
2643	DIN54233-3	11.u 		11.u	11.u	11.u	11.u	IN/A
2644	DIN54233-3	0.138						
2654	ISO105E04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2658	ISO105E04			0.65	1.25	0.47		
2665	prEN16711	<0.20	<0.20	<0.10	<0.20	<0.20	<0.10	< 0.02
2678	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2715	GB/T17593	0.060	0.048	0.015	0.050	0.014	0.101	0.000
3118								

3146 3153	DIN54233-3 ISO105E04	n.d. <1	n.d. <0.3	n.d. <0.03	n.d. <0.5	n.d. <1	n.d. <0.3	n.d. <0.01
3154	DIN54233-3	<u> </u>	<0.5	<0.03	<0.5 		<0.5	<0.01
3166	In house	<0.08	<0.01	< 0.009	< 0.02	0.007	0.015	< 0.003
3172	DIN54233-3	<0.00 <5	<0.01	<0.009	<0.02	<0.1	<0.1	<0.003
-		_	<0.05	<0.02	_	<0.1	<0.1	
3176	DIN54233-3							
3190	DIN54233-3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3197	DIN54233-3							
3210	ISO105E04	0.0231	<0.03	<0.03	0.112	<0.02	<0.2	< 0.02
3214	DIN54233-3	<1.0	<0.3	< 0.03	<0.5	<0.3	<0.1	< 0.01
3220	ISO105E04	n.d.	0.1	n.d.	n.d.	n.d.	n.d.	n.d.
3228	DIN54233-3	< 0.5	< 0.02	< 0.02	<0.5	<0.5	< 0.02	< 0.02
3232	ISO105E04	n.d	n.d	n.d	n.d	n.d	n.d	n.d
3237	In house	0.03	n.d.	n.d.	0.04	n.d.	n.d.	n.d.
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	36	34	35	37	35	33	30
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<5	<1	<0.1	<2	<4	<1	< 0.02
	st.dev. (n)	n.a.	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.	n.a.
	R(DIN54233-3:2010)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Results in Bold, Underlined and Italic are marked as "a possible false positive test result".

APPENDIX 2

Dilution ratio as reported by participants

labnrs	What ratio was used in gram textile per ml?	labnrs	What ratio was used in gram textile per ml?
110		2540	1 gram textile per 50 ml perspiration liquid
213	1 gram textile per 50 ml perspiration liquid	2545	1 gram textile per 50 ml perspiration liquid
362	1 gram textile per 50 ml perspiration liquid	2566	
551	1 gram textile per 50 ml perspiration liquid	2567	1 gram textile per 50 ml perspiration liquid
623	1 gram textile per 50 ml perspiration liquid	2572	1 gram textile per 50 ml perspiration liquid
840	1 gram textile per 50 ml perspiration liquid	2590	1 gram textile per 50 ml perspiration liquid
2108	1 gram textile per 50 ml perspiration liquid	2592	1 gram textile per 50 ml perspiration liquid
2115	1 gram textile per 50 ml perspiration liquid	2606	1 gram textile per 50 ml perspiration liquid
2120	1 gram textile per 50 ml perspiration liquid	2613	1 gram textile per 50 ml perspiration liquid
2121	1 gram textile per 50 ml perspiration liquid	2623	1 gram textile per 50 ml perspiration liquid
2129	0.4 gram textile per 20 ml perspiration liquid	2638	1 gram textile per 50 ml perspiration liquid
2139	1 gram textile per 50 ml perspiration liquid	2643	1 gram textile per 50 ml perspiration liquid
2165	1 gram textile per 50 ml perspiration liquid	2644	1 gram textile per 50 ml perspiration liquid
2172	1 gram textile per 50 ml perspiration liquid	2654	1 gram textile per 50 ml perspiration liquid
2184	1 gram textile per 50 ml perspiration liquid	2658	1 gram textile per 50 ml perspiration liquid
2190		2665	1 gram textile per 50 ml perspiration liquid
2201	1 gram textile per 50 ml perspiration liquid	2678	1 gram textile per 50 ml perspiration liquid
2232	1 gram textile per 50 ml perspiration liquid	2715	1 gram textile per 20 ml perspiration liquid
2241	1 gram textile per 50 ml perspiration liquid	3118	
2247	1 gram textile per 50 ml perspiration liquid	3146	1 gram textile per 50 ml perspiration liquid
2255	1 gram textile per 50 ml perspiration liquid	3153	1 gram textile per 50 ml perspiration liquid
2271	1 gram textile per 50 ml perspiration liquid	3154	1 gram textile per 50 ml perspiration liquid
2289		3166	
2299	1 gram textile per 50 ml perspiration liquid	3172	1 gram textile per 50 ml perspiration liquid
	1 gram textile per 50 ml perspiration liquid		1 gram textile per 50 ml perspiration liquid
2295	1 gram textile per 50 ml perspiration liquid	3176	1 gram textile per 50 ml perspiration liquid
2301	4 man toutile non 50 ml nonmination limited	3190	1 gram textile per 50 ml perspiration liquid
2310	1 gram textile per 50 ml perspiration liquid	3197	1 gram textile per 50 ml perspiration liquid
2311	1 gram textile per 50 ml perspiration liquid	3210	1 gram textile per 50 ml perspiration liquid
2350	1 gram textile per 50 ml perspiration liquid	3214	1 gram textile per 50 ml perspiration liquid
2357	1 gram textile per 50 ml perspiration liquid	3220	1 gram textile per 50 ml perspiration liquid
2358	1 gram textile per 50 ml perspiration liquid	3228	1 gram textile per 50 ml perspiration liquid
2363	1 gram textile per 50 ml perspiration liquid	3232	1 gram textile per 50 ml perspiration liquid
2365	1 gram textile per 50 ml perspiration liquid	3237	1 gram textile per 50 ml perspiration liquid
2369	1 gram textile per 50 ml perspiration liquid		
2370	1 gram textile per 50 ml perspiration liquid		
2375	1 gram textile per 50 ml perspiration liquid		
2379			
2380	1 gram textile per 50 ml perspiration liquid		
2385	1 gram textile per 50 ml perspiration liquid		
2390	1 gram textile per 50 ml perspiration liquid		
2410	1 gram textile per 50 ml perspiration liquid		
2432	1 gram textile per 50 ml perspiration liquid		
2442	1 gram textile per 50 ml perspiration liquid		
2449	1 gram textile per 50 ml perspiration liquid		
2459	1 gram textile per 50 ml perspiration liquid		
2472	1 gram textile per 50 ml perspiration liquid		
2482	1 gram textile per 50 ml perspiration liquid		
2483	1 gram textile per 50 ml perspiration liquid		
2488	0.5 gram textile per 25 ml perspiration liquid		
2492	1 gram textile per 20 ml perspiration liquid		
2495	1 gram textile per 50 ml perspiration liquid		
2496	1 gram textile per 50 ml perspiration liquid		
2497	1 gram textile per 50 ml perspiration liquid		
2511	1 gram textile per 50 ml perspiration liquid		
2532	0.3 gram textile per 15 ml perspiration liquid	1	

APPENDIX 3

Number of participants per country:

- 4 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in BULGARIA
- 1 lab in CAMBODIA
- 3 labs in FRANCE
- 7 labs in GERMANY
- 4 labs in HONG KONG
- 7 labs in INDIA
- 3 labs in INDONESIA
- 7 labs in ITALY
- 2 labs in MOROCCO
- 18 labs in P.R. of CHINA
- 4 labs in PAKISTAN
- 1 lab in PORTUGAL
- 1 lab in SINGAPORE
- 5 labs in SOUTH KOREA
- 3 labs in TAIWAN R.O.C.
- 1 lab in THAILAND
- 2 labs in TUNISIA
- 7 labs in TURKEY
- 1 lab in U.A.E.
- 2 labs in U.S.A.
- 3 labs in VIETNAM

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect 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 outlier test R(0.05) = straggler in Rosner outlier test

n.a. = not applicablen.d. = not detected

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