Results of Proficiency Test
Perspirated Metals in textile
October 2014

Organised by: Institute for Interlaboratory Studies
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CONTENTS

1 INTRODUCTION

Since the 1990's, many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known programs are for instance Milieukeur (the Netherlands), Oeko-Tex Standard 100 (Germany) 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 2014/2015.
In the 2014 interlaboratory study 86 laboratories in 24 different countries participated. See appendix 3 for the number of participants per country. In this report the results of the 2014 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 preparation and analyses were subcontracted.
It was decided to use 2 different textile samples, both positive on metals in this round and to request to use a solid/liquid ratio of 1/50 by preference (see paragraph 6 and the report iis07A05). Participants were requested to report results with 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 ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. This PT falls under the accredited scope. 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 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 can be downloaded from the iis website http://www.iisnl.com.

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, #14205 and #14206 were each well mixed and divided over 111 subsamples of 2.5-3.0 grams. The samples were labelled and tested for homogeneity on resp. 8 (#14205) and 4 (#14206) 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 #14205-1	59.6
Sample #14205-2	57.9
Sample #14205-3	55.7
Sample #14205-4	56.8
Sample #14205-5	58.4
Sample #14205-6	61.1
Sample #14205-7	58.0
Sample #14205-8	57.3

Table 1: homogeneity test results of subsamples #14205

	Perspirated Antimony in mg/kg	*Perspirated Cobalt in mg/kg*
Sample #14206-1	35.4	10.4
Sample #14206-2	36.8	9.8
Sample #14206-3	35.6	9.8
Sample #14206-4	33.8	10.4

Table 2: homogeneity test results of subsamples #14206

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
r (observed)	4.7
reference method	DIN54233-3:2010
0.3 x R (reference method)	7.8

Table 3: repeatabilities of subsamples #14205

	Perspirated Antimony in mg/kg	*Perspirated Cobalt in mg/kg*
r (observed)	3.5	1.0
reference method	DIN54233-3:2010	DIN54233-3:2010
0.3 x R (reference method)	5.9	1.1

Table 4: repeatabilities of subsamples #14206

The calculated repeatabilities are all in good agreement with 0.3 times the estimated target reproducibilities of the reference test method. Therefore, homogeneity of all subsamples was assumed.

To each of the participating laboratories, two samples, one of sample #14205 and one of sample #14206 were sent on October 15, 2014.

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 it 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, was sent together with each set of samples. In addition, a letter of instructions was added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after deadline, a reminder fax 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 3, no.16). 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 3; 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:

$z_{(target)}$ = (result - average of PT) / 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:

$\lvert z\rvert < 1$	good
$1 < \lvert z\rvert < 2$	satisfactory
$2 < \lvert z\rvert < 3$	questionable
$3 < \lvert z\rvert$	unsatisfactory

4 EVALUATION

During the execution of this proficiency test, no problems occurred with sample dispatch.
Eighteen laboratories reported test results after the final reporting date.
The 85 reporting laboratories did report in total 344 numerical results.
Observed were 10 statistical outlying results, which is 4.0% 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 does mention 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 METAL

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

For sample #14205

Copper: The determination of this metal was not problematic at a perspiration level of 57 mg/kg. Four 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).

For sample #14206

Antimony: The determination of this metal was not problematic at a perspiration level of 43.4 mg/kg. Four statistical outliers and two false negatives 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).

Cobalt: The determination of this metal was not problematic at a perspiration level of 10.5 mg/kg. Two 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).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	*Unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Perspirated Copper	mg/kg	81	57.05	16.69	25.56

Table 5: reproducibilities of perspirated metals in sample #14205

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Perspirated Antimony	mg/kg	79	43.36	19.88	24.28
Perspirated Cobalt	mg/kg	83	10.53	4.11	3.83

Table 6: reproducibilities of perspirated metals in sample #14206

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories has no difficulties with the analysis when compared with the target reproducibility of the reference test method E-DIN54233-3:10. See also the discussions in paragraphs 4.1 and 6.

5 COMPARISON WITH THE PREVIOUS PROFICIENCY TESTS

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	*October 2014*	*October 2013*	*October 2012*	*October 2011*	*October 2010*	*October 2009*	*November 2008*	*draft-DIN 54233-3*
Arsenic	--	--	--	--	--	--	--	20%
Antimony	16%	--	--	19%	--	15%	16%	20%
Cadmium	--	9%	14%	--	14%	--	--	10%
Chromium	--	15%	--	19%	--	--	--	15%
Cobalt	14%	--	11%	8%	11%	--	10%	13%
Copper	10%	10%	--	22%	--	16-17%	9%	16%
Lead	--	--	--	--	--	--	--	40%
Mercury	--	--	41%	--	--	--	--	31%
Nickel	--	11-13%	--	10-14%	7%	--	8%	10%

Table 7: 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 8), 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 many laboratories does not meet the requirements of the Standards (reported detection limit is often 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 8: 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 A.A.S or ICP”.
It should be noticed that for the results reported in this proficiency test, all participants have 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 E DIN 54233-3:2010.

Sample #14206 was used in a previous PT iis08A05 as sample #0866. When the assigned value of both PTs are compared the resemblance is good, see below table

	unit	n		Average		2.8 * sd	
		#0866	#14206	#0866	#14206	#0866	#14206
Total Antimony as Sb	mg/kg	47	79	42.06	43.36	19.20	19.88
Total Cobalt as Co	mg/kg	45	83	10.74	10.53	3.36	4.11

Table 9: comparison of samples #0866 and #14206

In general, it can be concluded from the reproducibilities of the Copper, Antimony and Cobalt determinations that the quality of the testing of these perspirated metals in textile is good.

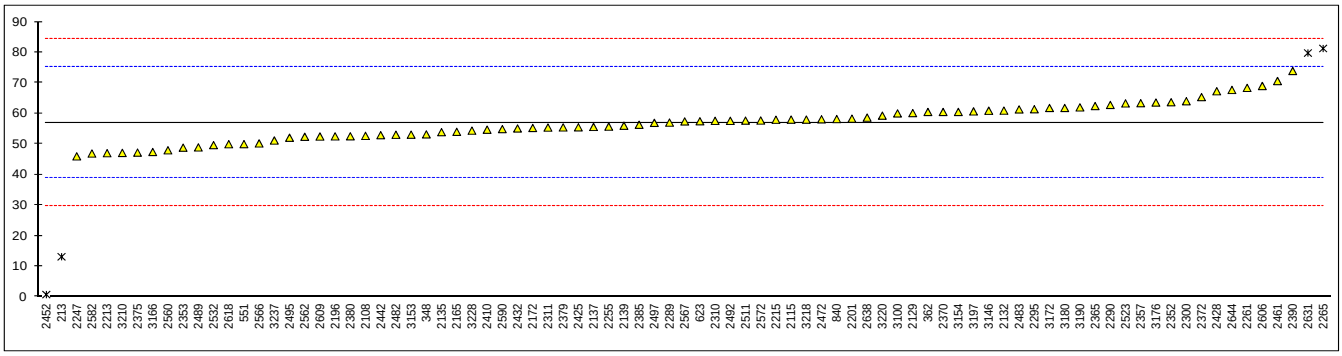
APPENDIX 1

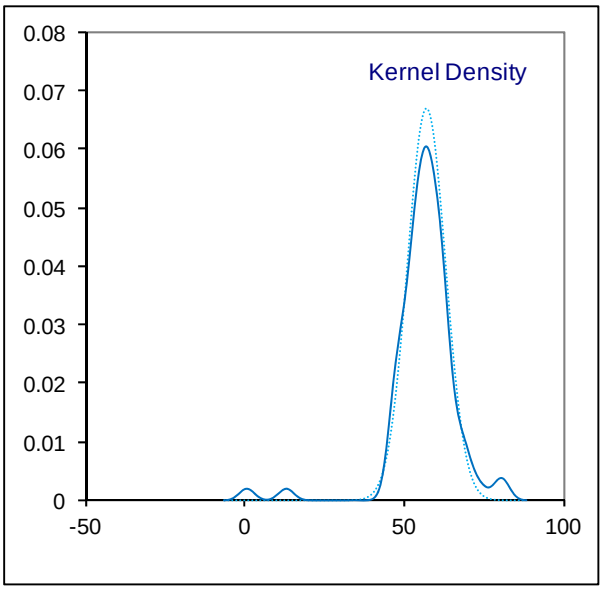
Determination of perspirated Copper on sample #14205; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	Oekotex100/200	13.09	R(0.01)	-4.82	
348	INH-7334	53.16		-0.43	
362	INH-103	60.5		0.38	
551	E104/ISO11085	49.98		-0.77	
623	ISO105E04	57.4563		0.04	
840	DIN54233	58.17		0.12	
2108	in house	52.67		-0.48	
2115	DIN54233-3	58.00		0.10	
2129	EN16711-2	60.128		0.34	
2132	E DIN54233-3	60.94		0.43	
2135	E DIN54233-3	53.9		-0.35	
2137	E DIN54233-3	55.6		-0.16	
2139	Oekotex100	56		-0.11	
2165	E DIN54233-3	54.00		-0.33	
2172	DIN54233-3	55.26		-0.20	
2196	DIN54233-3	52.5		-0.50	
2201	DIN54233-3	58.31		0.14	
2213	E DIN54233-3	47		-1.10	
2215	E DIN54233-3	57.95		0.10	
2247	ISO105E04	45.97		-1.21	
2255	ISO105E04	55.7		-0.15	
2261	GB/T17593	68.379		1.24	
2265	E DIN54233-3	81.28	R(0.05)	2.65	
2289	E DIN54233-3	57.02		0.00	
2290	ISO105E04	62.8		0.63	
2295	DIN54233-3	61.4		0.48	
2300	E DIN54233-3	64.02		0.76	
2310	DIN54233-3	57.6		0.06	
2311	DIN54233-3	55.4		-0.18	
2352	DIN54233-3	63.7		0.73	
2353	Oekotex	48.82		-0.90	
2357	E DIN54233-3	63.38		0.69	
2365	DIN54233-3	62.43		0.59	
2370	DIN54233-3	60.5		0.38	
2372	E DIN54233-3	65.37		0.91	
2375	E DIN54233-3	47.2		-1.08	
2379	E DIN54233-3	55.44		-0.18	
2380	E DIN54233-3	52.53		-0.50	
2385	E DIN54233-3	56.3		-0.08	
2390	ISO105E04	73.91		1.85	
2410	DIN54233-3	54.7		-0.26	
2425	DIN54233-3	55.48		-0.17	
2428	GB/T17593	67.28		1.12	
2432	ISO105E04	55.10		-0.21	
2442	in house	52.90		-0.45	
2452	E DIN54233-3	0.72	R(0.01)	-6.17	
2461	GB/T17593	70.625		1.49	
2472	GB/T17593	58.10		0.12	
2482	DIN54233-3	53.042		-0.44	
2483	ISO105E04	61.3400		0.47	
2489	E DIN54233-3	48.90		-0.89	
2492	in house	57.6		0.06	
2495	ISO17072-1	52.07		-0.55	
2497	DIN54233-3	56.94		-0.01	
2511	DIN54233-3	57.675		0.07	
2523	E DIN54233-3	63.30		0.68	
2532	E DIN54233-3	49.6581		-0.81	
2560	DIN54233-3	47.95		-1.00	
2562	E DIN54233-3	52.38		-0.51	
2566	ISO105E04	50.2		-0.75	
2567	ISO105E04	57.4		0.04	
2572	ISO105E04	57.7		0.07	
2582	ISO105E04	46.90		-1.11	
2590	ISO17072-1	54.90		-0.24	
2606	in house	69		1.31	
2609	GB/T17593	52.469		-0.50	
2614		-----		-----	
2618	in house	49.94		-0.78	
2631	KS K0731	79.792	R(0.05)	2.49	
2638	E DIN54233-3	58.65		0.18	
2644	DIN54233-3	67.7		1.17	
3100	E DIN54233-3	60.02		0.33	
3146	E DIN54233-3	60.9		0.42	
3153	ISO105E04	53.068		-0.44	
3154	DIN54233-3	60.51		0.38	

3166	INH-7040	47.4	-1.06
3172	DIN54233-3	61.8	0.52
3176	DIN54233-3	63.59	0.72
3180	DIN54233-3	61.8	0.52
3190	E DIN54233-3	62.0	0.54
3197	ISO105E04	60.68	0.40
3210	ISO17072-1	47.1	-1.09
3218	E DIN54233-3	58.0	0.10
3220	DIN54233	59.3	0.25
3228	E DIN54233-3	54.39	-0.29
3237	in house	51.157	-0.65

normality	OK
n	81
outliers	4
mean (n)	57.049
st.dev. (n)	5.9608
R(calc.)	16.690
R(DIN54233-3-2010)	25.558



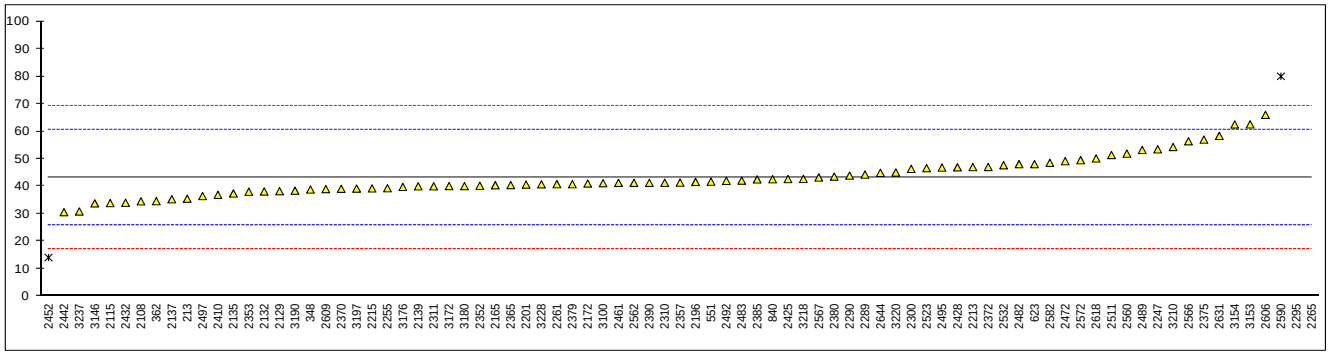


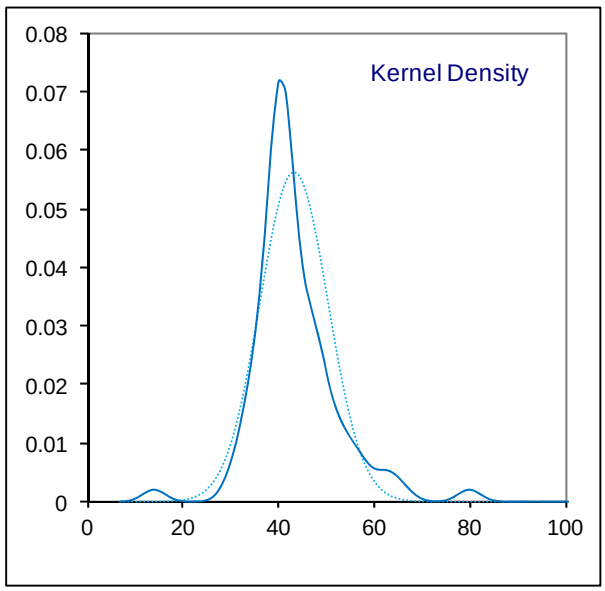
Determination of perspirated Antimony on sample #14206; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	Oekotex100/200	35.49		-0.91	
348	INH-7334	38.84		-0.52	
362	INH-103	34.6		-1.01	
551	E104/ISO11085	41.71	C	-0.19	First reported 375.10
623	ISO105E04	48.0769		0.54	
840	DIN54233	42.57		-0.09	
2108	in house	34.53		-1.02	
2115	DIN54233-3	33.90		-1.09	
2129	EN16711-2	38.235		-0.59	
2132	E DIN54233-3	38.13		-0.60	
2135	E DIN54233-3	37.4		-0.69	
2137	E DIN54233-3	35.3		-0.93	
2139	Oekotex100	40		-0.39	
2165	E DIN54233-3	40.40		-0.34	
2172	DIN54233-3	41.03		-0.27	
2196	DIN54233-3	41.6		-0.20	
2201	DIN54233-3	40.63		-0.31	
2213	E DIN54233-3	47		0.42	
2215	E DIN54233-3	39.24		-0.47	
2247	ISO105E04	53.46		1.16	
2255	ISO105E04	39.3		-0.47	
2261	GB/T17593	40.767		-0.30	
2265	E DIN54233-3	352.15	R(0.01)	35.61	
2289	E DIN54233-3	44.27		0.11	
2290	ISO105E04	43.9		0.06	
2295	DIN54233-3	183	C,R(0.01)	16.10	First reported 286.3
2300	E DIN54233-3	46.35		0.34	
2310	DIN54233-3	41.3		-0.24	
2311	DIN54233-3	40.0		-0.39	
2352	DIN54233-3	40.2		-0.36	
2353	Oekotex	38.0250		-0.62	
2357	E DIN54233-3	41.36		-0.23	
2365	DIN54233-3	40.45		-0.34	
2370	DIN54233-3	39.1		-0.49	
2372	E DIN54233-3	47.04		0.42	
2375	E DIN54233-3	57		1.57	
2379	E DIN54233-3	40.78		-0.30	
2380	E DIN54233-3	43.53		0.02	
2385	E DIN54233-3	42.5		-0.10	
2390	ISO105E04	41.27		-0.24	
2410	DIN54233-3	36.9		-0.74	
2425	DIN54233-3	42.64		-0.08	
2428	GB/T17593	46.90		0.41	
2432	ISO105E04	34.02		-1.08	
2442	in house	30.60		-1.47	
2452	E DIN54233-3	14.09	R(0.01)	-3.38	
2461	GB/T17593	41.250		-0.24	
2472	GB/T17593	49.15		0.67	
2482	DIN54233-3	48.068		0.54	
2483	ISO105E04	42.0700		-0.15	
2489	E DIN54233-3	53.23		1.14	
2492	in house	42.0		-0.16	
2495	ISO17072-1	46.82		0.40	
2497	DIN54233-3	36.46		-0.80	
2511	DIN54233-3	51.40		0.93	
2523	E DIN54233-3	46.62		0.38	
2532	E DIN54233-3	47.6765		0.50	
2560	DIN54233-3	51.89		0.98	
2562	E DIN54233-3	41.26		-0.24	
2566	ISO105E04	56.4		1.50	
2567	ISO105E04	43.2		-0.02	
2572	ISO105E04	49.5		0.71	
2582	ISO105E04	48.54		0.60	
2590	ISO17072-1	80.01	C, R(0.01)	4.23	First reported <0.001
2606	in house	66		2.61	
2609	GB/T17593	38.985		-0.50	
2614		-----		-----	
2618	in house	50.13		0.78	
2631	KS K0731	58.339		1.73	
2638	E DIN54233-3	n.d.		-----	False negative?
2644	DIN54233-3	44.9		0.18	
3100	E DIN54233-3	41.14		-0.26	
3146	E DIN54233-3	33.8		-1.10	
3153	ISO105E04	62.547		2.21	
3154	DIN54233-3	62.51		2.21	
3166	INH-7040	n.d.		-----	False negative?

3172	DIN54233-3	40.1	-0.38
3176	DIN54233-3	39.77	-0.41
3180	DIN54233-3	40.1	-0.38
3190	E DIN54233-3	38.4	-0.57
3197	ISO105E04	39.18	-0.48
3210	ISO17072-1	54.3	1.26
3218	E DIN54233-3	42.7	-0.08
3220	DIN54233	45.0	0.19
3228	E DIN54233-3	40.73	-0.30
3237	in house	30.85	-1.44

normality	not OK
n	79
outliers	4
mean (n)	43.359
st.dev. (n)	7.1008
R(calc.)	19.882
R(DIN54233-3-2010)	24.281



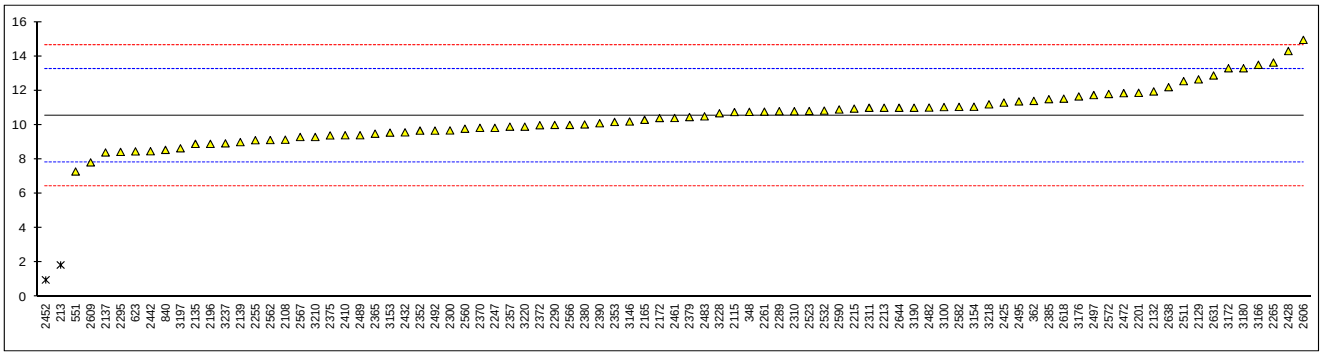


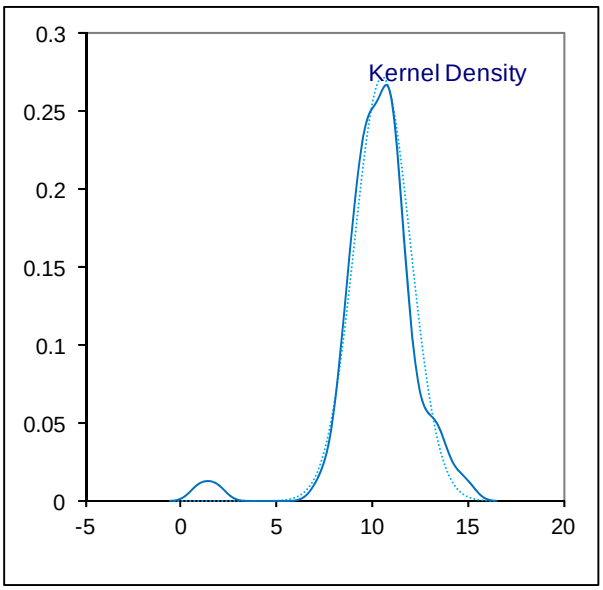
Determination of perspirated Cobalt on sample #14206; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	/Oekotex100/200	1.85	R(0.01)	-6.34	
348	INH-7334	10.76		0.17	
362	INH-103	11.4		0.64	
551	E104/ISO11085	7.29		-2.37	
623	ISO105E04	8.4615		-1.51	
840	DIN54233	8.55		-1.45	
2108	in house	9.14		-1.02	
2115	DIN54233-3	10.75		0.16	
2129	EN16711-2	12.652		1.55	
2132	E DIN54233-3	11.95		1.04	
2135	E DIN54233-3	8.9		-1.19	
2137	E DIN54233-3	8.4		-1.56	
2139	Oekotex100	9		-1.12	
2165	E DIN54233-3	10.30		-0.17	
2172	DIN54233-3	10.40		-0.10	
2196	DIN54233-3	8.9		-1.19	
2201	DIN54233-3	11.87		0.98	
2213	E DIN54233-3	11		0.34	
2215	E DIN54233-3	10.95		0.31	
2247	ISO105E04	9.83		-0.51	
2255	ISO105E04	9.11		-1.04	
2261	GB/T17593	10.773		0.18	
2265	E DIN54233-3	13.63		2.26	
2289	E DIN54233-3	10.80		0.20	
2290	ISO105E04	10.0		-0.39	
2295	DIN54233-3	8.43		-1.53	
2300	E DIN54233-3	9.68		-0.62	
2310	DIN54233-3	10.8		0.20	
2311	DIN54233-3	11.0		0.34	
2352	DIN54233-3	9.67		-0.63	
2353	Oekotex	10.1755		-0.26	
2357	E DIN54233-3	9.90		-0.46	
2365	DIN54233-3	9.491		-0.76	
2370	DIN54233-3	9.83		-0.51	
2372	E DIN54233-3	9.984		-0.40	
2375	E DIN54233-3	9.39	C	-0.83	First reported 9.39 as Copper
2379	E DIN54233-3	10.46		-0.05	
2380	E DIN54233-3	10.03		-0.37	
2385	E DIN54233-3	11.5		0.71	
2390	ISO105E04	10.10		-0.31	
2410	DIN54233-3	9.4		-0.83	
2425	DIN54233-3	11.30		0.56	
2428	GB/T17593	14.30		2.75	
2432	ISO105E04	9.57		-0.70	
2442	in house	8.47		-1.51	
2452	E DIN54233-3	0.98	R(0.01)	-6.98	
2461	GB/T17593	10.405		-0.09	
2472	GB/T17593	11.85		0.96	
2482	DIN54233-3	11.011		0.35	
2483	ISO105E04	10.5000		-0.02	
2489	E DIN54233-3	9.40		-0.83	
2492	in house	9.67		-0.63	
2495	ISO17072-1	11.37		0.61	
2497	DIN54233-3	11.74		0.88	
2511	DIN54233-3	12.55		1.48	
2523	E DIN54233-3	10.81		0.20	
2532	E DIN54233-3	10.8329		0.22	
2560	DIN54233-3	9.78		-0.55	
2562	E DIN54233-3	9.12		-1.03	
2566	ISO105E04	10.0		-0.39	
2567	ISO105E04	9.3		-0.90	
2572	ISO105E04	11.8		0.93	
2582	ISO105E04	11.05		0.38	
2590	ISO17072-1	10.90		0.27	
2606	in house	14.95		3.23	
2609	GB/T17593	7.822		-1.98	
2614		-----		-----	
2618	in house	11.53		0.73	
2631	KS K0731	12.878		1.71	
2638	E DIN54233-3	12.2		1.22	
2644	DIN54233-3	11.0		0.34	
3100	E DIN54233-3	11.04		0.37	
3146	E DIN54233-3	10.2		-0.24	
3153	ISO105E04	9.555		-0.71	
3154	DIN54233-3	11.06		0.39	

3166	INH-7040	13.5	2.17
3172	DIN54233-3	13.3	2.02
3176	DIN54233-3	11.66	0.83
3180	DIN54233-3	13.3	2.02
3190	E DIN54233-3	11.0	0.34
3197	ISO105E04	8.64	-1.38
3210	ISO17072-1	9.30	-0.90
3218	E DIN54233-3	11.2	0.49
3220	DIN54233	9.9	-0.46
3228	E DIN54233-3	10.68	0.11
3237	in house	8.93	-1.17

normality	OK
n	83
outliers	2
mean (n)	10.530
st.dev. (n)	1.4669
R(calc.)	4.107
R(DIN54233-3-2010)	3.833





APPENDIX 2

Determination of other perspirated metals on sample #14205 and #14206; results in mg/kg

	#14205								#14206						
lab	As	Sb	Cd	Cr	Co	Pb	Hg	Ni	As	Cd	Cr	Cu	Pb	Hg	Ni
213							0.44	0.25						0.14	0.15
348															
362												0.545			
551															
623															
840															
2108								0.153							0.100
2115		0.38		0.06				0.19			0.12	0.24			0.07
2129								0.274				0.934			0.258
2132															
2135															
2137															
2139															
2165															
2172															
2196															
2201															
2213												**47**			
2215															
2247															
2255															
2261															
2265				0.45	0.62			1.24		0.03	0.29	2.40			1.47
2289															
2290															
2295															
2300															
2310															
2311															
2352															
2353	0.0285	0.0113	0.0034	0.0505	0.0024		0.0010	0.1922	0.0790	0.0050	0.0506	0.3774	0.0047		0.1601
2357															
2365															
2370															
2372															
2375															
2379															
2380															
2385															
2390															
2410															
2425															
2428															
2432															
2442															
2452				0.41		2.06		0.076			0.46	0.23	1.9		0.08

2461															
2472															
2482															
2483															
2489															
2492															
2495															
2497	0.0011	0.112	0.0025	0.111	0.012	0.048	0.0091	0.361	0.0009	0.016	0.232	0.001	0.123	0.0081	0.118
2511															0.518
2523								0.2102				0.3186			0.1566
2532			0.02					0.1138				0.35			0.1483
2560															
2562															
2566															
2567															
2572												2.1			
2582															
2590				0.41											
2606								0.19						0.039	0.28
2609															
2614															
2618															
2631	0.012					0.145		0.125			0.043	0.373	0.043		0.100
2638															
2644								0.31				0.51			0.37
3100															
3146															
3153															
3154															
3166									**65.2**						
3172															
3176															
3180															
3190															
3197												0.19			
3210															
3218															
3220	1.0			1.0		0.5		0.5		0.5	1.2				
3228															
3237															

Results in bold and underlined are false positive results

APPENDIX 3

Number of participants per country:

6 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
1 lab in	FRANCE
8 labs in	GERMANY
5 labs in	HONG KONG
10 labs in	INDIA
1 lab in	INDONESIA
6 labs in	ITALY
4 labs in	KOREA
1 lab in	MOROCCO
19 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	SPAIN
1 lab in	SRI LANKA
1 lab in	SWITZERLAND
4 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
2 labs 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 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 applicable
n.d. = not detected

Literature:

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17 E DIN 54233-3:2010 (entwurf)
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