Results of Proficiency Test
Perspirated Metals in textile
October 2011

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. Also this year this scheme is part of the proficiency testing program 2011/2012.
In this interlaboratory study 70 laboratories in 23 different countries participated. See appendix 3 for the number of participants per country. In this report the results of this proficiency test are presented and discussed.

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 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 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO17043:2010.
This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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 January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

All data present 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 a subcontractor. The two finely cut textile samples, an orange fabric (sample #11085) and a yellow fabric (sample #11088) were each well mixed and divided over 130 subsamples of approx. 3.5 grams. The samples were labelled and tested for homogeneity on 8 randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17025 accredited laboratory.
See the following tables for the homogeneity test results.

	Perspirated Nickel in mg/kg	*Perspirated Chromium in mg/kg*
Sample #11085-1	15.56	3.90
Sample #11085-2	15.48	3.69
Sample #11085-3	15.50	3.43
Sample #11085-4	15.48	3.86
Sample #11085-5	15.54	3.50
Sample #11085-6	15.59	3.42
Sample #11085-7	15.23	3.42
Sample #11085-8	15.47	3.62

table 1: homogeneity test results of subsamples #11085

	Perspirated Antimony in mg/kg	*Perspirated Cobalt in mg/kg*
Sample #11088-1	31.7	98.9
Sample #11088-2	30.8	97.1
Sample #11088-3	31.4	101.7
Sample #11088-4	33.0	99.1
Sample #11088-5	33.0	96.3
Sample #11085-6	33.0	101.5
Sample #11085-7	31.8	97.8
Sample #11085-8	31.3	99.7

table 2: homogeneity test results of subsamples #11088

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 Nickel in mg/kg	*Perspirated Chromium in mg/kg*
r (observed)	0.3	0.5
reference method	Horwitz	Horwitz
0.3 x R (reference method)	1.4	0.4

Table 3: repeatabilities of subsamples #11085

	Perspirated Antimony in mg/kg	*Perspirated Cobalt in mg/kg*
r (observed)	2.5	5.4
reference method	Horwitz	Horwitz
0.3 x R (reference method)	2.6	6.7

Table 4: repeatabilities of subsamples #11088

The calculated repeatabilities are both in good agreement with 0.3 times the estimated target reproducibilities, calculated using the Horwitz equation. Therefore, homogeneity of all subsamples was assumed.

In total approx. 3.5 grams of each of the samples #11085 and #11088 were sent to the participating laboratories on October 12, 2011.

2.5 ANALYSES

The participants were asked to determine the concentrations of 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 to use preferably a solid/liquid ration of 1/50 g/ml as prescribed in E-DIN54233-3:2010. To get comparable results, detailed report forms were sent together with each set of samples. On the report forms the requested heavy metals, including the units and questions about the analytical details, were pre-printed. Also a letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the 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, see lit.5) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 3.

3.1 STATISTICS

Statistical calculations were performed as described in the report ’iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of January 2010 (iis-protocol, version 3.2).
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.
Before further calculations, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. In the case of an anormal distribution, the statistical evaluation should be used with care.

According to ISO 5725 (1986 and 1994, lit.8 and 9) the original results per determination were submitted subsequently to Dixon’s and Grubbs' 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. Stragglers are

marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' 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 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 method is producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nos.14 and 15).

3.3 Z-SCORES

To evaluate the performance of the individual participating laboratories the z-scores were calculated.
In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The $z_{(target)}$-scores were calculated according to:

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

The $z_{(target)}$-scores are listed in the result tables in appendix 1.

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 the fit-for-useness of the reported test result.

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

During the execution of this proficiency test, no problems occurred with sample dispatch and with the reporting of the test results. However, three laboratories decided not to report any test results due to various reasons.
Ten laboratories reported test results after the final reporting date.
Finally, the 67 reporting laboratories did report in total 340 numerical results.
All participating laboratories, except one, did use a solid/liquid ratio of 1/50 as requested. The deviating laboratory used as solid/liquid ratios 1/500, 1/1000 and 1/2500 g/mL.
Observed were 22 statistical outlying results, which is 6.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets, except for Cobalt (#11088) proved to have a non-Gaussian distribution. Therefore, the statistical evaluation for these determinations should be used with care.

The majority of the participating laboratories used ISO 1505-E04, a method that regretfully does not mention any reproducibility. In 2010 the draft method DIN 54233-3 was issued. This method does mention one reproducibility for one concentration per metal (varying from 13-40%). As the actual reproducibility will be concentration dependent and as the concentrations in this PT are significantly different from the ones mentioned in DIN54233-3, the reproducibilities, estimated by the Horwitz equation, were used in this PT for evaluation.

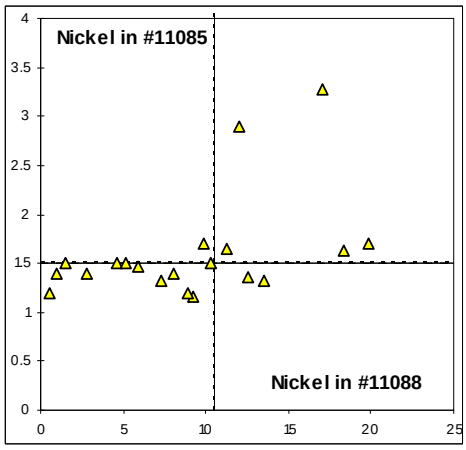
4.1 EVALUATION PER METAL

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

Chromium: The determination of this metal was problematic for sample #11085 at a perspiration level of 5.1 mg/kg.
Only two statistical outliers were observed. However, the observed reproducibility after rejection of the statistical outliers, is not in agreement with the estimated target reproducibility calculated using the Horwitz equation.

Nickel: The determination of this metal was only problematic for a number of participating laboratories. For sample #11085 at a perspiration level of 25.3 mg/kg, five statistical outliers were observed and for sample #11088, six statistical outliers were observed.

Both calculated reproducibilities are after rejection of the statistical outliers in full agreement with the estimated target reproducibilities calculated using the Horwitz equation. A significant correlation of the Nickel results on the two samples #11085 and #11088 was not detected, see below graph.



Antimony: The determination of this metal was very problematic for sample #11088 at a perspiration level of 49.3 mg/kg. Four statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers, is not at all in agreement with the estimated target reproducibility calculated using the Horwitz equation.

Cobalt: The determination of this metal was not problematic for sample #11088 at a perspiration level of 104.9 mg/kg. Only two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers, is in full agreement with the estimated target reproducibility calculated using the Horwitz equation.

Copper: The determination of this metal was problematic for sample #11088 at a perspiration level of 1.8 mg/kg, which may be below or near the detection limit. Three statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers, is not in agreement with the estimated target reproducibility calculated using the Horwitz equation.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the strict calculated reproducibilities using the Horwitz equation 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 (Horwitz equation), are compared in the next two tables.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Perspirated Chromium	mg/kg	59	5.12	2.70	1.79
Perspirated Nickel	mg/kg	59	25.3	7.2	7.0

table 5: reproducibilities of perspirated metals in sample #11085

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Perspirated Antimony	mg/kg	60	49.3	25.7	12.3
Perspirated Cobalt	mg/kg	61	104.9	24.2	23.3
Perspirated Copper	mg/kg	31	1.78	1.08	0.73
Perspirated Nickel	mg/kg	48	1.48	0.57	0.62

table 6: reproducibilities of perspirated metals in sample #11088

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories have no difficulties with the analysis when compared with the strict target results calculated with the Horwitz equation. See also the discussions in paragraphs 4.1 and 6.

5 COMPARISON WITH THE PREVIOUS PROFICIENCY TESTS

The spreads that were found in the results during the present round are in agreement with the spreads as observed in previous rounds (see below table).

Parameter	*October 2011*	*October 2010*	*October 2009*	*November 2008*	*November 2007*	*November 2006*	*November 2005*
Arsenic	--	--	--	--	66%	--	--
Antimony	52%	--	41%	46%	--	--	--
Cadmium	--	40%	--	--	--	85%	57%
Chromium	53%	--	--	--	114%	47%	83%
Cobalt	23%	31%	--	28%	--	--	50%
Copper	61%	--	48-46%	26%	64-80%	93-39	66%
Lead	--	--	--	--	375%	138-172%	--
Mercury	--	--	--	--	--	134%	--
Nickel	29-38%	19%	--	23%	41%	--	88-39%

table 7: Comparison of observed relative reproducibilities (since 2008 s/l ratio prescribed on 1:50)

The improvement of the results in the last four rounds is most probably caused by the use of a more uniform solid/liquid ratio then in rounds before 2008 (>90% of all participating laboratories reported results after use of a liquor ratio of 1:50 since 2008).

6 DISCUSSION

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (table 9), it could be 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	EU-adult clothes	EU-baby clothes	Öko-Tex 103 non skin contact	Öko-Tex 103 direct skin contact	Öko-Tex 106 baby clothes
Arsenic (As) mg/kg	1)	1)	1.0	1.0	0.2
Antimony (Sb) mg/kg	1)	1)	30.0	30.0	30.0
Cadmium (Cd) mg/kg	1)	1)	0.1	0.1	0.1
Chromium (Cr) mg/kg	1)	1)	2.0	2.0	1.0
Cobalt (Co) mg/kg	1)	1)	4.0	4.0	1.0
Copper (Cu) mg/kg	1)	1)	50.0	50.0	25.0
Lead (Pb) mg/kg	1)	1)	1.0	1.0	0.2
Mercury (Hg) mg/kg	1)	1)	0.02	0.02	0.02
Nickel (Ni) mg/kg	1)	1)	4.0	4.0	1.0

table 8: Ecolabelling Standards and Requirements for Textiles in EU

1) No use of metals in dyes and pigments

Methods for determination of these Heavy Metals are specified in the Standards of the Ecolabelling Institutes. Unfortunately, only test methods for the release of heavy metals via perspiration is mentioned. 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. However, the liquor ratio (ml of perspiration liquid / gram of textile) appeared to be a parameter of utmost importance and without mentioning this ratio (or the respective test method), the test results may have little value (see previous reports iis07A05 and iis08A05 on “Perspirated Metals in Textile”). Therefore in this proficiency test the participating laboratories were advised to use preferably a ratio of 1:50 as in the latest available draft test method E DIN 54233-3:2010.

General

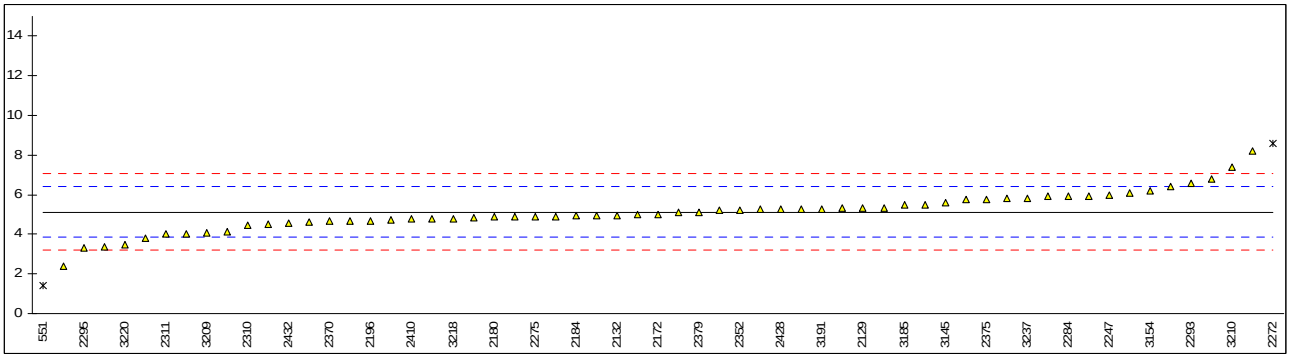
The spreads observed in this interlaboratory study are not caused by just one critical point in the analysis. Consequently, the reproducibilities cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus rise of the quality of the analytical results.

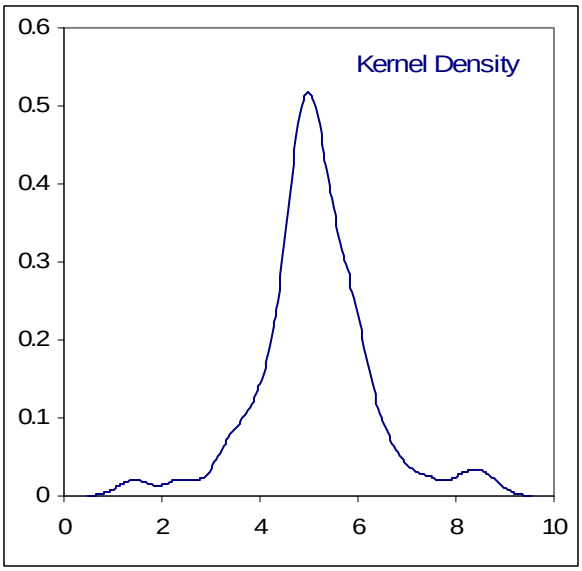
APPENDIX 1

Determination of perspirated Chromium (s/l ratio = 1/50) on sample #11085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	3.36		-2.74	
551		1.43	G(0.05)	-5.76	
1179	DIN54233-3	4.94		-0.28	
2120	DIN54233-3	6.1		1.53	
2129	ISO105-E04	5.3		0.29	
2132	in house	4.95		-0.26	
2137	ISO105-E04	5.9		1.22	
2165	DIN54233	5.0		-0.18	
2172	ISO105-E04	5.02		-0.15	
2180	DIN54233-3	4.9		-0.34	
2181	in house	n.d.		-----	
2184	DIN54233	4.93		-0.29	
2196	DIN54233-3	4.7		-0.65	
2201	DIN54233	4.8		-0.50	
2247	ISO105-E04	6		1.38	
2256	ISO105-E04	4.90		-0.34	
2265	DIN54233-3	2.39		-4.26	
2272	ISO17071-1	8.6	G(0.05)	5.44	
2275	DIN54233-3	4.9		-0.34	
2284	DIN54233	5.9		1.22	
2285	DIN54233-3	5.3		0.29	
2293	ISO105-E04	6.569		2.27	
2295	in house	3.3		-2.84	
2303	in house	5.3		0.29	
2310	ISO105-E04	4.44		-1.06	
2311	ISO105-E04	4.0		-1.74	
2352	OEKO-Tex	5.2		0.13	
2357	ISO105-E04	4.86		-0.40	
2358	OEKO-Tex	4.5		-0.96	
2363	ISO105-E04	5.1		-0.03	
2365	ISO105-E04	4.6		-0.81	
2366		-----		-----	
2370	ISO105-E04	4.66		-0.71	
2372	ISO105-E04	6.40		2.00	
2375	INH-103	5.76		1.00	
2379	ISO105-E04	5.12		0.00	
2380	ISO105-E04	n.d.		-----	
2385	DIN54233-3	5.9		1.22	
2390	ISO105-E04	5.2		0.13	
2410	in house	4.8		-0.50	
2428		5.27		0.24	
2432	in house	4.54		-0.90	
2442	in house	4.0365		-1.69	
2452	DIN54233-3	6.82		2.66	
2459	ISO105-E04	n.d.		-----	
2461		-----		-----	Reported 4.63 mg/kg for s/l=1/500 (z=-0.68)
3117	OEKO-Tex	4.73		-0.60	
3124		-----		-----	
3145	INH-10	5.6		0.75	
3153	ISO105-E04	5.5		0.60	
3154	ISO71072-1	6.186		1.67	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	5.76		1.00	
3185	ISO105-E04	5.5		0.60	
3190	OEKO-Tex	4.7		-0.65	
3191	OEKO-Tex	5.29		0.27	
3197	ISO105-E04	5.8		1.07	
3200	in house	3.8		-2.06	
3203	DIN54233	4.13		-1.54	
3204	DIN54233-3	5.28		0.25	
3209	in house	4.1		-1.59	
3210	EN17072-1	7.40		3.56	
3214	DIN54233-3	5.27	C	0.24	First reported 7.27
3218	DIN54233-3	4.8		-0.50	
3220	ISO105-E04	3.5		-2.53	
3222	in house	8.2		4.81	
3226		-----		-----	
3228	in house	4.9		-0.34	
3237	in house	5.81		1.08	

normality	not OK
n	59
outliers	2
mean (n)	5.117
st.dev. (n)	0.9652
R(calc.)	2.703
R(Horwitz)	1.793

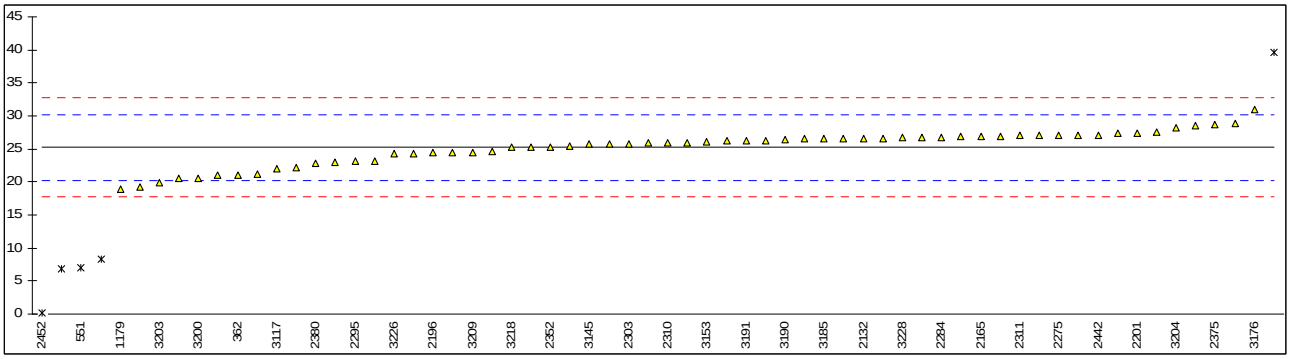


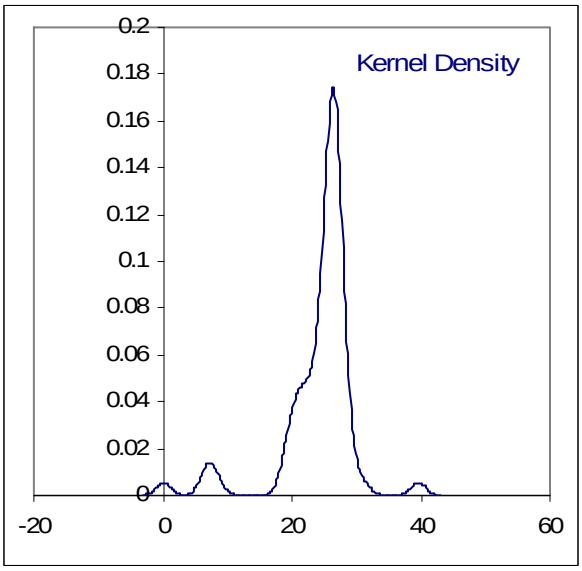


Determination of perspirated Nickel (s/l ratio = 1/50) on sample #11085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	21.1		-1.67	
551		7.01	G(0.01)	-7.34	
1179	DIN54233-3	18.9		-2.56	
2120	DIN54233-3	26		0.30	
2129	ISO105-E04	28.6		1.35	
2132	in house	26.55		0.52	
2137	ISO105-E04	24.7	C	-0.22	First reported 11.0
2165	DIN54233	26.9		0.66	
2172	ISO105-E04	25.3		0.02	
2180	DIN54233-3	20.6		-1.87	
2181	in house	19.2476		-2.42	
2184	DIN54233	26.32		0.43	
2196	DIN54233-3	24.4		-0.34	
2201	DIN54233	27.4		0.87	
2247	ISO105-E04	26		0.30	
2256	ISO105-E04	21.18		-1.64	
2265	DIN54233-3	8.36	G(0.01)	-6.80	
2272	ISO17071-1	23.0		-0.91	
2275	DIN54233-3	27.1		0.74	
2284	DIN54233	26.8		0.62	
2285	DIN54233-3	23.1		-0.87	
2293	ISO105-E04	27.323		0.83	
2295	in house	23.1		-0.87	
2303	in house	25.8		0.22	
2310	ISO105-E04	26		0.30	
2311	ISO105-E04	27.0		0.70	
2352	OEKO-Tex	25.3		0.02	
2357	ISO105-E04	26.50		0.50	
2358	OEKO-Tex	26.5		0.50	
2363	ISO105-E04	25.5		0.10	
2365	ISO105-E04	25.8		0.22	
2366		-----		-----	
2370	ISO105-E04	24.3		-0.38	
2372	ISO105-E04	24.5		-0.30	
2375	INH-103	28.66		1.37	
2379	ISO105-E04	26.64		0.56	
2380	ISO105-E04	22.75		-1.01	
2385	DIN54233-3	26.2		0.38	
2390	ISO105-E04	22.2		-1.23	
2410	in house	21		-1.71	
2428		27.14		0.76	
2432	in house	27.48		0.90	
2442	in house	27.146		0.76	
2452	DIN54233-3	0.11	G(0.01)	-10.12	
2459	ISO105-E04	6.8	G(0.01)	-7.42	
2461		-----		-----	Reported 23.62 mg/kg for s/l=1/1000 (z=-0.66)
3117	OEKO-Tex	21.97		-1.32	
3124		-----		-----	
3145	INH-10	25.7		0.18	
3153	ISO105-E04	26.1		0.34	
3154	ISO71072-1	26.86		0.65	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	31	C	2.31	First reported 41.5
3185	ISO105-E04	26.5		0.50	
3190	OEKO-Tex	26.4		0.46	
3191	OEKO-Tex	26.24		0.40	
3197	ISO105-E04	28.8		1.43	
3200	in house	20.6		-1.87	
3203	DIN54233	19.85		-2.17	
3204	DIN54233-3	28.2		1.19	
3209	in house	24.5		-0.30	
3210	EN17072-1	<1		-----	
3214	DIN54233-3	26.96		0.69	
3218	DIN54233-3	25.2		-0.02	
3220	ISO105-E04	26.8	C	0.62	First reported 11.7
3222	in house	39.6	G(0.01)	5.77	
3226	DIN54020	24.24		-0.41	
3228	in house	26.8		0.62	
3237	in house	27.01		0.71	

normality	not OK
n	59
outliers	5
mean (n)	25.25
st.dev. (n)	2.575
R(calc.)	7.21
R(Horwitz)	6.96

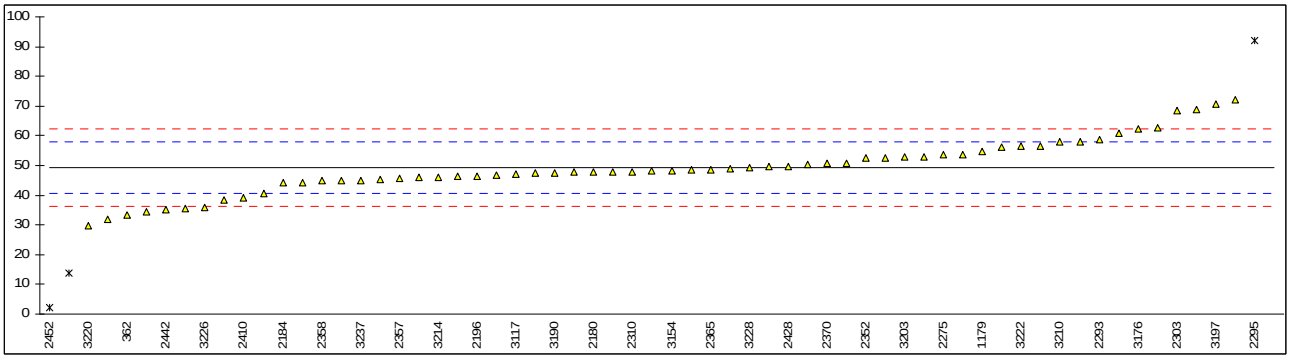


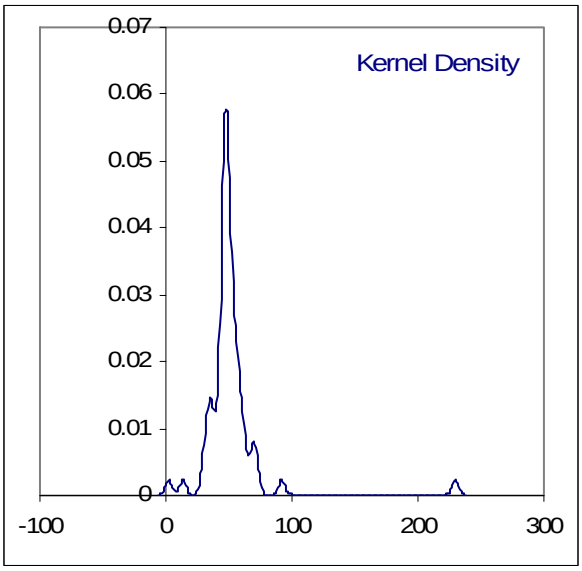


Determination of perspirated Antimony (s/l ratio = 1/50) on sample #11088; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	33.2		-3.67	
551		13.67	G(0.05)	-8.12	
1179	DIN54233-3	54.6		1.21	
2120	DIN54233-3	48		-0.30	
2129	ISO105-E04	40.6		-1.99	
2132	in house	45.25		-0.93	
2137	ISO105-E04	38.3		-2.51	
2165	DIN54233	48.4		-0.21	
2172	ISO105-E04	50.3		0.23	
2180	DIN54233-3	48		-0.30	
2181	in house	56.0387		1.53	
2184	DIN54233	44.21		-1.16	
2196	DIN54233-3	46.4		-0.66	
2201	DIN54233	48.1		-0.28	
2247	ISO105-E04	69		4.49	
2256	ISO105-E04	44.38		-1.12	
2265	DIN54233-3	229.99	G(0.01)	41.18	
2272	ISO17071-1	61.0		2.66	
2275	DIN54233-3	53.6		0.98	
2284	DIN54233	56.7		1.68	
2285	DIN54233-3	46.2		-0.71	
2293	ISO105-E04	58.810	C	2.16	First reported 186.0
2295	in house	92	C,G(0.05)	9.73	First reported 100
2303	in house	68.6		4.40	
2310	ISO105-E04	48		-0.30	
2311	ISO105-E04	46.0		-0.75	
2352	OEKO-Tex	52.4		0.70	
2357	ISO105-E04	45.53		-0.86	
2358	OEKO-Tex	44.8		-1.03	
2363	ISO105-E04	52.8		0.80	
2365	ISO105-E04	48.7		-0.14	
2366		-----		-----	
2370	ISO105-E04	50.8		0.34	
2372	ISO105-E04	52.4		0.70	
2375	INH-103	72.23		5.22	
2379	ISO105-E04	47.81		-0.34	
2380	ISO105-E04	48.95		-0.08	
2385	DIN54233-3	58.1		2.00	
2390	ISO105-E04	34.4		-3.40	
2410	in house	39		-2.35	
2428		49.51	C	0.05	First reported 77.73
2432	in house	35.52		-3.14	
2442	in house	35.2345		-3.21	
2452	DIN54233-3	2.15	G(0.01)	-10.75	
2459	ISO105-E04	n.d.		-----	
2461		-----		-----	Reported 48.55 mg/kg for s/l=1/2500 (z=-0.15)
3117	OEKO-Tex	47.01		-0.52	
3124		-----		-----	
3145	INH-10	53.8		1.02	
3153	ISO105-E04	62.6		3.03	
3154	ISO71072-1	48.11		-0.27	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	62.3	C	2.96	First reported 108.9
3185	ISO105-E04	46.7		-0.60	
3190	OEKO-Tex	47.5		-0.41	
3191	OEKO-Tex	50.82		0.34	
3197	ISO105-E04	70.8		4.90	
3200	in house	32.0		-3.95	
3203	DIN54233	52.77		0.79	
3204	DIN54233-3	49.5		0.04	
3209	in house	47.5		-0.41	
3210	EN17072-1	58		1.98	
3214	DIN54233-3	46.09		-0.73	
3218	DIN54233-3	45.0		-0.98	
3220	ISO105-E04	29.8		-4.45	
3222		56.4		1.62	
3226	DIN54020	35.88		-3.06	
3228	in house	49.2		-0.03	
3237	in house	45.01		-0.98	

normality	not OK
n	60
outliers	4
mean (n)	49.31
st.dev. (n)	9.187
R(calc.)	25.72
R(Horwitz)	12.29

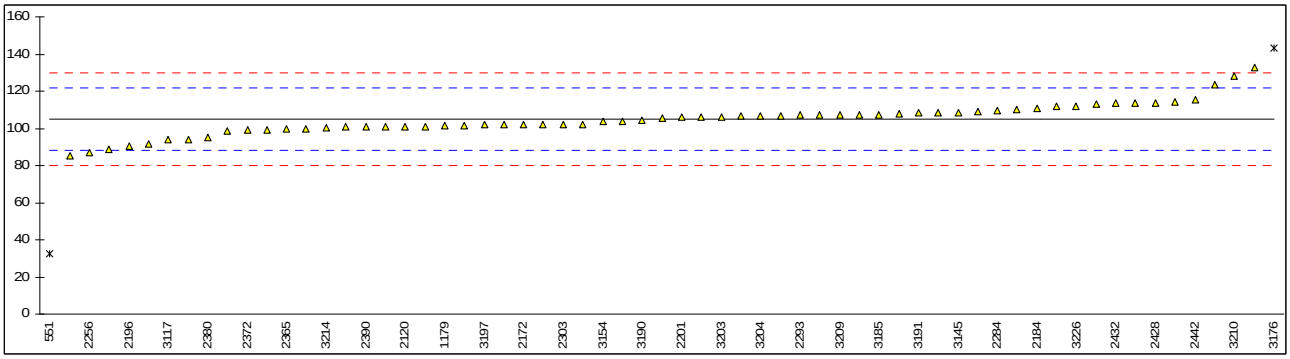


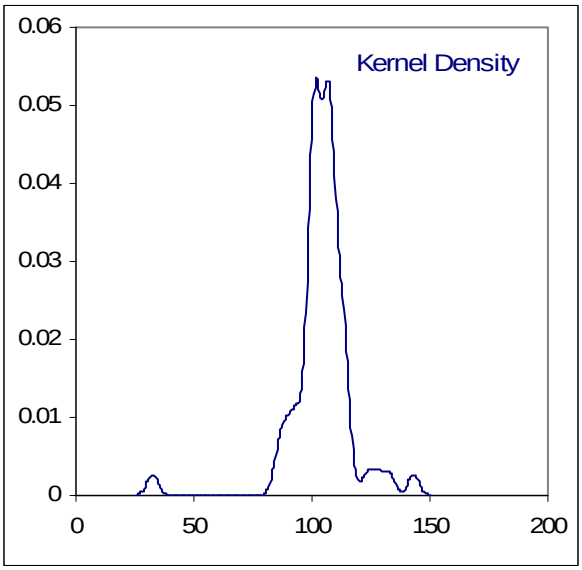


Determination of perspirated Cobalt (s/l ratio = 1/50) on sample #11088; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	94.0		-1.30	
551		32.58	G(0.01)	-8.68	
1179	DIN54233-3	101.2		-0.44	
2120	DIN54233-3	101		-0.46	
2129	ISO105-E04	113.7		1.06	
2132	in house	107.65		0.33	
2137	ISO105-E04	88.5	C	-1.96	First reported 64.5
2165	DIN54233	108.5		0.44	
2172	ISO105-E04	102		-0.34	
2180	DIN54233-3	112		0.86	
2181	in house	123.4377		2.23	
2184	DIN54233	110.57		0.69	
2196	DIN54233-3	90.3		-1.75	
2201	DIN54233	106.0		0.14	
2247	ISO105-E04	102		-0.34	
2256	ISO105-E04	87.17		-2.12	
2265	DIN54233-3	98.52		-0.76	
2272	ISO17071-1	101		-0.46	
2275	DIN54233-3	106.5		0.20	
2284	DIN54233	109.3		0.53	
2285	DIN54233-3	100.9		-0.48	
2293	ISO105-E04	107.133	C	0.27	First reported 303.5
2295	in house	101.3		-0.43	
2303	in house	102		-0.34	
2310	ISO105-E04	104		-0.10	
2311	ISO105-E04	108.9		0.48	
2352	OEKO-Tex	101.1		-0.45	
2357	ISO105-E04	107.37		0.30	
2358	OEKO-Tex	105.5		0.08	
2363	ISO105-E04	102.0		-0.34	
2365	ISO105-E04	99.5		-0.64	
2366		-----		-----	
2370	ISO105-E04	106		0.14	
2372	ISO105-E04	98.9		-0.72	
2375	INH-103	114.30	C	1.13	First reported 11.43
2379	ISO105-E04	10.6.85		-----	
2380	ISO105-E04	95.23		-1.16	
2385	DIN54233-3	110		0.62	
2390	ISO105-E04	101.0	C	-0.46	First reported 63.2
2410	in house	100	C	-0.58	First reported 67
2428		113.70		1.06	
2432	in house	113.36		1.02	
2442	in house	115.42		1.27	
2452	DIN54233-3	132.92		3.37	
2459	ISO105-E04	n.d.		-----	
2461		-----		-----	Reported 99.55 mg/kg for s/l=1/5000 (z=-0.64)
3117	OEKO-Tex	93.76		-1.33	
3124		-----		-----	
3145	INH-10	108.5		0.44	
3153	ISO105-E04	102.1		-0.33	
3154	ISO71072-1	103.5		-0.16	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	143.2	C,G(0.01)	4.60	First reported 156.8
3185	ISO105-E04	107.4		0.30	
3190	OEKO-Tex	104.5		-0.04	
3191	OEKO-Tex	108.2		0.40	
3197	ISO105-E04	102.0		-0.34	
3200	in house	85.3		-2.35	
3203	DIN54233	106.25		0.17	
3204	DIN54233-3	106.5		0.20	
3209	in house	107.2		0.28	
3210	EN17072-1	128		2.78	
3214	DIN54233-3	100.01		-0.58	
3218	DIN54233-3	99.3		-0.67	
3220	ISO105-E04	91.6	C	-1.59	First reported 37.8
3222	in house	106.4		0.18	
3226	DIN54020	112.09		0.87	
3228	in house	107.2		0.28	
3237	in house	112.9		0.97	

normality	OK
n	61
outliers	2
mean (n)	104.86
st.dev. (n)	8.628
R(calc.)	24.16
R(Horwitz)	23.32

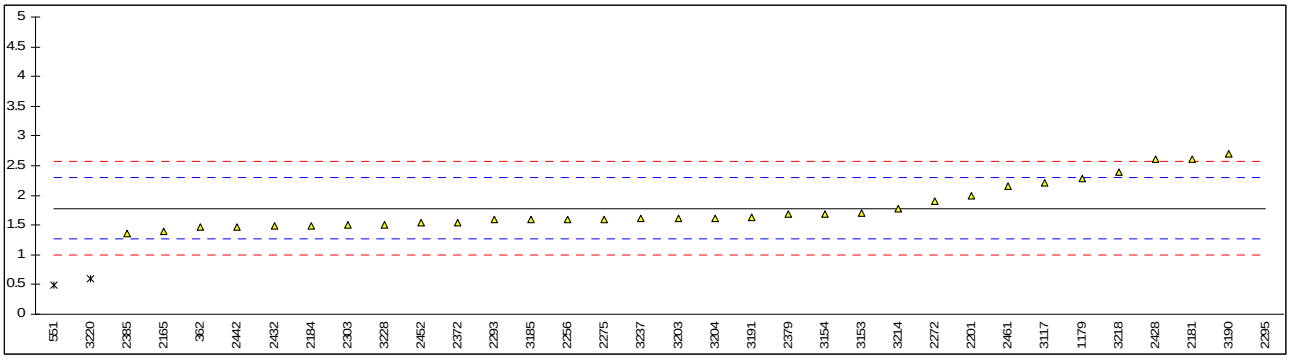


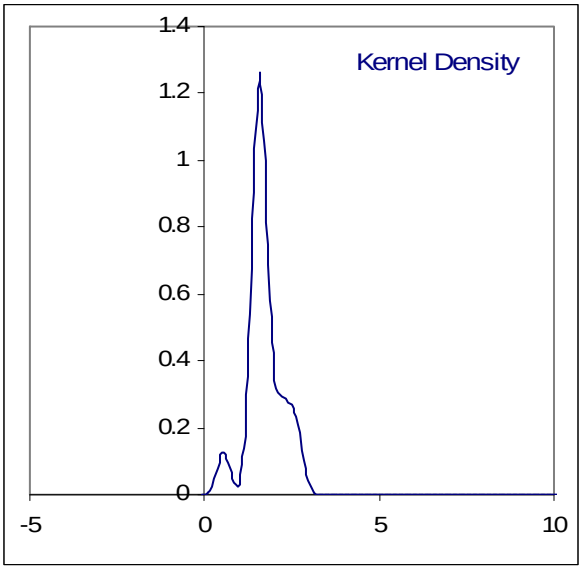


Determination of perspirated Copper (s/l ratio = 1/50) on sample #11088; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	1.46		-1.24	
551		0.49	G(0.01)	-4.95	
1179	DIN54233-3	2.29		1.93	
2120	DIN54233-3	<2.5		-----	
2129	ISO105-E04	<5.0		-----	
2132	in house	<2.0		-----	
2137	ISO105-E04	n.d.		-----	
2165	DIN54233	1.4		-1.47	
2172	ISO105-E04	<5.0		-----	
2180		-----		-----	
2181	in house	2.6053		3.14	
2184	DIN54233	1.49		-1.12	
2196	DIN54233-3	<0.5		<-4.96	False negative?
2201	DIN54233	2.0		0.83	
2247		-----		-----	
2256	ISO105-E04	1.60		-0.70	
2265	DIN54233-3	n.d.		-----	
2272	ISO17071-1	1.9		0.44	
2275	DIN54233-3	1.6		-0.70	
2284	DIN54233	<5.0		-----	
2285		-----		-----	
2293	ISO105-E04	1.591	C	-0.74	First reported 6.838
2295	in house	20.2	G(0.01)	70.39	
2303	in house	1.5		-1.09	
2310	ISO105-E04	n.d.		-----	
2311	ISO105-E04	<5.0		-----	
2352	OEKO-Tex	n.d.		-----	
2357	ISO105-E04	n.d.		-----	
2358	OEKO-Tex	<5.0		-----	
2363	ISO105-E04	<5.0		-----	
2365	ISO105-E04	n.d.		-----	
2366		-----		-----	
2370	ISO105-E04	n.d.		-----	
2372	ISO105-E04	1.54		-0.93	
2375	INH-103	n.d.		-----	
2379	ISO105-E04	1.68		-0.40	
2380	ISO105-E04	n.d.		-----	
2385	DIN54233-3	1.36		-1.62	
2390		-----		-----	
2410	in house	n.d.		-----	
2428		2.60		3.12	
2432	in house	1.48		-1.16	
2442	in house	1.46		-1.24	
2452	DIN54233-3	1.54		-0.93	
2459	ISO105-E04	n.d.		-----	
2461	INH-17593	2.16		1.44	
3117	OEKO-Tex	2.21		1.63	
3124		-----		-----	
3145		-----		-----	
3153	ISO105-E04	1.7		-0.32	
3154	ISO71072-1	1.685		-0.38	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	n.d.		-----	
3185	ISO105-E04	1.6		-0.70	
3190	OEKO-Tex	2.7		3.50	
3191	OEKO-Tex	1.63		-0.59	
3197		-----		-----	
3200	in house	<2.0		-----	
3203	DIN54233	1.61		-0.66	
3204	DIN54233-3	1.62		-0.63	
3209	in house	<2.0		-----	
3210	EN17072-1	<25		-----	
3214	DIN54233-3	1.78		-0.01	
3218	DIN54233-3	2.4		2.35	
3220	ISO105-E04	0.6	G(0.01)	-4.53	
3222		-----		-----	
3226		-----		-----	
3228	in house	1.5		-1.09	
3237	in house	1.61		-0.66	

normality	not OK
n	31
outliers	3
mean (n)	1.784
st.dev. (n)	0.3861
R(calc.)	1.081
R(Horwitz)	0.733

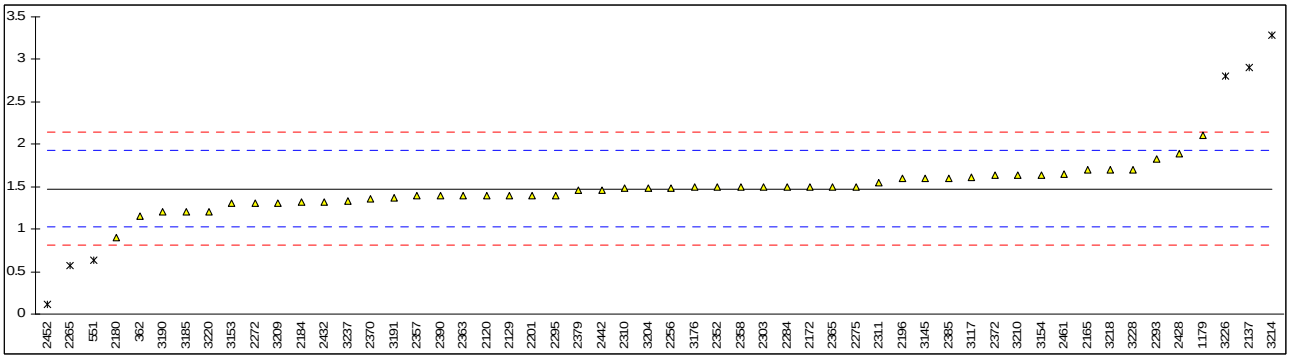


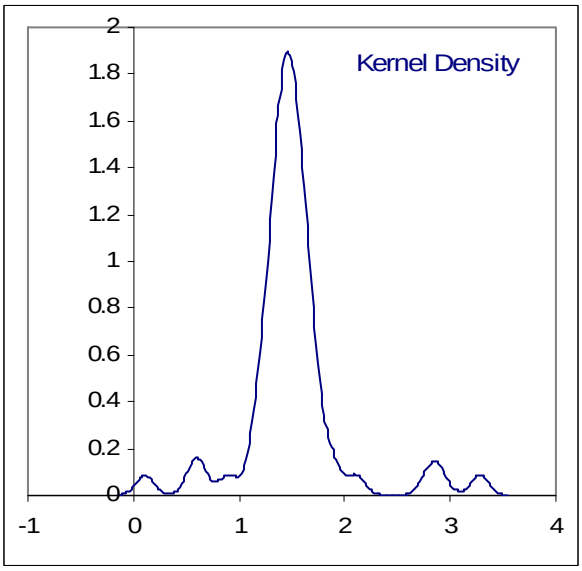


Determination of perspirated Nickel (s/l ratio = 1/50) on sample #11088; results in mg/kg

lab	method	value	mark	z(targ)	remarks
362	INH-103	1.15		-1.46	
551		0.63	G(0.01)	-3.80	
1179	DIN54233-3	2.11		2.85	
2120	DIN54233-3	1.4		-0.34	
2129	ISO105-E04	1.4		-0.34	
2132	in house	<2.0		-----	
2137	ISO105-E04	2.9	G(0.05)	6.39	
2165	DIN54233	1.7		1.01	
2172	ISO105-E04	1.50		0.11	
2180	DIN54233-3	0.9		-2.59	
2181	in house	n.d.		-----	
2184	DIN54233	1.32		-0.70	
2196	DIN54233-3	1.6		0.56	
2201	DIN54233	1.4		-0.34	
2247		-----		-----	
2256	ISO105-E04	1.49		0.06	
2265	DIN54233-3	0.57	G(0.05)	-4.07	
2272	ISO17071-1	1.3		-0.79	
2275	DIN54233-3	1.5		0.11	
2284	DIN54233	1.5		0.11	
2285		-----		-----	
2293	ISO105-E04	1.828	C	1.58	First reported 5.678
2295	in house	1.4		-0.34	
2303	in house	1.5		0.11	
2310	ISO105-E04	1.48		0.02	
2311	ISO105-E04	1.55		0.33	
2352	OEKO-Tex	1.5		0.11	
2357	ISO105-E04	1.40		-0.34	
2358	OEKO-Tex	1.5		0.11	
2363	ISO105-E04	1.4		-0.34	
2365	ISO105-E04	1.5		0.11	
2366		-----		-----	
2370	ISO105-E04	1.36		-0.52	
2372	ISO105-E04	1.63		0.69	
2375	INH-103	n.d.		-----	
2379	ISO105-E04	1.46		-0.07	
2380	ISO105-E04	n.d.		-----	
2385	DIN54233-3	1.60		0.56	
2390	ISO105-E04	1.4		-0.34	
2410	in house	n.d.		-----	
2428		1.89		1.86	
2432	in house	1.32		-0.70	
2442	in house	1.46		-0.07	
2452	DIN54233-3	0.11	G(0.05)	-6.13	
2459	ISO105-E04	n.d.		-----	
2461	INH-17593	1.65		0.78	
3117	OEKO-Tex	1.61		0.60	
3124		-----		-----	
3145	INH-10	1.6		0.56	
3153	ISO105-E04	1.3		-0.79	
3154	ISO71072-1	1.636		0.72	
3166		-----		-----	
3172		-----		-----	
3176	DIN54233-3	1.5		0.11	
3185	ISO105-E04	1.2		-1.24	
3190	OEKO-Tex	1.2		-1.24	
3191	OEKO-Tex	1.37		-0.48	
3197		-----		-----	
3200	in house	<2.0		-----	
3203		-----		-----	
3204	DIN54233-3	1.48		0.02	
3209	in house	1.3		-0.79	
3210	EN17072-1	1.63		0.69	
3214	DIN54233-3	3.28	G(0.01)	8.10	
3218	DIN54233-3	1.7		1.01	
3220	ISO105-E04	1.2	C	-1.24	First reported 0.5
3222		-----		-----	
3226	DIN54020	2.80	G(0.01)	5.94	
3228	in house	1.7		1.01	
3237	in house	1.33		-0.66	

normality	not OK
n	48
outliers	6
mean (n)	1.476
st.dev. (n)	0.2021
R(calc.)	0.566
R(Horwitz)	0.624





Determination of other perspirated metals on sample #11085; results in mg/kg

lab	As	Sb	Cd	Co	Cu	Pb	Hg
362	--	--	--	--	0.36	--	--
551	--	--	--	--	0.12	--	--
1179	0.037	--	--	0.012	0.239	--	--
2120	--	--	--	--	--	--	--
2129	--	--	--	--	--	--	--
2132	--	--	--	--	--	--	--
2137	--	--	--	--	--	--	--
2165	--	--	--	--	--	--	--
2172	--	--	--	--	--	--	--
2180	--	--	--	--	--	--	--
2181	--	--	--	--	0.6659	--	--
2184	--	--	--	--	--	--	--
2196	--	--	--	--	--	--	--
2201	--	--	--	--	--	--	--
2247	--	--	--	--	--	--	--
2256	--	--	--	--	0.34	--	--
2265	--	15.00	--	--	--	0.18	--
2272	--	--	--	--	--	--	--
2275	--	--	--	--	--	--	--
2284	--	--	--	--	--	--	--
2285	--	--	--	--	--	--	--
2293	--	--	--	--	0.329	--	--
2295	--	--	--	0.96	--	--	--
2303	--	--	--	0.9	--	--	--
2310	--	--	--	--	--	--	--
2311	--	--	--	--	--	--	--
2352	--	--	--	--	--	--	--
2357	--	--	--	--	--	--	--
2358	--	--	--	--	--	--	--
2363	--	--	--	--	--	--	--
2365	--	--	--	--	--	--	--
2366	--	--	--	--	--	--	--
2370	--	--	--	--	--	--	--
2372	--	--	--	--	--	--	--
2375	--	--	--	--	--	--	--
2379	--	0.03	--	0.06	--	0.01	--
2380	--	--	--	--	--	--	--
2385	--	--	--	--	0.28	--	--
2390	--	--	--	--	--	--	--
2410	--	--	--	--	--	--	--
2428	0.47	--	0.23	0.04	0.81	1.40	--
2432	--	--	--	--	--	--	--
2442	--	--	--	--	--	--	--
2452	0.06	0.12	002	0.04	0.31	0.07	0.02
2459	--	--	--	--	--	--	--
2461	--	--	--	--	--	--	--
3117	--	--	--	--	--	--	--
3124	--	--	--	--	--	--	--
3145	--	--	--	--	--	--	--
3153	--	--	--	--	--	--	--
3154	--	--	--	--	--	--	--
3166	--	--	--	--	--	--	--
3172	--	--	--	--	--	--	--
3176	--	--	--	--	--	--	--
3185	--	--	--	--	--	--	--
3190	--	--	--	--	2.2	--	--
3191	--	0.02	--	0.01	0.38	0.05	0.04
3197	--	--	--	--	--	--	--
3200	--	--	--	--	--	--	--
3203	--	0.25	--		0.40	0.11	--
3204	0.016	0.044	--	0.026	0.354	0.022	--
3209	--	--	--	--	--	--	--
3210	--	--	--	--	--	--	--
3214	0.103	--	--	--	1.49	--	--
3218	--	--	--	--	--	--	--
3220	--	--	--	--	--	0.05	--
3222	--	--	0.2	--	--	--	--
3226	--	--	0.009	0.043	--	--	--
3228	--	--	--	--	--	--	--
3237	--	--	--	--	0.34	--	--

Determination of other perspirated metals on sample #11088; results in mg/kg

lab	As	Cd	Cr	Pb	Hg
362	0.042	--	0.011	--	--
551	--	--	--	--	--
1179	--	--	--	--	--
2120	--	--	--	--	--
2129	--	--	--	--	--
2132	--	--	--	--	--
2137	--	--	--	--	--
2165	--	--	--	--	--
2172	--	--	--	--	--
2180	--	--	--	--	--
2181	--	--	--	1.1562	--
2184	0.09	--	--	--	--
2196	--	--	--	--	--
2201	--	--	--	--	--
2247	--	--	--	--	--
2256	0.87	--	--	--	--
2265		--	--	0.16	--
2272		--	--	--	--
2275		--	--	--	--
2284		--	--	--	--
2285		--	--	--	--
2293		--	1.753	--	--
2295		--	--	--	--
2303		--	--	--	1.1
2310		--	--	--	--
2311		--	--	--	--
2352		--	--	--	--
2357		--	--	--	--
2358		--	--	--	--
2363		--	--	--	--
2365		--	--	--	--
2366		--	--	--	--
2370		--	--	--	--
2372		--	--	--	--
2375		--	--	--	--
2379	1.52	0.01	--	--	0.02
2380	--	--	--	--	--
2385	--	--	--	--	--
2390	--	--	--	--	--
2410	--	--	--	--	--
2428	--	--	0.10	0.13	--
2432	--	--	--	--	--
2442	--	--	--	--	--
2452	0.06	0.02	0.08	0.07	0.02
2459	--	--	--	--	--
2461	--	--	--	--	--
3117	--	--	--	--	--
3124	--	--	--	--	--
3145	--	--	--	--	--
3153	--	--	--	--	--
3154	--	--	--	--	--
3166	--	--	--	--	--
3172	--	--	--	--	--
3176	--	--	--	--	--
3185	--	--	--	--	--
3190	--	--	--	--	--
3191	0.07	0.01	--	--	0.03
3197	--	--	--	--	--
3200	--	--	--	--	--
3203	--	--	--	--	--
3204	0.0648	0.0064	0.0363	0.0162	0.0022
3209	--	--	--	--	--
3210	--	--	2.01	--	--
3214	3.19	--	--	--	--
3218	--	--	--	--	--
3220	--	--	0.6	--	0.03
3222	--	0.3	--	--	--
3226	--	0.016	--	--	--
3228	--	--	--	--	--
3237	--	--	--	--	--

APPENDIX 2

Analytical details of determination

lab	Methods used	Quantification technique	remark
362	RSTS-SL-103-1	ICP-OES	
551	--	--	
1179	DIN 54233-3:2010	ICP-MS	
2120	DIN 54233	ICP-MS	
2129	ISO 105-E04, ISO 17294-2		
2132	In house	ICP-OES	
2137	ISO 105-E04	ICP	
2165	DIN 54233	ICP-MS	
2172	ISO 105-E04, ISO 17294-2	ICP-MS	
2180	DIN 54233-3	ICP, G-AAS, CV-AAS	
2181	DIN 54233-3	Flame-AAS, CV-AAS	
2184	DIN 54233	ICP-MS	
2196	DIN 54233-3:2010	ICP-OES	
2201	DIN 54233	ICP-MS	
2247	ISO 105-E04	ICP	
2256	ISO 105-E04	ICP-MS	
2265	DIN 54233-3	AAS	
2272	ISO 17071-1:2011	ICP-OES	
2275	DIN 54233-3	ICP	
2284	DIN 54233	ICP	
2285	DIN 54233-3	ICP-MS	
2293	ISO 105-E04	ICP-OES	
2295	In house	ICP-MS	
2303	In house		
2310	ISO 105-E04	ICP-MS	
2311	ISO 105-E04	ICP, AAS	
2352	ISO 105-E04	ICP-OES, AAS	
2357	ISO 105-E04	ICP, GF-AAS, AFS	
2358	ISO 105-E04	ICP-MS	
2363	ISO 105-E04	ICP-MS	
2365	ISO 105-E04	ICP-MS	
2366	--	--	
2370	ISO 105-E04	ICP-OES	
2372	ISO 105-E04	ICP	
2375	RSTS-SL-103-1	ICP-MS	
2379	ISO 105-E04	ICP	
2380	ISO 105-E04	ICP-OES	
2385	DIN 54233-3	ICP-MS	
2390	ISO 105-E04, DIN 5402	ICP-OES	
2410	ISO 105-E04	ICP-MS	
2428	--	--	
2432	In house	ICP-MS	
2442	In house	ICP	
2452	DIN 54233-3	AAS	
2459	ISO 105-E04	AAS	
2461	GB-T17593.1/4-2006	AAS, AFS	
3117	ISO 105-E04	ICP-OES, AFS	
3124	--	--	
3145	In house	AAS, ICP-OES	
3153	ISO 105-E04	ICP-MS	
3154	ISO 17072-1	ICP-OES	
3166	--	--	
3172	--	--	
3176	DIN 54233-3	ICP-MS	
3185	ISO 105-E04	ICP-MS	
3190	ISO 105-E04	ICP-OES	
3191	ISO 105-E04	ICP-MS	
3197	ISO 105-E04	AAS	
3200	In house	ICP-OES, ICP-MS	
3203	DIN 54233	ICP	
3204	DIN 54233-3:2010	ICP-MS	
3209	In house	ICP-OES, ICP-MS	
3210	ISO 17072-1	ICP-MS	
3214	DIN 54233-3	ICP-MS	
3218	DIN 54233-3	ICP	
3220	ISO 105-E04	ICP-MS	
3222	In house	AAS	
3226	DIN 54020	ICP-MS	
3228	ISO 105-E04	ICP-MS	
3237	In house	ICP-MS	

APPENDIX 3

Number of participants per country:

2 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	DENMARK
1 lab in	FRANCE
6 labs in	GERMANY
1 lab in	GUATEMALA
5 labs in	HONG KONG
4 labs in	INDIA
3 labs in	ITALY
2 labs in	KOREA
25 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	POLAND
1 lab in	PORTUGAL
1 lab in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
5 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM

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
n.a.	= not applicable
n.d.	= not detected

Literature:

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