Results of Proficiency Test
Perspirated Metals in textile
October 2010

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), Öko-Tex Standard 100 (Germany) and Thai Green Label (Thailand).
Since several years the Institute for Interlaboratory Studies (iis) organises a proficiency scheme for perspirated metals in textile. Also this year this scheme is part of the proficiency testing program 2010/2011.
In this interlaboratory study 65 laboratories in 22 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 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, a orange fabric (sample #1076) and a pink fabric (sample #1077) were each well mixed and divided over 84 subsamples of approx. 3.5 grams. The samples were labelled and tested for homogeneity on 5 randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17025 accredited laboratory.

See the following tables for the homogeneity test results.

Orange fabric	*Perspirated Cobalt in mg/kg*
Sample #1076-1	39.5
Sample #1076-2	40.0
Sample #1076-3	39.5
Sample #1076-4	39.8
Sample #1076-5	40.1

table 1: homogeneity test of subsamples #1076

Pink fabric	*Perspirated Nickel in mg/kg*
Sample #1077-1	84.1
Sample #1077-2	86.0
Sample #1077-3	82.7
Sample #1077-4	83.5
Sample #1077-5	84.4

table 2: homogeneity test of subsamples #1077

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 Cobalt in #1076	*Perspirated Nickel in #1077*
r (observed)	0.8	3.4
reference method	Horwitz	Horwitz
0.3 x R (reference method)	3.1	5.8

Table 3: : repeatabilities of subsamples #1076 and #1077

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 #1076 and #1077 were sent to the participating laboratories on October 7, 2010.

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 as prescribed in E DIN 54233-3:2010. 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.

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 3; nr.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.

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, some problems occurred with sample dispatch and with the reporting of the test results. Two laboratories decided not to report any test results due to various reasons and one laboratory did withdraw its test results because it did use test method EN71-3, thus finding incomparable test results.
Four laboratories reported test results after the final reporting date.
Finally, the 62 reporting laboratories did report in total 194 numerical results. The vast majority of the participating laboratories did use a solid/liquid ratio of 1/50 as requested. Only two laboratories used (also) different solid/liquid ratios, being 1/10 and 1/20 g/mL.

Observed were 14 statistical outlying results, which is 7.2% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal distribution.

Most 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 DIN 54233-3, 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.
Only for Cadmium and Cobalt in sample #1076 and Nickel in sample #1077 a sufficient number of numerical results were reported in order to perform a statistical evaluation.
For all other elements of both samples, no significant conclusions could be drawn as only a few participants reported a numerical result, which were probably near or below the detection limit of the methods used. All other participants reported “not detected” in these cases.

Cadmium: The determination of this metal was problematic for sample #1076 at a perspiration level of 10.3 mg/kg.
Six statistical outliers were observed in the test results, determined with a s/l ratio of 1/50.
The observed reproducibility is, even after rejection of the six statistical outliers, not in agreement with the estimated target reproducibility calculated using the Horwitz equation.

Cobalt: The determination of this metal was only problematic for a number of participating laboratories for sample #1076 at a perspiration level of 11.8 mg/kg. Three statistical outliers were observed in the test results, determined with a s/l ratio of 1/50.
The observed reproducibility is, after rejection of the three statistical outliers, in full 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 #1077 at a perspiration level of 26.94 mg/kg. Five statistical outliers were observed in the test results, determined with a s/l ratio of 1/50.
The observed reproducibility is, after rejection of the five statistical outliers, in full 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 very 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 cadmium	mg/kg	55	10.29	4.09	3.25
Perspirated Cobalt	mg/kg	58	11.79	3.61	3.64

table 4: reproducibilities of sample #1076

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Perspirated Nickel	mg/kg	56	26.94	5.24	7.35

table 5: reproducibilities of sample #1077

From the above tables it can be concluded that, without statistical calculations, the group of participating laboratories have little 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 2010*	*October 2009*	*November 2008*	*November 2007*	*November 2006*	*November 2005*	*November 2004*
Arsenic	--	--	--	66%	--	--	62%
Antimony	--	41%	46%	--	--	--	--
Cadmium	40%	--	--	--	85%	57%	--
Chromium	--	--	--	114%	47%	83%	--
Cobalt	31%	--	28%	--	--	50%	--
Copper	--	48-46%	26%	64-80%	93-39	66%	71%
Lead	--	--	--	375%	138-172%	--	245%
Mercury	--	--	--	--	134%	--	--
Nickel	19%	--	23%	41%	--	88-39%	28%

table 6: Comparison of observed relative reproducibilities (since 2008 s/l ratio prescribed on 1:50)

The improvement of the results in the last three 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).

In this proficiency test for Perspiration of Heavy Metals in Textile, the samples from proficiency tests November 2005 (iis05A03) were reused. An overview of the differences in results is summarized in table 7 and 8.

		iis10A05			iis05A03		
Parameter	Unit	n	average	2.8 * sd	n	average	2.8 * sd
Perspirated Cadmium #1076	mg/kg	55	10.29	4.09	32	9.94	5.67
Perspirated Cobalt #1076	mg/kg	58	11.79	3.61	31	11.45	5.69

table 7: comparison results of PT iis10A05 and iis05A03

		iis10A05			iis05A03		
Parameter	Unit	n	average	2.8 * sd	n	average	2.8 * sd
Perspirated Nickel #1077	mg/kg	56	26.94	5.24	33	27.08	10.54

table 8: comparison results of PT iis10A05 and iis05A03

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 (Detection limit is 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 9: 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

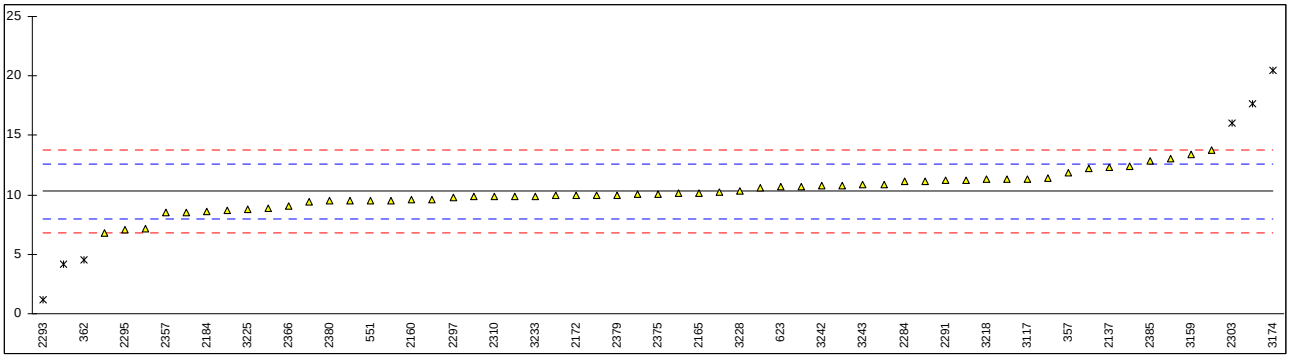
The spreads observed in this interlaboratory study are not caused by just one critical point in the analysis. Consequently, the reproducibilities cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective 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 Cadmium (s/l ratio = 1/50) on sample #1076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
357	ISO105-E04	11.9		1.39	
362	RSTS-SL-103-1	4.5	DG(0.01)	-5.00	
551	in house	9.53		-0.66	
623	ISO105-04	10.69		0.34	
840	ISO105-E04	9.54		-0.65	
1179		-----		-----	
2120	OEKO-TEX	10.1		-0.16	
2129	ISO105-E04/EN17294-2	12.4		1.82	
2132	ISO105-E04	8.84		-1.25	
2137	ISO105-E04	12.3		1.73	
2160	in house	9.58		-0.61	
2165	in house	10.1		-0.16	
2172	ISO105-E04/EN17294-2	10.0		-0.25	
2182	ISO105-E04/DIN5402	8.7		-1.37	
2184	ISO105-E04	8.6		-1.46	
2196	OEKO-TEX	6.8		-3.01	
2215	OEKO-TEX	11.1		0.70	
2224		-----		-----	reported 7.0 mg/kg for s/l=1/20 (z=-2.84)
2241	OEKO-TEX	11.4		0.96	
2265	DIN54233-3	7.18		-2.68	
2271	in house	10.6		0.27	
2284	ISO105-E04	11.1		0.70	
2290	EN 71-3	-----	W	-----	result was withdrawn as used test method is not comparable
2291	OEKO-TEX	11.20		0.78	
2293	DIN54020	1.2	G(0.05)	-7.84	
2295	Extraction	7.1		-2.75	
2297	ISO105-E04	9.8		-0.42	
2303	in house	16.0	G(0.05)	4.92	
2310	ISO105-E04	9.86		-0.37	
2311	ISO105-E04	10.02		-0.23	
2352	ISO105-E04	9.6		-0.60	
2357	ISO105-E04	8.5		-1.54	
2358	OEKO-TEX	10.0		-0.25	
2363	ISO105-E04	10.2		-0.08	
2365	ISO105-E04	8.51		-1.54	
2366	ISO105-E04	9.1		-1.03	
2370	ISO105-E04	9.49		-0.69	
2372	ISO105-E04	9.38		-0.79	
2375	ISO105-E04	10.06		-0.20	
2379	ISO105-E04	10.00		-0.25	
2380	ISO105-E04	9.47		-0.71	
2385	OEKO-TEX	12.9		2.25	reported also 9.3 mg/kg for s/l=1/10 (z=-0.85)
3117	OEKO-TEX	11.35		0.91	
3124	ISO105-E04	13.8		3.03	
3146	in house	10.7		0.35	
3153	ISO105-E04	9.9		-0.34	
3154	in house	9.95		-0.29	
3159	ISO105-E04	13.40		2.68	
3170	in house	17.66	G(0.05)	6.36	
3172		-----		-----	
3174	ISO105-E04	20.46	G(0.01)	8.77	
3176	DIN54020/ISO17294	12.19		1.64	
3180	in house	13.0		2.34	
3185	in house	10.9		0.53	
3190	OEKO-TEX	11.24		0.82	
3200	in house	10.8		0.44	
3209	DIN54020	9.86		-0.37	
3210	in house	4.2	DG(0.01)	-5.25	
3218	ISO105-E04	11.3		0.87	
3225	in house	8.8		-1.29	
3228	in house	10.3		0.01	
3233	OEKO-TEX	9.9		-0.34	
3237	in house	11.3		0.87	
3242	ISO105-E04	10.77		0.41	
3243	DIN54020	10.9		0.53	

normality	OK
n	55
outliers	6
mean (n)	10.291
st.dev. (n)	1.4607
R(calc.)	4.090
R(Horwitz)	3.246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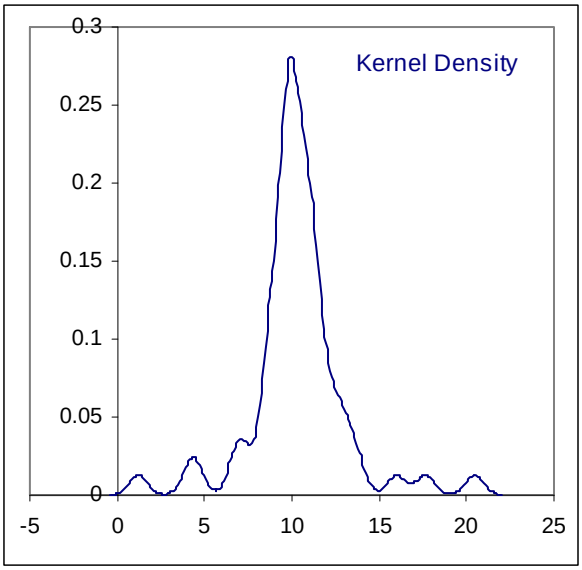
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-5
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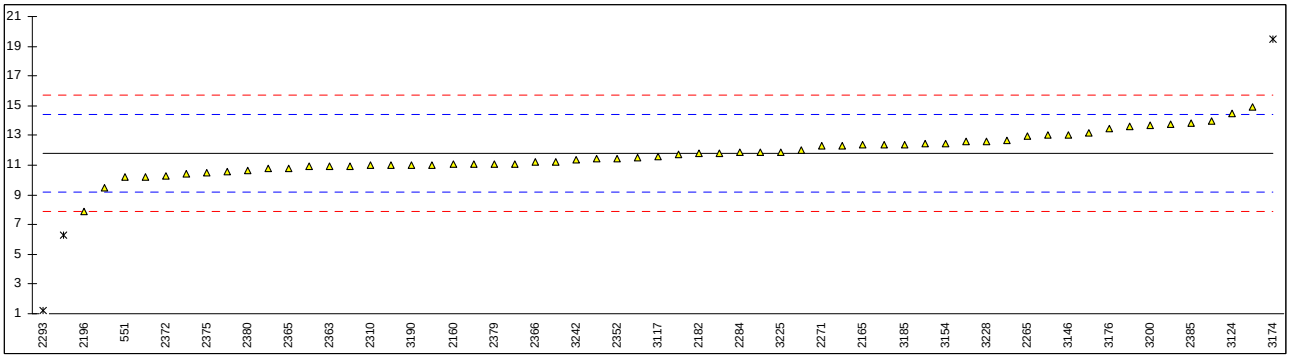
5
10
15
20
25

Determination of perspirated Cobalt (s/l ratio = 1/50) on sample #1076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
357	ISO105-E04	13.0		0.93	
362	RSTS-SL-103-1	10.9		-0.69	
551	in house	10.17		-1.25	
623	ISO105-04	10.19		-1.23	
840	ISO105-E04	11.7		-0.07	
1179		-----		-----	
2120	OEKO-TEX	10.4		-1.07	
2129	ISO105-E04/EN17294-2	13.2		1.08	
2132	ISO105-E04	12.42		0.48	
2137	ISO105-E04	9.5		-1.76	
2160	in house	11.09		-0.54	
2165	in house	12.4		0.47	
2172	ISO105-E04/EN17294-2	10.6		-0.92	
2182	ISO105-E04/DIN5402	11.8		0.01	
2184	ISO105-E04	12.3		0.39	
2196	OEKO-TEX	7.9		-2.99	
2215	OEKO-TEX	11.1		-0.53	
2224		-----		-----	reported 11.9 mg/kg for s/l=1/20 (z=+0.08)
2241	OEKO-TEX	12.7		0.70	
2265	DIN54233-3	12.97		0.90	
2271	in house	12.3		0.39	
2284	ISO105-E04	11.9		0.08	
2290	EN 71-3	-----	W	-----	result was withdrawn as used test method is not comparable
2291	OEKO-TEX	11.50		-0.22	
2293	DIN54020	1.2	G(0.01)	-8.14	
2295	Extraction	12		0.16	
2297	ISO105-E04	11.1		-0.53	
2303	in house	14.0		1.70	
2310	ISO105-E04	10.98		-0.62	
2311	ISO105-E04	11.0		-0.61	
2352	ISO105-E04	11.4		-0.30	
2357	ISO105-E04	10.8		-0.76	
2358	OEKO-TEX	11.4		-0.30	
2363	ISO105-E04	10.9		-0.69	
2365	ISO105-E04	10.8		-0.76	
2366	ISO105-E04	11.2		-0.46	
2370	ISO105-E04	10.9		-0.69	
2372	ISO105-E04	10.3		-1.15	
2375	ISO105-E04	10.50		-0.99	
2379	ISO105-E04	11.10		-0.53	
2380	ISO105-E04	10.65		-0.88	
2385	OEKO-TEX	13.8		1.54	reported also 11.9 mg/kg for s/l=1/10 (z=+0.08)
3117	OEKO-TEX	11.59		-0.16	
3124	ISO105-E04	14.5		2.08	
3146	in house	13.0		0.93	
3153	ISO105-E04	11.2		-0.46	
3154	in house	12.45		0.51	
3159	ISO105-E04	13.77		1.52	
3170	in house	13.58		1.37	
3172		-----		-----	
3174	ISO105-E04	19.45	G(0.05)	5.88	
3176	DIN54020/ISO17294	13.49		1.30	
3180	in house	11.0		-0.61	
3185	in house	12.4		0.47	
3190	OEKO-TEX	11.0		-0.61	
3200	in house	13.7		1.47	
3209	DIN54020	12.57		0.60	
3210	in house	6.3	G(0.01)	-4.22	
3218	ISO105-E04	11.9		0.08	
3225	in house	11.9		0.08	
3228	in house	12.6		0.62	
3233	OEKO-TEX	14.9		2.39	
3237	in house	11.8		0.01	
3242	ISO105-E04	11.35		-0.34	
3243	DIN54020	12.4		0.47	

normality	OK
n	58
outliers	3
mean (n)	11.793
st.dev. (n)	1.2893
R(calc.)	3.610
R(Horwitz)	3.644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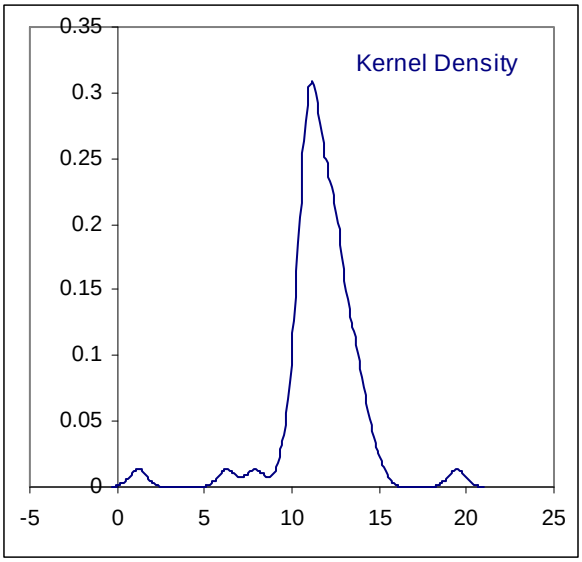
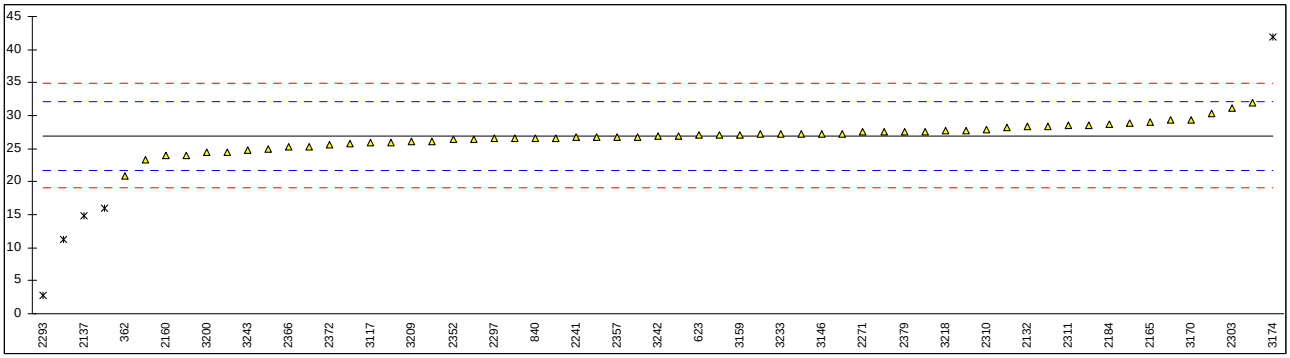
2379
2366
3242
2352
3117
2182
2284
3225
2271
2165
3185
3154
3228
2265
3146
3176
3200
2385
3124
3174

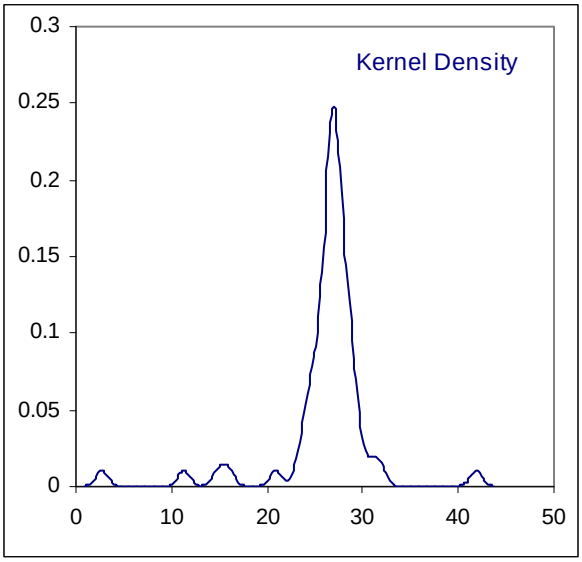

Kernel Density
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-5
0
5
10
15
20
25

Determination of perspirated Nickel (s/l ratio = 1/50) on sample #1077; results in mg/kg

lab	method	value	mark	z(targ)	remarks
357	ISO105-E04	28.8		0.71	
362	RSTS-SL-103-1	20.9		-2.30	
551	in house	24.53		-0.92	
623	ISO105-04	27.06		0.05	
840	ISO105-E04	26.6		-0.13	
1179		-----		-----	
2120	OEKO-TEX	27.2		0.10	
2129	ISO105-E04/EN17294-2	26.1		-0.32	
2132	ISO105-E04	28.37		0.55	
2137	ISO105-E04	14.9	C,G(0.01)	-4.58	first reported 21.0 mg/kg
2160	in house	23.96		-1.13	
2165	in house	29.0		0.79	
2172	ISO105-E04/EN17294-2	27.6		0.25	
2182	ISO105-E04/DIN5402	28.4		0.56	
2184	ISO105-E04	28.7		0.67	
2196	OEKO-TEX	23.3		-1.39	
2215	OEKO-TEX	26.8		-0.05	
2224		-----		-----	reported 29.3 mg/kg for s/l=1/20 (z=+0.90)
2241	OEKO-TEX	26.7		-0.09	
2265	DIN54233-3	11.31	G(0.01)	-5.95	
2271	in house	27.5		0.21	
2284	ISO105-E04	27.5		0.21	
2290	EN 71-3	-----	W	-----	result was withdrawn as used test method is not comparable
2291	OEKO-TEX	24.00		-1.12	
2293	DIN54020	2.7	G(0.01)	-9.23	
2295	Extraction	29.3		0.90	
2297	ISO105-E04	26.5		-0.17	
2303	in house	31.2		1.62	
2310	ISO105-E04	27.95		0.39	
2311	ISO105-E04	28.47		0.58	
2352	ISO105-E04	26.4		-0.20	
2357	ISO105-E04	26.8		-0.05	
2358	OEKO-TEX	25.8		-0.43	
2363	ISO105-E04	26.8		-0.05	
2365	ISO105-E04	26.4		-0.20	
2366	ISO105-E04	25.2		-0.66	
2370	ISO105-E04	26.0		-0.36	
2372	ISO105-E04	25.6		-0.51	
2375	ISO105-E04	27.72		0.30	
2379	ISO105-E04	27.56		0.24	
2380	ISO105-E04	27.15		0.08	
2385	OEKO-TEX	27.3		0.14	reported also 29.2 mg/kg for s/l=1/10 (z=+0.86)
3117	OEKO-TEX	25.94		-0.38	
3124	ISO105-E04	27.1		0.06	
3146	in house	27.3		0.14	
3153	ISO105-E04	26.9		-0.01	
3154	in house	26.64		-0.11	
3159	ISO105-E04	27.14		0.08	
3170	in house	29.31		0.90	
3172		-----		-----	
3174	ISO105-E04	41.89	G(0.01)	5.70	
3176	DIN54020/ISO17294	31.96	C	1.91	first reported 34.59 mg/kg
3180	in house	25.0		-0.74	
3185	in house	28.6		0.63	
3190	OEKO-TEX	25.3		-0.62	
3200	in house	24.5		-0.93	
3209	DIN54020	26.01		-0.35	
3210	in house	16.0	G(0.01)	-4.17	
3218	ISO105-E04	27.7		0.29	
3225	in house	26.5		-0.17	
3228	in house	28.2		0.48	
3233	OEKO-TEX	27.2		0.10	
3237	in house	30.3		1.28	
3242	ISO105-E04	26.83		-0.04	
3243	DIN54020	24.84		-0.80	

normality	OK
n	56
outliers	5
mean (n)	26.936
st.dev. (n)	1.8704
R(calc.)	5.237
R(Horwitz)	7.351


45
40
35
30
25
20
15
10
5
0
2293
2137
362
2160
3200
3243
2366
2372
3117
3209
2352
2297
840
2241
2357
3242
623
3159
3233
3146
2271
2379
3218
2310
2132
2311
2184
2165
3170
2303
3174


Kernel Density
0.3
0.25
0.2
0.15
0.1
0.05
0
0
10
20
30
40
50

Determination of other perspirated metals on sample #1076; results in mg/kg

lab	As	Sb	Cr	Cu	Pb	Hg	Ni
357	<2	<2	<2	<2	<2	<0.5	<2
362	<0.05	<0.05	0.07	0.42	<0.05	<0.05	0.11
551	-----	-----	-----	-----	-----	-----	-----
623	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
1179	-----	-----	-----	-----	-----	-----	-----
2120	<0.05	<12.5	<0.5	<12.5	<0.05	-----	<0.5
2129	<0.1	<0.2	<0.2	<5.0	<0.1	<0.01	<0.4
2132	<2.5	<2.5	<2.0	<2.0	<3.0	<1.0	<2.0
2137	-----	-----	-----	-----	-----	-----	-----
2160	-----	-----	<0.5	<0.5	<0.5	<0.02	<0.5
2165	-----	-----	-----	-----	-----	-----	-----
2172	<0.02	<0.5	<0.1	<5	<0.1	<0.005	<0.1
2182	-----	0.3	-----	0.7	-----	-----	-----
2184	<0.02	<0.5	<0.5	<0.5	<0.02	<0.02	<0.5
2196	<0.5	<0.5	<0.5	1.7	<0.5	<0.5	<0.5
2215	<0.1	<1.0	<0.1	<5.0	<0.1	<0.02	<0.1
2224	<0.2	<5	<1	<5	<0.2	<0.02	<1
2241	-----	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----	0.49
2271	<1.0	<2.5	<1.0	<2.0	<1.0	<0.05	<1.0
2284	-----	-----	-----	-----	-----	-----	-----
2290	-----	-----	-----	-----	-----	-----	-----
2291	<0.20	<3.00	<0.50	1.45	<0.20	<0.01	<1.00
2293	-----	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----	-----
2297	-----	-----	-----	-----	-----	-----	-----
2303	0.6	0.8	0.5	1.6	0.6	3.3	0.6
2310	-----	-----	-----	-----	-----	-----	-----
2311	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----
2358	-----	-----	-----	-----	-----	-----	-----
2363	-----	-----	-----	-----	-----	-----	-----
2365	-----	-----	-----	-----	-----	-----	-----
2366	-----	-----	-----	-----	-----	-----	-----
2370	-----	-----	-----	-----	-----	-----	-----
2372	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2379	-----	-----	-----	-----	-----	-----	-----
2380	-----	-----	-----	-----	-----	-----	-----
2385	<0.1	<0.1	<0.1	0.4	<0.1	<0.01	<0.4
3117	-----	-----	-----	-----	-----	-----	-----
3124	-----	-----	-----	-----	-----	-----	-----
3146	<0.2	<1	<0.5	<1	<0.2	<0.02	<0.5
3153	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----
3159	-----	-----	-----	-----	-----	-----	-----
3170	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----
3174	-----	-----	0.02	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----
3180	-----	-----	-----	-----	-----	-----	-----
3185	-----	-----	-----	-----	-----	-----	-----
3190	-----	-----	-----	-----	-----	-----	-----
3200	-----	-----	-----	-----	-----	-----	-----
3209	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----
3218	-----	-----	-----	-----	-----	-----	-----
3225	<2.5	<2.5	<2.0	<2.0	<3.0	<0.05	<2.0
3228	-----	-----	-----	-----	-----	-----	-----
3233	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----
3242	-----	-----	-----	-----	-----	-----	-----
3243	0.050	0.023	0.078	0.224	0.015	0.010	0.636

Determination of other perspirated metals on sample #1077; results in mg/kg

lab	As	Sb	Cd	Cr	Co	Cu	Pb	Hg
357	<2	<2	<2	<2	<2	<2	<2	<0.5
362	<0.05	<0.05	<0.05	0.19	<0.05	0.69	<0.05	<0.05
551	-----	-----	-----	-----	-----	-----	-----	-----
623	-----	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----	-----
1179	-----	-----	-----	-----	-----	-----	-----	-----
2120	<0.05	<12.5	<0.05	<0.5	<0.5	<12.5	<0.05	-----
2129	<0.1	<0.2	<0.05	<0.2	<0.2	<5	<0.1	<0.01
2132	<2.5	<2.5	<0.30	<2.0	<2.0	<2.0	<3.0	<1.0
2137	-----	-----	-----	-----	-----	-----	-----	-----
2160	-----	-----	<0.5	<0.5	<0.5	<0.5	<0.5	<0.02
2165	-----	-----	-----	-----	-----	-----	-----	-----
2172	<0.02	<0.5	<0.02	<0.1	<0.1	<5	<0.1	<0.005
2182	-----	-----	-----	-----	-----	1.0	-----	-----
2184	<0.02	<0.5	<0.02	<0.5	<0.5	<0.5	<0.02	<0.02
2196	<0.5	<0.5	<0.5	<0.5	<0.5	1.9	<0.5	<0.5
2215	<0.1	<1.0	<0.1	<0.1	<0.1	<5.0	<0.1	<0.02
2224	<0.2	<5	<1	<1	<1	<5	<0.2	<0.02
2241	-----	-----	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----	-----	-----
2271	<1.0	<2.5	<0.3	<1.0	<1.0	<2.0	<1.0	<0.05
2284	-----	-----	-----	-----	-----	-----	-----	-----
2290	-----	-----	-----	-----	-----	-----	-----	-----
2291	<0.20	<3.00	<0.10	<0.50	<1.00	1.60	<0.20	<0.01
2293	-----	-----	-----	-----	-----	0.1	-----	-----
2295	-----	-----	-----	-----	-----	-----	-----	-----
2297	-----	-----	-----	-----	-----	-----	-----	-----
2303	0.5	0.6	0.5	0.6	0.5	2.6	0.5	0.9
2310	-----	-----	-----	-----	-----	-----	-----	-----
2311	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	-----	-----	-----	-----	-----	-----	-----	-----
2363	-----	-----	-----	-----	-----	-----	-----	-----
2365	-----	-----	-----	-----	-----	-----	-----	-----
2366	-----	-----	-----	-----	-----	-----	-----	-----
2370	-----	-----	-----	-----	-----	-----	-----	-----
2372	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----
2379	-----	-----	-----	-----	-----	-----	-----	-----
2380	-----	-----	-----	-----	-----	-----	-----	-----
2385	<0.1	<0.1	<0.05	<0.1	<0.1	0.7	<0.1	<0.01
3117	-----	-----	-----	-----	-----	-----	-----	-----
3124	-----	-----	-----	-----	-----	-----	-----	-----
3146	<0.2	<1	<0.1	<0.5	<0.5	<1	<0.2	<0.02
3153	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3159	-----	-----	-----	-----	-----	-----	-----	-----
3170	-----	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----	-----
3174	-----	-----	-----	0.05	0.02	0.14	-----	-----
3176	-----	-----	-----	0.62	0.15	-----	0.15	-----
3180	-----	-----	-----	-----	-----	-----	-----	-----
3185	-----	-----	-----	-----	-----	-----	-----	-----
3190	-----	-----	-----	-----	-----	1.2	-----	-----
3200	-----	-----	-----	-----	-----	-----	-----	-----
3209	-----	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----	-----
3218	-----	-----	-----	-----	-----	-----	-----	-----
3225	<2.5	<2.5	<0.3	<2.0	<2.0	<2.0	<3.0	<0.05
3228	-----	-----	-----	-----	-----	-----	-----	-----
3233	-----	-----	-----	-----	0.1	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----
3242	-----	-----	-----	-----	-----	-----	-----	-----
3243	0.050	0.025	0.024	0.029	0.048	0.420	0.025	0.010

APPENDIX 2

Number of participants in alphabetic country order:

1 lab in	BANGLADESH
1 lab in	BRASIL
1 lab in	BULGARIA
1 lab in	DENMARK
1 lab in	FINLAND
2 labs in	FRANCE
6 labs in	GERMANY
1 lab in	GUATEMALA
7 labs in	HONG KONG
4 labs in	INDIA
1 lab in	INDONESIA
1 lab in	ITALY
1 lab in	KOREA
22 labs in	P.R. of CHINA
1 lab in	PORTUGAL
1 lab in	SWITZERLAND
2 labs in	TAIWAN R.O.C.
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
5 labs in	TURKEY
1 lab in	UNITED KINGDOM
2 labs in	VIETNAM

APPENDIX 3

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
n.r.	= not reported

Literature:

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2 Öko-Tex Standard 100; January 2008
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9 ISO 5725. parts 1-6. (1994)
10 ISO105 E4: 1994
11 ISO14184-1: 1994
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13 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
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15 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/).
16 Official Journal of the European Communities L133/29 : May 2002
17 E DIN 54233-3:2010