Results of Proficiency Test
Nickel Release
May 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Nickel has always been used in various applications, as a pure metal, as a plated substance on another metal or as an alloy. Nickel applications usually do not give problems, but when nickel comes into prolonged and direct contact with the human skin, sensitization can occur. When a person becomes sensitive to nickel, even the smallest amounts can provoke an allergic reaction. By this, nickel is the most frequent cause of contact allergy in Europe. Both the contact itself (sometimes enhanced by damaged skin) and skin conditions as sweat can cause the body to be exposed by nickel. In order to decrease the amount of people that become sensitized, nickel containing items that are used in prolonged human contact are tested for nickel release. These products involve products like jewellery in piercings (ear rings), other jewellery, watches or clothes fasteners, such as buttons and belts.

On request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for the determination of nickel release in the 2013-2014 PT program. In the interlaboratory study of May 2014, 112 laboratories from 26 different countries have participated (see appendix 4). 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 organizer of this proficiency test. It was decided to send 2 different samples (labelled #14075 and #14076), both positive on nickel release. The analyses for fit-for-use and for homogeneity testing were subcontracted.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation. Also an inventory was made of the analytical details of the used test method, by means of a questionnaire, which was included in the report form.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. 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 a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed during the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two samples were used in this proficiency test, both positive on nickel release.

The first sample consisted of square metal pieces (#14075), which were purchased from a local supplier. Eight stratified randomly selected samples were tested using EN1811:2011 to check the homogeneity of the batch.
The dimensions of each sample were approximately (l x w x h): 1.000 x 1.015 x 0.210 cm.

The second batch of samples of an artificial alloy of copper and nickel was prepared and tested for homogeneity by Mrs. Xu FeiFei of the Zhejiang Academy of Science and Technology for Inspection and Quarantine, located in Zhejiang District, P. R. of China.
This batch consisted of thin circular metal disks (#14076). Ten stratified randomly selected samples were tested using EN1811:2011 to check the homogeneity of the batch.
The dimensions of each disk were approximately: a diameter of 1.94 cm and a thickness of 0.12 cm.

The test results of the homogeneity tests are shown in table 1.

Nickel release	*in #14075 (µg/cm^2/week)*	*in #14076 (µg/cm^2/week)*
sample 1	0.440	15.237
sample 2	0.491	14.963
sample 3	0.529	14.607
sample 4	0.512	16.700
sample 5	0.511	15.077
sample 6	0.403	14.200
sample 7	0.472	15.770
sample 8	0.511	15.677
sample 9	--	13.343
sample 10	--	14.337

table 1: homogeneity test results of samples #14075 and #14076

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility in agreement with the procedure of ISO13528, Annex B2, in the next table:

Nickel release	*in #14075 (µg/cm^2/week)*	*in #14076 (µg/cm^2/week)*
r (observed)	0.0432	0.9418
reference method	EN1811:2001	EN1811:2001
0.3 x R (reference method)	0.0483	1.4976

table 2: evaluation of the repeatability of samples #14075 and #14076

The repeatabilities of the results of the homogeneity tests for nickel release of sample #14075 and sample #14076 were in agreement with 0.3 times the reproducibility mentioned in the reference method EN1811:2011. Therefore, homogeneity of the subsamples was assumed for both sample batches.

Three plates of sample #14075 and one plate of sample #14076 were sent to each of the participating laboratories on May 14, 2014.

2.5 ANALYSES

The participants were requested to determine the nickel release of two different materials, applying the analysis procedure that is routinely used in the laboratory. To get comparable results reported, a detailed report form was sent together with the samples. The report form included a questionnaire about the test performance, in order to identify, if possible, analytical details that might have influence on the results of the test.
Also a letter of instructions was sent with the samples.

3 RESULTS

During the four weeks after sample despatch, the test 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) 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 5.

3.1 STATISTICS

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

According to ISO 5725 (1986 and 1994, lit. 7 and 8) the original results per determination were submitted subsequently to Dixon’s, Grubbs' and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner General ESD test (ref. 13). Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner General ESD test (ref. 13). 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 is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 5, no 11). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This 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 $z_{(target)}$-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 no significant problems were encountered. From the 112 participants, 22 participants reported results after the deadline for reporting and one participant did not report any test results at all. Finally, the 111 reporting laboratories submitted 222 numerical results. Observed were 4 outlying results, which is 1.8%. 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, see also paragraph 3.1.

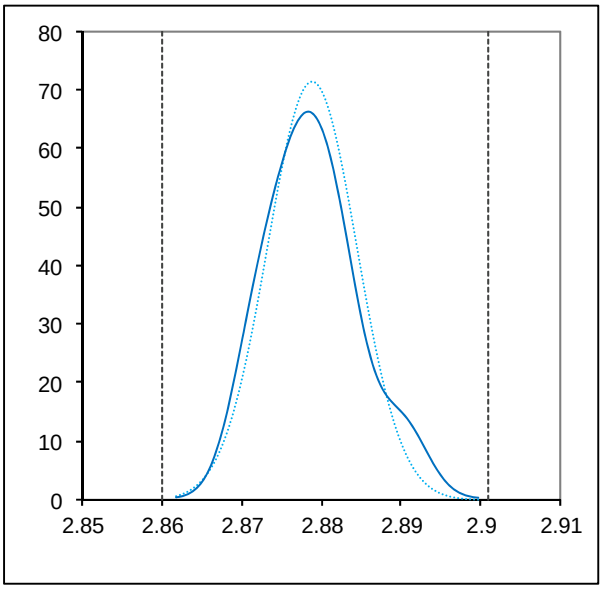
The reproducibility used in this PT report was taken from Annex B of the test method EN1811:2011. It states: the relative method reproducibility in this ILC was 33.3%.

4.1 ANALYTICAL AND METHOD DETAILS FROM THE REPORT FORM QUESTIONNAIRE

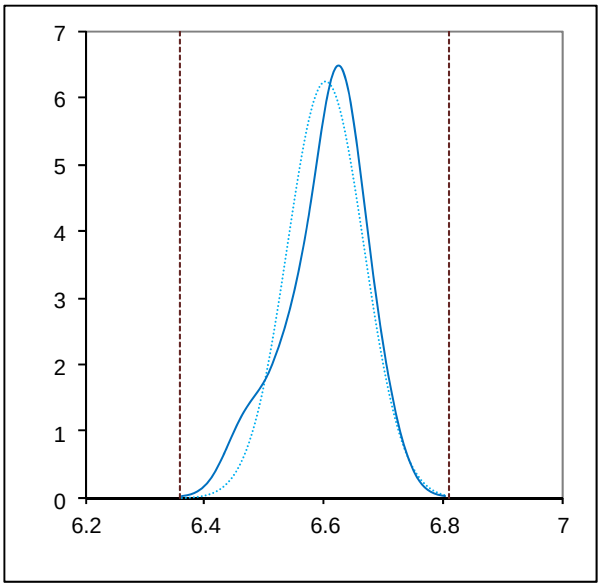
All data involved in this paragraph has been summarized in Appendix 2 and 3. On the report form the test results of the various analytical steps could be reported, like the initial volume used of the release solution, the area used and the nickel concentration in the release solution after one week. Unfortunately, the final volume of the test solution (or the release solution

diluted with dilute nitric acid before measurement) was not requested. Also included in the report form was a questionnaire about the performance of the method.

Analytical result: Calculation of surface area:
Ten random samples of the batch were measured to determine the average area of the plates. These values were plotted as a normal distribution. From this normal distribution curve, limits could be determined as to the area value that would belong to the normal distributions (see figures below).



#14075 acceptable area range: 2.86-2.90 cm^2



#14076 acceptable area range: 6.35-6.81 cm^2

The average surface area for sample #14075 is 2.88 ± 0.02 cm^2 and for sample #14076 it is 6.60 ± 0.20 cm^2.

A large part of the laboratories did not report an area within the limits mentioned above. For sample #14075 around 80% reported an area outside these limits. Around around 25% of the participants reported an area of 2.8 cm^2, which would be the area if the plates were measured with a ruler instead of a sliding calliper: 1 x 1 x 0.2 cm.
For sample #14076 around 20% reported an area outside of the limits for this sample.

For both samples some participants found a significantly higher area than iis calculated. Also some participants reported a smaller value, this could be due to a wrong measurement or calculation, but the method also allows the use of wax or lacquer to decrease the area exposed to the sweat solution. Some laboratories might have done this.

iis statistically evaluated the values of the area, reported by the participants. Using the standard deviation and average, a relative standard deviation (RSD) could be calculated. The RSD_{area} is 9% for sample #14075 and the RSD_{area} is 10% for sample #14076. This means that the reported values for area show a significant spread.
From the 2014 PT results, the relative standard deviation for nickel release could be calculated. The $RSD_{nickel\ release}$ is 27% for #14075 and the $RSD_{nickel\ release}$ is 31% for #14076.
As these figures show, one third of the spread in nickel release may be caused by the value of the area. Since the area is used to determine the nickel release, it is of the utmost importance that this is measured and calculated correctly.

Analytical result: Volume of test solution (initial):
The method of EN1811:2011 prescribes that the initial test solution used should be 1 ml/cm^2 surface area. Since only one volume of test solution was requested on the report form, the volumes reported by the participants may be the initial or the final volumes. Still it appears that some of the participants may have used a higher ratio than 1. This may also be a cause for a higher spread in the nickel release test results.

Analytical result: Final volume of test solution:
After the sample has been stored in the release solution for a week, the sample is taken out and the solution is diluted with nitric acid solution. The volume of this step was not requested on the report form, but it could be calculated for most participants by iis, using the other test results from the report form. The calculated dilution volumes showed a lot of variation, from 10 or 25 ml to 1000 or 5000 ml. A very high dilution of the test solution may introduce extra spread on the nickel release result. The measurement may become more difficult if a lower concentration is diluted with a high volume.

Method detail: pre-treatment of vessel:
The vessel, used for leaving the sample in the sweat solution for a week, should be pre-treated with dilute nitric acid for 4 hrs. This is done to remove any nickel still present from an earlier test. About half of the participants reported to have done this pre-treatment. Some stored the vessels in dilute nitric acid. Some only flushed the vessel or used distilled water, soap or sweat solution to clean it. When no pre-treatment is used, there will be a risk that the test result for nickel release will be higher.

Method detail: sample degreasing:
The majority of the participants reported to have degreased the sample with a mild soap solution.

Method detail: composition of the sweat solution:
Three participants used ammonia to increase the pH value of the sweat solution instead of a solution of sodium hydroxide. This is not according to the latest version of EN1811:2011, but was part of the previous versions of this test method.
Two participants adjusted the pH value of the solution to 5.5 instead of 6.5.
One participant used a lower amount of sodium chloride for the sweat solution.
Because in all cases above the composition of the sweat solution was not made as prescribed in the method, the test results of these six laboratories were excluded from the statistical evaluation.

Method detail: analysis technique:
The majority of the participants used ICP-OES to measure the nickel in the sweat solution. Others used ICP-MS and some used (GF)AAS. No significant differences could be found between using the different techniques.

Method detail: use of replicates to determine nickel:
Most participants replicated the nickel determination from the sweat solution, as is stated in the test method.

4.2 EVALUATION PER SAMPLE

In this section, the determination is discussed. All statistical results reported on the samples are summarised in appendix 1.

Sample #14075:

Nickel release: The determination of nickel release at a concentration level of 0.5 µg/cm^2/week was problematic. Three statistical outliers were observed and the test results of six laboratories were excluded (see paragraph 4.1). The calculated reproducibility, after rejection of the suspect data, is not in agreement with EN1811:2011.

Sample #14076:

Nickel release: The determination of nickel release at a concentration level of 13 µg/cm^2/week was very problematic. One statistical outlier was observed and the test results of six laboratories were excluded (see paragraph 4.1). The calculated reproducibility, after rejection of the suspect data, is not at all in agreement with EN1811:2011.

4.3 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as found for the group of participating laboratories and the estimated reproducibilities of EN1811:2011 (R_{target}) in the next tables:

Element	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Nickel	µg/cm^2/week	102	0.511	0.384	0.170

Table 3: reproducibilities of test results in sample #14075

Element	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Nickel	µg/cm^2/week	104	12.982	11.313	4.323

Table 4: reproducibilities of test results in sample #14076

From the above table, it can be concluded, without further statistical calculations, that the participating laboratories have problems with the analysis of nickel release, when compared to the target reproducibility results of the EN1811 method.

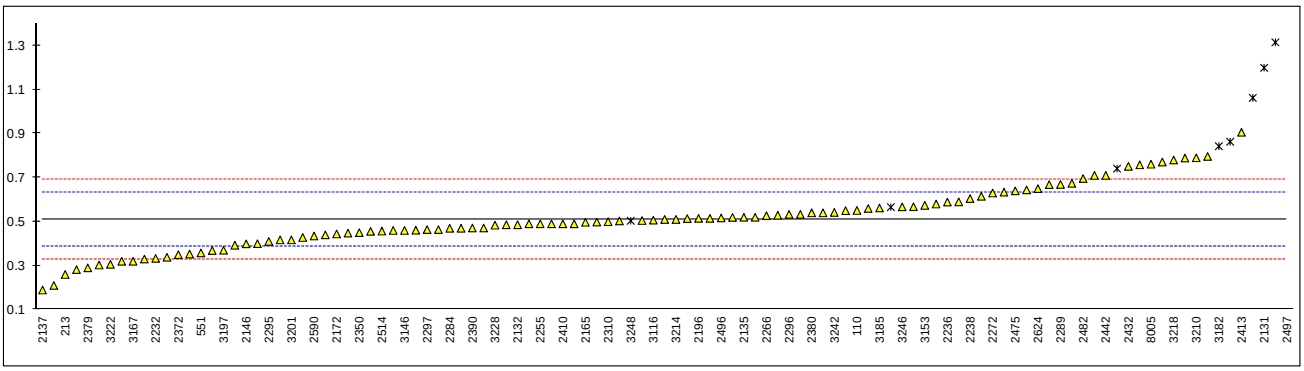
APPENDIX 1

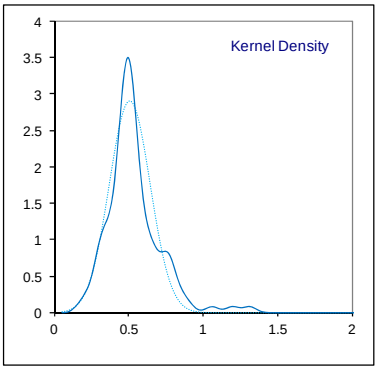
Determination of Nickel Release sample #14075; results in µg/cm^2/week

lab	method	value	mark	z(targ)	remarks
110	EN1811:2011 + AC:2012	0.55077		0.66	
213	EN1811:2011 + AC:2012	0.26		-4.13	
310	EN1811:2011 + AC	0.486		-0.41	
330		0.51		-0.01	
362		0.303		-3.42	
551	EN1811:2011 + AC:2012	0.3580		-2.52	
840	EN1811:2011 + AC:2012	0.55		0.64	
1911	EN1811:2011	0.282		-3.77	
2108	EN1811:2011 + AC:2012	0.417		-1.54	
2115	EN1811	0.590		1.30	
2121	EN1811:2011	1.062	R(0.05)	9.07	
2129	EN1811	0.440		-1.17	
2131	EN1811	1.199	ex	11.33	See §4.1, different composition sweat solution
2132	EN1811:2011 + AC:2012	0.486		-0.41	
2135	EN1811:2011 + AC:2012	0.52		0.15	
2137	EN1811:2011 + AC:2012	0.1895		-5.29	
2139	EN1811:2011	0.40		-1.82	
2146	EN1811:2011	0.399		-1.84	
2156	EN1811	0.71		3.28	
2165	EN1811:2011 + AC:2012	0.497		-0.23	
2172	EN1811:2011 + AC:2012	0.444		-1.10	
2190	EN1811:2011	0.32		-3.14	
2196	EN1811:2011 + AC:2012	0.515		0.07	
2201	EN1811:2011	0.644		2.19	
2215	EN1811:2011	0.505085		-0.09	
2229	EN1811:2011	0.456		-0.90	
2230	EN1811:2011	0.498		-0.21	
2232	EN1811:2011	0.333		-2.93	
2236	EN1811:2011 + AC:2012	0.589	C	1.29	First reported: 1.216
2238	EN1811:2011 + AC:2012	0.605		1.55	
2241	EN1811:2011 + AC:2012	0.560		0.81	
2247	EN1811:2011	0.448		-1.03	
2255	EN1811:2011	0.49		-0.34	
2256	EN1811:2011	0.393		-1.94	
2266	EN1811	0.527		0.27	
2272	EN1811:2011	0.63		1.96	
2284	EN1811:2011 + AC:2012	0.470		-0.67	
2289	EN1811:2011	0.669		2.60	
2290	EN1811:2011 + AC:2012	0.568		0.94	
2295	EN1811	0.41	C	-1.66	First reported: 0.11
2296	EN1811:2011	0.533		0.36	
2297	EN1811:2011 + AC:2012	0.464		-0.77	
2301	EN1811	0.490		-0.34	
2310	EN1811:2011 + AC:2012	0.50		-0.18	
2311	EN1811:2011 + AC:2012	0.52		0.15	
2350	EN1811:2011 + AC:2012	0.450		-1.00	
2352	EN1811:2011 + AC:2012	0.5150		0.07	
2353	EN1811	0.5140		0.05	
2357	EN1811:2011 + AC:2012	0.49		-0.34	
2359	EN1811:2011 + AC:2012	0.529		0.30	
2365	EN1811:2011 + AC:2012	0.758		4.07	
2366	EN1811:2011 + AC:2012	0.519		0.13	
2370	EN1811	0.789		4.58	
2372	EN1811	0.3501		-2.65	
2375	EN1811:2011 + AC:2012	0.21		-4.95	
2379	EN1811:2011 + AC:2012	0.29		-3.63	
2380	EN1811:2011 + AC:2012	0.54		0.48	
2385	EN1811	0.863	ex	5.80	See §4.1, pH of sweat solution too low
2390	EN1811	0.471		-0.66	
2403	EN1811:2011 + AC:2012	0.470		-0.67	
2410	EN1811:2011	0.49		-0.34	
2413	EN1811:2011 + AC:2012	0.906		6.50	
2425	EN1811:2011 + AC:2012	0.54		0.48	
2432	EN1811:2011	0.751		3.95	
2440	GB/T19719:2005	0.741	C, ex	3.79	First reported: 0.926; see §4.1, ammonia used to adjust pH
2442	EN1811	0.71		3.28	
2459	EN1811	0.369		-2.33	
2475	EN1811	0.64		2.13	
2482	EN1811:2011 + AC:2012	0.6959		3.05	
2488	EN1811:2011	0.503		-0.13	
2492	EN1811	0.634		2.03	
2495	EN1811:2011	0.471		-0.66	
2496	EN1811:2011	0.5170		0.10	
2497	EN1811:2011 + AC:2012	2.74	C, R(0.01)	36.69	First reported: 1.74
2500	EN1811:2011	0.58		1.14	
2508	EN1811:2011	0.796		4.69	

2511	EN1811:2011	0.533		0.36	
2514	EN1811:2011	0.457		-0.89	
2522		0.46		-0.84	
2549	EN1811:2011	0.771		4.28	
2566	EN1811	0.338		-2.84	
2573	EN1811:2011 + AC:2012	0.461		-0.82	
2590	EN1811	0.435		-1.25	
2605	EN1811	0.464		-0.77	
2624	EN1811:2011	0.650		2.29	
3100	EN1811:2011	0.428		-1.36	
3116	EN1811:2011	0.507		-0.06	
3146	EN1811:2011 + AC:2012	0.46		-0.84	
3151	EN1811:2011 + AC:2012	0.615		1.71	
3153	EN1811:2011 + AC:2012	0.574		1.04	
3167	EN1811:2011	0.32		-3.14	
3172	EN1811:2011	0.49		-0.34	
3180	EN1811:2011	0.566	ex	0.91	See §4.1, ammonia used to adjust pH
3182	in house	0.8426	ex	5.46	See §4.1, pH of sweat solution too low
3185	EN1811:2011 + AC:2012	0.562		0.84	
3190	EN1811:2011 + AC:2012	0.668		2.59	
3197	EN1811	0.37		-2.32	
3201	EN1811:2012	0.417		-1.54	
3203		-----		-----	
3210	EN1811	0.79		4.60	
3214	EN1811:2011 + AC:2012	0.51		-0.01	
3218	EN1811:2011	0.78		4.43	
3220	DIN12472/1811	0.33		-2.98	
3222	EN1811:2011	0.306		-3.37	
3225	EN1811	0.674		2.69	
3228	EN1811:2011 + AC:2012	0.484		-0.44	
3237	EN1811:2011	0.353		-2.60	
3242	EN1811:2011 + AC:2012	0.542		0.51	
3243	EN1811	1.31454	C, R(0.01)	13.23	First reported: 1.15901
3246	EN1811:2011	0.5670		0.92	
3248	EN1811:2011	0.504	ex	-0.11	See §4.1, ammonia used to adjust pH
8005	EN1811:2011	0.761		4.12	

		All data	
normality	OK	OK	
n	102	107	
outliers	3 (+6ex)	4	
mean (n)	0.5108	0.5198	
st.dev. (n)	0.13704	0.14335	
R(calc.)	0.3837	0.4014	
R(EN1811:2011)	0.1701	0.1731	Compare R(Horwitz) = 0.2532



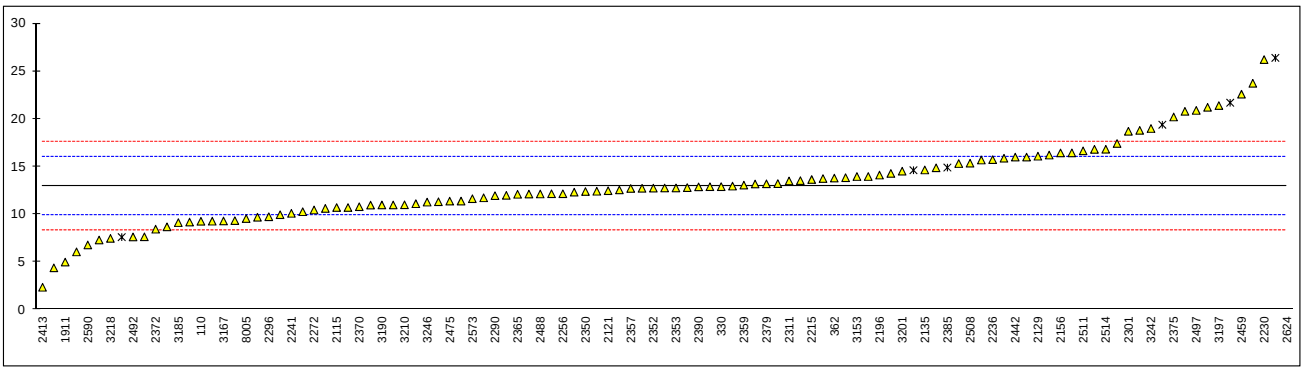


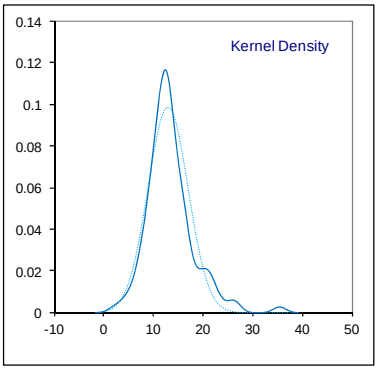
Determination of Nickel Release sample #14076; results in µg/cm^2/week

lab	method	value	mark	z(targ)	remarks
110	EN1811:2011 + AC:2012	9.2699		-2.40	
213	EN1811:2011 + AC:2012	16.21		2.09	
310	EN1811:2011 + AC	13.53		0.36	
330		12.9		-0.05	
362		13.799		0.53	
551	EN1811:2011 + AC:2012	8.6890		-2.78	
840	EN1811:2011 + AC:2012	15.69		1.75	
1911	EN1811:2011	4.993		-5.17	
2108	EN1811:2011 + AC:2012	9.961		-1.96	
2115		10.710		-1.47	
2121	EN1811:2011	12.473		-0.33	
2129	EN1811	16.09		2.01	
2131	EN1811	26.4	ex	8.69	See §4.1, different composition sweat solution
2132	EN1811:2011 + AC:2012	14.870		1.22	
2135	EN1811:2011 + AC:2012	14.65		1.08	
2137	EN1811:2011 + AC:2012	10.7143		-1.47	
2139	EN1811:2011	13.84		0.56	
2146	EN1811:2011	4.39		-5.56	
2156	EN1811	16.43		2.23	
2165	EN1811:2011 + AC:2012	12.151		-0.54	
2172	EN1811:2011 + AC:2012	11.99		-0.64	
2190	EN1811:2011	6.06		-4.48	
2196	EN1811:2011 + AC:2012	14.11		0.73	
2201	EN1811:2011	11.39		-1.03	
2215	EN1811:2011	13.6463		0.43	
2229	EN1811:2011	12.122		-0.56	
2230	EN1811:2011	26.22		8.57	
2232	EN1811:2011	21.217		5.33	
2236	EN1811:2011 + AC:2012	15.73	C	1.78	First reported: 31.50
2238	EN1811:2011 + AC:2012	11.321		-1.08	
2241	EN1811:2011 + AC:2012	10.102		-1.87	
2247	EN1811:2011	16.44		2.24	
2255	EN1811:2011	12.89		-0.06	
2256	EN1811:2011	12.151		-0.54	
2266	EN1811	23.738		6.97	
2272	EN1811:2011	10.455		-1.64	
2284	EN1811:2011 + AC:2012	10.62		-1.53	
2289	EN1811:2011	9.697		-2.13	
2290	EN1811:2011 + AC:2012	11.956		-0.66	
2295	EN1811	9.34	C	-2.36	First reported: 2.06
2296	EN1811:2011	9.747		-2.10	
2297	EN1811:2011 + AC:2012	12.76		-0.14	
2301	EN1811	18.705		3.71	
2310	EN1811:2011 + AC:2012	12.8		-0.12	
2311	EN1811:2011 + AC:2012	13.5		0.34	
2350	EN1811:2011 + AC:2012	12.39		-0.38	
2352	EN1811:2011 + AC:2012	12.7462		-0.15	
2353	EN1811	12.7653		-0.14	
2357	EN1811:2011 + AC:2012	12.71		-0.18	
2359	EN1811:2011 + AC:2012	13.061		0.05	
2365	EN1811:2011 + AC:2012	12.099		-0.57	
2366	EN1811:2011 + AC:2012	13.75		0.50	
2370	EN1811	10.8		-1.41	
2372	EN1811	8.4403		-2.94	
2375	EN1811:2011 + AC:2012	20.2		4.68	
2379	EN1811:2011 + AC:2012	13.20		0.14	
2380	EN1811:2011 + AC:2012	13.22		0.15	
2385	EN1811	14.90	ex	1.24	See §4.1, pH of sweat solution too low
2390	EN1811	12.88		-0.07	
2403	EN1811:2011 + AC:2012	14.284		0.84	
2410	EN1811:2011	10.984		-1.29	
2413	EN1811:2011 + AC:2012	2.352		-6.88	
2425	EN1811:2011 + AC:2012	13.18		0.13	
2432	EN1811:2011	17.422		2.88	
2440	GB/T19719:2005	21.695	ex	5.64	See §4.1, ammonia used to adjust pH
2442	EN1811	16.00		1.96	
2459	EN1811	22.593		6.23	
2475	EN1811	11.38		-1.04	
2482	EN1811:2011 + AC:2012	12.4161		-0.37	
2488	EN1811:2011	12.131		-0.55	
2492	EN1811	7.632		-3.47	
2495	EN1811:2011	12.955		-0.02	
2496	EN1811:2011	12.56		-0.27	
2497	EN1811:2011 + AC:2012	20.89		5.12	
2500	EN1811:2011	12.32		-0.43	
2508	EN1811:2011	15.36		1.54	

2511	EN1811:2011	16.657		2.38	
2514	EN1811:2011	16.82		2.49	
2522		12.73	C	-0.16	First reported: 44.65
2549	EN1811	18.80	C	3.77	First reported: 31.333
2566	EN1811	16.81	C	2.48	First reported: 33.86
2573	EN1811:2011 + AC:2012	11.634		-0.87	
2590	EN1811	6.792		-4.01	
2605	EN1811	7.31		-3.67	
2624	EN1811:2011	35.45	R(0.01)	14.55	
3100	EN1811:2011	7.64		-3.46	
3116	EN1811:2011	9.19		-2.46	
3146	EN1811:2011 + AC:2012	10.97		-1.30	
3151	EN1811:2011 + AC:2012	11.11		-1.21	
3153	EN1811:2011 + AC:2012	13.96		0.63	
3167	EN1811:2011	9.3		-2.38	
3172	EN1811:2011	10.27		-1.76	
3180	EN1811:2011	19.392	ex	4.15	See §4.1, ammonia used to adjust pH
3182	in house	7.6089	ex	-3.48	See §4.1, pH of sweat solution too low
3185	EN1811:2011 + AC:2012	9.131		-2.49	
3190	EN1811:2011 + AC:2012	10.978		-1.30	
3197	EN1811	21.40		5.45	
3201	EN1811:2012	14.522	C	1.00	First reported: 7.261
3203		-----		-----	
3210	EN1811	11.0		-1.28	
3214	EN1811:2011 + AC:2012	9.28		-2.40	
3218	EN1811:2011	7.48		-3.56	
3220	DIN12472/1811	16.0		1.96	
3222	EN1811:2011	15.320		1.51	
3225	EN1811	15.879		1.88	
3228	EN1811:2011 + AC:2012	11.73		-0.81	
3237	EN1811:2011	13.961		0.63	
3242	EN1811:2011 + AC:2012	18.992		3.89	
3243	EN1811	20.8		5.06	
3246	EN1811:2011	11.2778		-1.10	
3248	EN1811:2011	14.622	ex	1.06	See §4.1, ammonia used to adjust pH
8005	EN1811:2011	9.56		-2.22	

		All data:	
normality	suspect	suspect	
n	104	110	
outliers	1 +(6 ex)	1	
mean (n)	12.9817	13.2246	
st.dev. (n)	4.04030	4.29122	
R(calc.)	11.3129	12.0154	
R(EN1811:2011)	4.3229	4.4038	Compare R(Horwitz) = 3.9540





APPENDIX 2

Analytical details for samples #14075 and #14076:

lab	#14075 volume (ml)	area (cm^2)	Ni-conc. (µg/l)	#14076 volume (ml)	area (cm^2)	Ni-conc. (µg/l)	remarks
110	3	**3**	330.467	7.25	**7.22**	6692.9	#14076: area higher than possible
213	100	**2.825**	7.22667	10000	**2.43**	3.94	#14076: possibly part was covered?
310	3	**2.85**	138.433	6	**6.32**	8566	
330	4	**3.34**		7	6.69		#14075: area higher than possible
362	10	**2.83**	86	10	6.57	9066	
551	25	**2.84**	40.6667	25	6.56	2280	
840		**2.83**	0.155		6.68	5.24	
1911	2.9	**2.840**	32.1134	6.5	6.499	649.0	
2108	25	2.88	48	25	6.55	2610	
2115	3	**2.8**	166.667	6.3	**6.3**	3400	
2121	25	**2.85**	60.6	20	6.60	1646.2	
2129	2.5	2.871	507.337	13.0	6.791	8406.712	
2131	5	2.88	690.667	10	6.77	17882	
2132	3	**2.85**	277	7	6.60	7010	
2135	25	**2.8**	59.0	25	**5.91**	3460	
2137	3.0	**2.8**	53.0667	7.0	**6.3**	1350	
2139	10	**2.8**	112	10	**6.9**	9550	
2146	2	**2.83**	113.0	4	6.61	2903	
2156	10	**2.8**	198	25	6.5	4273	
2165	3.0	**2.81**	93.0667	6.5	6.52	2440	
2172	2.80	**2.80**	249.033	6.57	6.57	7874	
2190	5	**2.823**	180	5	**3.71**	4500	#14076: possibly part was covered?
2196	10	**2.83**	145.87	25	6.63	3742.6	
2201	2.8667	2.8666667	184.667	6.7	6.7	1526	
2215	5.00	**2.92**	147.333	10.0	6.66	3635	
2229	2.8	**2.800**	456.10	6.5	6.518	12156	
2230	5	**2.84**	283	10	6.72	17620	
2232	8.0	**2.42**	80.4667	10.0	**6.136**	5207.2	
2236	2.82	**2.82**	171.333	10	**5.99**	3770	
2238	2.8	**2.8**	605.333	6.6	6.6	11321	
2241	2.853	**2.853**	159.733	6.66	6.66	6727.8	
2247	10	**2.82**	126.433	25	6.60	4342	
2255	10	**2.81**	137.667	25	6.57	3387.0	
2256	10	**2.83**	111.333	25	6.563	3190	
2266	10	2.9	150.4	25	**7**	66.030	#14076: area higher than possible
2272	10	**2.84**	179.1	25	6.59	2756	
2284	3.00	2.87	453.667	4	6.62	1758	
2289	2.8	**2.8**	187.333	6.6	6.6	2560	
2290	2.84	**2.84**	161.333	6.67	6.67	3190	
2295	5	2.87		10	6.67		
2296	2	2.901	154.67	5	6.614	3223	
2297	3.0	**2.8**	132	6.5	6.4	3263	
2301	10	**2.6578**	128.33	10	6.4668	12.096	
2310	3	**2.84**	141	6	**6.33**	162	
2311	5	**2.8**	144.667	10	6.5	175	
2350	10	**2.828**		25	6.563		
2352	2.85	**2.84**	146.267	6.60	6.58	336.5	
2353	4	**3.34**	0.513	7	6.69	12.77	#14075: area higher than possible
2357	3	**2.85**	140.533	7	6.61	3361	
2359	3	**2.83**	150.000	7	6.61	3453	
2365	2.80	**2.80**	212.333	6.67	6.67	1614	
2366	3.0	**2.83**	0.29333	7.0	6.64	9.11	
2370	10	**2.84**	224.333	10	6.64	7170	
2372	10	2.88	100.867	10	6.59	5562	
2375	10	**2.8**	3	10	6.6	7	
2379	2.84	**2.84**	167.7	6.45	6.45	8530	
2380	3.00	**2.79**	302.7	7.00	6.52	8620	
2385	3	**2.85**	807.0	7	6.65	13940	
2390	2.9	2.86	135.0	6.7	6.69	431	

Analytical details for samples #14075 and #14076, continued:

lab	#14075 volume (ml)	area (cm^2)	Ni-conc. (µg/l)	#14076 volume (ml)	area (cm^2)	Ni-conc. (µg/l)	remarks
2403	10	**2.8**	131.667	25	6.6	3771	
2410	3	**2.8**	136.267	7	6.6	2899.7	
2413	10	**2.02**	73.2	10	**5.91**	556.0	
2425	5	**2.783**	299.6	10	6.6	8708.90	
2432	10	**2.85**	214.1	11	6.49	10279.2	
2440	5.0	**2.84**	105.2	8.0	6.64	900.362	
2442	10	**2.84**	201.5	10	6.63	10610.00	
2459	5	**2.85**	207.667	7	6.59	21270	
2475	10	2.88	207.233	20	6.58	3767.96	
2482	2.8267	**2.8266667**	69.8333	6.58	6.58	1238	
2488	3	**2.84**	143.081	10	6.7	813.148	
2492	2.15	2.89	73.0433	3.36	**6.324**	1930.258	
2495	5.00	2.860	269.333	5.00	**3.687**	9553	#14076: possibly part was covered?
2496	25.0	**2.84**	58.6667	25.0	6.62	3325.0	
2497	12	**2.34**	338.133	12	6.60	11490	
2500	5	**2.83**	330.333	10	6.63	8195	
2508	10	**2.752**	212.333	10	6.579	9909	
2511	5	**2.8**	300.104	10	**5.91**	9894.500	
2514	10	**2.82**	128.5	10	6.65	11185.0	
2522	5	**4.92**	223	7	6.6	8400	#14075: area higher than possible
2549	10	2.89	225.743	10	6.62	85.84	
2566	5	**2.65**	179.667	10	**6.32**	21400	
2573	2.84	**2.84**	1310.33	6.54	6.54	76080	
2590	2	**2.85**	125.0	5	6.8	4593	
2605	2.9	2.9	134.667	6.6	6.6	1930	
2624	2.9	2.89	189.033	6.6	6.61	11848	
3100	2.8	**2.8**	119.8	6.5	6.5	1985.71	
3116	3	**2.84**	144	7	6.64	2440	
3146	2.84	**2.84**	130.667	6.39	6.39	5400	
3151	3	2.88	614.667	7	6.7	11110	
3153	2.9	2.883	165.267	6.7	6.68	186.5	
3167	5	**2.8**	179	6.28	**6.28**	9300	
3172	4.000	2.870	355.987	6.650	6.650	10268.37	
3180	6.0	**2.8**	264.267	7.0	6.4	17730	
3182	10	**2.8**	236	10	**3.61**	2747	#14076: possibly part was covered?
3185	3.0	**2.8**	524.467	7.0	6.5	8479	
3190	2.8	**2.8**	667.667	6.7	6.7	10978	
3197	2.9	2.88	106.333	6.6	6.56	5615	
3201	10	**2.8**	116.667	20	**6.9**	5010	
3203							
3210	10	**2.84**	223.333	10	6.63	7260	
3214	10	**2.8**	142.667	25	6.52	2420	
3218	2.8	**2.8**	216.867	6.5	6.5	1944.7	
3220	10	**2**	6.63333	10	**2.954**	472.6	Possibly part was covered?
3222	3	**2.80**	85.7533	10	6.48	4963.68	
3225	3	2.9	191.333	6.7	**7**	10480	#14076: area higher than possible
3228	2.89	2.89	140	6.63	6.63	3110	
3237	5	2.864	10.1213	10	6.671	37.254	
3242	50	**2.827**	30.6667	50	**2.83**	1075	Same value for both: typing error?
3243	2.84	**2.84**	1159.01	6.517	6.517	20800	
3246	5.00	**2.848**	323	10	6.511	7343	
3248	2.8	2.86	57.6667	6.5	6.54	3825	
8005	3	**2.83**	215.333	7	6.64	2540	

Figures in bold: Value of area outside normal distribution found for the area of the sample
Normal distribution for #14075 2.86 - 2.90 cm^2 and for #14076 6.35 - 6.81 cm^2.
See §4.1: Calculation of surface area

APPENDIX 3

Test method procedure details for samples #14075 and #14076:

	Test vessel material	pretreat. vessel	with	sample degreas.	with	date of test sol. prep.	pH	Urea	NaCl	lactic acid	NaOH	analysis technique	use replicates?
110	Polypropylene	No		No		30-5-2014	6.5	Yes	Yes	Yes	Yes	ICP-MS	No
213	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfonic acid	16-5-2014	6.5	0.1%	0.5%	0.1%		ICP-MS	No
310	Glass	No		Yes	Mild soap	26-5-2014	6.45	Yes	Yes	Yes	Yes	ICP-MS	No
330												ICP-MS	No
362	Glass	Yes	5% Nitric Acid for 10 hrs.	No		31-05-2014	6.50	Yes	Yes	Yes		ICP-OES	Yes
551	Glass	Yes	20% Nitric Acid for 5 hrs.	Yes	Sodium laureth sulfate	26-05-2014	6.51	Yes	Yes	Yes		ICP-MS/ICP-OES	Yes
840	Glass	No		Yes	Degreasing solution	19-05-2014	6.5					ICP-OES	Yes
1911	Glass / LDPE	Yes	5% Nitric Acid for 5 hrs.	Yes	sodium dodecylbenzene sulfate sol.	02-06-2014	6.496	0.1%	0.5%	0.1%	Yes	GFAAS / FAAS	Yes
2108	Polypropylene	No		Yes	0.5% sodium dodecyl sulfate	16-05-2014	6.48	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
2115	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfate	22-05-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2121	Glass	Yes	2% Nitric Acid for 8 hrs	Yes	0.5% sodium dodecyl sulfate	28-05-2014	6.49	Yes	Yes	Yes	Yes	ICP-MS	Yes
2129	Polystyrene / Glass	No		Yes	0.5% detergent	30-05-2014	6.490-6.509	0.1%	0.5%	0.1%			Yes
2131	Polypropylene	No		Yes	sodium dodecyl sulfate	26-05-2014		0.1%	**0.1%**	0.1%		ICP-MS	Yes
2132	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfate	23-05-2014	6.5	0.1%	0.5%	0.1%	Yes	AAS	Yes
2135	Glass	No		Yes	Washing up liquid	19-05-2014	6.55	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2137	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	sodium dodecylbenzene sulfate sol.	02-06-2014	6.5	0.1%	0.5%	0.1%		ICP-OES	Yes
2139	Polypropylene	No		No		05-06-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2146	Glass	No		Yes	0.5% Extran	20-05-2014	6.47	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2156	Polypropylene	Yes	5% Nitric Acid for 4 hrs	Yes	1% detergent	27-05-2014	6.5	0.1%	0.5%	0.1%		ICP-OES	Yes
2165	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfate	30-05-2014	6.52	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2172	Polypropylene	Yes	10% Nitric Acid for 24 hrs	Yes	degrease solution	21-05-2014	6.51	0.1%	0.5%	0.1%		ICP-OES	Yes
2190	Glass	No		No			6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	No
2196	Glass	Yes	5% Nitric Acid for 4 hrs	Yes	0.5% sodium dodecylbenzene sulfate	29-05-2014	6.51	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2201	Polypropylene	Yes	15% Nitric Acid for 12 hrs	Yes	Degreasing solution	29-05-2014	6.45-6.49	0.1%	0.5%	0.1%	Yes	ICP-MS/ICP-OES	Yes
2215	Polypropylene	Yes	10% Nitric Acid for 24 hrs	No		03-06-2014	6.50	Yes	Yes	Yes	Yes	ICP-OES	Yes
2229	Polypropylene	No		Yes	Detergent	27-05-2014	6.527	Yes	Yes	Yes	Yes	ICP-MS	No
2230	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	sodium dodecylbenzene sulfonate	16-06-2014	6.49-6.51	Yes	Yes	Yes		ICP-OES	Yes
2232	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	sodium dodecylbenzene sulfonate	12-06-2014	6.51	0.1%	0.5%	0.1%		ICP-OES	Yes
2236	Glass	No		Yes	5% dodecyl sulfonic acid	03-06-2014	6.49	0.1%	0.5%	0.1%		ICP-OES	Yes
2238	Glass	Yes	5% Nitric Acid for 24 hrs	Yes	0.5% sodium dodecylbenzene sulfate	06-06-2014	6.51	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2241	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfate	29-05-2014	6.50-6.52	0.1%	0.5%	0.1%		ICP-OES	Yes
2247	Polypropylene	No		Yes	0.5% detergent	22-05-2014	6.49	0.1%	0.5%	0.1%		ICP-OES	Yes
2255	Glass	Yes	5% Nitric Acid for 4 hrs	Yes	Degreasing solution	06-06-2014	6.5	0.1%	0.5%	0.1%		ICP-MS	No
2256	Glass	No		Yes	Detergent	29-05-2014	6.49	Yes	Yes	Yes		ICP-OES	Yes
2266	Polypropylene	No		Yes	dodecyl sulfate solution	21-05-2014	6.51		Acidified Artificial Sweat			ICP-OES	Yes
2272	Polypropylene	No		Yes	Degreasing solution	23-05-2014	6.50	Yes	Yes	Yes		ICP-OES	Yes
2284	Polypropylene	Yes	10% Nitric Acid for 24 hrs	Yes	Detergent	21-05-2014	6.51	0.1%	0.5%	0.1%		ICP-OES	Yes

	Test vessel material	pretreat. vessel	with	sample degreas.	with	test sol prep	pH	Urea	NaCl	lactic acid	NaOH	analysis technique	use replicates?
2289	Polypropylene	Yes	5% Nitric Acid for 12 hrs	Yes	0.5% dodecylbenzene sulfonate	05-06-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2290	Glass	Yes	10% Nitric Acid for 24 hrs	Yes	1% sodium dodecylbenzen sulfonate	26-05-2014	6.48	0.1%	0.5%	0.1%		ICP-MS	Yes
2295													
2296	Polypropylene	No		Yes	Tri-sodium di phosphate decahydrate	29-05-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2297	Polyethylene	Yes	5% Nitric Acid for 24 hrs	Yes	0.1% sodium dodecylbenzene sulfonate	22-05-2014	6.51	Yes	Yes	Yes	Yes	ICP-OES	Yes
2301	Glass	No				26-05-2014	6.52	0.1%	0.5%	0.1%	Yes	ICP-OES	No
2310	Glass	Yes	5% Nitric Acid for 4 hrs	Yes	0.5% sodium dodecylbenzene sulfonic acid	05-06-2014	6.50	0.1%	0.5%	0.1%	Yes	AAS	Yes
2311	Glass	Yes	5% Nitric Acid for 4 hrs	No		20-05-2014	6.48-6.50	0.1%	0.5%	0.1%	Yes	AAS	Yes
2350	Glass	Yes	5% Nitric Acid for 4 hrs	Yes	dodecylbenzen sulfate solution	21-05-2014	6.52	Yes	Yes	Yes	Yes	ICP-OES	Yes
2352	Polypropylene	Yes	Nitric Acid for 24 hrs	No		23-05-2014	6.48-6.51	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2353	Glass	Yes	5% Nitric Acid for 8 hrs	Yes	0.5% sodium dodecylbenzene sulfate	16-05-2014	6.49	0.1%	0.5%	0.1%	Yes	ICP-OES	No
2357	Polypropylene	No		No			6.50	0.1%	0.5%	0.1%		ICP-OES	Yes
2359	Polypropylene	Yes	5% Nitric Acid for 5 hrs.	Yes	0.5% sodium dodecylbenzene sulfonate	22-05-2014	6.53	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2365	Polypropylene	No		Yes	5% sodium dodecylbenzene sulfate	04-06-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2366	Polyethylene	Yes	1:1 Hydrochloric Acid for 96 hrs	Yes	household detergent	19-05-2014	6.50	Yes	Yes	Yes		ICP-OES	Yes
2370	Other	Yes	Nitric Acid for 1/3 hrs	No		28-05-2014	6.51	Yes	Yes	Yes		ICP-OES	Yes
2372	Polypropylene	No		Yes	sodium dodecylbenzene sulfonate	27-05-2014	6.51	Yes	Yes	Yes	Yes	ICP-OES	No
2375	Polypropylene	No		Yes	EN1811:2011+AC2012	02-06-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-MS/ICP-OES	Yes
2379	Glass	Yes	20% Nitric Acid for 12hrs	Yes	Teepol	23-05-2014	6.58	0.1%	0.5%	0.1%		ICP-OES	Yes
2380	Glass	Yes	5% Nitric Acid for 12 hrs	Yes	5% dodecyl sulfonic acid	01-06-2014	6.46	Yes	Yes	Yes	Yes	AAS	Yes
2385	Polypropylene	No		Yes	0.1% dodecylsulfate solution	04-06-2014	**5.5**	Yes	Yes	Yes	Yes	ICP-OES	Yes
2390	Polypropylene	Yes	5% Nitric Acid for 4 hrs	No		27-05-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2403	Glass	Yes	5% Nitric Acid for 24 hrs	Yes	0.5% anion surfactant	29-05-2014	6.48	0.1%	0.5%	0.1%		ICP-OES	No
2410	Polypropylene	No		No		22-05-2014	6.5	0.1%	0.5%	0.1%		ICP-OES	Yes
2413	Glass	No		No		09-06-2014	6.5	artificial sweat				ICP-OES	No
2425	Glass	No		Yes	0.50% detergent solution	05-06-2014	6.5	0.1%	0.5%	0.1%		ICP-OES	No
2432	Polypropylene	Yes	5% Nitric Acid for 4 hrs	No		19-05-2014		0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
2440	Polypropylene	No		Yes	0.5% sodium dodecylbenzene sulfonate sol.	04-06-2014	6.48	0.1%	0.5%	0.1%	**NH3**	ICP-OES	Yes
2442	Polypropylene	No		No		29-05-2014	6.5	0.1%	0.5%	0.1%		ICP-MS	Yes
2459	Glass	Yes	Sodium dodecylbenzene sulfonated for 1/2 hrs	Yes	sodium dodecylbenzene sulfonate	18-06-2014	6.52	Yes	Yes	Yes	Yes	AAS	Yes
2475	Glass	Yes	5% Nitric Acid for 24 hrs	Yes	sodium dodecylbenzene sulfonate	04-06-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
2482	Polypropylene	No		No		02-06-2014	6.5	Yes	Yes	Yes	Yes	ICP-OES	No
2488	Glass	no		Yes	0.5% sodium dodecyl sulfate	05-06-2014	6.51	0.1%	0.5%	0.1%		ICP-MS	Yes
2492	Polypropylene	No		Yes	sodium dodecylbenzene sulfonate	16-05-2014	6.509	0.1%	0.5%	0.1%		ICP-MS	Yes
2495	Polypropylene	No		Yes	0.1% sodium dodecylsulfate	28-05-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2496	Polypropylene	Yes	5% Nitric Acid for 4 hrs	Yes	0.5% sodium dodecylbenzene sulfate	29-05-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2497	Glass	Yes	Dilute Nitric Acid for 4 hrs	No		20-05-2014	6.51	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
2500	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	5% dodecyl benzene sulfonate	05-06-2014	6.48-6.51	0.1%	0.5%	0.1%		ICP-OES	Yes
2508													

2511	polytetrafluoroethylene	No		Yes	0.5% sodium dodecyl benzene sulfonate	17-06-2014	6.51	0.1%	0.5%	0.1%		ICP-MS	No

	Test vessel material	pretreat. vessel	with	sample degreas.	with	test sol prep	pH	Urea	NaCl	lactic acid	NaOH	analysis technique	use replicates?
2514	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	Degreasing solution	04-06-2014	6.5	0.1%	0.5%	0.1%			No
2522	Polypropylene	Yes	distilled water for 1/2 hrs	Yes	Degreasing solution	06-06-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2549	Polypropylene	Yes	for 2 hrs	Yes	commercial detergent powder	26-05-2014	6.50	0.1%	0.5%	0.1%		ICP-MS	Yes
2566	Polypropylene	No		Yes	dodecylbenzene sulfonated	29-05-2014	6.5	Yes	Yes	Yes		ICP-OES	Yes
2573	Glass	Yes	1% Nitric Acid for 0.25 hrs	Yes	0.5% sodium alkylaryl sulfonate	23-05-2014	6.53	0.1%	0.5%	0.1%		ICP-OES	Yes
2590	Polypropylene	No		No		16-05-2014	6.4	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
2605	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	0.5% sodium dodecylbenzene sulfate	30-05-2014	6.51	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
2624	Polypropylene	Yes	5% Nitric Acid for 4 hrs	Yes	0.5% sodium alkyl sulfate	04-06-2014	6.49	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
3100	Glass	Yes	Dilute Nitric Acid for 4 hrs	Yes	Degreasing solution	27-05-2014	6.50-6.52	0.1%	0.5%	0.1%	Yes	ICP-OES	No
3116	Polypropylene	No		Yes	Degreasing solution	05-06-2014	6.5	Yes	Yes	Yes		ICP-OES	Yes
3146	Polypropylene	Yes	5% Nitric Acid for 4 hrs	Yes	sodium dodecyl sulfate	23-05-2014	6.54	Yes	Yes	Yes		ICP-OES	Yes
3151	Polypropylene	No		Yes	0.5% sodium dodecyl sulfate	16-05-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
3153	Glass	Yes	5% Nitric Acid for 24 hrs	Yes	Sodium dodecylbenzene sulfonate sol	28-05-2014	6.5	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
3167	Glass	Yes	5% Nitric Acid for 24 hrs	No		03-06-2014	6.50	0.1%	0.5%	0.1%		ICP-OES	No
3172	Polypropylene	No		Yes	sodium dodecylbenzene sulfate sol.	03-06-2014	6.56	Yes	Yes	Yes	Yes	ICP-MS	Yes
3180	Glass	No		Yes	sodium dodecyl sulfate sol	20-05-2014	6.5	0.1%	0.5%	0.1%	**NH3**	GFAAS	Yes
3182	Glass	Yes	10% Nitric Acid for 2 hrs	No		05-06-2014	**5.4**					ICP-MS	Yes
3185	Glass	No		Yes	5% sodium dodecylbenzene sulfate	03-06-2014	6.49	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
3190	Polypropylene	Yes	20% Nitric Acid for 24 hrs	Yes	0.5% sodium dodecylbenzene sulfate	30-05-2014	6.51	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
3197	Glass	Yes	5% Nitric Acid for 4 hrs	Yes	Degreasing solution	03-06-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
3201	PMP Glass/PMP	Yes	flushed with dil. nitric acid	Yes	Diluted dish liquid	21-05-2014	6.5	Yes	Yes	Yes	Yes	GFAAS	Yes
3203													
3210	Polystyrene	No		Yes		20-05-2014	6.15-6.57	0.1%	0.5%	0.1%		ICP-OES	No
3214	Glass	Yes	5% Nitric Acid for 18 hrs	Yes	Acid detergent (Citranox)	05-06-2014	6.47	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
3218	Polypropylene	Yes	5% Nitric Acid for 5 hrs.	Yes	0.5% sodium dodecylbenzene sulfate	03-06-2014	6.51	0.1%	0.5%	0.1%		ICP-MS	Yes
3220	Glass	Yes	test solution for 1 hr	Yes	0.5% sodium dodecylbenzene sulfonate	04-06-2014	6.5	Yes	Yes	Yes		ICP-MS	No
3222	Glass	Yes	3% Nitric Acid (storage condition)	Yes	0.5% sodium dodecylbenzene sulfate	21-05-2014	6.52	0.1%	0.5%	0.1%	Yes	GFAAS	Yes
3225	Glass	Yes	5% Nitric Acid for 12 hrs	Yes	sodium dodecylsulfate	03-06-2014	6.5	0.1%	0.5%	0.1%		ICP-OES	Yes
3228	Polypropylene	No		Yes	sodium dodecylbenzene sulfate	21-05-2014	6.47	0.1%	0.5%	0.1%	Yes	ICP-OES	Yes
3237	Polypropylene	No		Yes		20-05-2014	6.47	0.1%	0.5%	0.1%	Yes	ICP-MS	Yes
3242	Polypropylene	Yes	rinsed with buffer solution	Yes	Degreasing solution	19-05-2014	6.55	0.1%	0.5%	0.1%	Yes	ICP-OES	No
3243	Polypropylene	Yes	5% Nitric Acid for 4 hrs	Yes	Fit solution	19-05-2014	6.53	0.1%	0.5%	0.1%	Yes	GFAAS/ICP-OES	Yes
3246	Polypropylene	No		Yes	Aceton	03-06-2014	6.50	0.1%	0.5%	0.1%	Yes	ICP-OES	No
3248	Glass	Yes	Artifical Sweat solution for 1 hrs	No		29-05-2014	6.4	0.1%	0.5%	0.1%	**NH3**	ICP-OES	Yes
8005	Polypropylene	No		Yes	Degreasing solution	06-05-2014	6.5	Yes	Yes	Yes		ICP-OES	Yes

APPENDIX 4

Number of participants per country

5 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in FINLAND
6 labs in FRANCE
10 labs in GERMANY
8 labs in HONG KONG
7 labs in INDIA
1 lab in INDONESIA
7 labs in ITALY
4 labs in KOREA
1 lab in MALAYSIA
1 lab in MOROCCO
33 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in POLAND
1 lab in SINGAPORE
2 labs in SWITZERLAND
4 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in THE NETHERLANDS
1 lab in TUNISIA
5 labs in TURKEY
3 labs in U.S.A.
1 lab in UNITED KINGDOM
3 labs in VIETNAM

APPENDIX 5

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
W	= result withdrawn on request of participant
ex	= excluded from calculations
fr.	= first reported result

Literature:

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