Results of Proficiency Test
Nickel Release
June 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. R.G.Visser & ing. N. Boelhouwer
Report: iis15V03

August 2015

CONTENTS

Appendices:

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 to 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 annual testing program since 2014.
In the interlaboratory study of June 2015, 119 laboratories from 29 different countries have participated (see appendix 4). In this report, the results of the 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organizer of this proficiency test. It was decided to send three pieces of one sample (labelled #15075), 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

The sample consisted of square metal pieces (#15075) with a hole in one of the corners, which were purchased from a local supplier. The pieces were massive, prepared from one alloy and not plated. The dimensions of each sample were approximately 1.2 x 1.2 x 0.2 cm and the hole had a diameter of approx 4 mm.
Six stratified randomly selected samples were tested using EN1811:2011 to check the homogeneity of the batch.

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

Nickel release	*in #15075 (µg/cm^2/week)*
sample 1	2.7
sample 2	2.5
sample 3	2.5
sample 4	2.5
sample 5	2.7
sample 6	2.5

table 1: homogeneity test results of samples #15075

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 #15075 (µg/cm^2/week)*
r (observed)	0.29
reference method	EN1811:2001
0.3 x R (reference method)	0.26

table 2: evaluation of the repeatability of samples #15075

The repeatability of the results of the homogeneity tests for nickel release of sample #15075 was in agreement with 0.3 times the reproducibility mentioned in the reference method EN1811:2011. Therefore, homogeneity of the subsamples was assumed for this sample.

Three pieces of sample #15075 were sent to each of the participating laboratories on May 13, 2015.

2.5 ANALYSES

The participants were requested to determine the nickel release on the sample, 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 may 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 123 participants, 16 participants reported results after the deadline for reporting and four participants did not report any test results at all. Finally, the 119 reporting laboratories all submitted an average numerical result. Observed were 11 outlying results, which is 9.8%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

The original data set did not proof to have a normal Gaussian distribution. The statistical evaluation of this data set 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 EVALUATION PER SAMPLE

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

Sample #15075:

Nickel release: The determination of nickel release at a concentration level of 1.33 µg/cm^2/week was very problematic. Eleven statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with EN1811:2011.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Element	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Nickel	µg/cm^2/week	108	1.331	1.042	0.443

Table 3: reproducibilities of test results in sample #15075

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2015 WITH PREVIOUS PTS

	May 2015	*May 2014*
Number of reporting labs	123	111
Number of results reported	119	222
Statistical outliers	11	4
Percentage outliers	9.8	1.8

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

In table 5 the observed uncertainty in this PT are compared with the uncertainties as observed in the previous PT.

	May 2015	*May 2014*	*target EN1811*
Nickel Release	28%	27-31%	11%

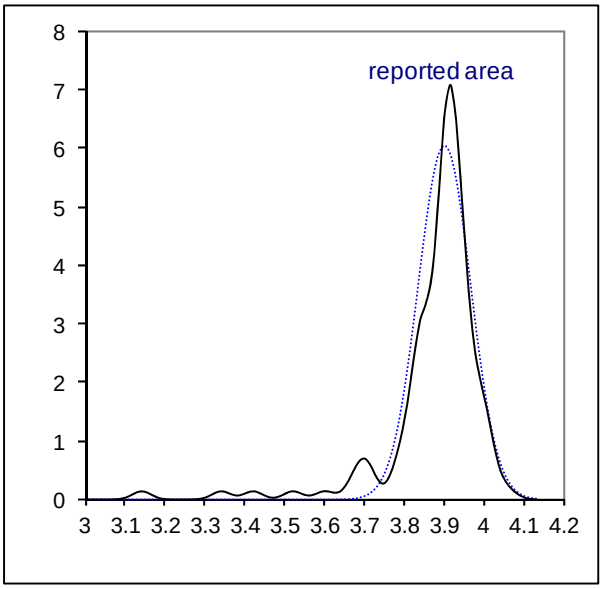
Table 5: Comparison of uncertainties (relative in %) of Nickel Release in this PT and previous PTs

No quality improvement is visible as the uncertainty did not decrease since 2014 and is still much larger than the target derived from EN1811.

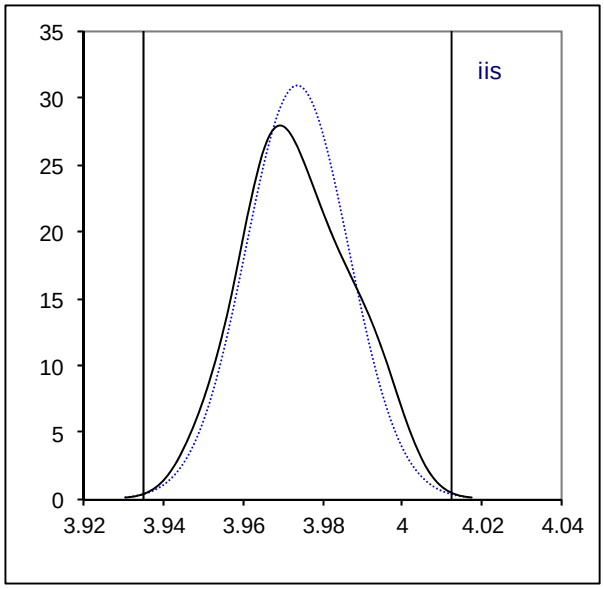
5 DISCUSSION OF REPORTED METHOD DETAILS

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 of the test solution, the final volume of the test solution, the contact area used and the nickel concentration in the release solution after one week.

Area of contact surface:
In total 120 laboratories reported the surface area used, see appendix 2 and below left graph:



range of reported area in cm^2 on sample #15075



range of area in cm^2 determined by iis on sample #15075

The reported surface area for sample #15075 varied from 0.28 (!) – 4.06 cm^2.
After exclusion of 8 statistical outlying data (all surfaces below 3.68 cm^2), the range narrowed to 3.34 – 4.06 cm^2. The average surface is 3.90 cm^2, with a standard deviation of 0.07 cm^2, thus RSD = 1.7%, which is much less than in the previous PT when 9-10% was found for the relative standard deviation in the reported surface area.
The dimensions measured on 10 test items by iis are: 1.209 x 1.210 x 0.021 cm for the square surface and a diameter of 0.395 cm for the hole. This leads to a contact surface of 3.974 cm^2 for the test item (hole included), see the right graph on the previous page. The effect on the area by the presence of the hole is minimal (+0.024 cm^2), because the radius of the hole is almost equal to the thickness of the plate.
From the 2014 PT results, the relative standard deviation for nickel release was calculated. The $RSD_{nickel\ release}$ was 27% for #15075 and the $RSD_{nickel\ release}$ was 31% for #14076. In this PT for sample #15075 the $RSD_{nickel\ release}$ is 28%. Regretfully the observed decrease in the spread of the estimation of the contact surface to only 1.7% in this PT, is not visible as a decrease in spread in the final nickel release test result.

Volume of the test solution (initial):
In total 116 laboratories reported the initial volume of the test solution used, see appendix 2. The test method of EN1811:2011 prescribes that the initial test solution used should be 1 ml per cm^2 surface area. The vast majority of the participants did use a ratio of 1 ml/cm^2, but the total range of ratios was 0.5 – 35.4 (!) ml/cm^2. The range of initial volumes was 2 – 25 ml. After exclusion of 12 statistically outlying ratios (of which 10 ratios above 2.49 ml/cm^2), the ratio range narrowed to 0.78 – 2.04 cm^2. The average ratio is 1.10 ml/cm^2, with a standard deviation of 0.24 ml/cm^2, thus RSD = 22%. Obviously some improvement may be available for this parameter.

Final volume of the test solution:
In total 118 laboratories reported the final volume of the test solution used, see appendix 2. 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. 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 to a large volume. The vast majority of the participants did use 10 ml as final volume, but the total range of volumes was 3 (!) – 50 ml. After exclusion of 6 statistically outlying data (all above 40 ml, a dilution of >10 times), the volume range narrowed to 3 – 30 ml. The average volume is 11.1 ml, with a standard deviation of 6.2 ml, thus RSD = 55%. Possibly some improvement may be available for this parameter.

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 at least 4 hrs, see paragraph 6.4 of EN1811:2011. This is done to remove any nickel present from an earlier test. About half of the participants reported to have done this pre-treatment, but 40% of the participants did not use any pre-treatment. When no pre-treatment is used, there will be a risk that the test result for nickel release will be higher. To check whether some effect is visible, the test results of the laboratories that did not use any pre-treatment were compared with the test results after acid treatment of at least 4 hrs., see table 6.

	No pre-treatment	*Acid pre-treatment> 4hrs*
Number of test results	48	59
Statistical outliers	2	2
Average in	1.56 µg/cm^2/week	1.26 µg/cm^2/week
Standard deviation	0.762 µg/cm^2/week	0.353 µg/cm^2/week
RSD%	49%	28%

Table 6: influence of pre-treatment of test vessel

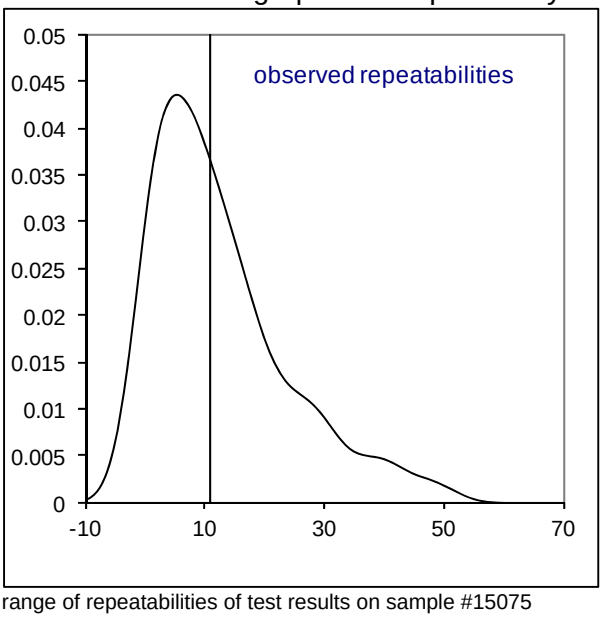
Indeed the effect of the acid pre-treatment is visible. The test results from a vessel that was not pre-treated are slightly higher that the test results from a pre-treated test vessel. Also the precision of the test results from both data sets differs about a factor 2. Clearly a significant quality improvement may be available for this parameter.

Sample degreasing:
The majority of the participants degreased the sample with detergent solution.

Composition of the sweat solution:
Not all participants adjusted the pH value of the solution to 6.5. The actual pH reported varies from 2.8 (!) – 6.63. The extremely low pH = 2.8 resulted in a significantly high nickel release which was found to be a statistical outlier.

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 were observed between using the different techniques.

Use of replicates:
Almost all participants that reported test results did report test results for the 3 sample pieces. From the 3 intermediate test results, the repeatability per laboratory was calculated. The repeatabilities vary strongly from a very small 0.2% to a very large 49%. Only 66 laboratories had a repeatability in agreement with the target repeatability of 11%, estimated from EN1811, see also the below graph. The repeatability of 52 laboratories was larger than 11%.



range of repeatabilities of test results on sample #15075

APPENDIX 1

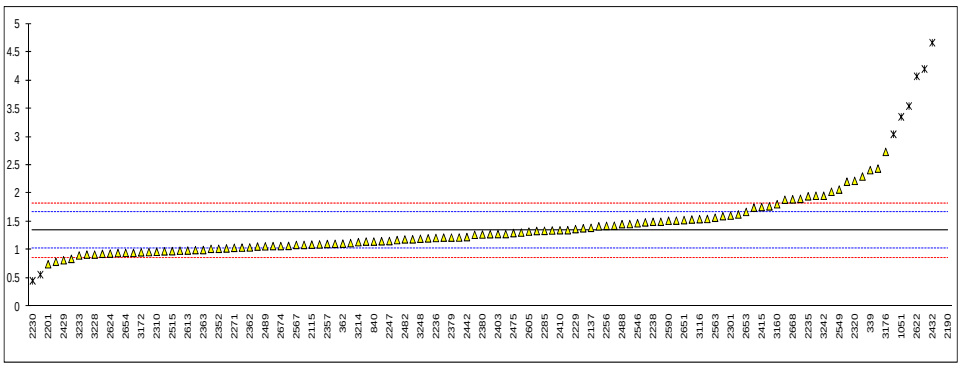
Determination of Nickel Release sample #15075; results in µg/cm^2/week

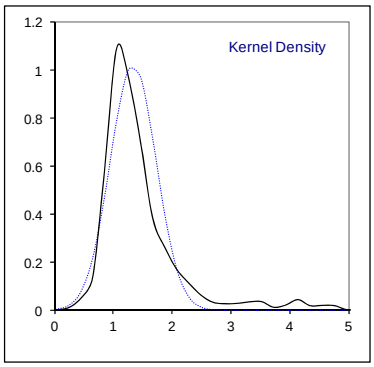
lab	method	value	mark	z(targ)	remarks
110	EN1811:2011 + AC:2012	3.5446	R(0.01)	13.99	
310	EN1811	2.44	C	7.01	first reported: 24.78
330	EN 12472 + EN 1811	1.19		-0.89	
339	EN1811:2011	2.413	C	6.84	first reported: 3.274
362	EN1811:2011	1.114		-1.37	
551	EN1811:2011	0.565	R(0.01)	-4.84	
623		-----		-----	
840	EN1811:2011 + AC:2012	1.15		-1.14	
1051	EN1811	3.354	R(0.01)	12.79	
2115	EN1811:2011 + EC2012 + EC 2013	1.097		-1.48	
2121		3.046	R(0.01)	10.84	
2129	EN1811:2012-10	1.177		-0.97	
2137	EN1811:2012	1.393		0.39	
2139	in house	1.96		3.98	
2156	EN1811:2011	1.091		-1.51	
2165	EN1811:2011 + AC:2012	1.029		-1.91	
2172	EN1811:2011 + AC:2012	1.890		3.54	
2184	EN1811:2011 + AC:2012	1.022		-1.95	
2190	EN1811:2011	52	C,R(0.01)	320.22	first reported: 0.052
2201	EN1811:2011 + AC:2012	0.7499		-3.67	
2229	EN1811:2011 + AC:2012	1.37		0.25	
2230	EN1811:2011	0.4568	R(0.01)	-5.52	
2232	EN1811:2011	1.599		1.70	
2235	EN1811:2011	1.95		3.92	
2236	EN1811:2011 + AC:2012	1.2137		-0.74	
2238	EN1811:2011 + AC:2012	1.50		1.07	
2241	EN1811:2011 + AC:2012	0.793		-3.40	
2247		1.159		-1.08	
2255	EN1811:2011	1.43		0.63	
2256	EN1811:2011	1.428		0.62	
2271	EN1811:2011 + AC:2012	1.04		-1.84	
2285	EN1811:2011 + AC:2012	1.34		0.06	
2289		0.844		-3.07	
2290	EN1811:2011	2.031		4.43	
2295	EN1811	0.934	C	-2.51	first reported: 2.60
2296		1.282		-0.31	
2300	EN1811:2011 + AC:2012	0.965		-2.31	
2301	EN1811:2011	1.6098		1.77	
2309	EN1811:2011 + AC:2012	1.15		-1.14	
2310	EN1811:2011 + AC:2012	0.97		-2.28	
2311	EN1811:2011 + AC:2012	1.1		-1.46	
2320	in house	2.223		5.64	
2347		1.0442		-1.81	
2352		1.0222		-1.95	
2357		1.107		-1.41	
2362	EN1811	1.047		-1.79	
2363		1.000		-2.09	
2365		1.073		-1.63	
2366		1.062		-1.70	
2370	EN1811:2011 + AC:2012	0.999		-2.10	
2375	EN1811:2011 + AC:2012	1.22		-0.70	
2379		1.22		-0.70	
2380	EN1811:2011 + AC:2012	1.276		-0.34	
2385	EN1811:2011	2.30		6.13	
2390		1.31		-0.13	
2403		1.282		-0.31	
2410	EN1811:2011 + AC:2012	1.35		0.12	
2415	EN1811:2011 + AC:2012	1.76		2.71	
2429	EN1811:2011	0.82		-3.23	
2432	EN1811:2011	4.664	R(0.01)	21.07	
2442		1.23		-0.64	
2459	EN1811:2011 + AC:2012	0.990		-2.15	
2468	EN1811:2011	2.208		5.55	
2475	EN1811:2011 + AC:2012	1.30		-0.19	
2482	EN1811:2011	1.1886		-0.90	
2485	EN1811:2011	0.978		-2.23	
2488	EN1811	1.458		0.81	
2489		1.066		-1.67	
2492		1.772		2.79	
2495		-----	W	-----	first reported: 3.398
2497	EN1811:2011 + AC:2012	1.351		0.13	
2504	EN1811:2011	1.270		-0.38	

2511	EN1811:2011 + AC:2012	0.948		-2.42	
2514	EN1811:2011	1.50		1.07	
2515		0.981		-2.21	
2516	EN1811:2011	1.489998		1.01	
2546		1.474		0.91	
2549	EN1811	2.07		4.67	
2560		1.55		1.39	
2563	EN1811	1.572		1.53	
2566		1.46		0.82	
2567	EN1811:2011	1.09		-1.52	
2590		1.519		1.19	
2605		1.327		-0.02	
2613		0.9913		-2.14	
2618	EN1811:2011 + AC:2012	1.222		-0.69	
2622	EN1811:2011	4.070	R(0.01)	17.31	
2624	EN1811:2011	0.938		-2.48	
2637	EN1811:2011 + AC:2012	5.45	R(0.01)	26.03	
2643	EN1811:2011 + AC:2012	1.1557		-1.10	
2649	EN1811:2011	1.75		2.65	
2651	EN1811:2011 + AC:2012	1.5296		1.26	
2653		1.6744		2.17	
2654	EN1811 + A1:2008	0.95	C	-2.40	first reported: 0.095
2657		1.07		-1.65	
2668		1.8969		3.58	
2674		1.0705		-1.64	
3100	EN1811:2011	1.207		-0.78	
3116	EN1811	1.546		1.36	
3118		-----		-----	
3124		-----		-----	
3145	in house	1.905		3.63	
3146	EN1811:2012-10	1.54		1.32	
3153	EN1811:2011 + AC:2012	1.28		-0.32	
3160	EN1811:2011	1.81		3.03	
3172	EN1811	0.96		-2.34	
3176	EN1811:2011 + AC:2012	2.735	C,R(0.01)	8.88	first reported: 3.60
3182	EN1811:2011	1.337		0.04	
3185	EN1811:2011 + AC:2012	1.123		-1.31	
3190	EN1811:2011 + AC:2012	0.92		-2.59	
3197	EN1811:2011	1.52		1.20	
3200	EN1811:2011 + AC:2012	0.950		-2.40	
3210		4.20	R(0.01)	18.13	
3214	EN1811:2011 + AC:2012	1.14		-1.20	
3218	EN1811:2011	1.11		-1.39	
3220		1.629		1.89	
3225	EN1811:2011 + AC:2012	1.42		0.57	
3228		0.92		-2.59	
3233	EN1811	0.905		-2.69	
3237	EN1811:2011	1.385		0.34	
3242	EN1811:2011 + AC:2012	1.96		3.98	
3243		1.34667		0.10	
3248	EN1811:2011	1.2		-0.82	

normality	suspect
n	108
outliers	11
mean (n)	1.3305
st.dev. (n)	0.37207
R(calc.)	1.0418
R(EN1811:2011)	0.4431

Compare R(Horwitz): 0.5710





APPENDIX 2

Calculated ratios and calculated relative repeatability standard deviations

lab	ratio ml/cm^2 (EN1811 target = 1)	RSDr in % (EN1811 target = 11.1%)
110	1.00	7.9
310	1.09	48.7
330	---	---
339	2.78	1.4
362	1.00	11.3
551	6.35	13.0
623	---	---
840	1.01	13.9
1051	1.00	10.4
2115	1.00	10.8
2121	0.99	37.5
2129	0.63	42.8
2137	1.08	39.7
2139	1.30	22.5
2156	1.27	7.4
2165	1.04	11.2
2172	1.03	14.3
2184	0.99	14.0
2190	0.99	12.5
2201	1.00	1.7
2229	1.04	7.6
2230	1.30	3.1
2232	2.54	0.2
2235	2.02	0.8
2236	1.00	9.9
2238	1.00	16.4
2241	1.00	8.6
2247	1.02	0.6
2255	1.03	25.1
2256	---	2.4
2271	1.02	5.8
2285	2.03	3.9
2289	1.00	9.1
2290	1.00	3.8
2295	1.28	0.9
2296	0.51	29.3
2300	1.03	3.0
2301	1.06	21.1
2309	1.03	0.5
2310	1.35	3.3
2311	1.28	5.8
2320	1.00	14.3
2347	1.00	8.1
2352	1.00	13.1
2357	1.03	14.6
2362	1.02	14.9
2363	0.99	4.4
2365	1.00	8.2
2366	1.03	17.1
2370	1.01	5.0
2375	1.00	7.6
2379	1.00	0.5
2380	1.03	18.3
2385	1.27	19.6
2390	1.00	38.4
2403	1.00	1.4
2410	1.00	20.3
2415	1.02	25.3
2429	1.00	3.1

2432	1.09	2.6
2442	0.88	16.0
2459	1.02	7.6
2468	1.00	26.3
2475	1.00	40.7
2482	1.00	19.5
2485	2.60	21.4
2488	2.42	9.3
2489	1.05	1.4
2492	1.01	25.1
2495	1.04	---
2497	1.28	1.5
2504	---	1.4
2511	1.03	5.2
2514	1.05	7.1
2515	---	7.5
2516	1.00	31.8
2546	0.78	16.1
2549	1.30	8.5
2560	1.04	15.4
2563	4.08	24.9
2566	1.14	0.7
2567	1.03	7.8
2590	2.53	3.7
2605	1.00	9.2
2613	1.01	2.1
2618	1.02	9.6
2622	0.78	15.8
2624	1.02	19.7
2637	1.04	1.9
2643	1.05	14.5
2649	1.54	2.9
2651	1.04	0.9
2653	1.53	12.1
2654	1.00	9.6
2657	1.03	6.1
2668	1.00	10.6
2674	1.00	18.1
3100	1.00	2.9
3116	1.02	5.3
3118	---	---
3124	---	---
3145	3.91	28.2
3146	1.00	34.6
3153	0.99	10.2
3160	1.89	19.8
3172	1.03	29.4
3176	2.04	1.8
3182	1.02	6.9
3185	1.00	9.4
3190	1.00	28.5
3197	1.00	30.6
3200	1.00	0.9
3210	3.18	28.7
3214	1.00	5.7
3218	1.00	6.8
3220	35.43	6.9
3225	1.02	1.1
3228	1.00	13.6
3233	1.00	13.3
3237	2.03	48.1
3242	1.50	0.3
3243	1.00	15.7
3248	1.02	3.0

APPENDIX 3

Analytical details for sample #15075-1:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
110	3.85	6-Jun-15	12-Jun-15	3.85	10	1268.1	3.2938
310	4	4-Jun-15	11-Jun-15	3.68	10	10090	1.57
330				3.94	4		-----
339	10	8-Jun-15	16-Jun-15	3.6	20	434.34	2.413
362	3.90	2-Jun-15	9-Jun-15	3.9		976	0.976
551	25	3-Jun-15	10-Jun-15	3.9363	50	77	0.489
623							-----
840	4	29-May-15	5-Jun-15	3.95	10	389	0.97
1051	4	3-Jun-15	10-Jun-15	4	10	1490.951667	3.727
2115	3.85	28-May-15	4-Jun-15	3.84	10	445.202	1.158
2121	4	5-Jun-15	12-Jun-15	4.06	15	1156.3	4.272
2129	2.5	20-May-15	27-May-15	3.95	3	2317.099	1.759
2137	4.0	2-Jun-15	9-Jun-15	3.7	25.0	171	1.151
2139	5	5-Jun-15	12-Jun-15	3.84	50	1.766	2.30
2156	5	2-Jun-15	9-Jun-15	3.9233	5	890.5	1.135
2165	4.0	1-Jun-15	1-Jun-15	3.86	5.0	758.03	0.982
2172	4	26-May-15	2-Jun-15	3.89	10	640.9	1.648
2184	4	19-May-15	26-May-15	4.03	10	389	0.963
2190	3.8	29-May-15	5-Jun-15	3.82	10	22400	58
2201	3.9	28-May-15	4-Jun-15	3.9	25	114.7	0.7352
2229	4	20-May-15	27-May-15	3.84	4	1267.5	1.32
2230	5	21-May-15	28-May-15	3.86	5	340.151	0.4406
2232	10.0	19-May-15	26-May-15	3.93	25.0	0.251	1.598
2235	8	22-May-15	29-May-15	3.96	10	30.7	1.94
2236	3.93	3-Jun-15	10-Jun-15	3.93	10	429	1.0916
2238	3.9	25-May-15	1-Jun-15	3.9	10	694	1.78
2241	3.93	1-Jun-15	8-Jun-15	3.93	10	292.2	0.745
2247	4	27-May-15	2-Jun-15	3.92	10	457.4	1.167
2255	4	9-Jun-15	16-Jun-15	3.9	10	703	1.80
2256				3.893	10	571	1.467
2271	4.0			3.91	5.0	0.8626	1.10
2285	8.0	26-May-15	2-Jun-15	3.9394	10.00	550.8	1.40
2289	4.0	28-May-15	4-Jun-15	4.0	10.0	372.1	0.930
2290	3.9	1-Jun-15	8-Jun-15	3.9	10	821.3	2.106
2295	5	26-May-15	2-Jun-15	3.91	5	1902	0.941
2296	2	1-Jun-15	8-Jun-15	3.93	50	134	1.706
2300	4	14-May-15	21-May-15	3.89	5	756.0	0.994
2301	4	1-Jun-15	7-Jun-15	3.78	5	1.4968	1.9810
2309	4	4-Jun-15	11-Jun-15	3.89	10	446	1.14
2310	5	22-May-15	29-May-15	3.71	10	346	0.93
2311	5	28-May-15	4-Jun-15	3.9	10	402.252	1.03
2320	3.854	29-May-15	5-Jun-15	3.854	5.0	2.52	2.524
2347	3.96	26-May-15	2-Jun-15	3.96	10	448.64	1.1329
2352	3.95	26-May-15	2-Jun-15	3.95	10	0.4177	1.0575
2357	4	28-May-15	4-Jun-15	3.88	10	3840	0.990
2362	4	20-May-15	27-May-15	3.94	10	345	0.876
2363	3.90	22-May-15	29-May-15	3.93	4.88	821	1.019
2365	3.91	28-May-15	4-Jun-15	3.91	10	410	1.049
2366	4.0	26-May-15	2-Jun-15	3.90	5.0	724	0.926
2370	4	21-May-15	28-May-15	3.946	10	0.375	0.951
2375	3.84	27-May-15	3-Jun-15	3.84	10	479.2	1.25
2379	3.84	20-May-15	27-May-15	3.84	5	941	1.22
2380	4.00	1-Jun-15	8-Jun-15	3.90	10.00	596.5 (119.3 x 5)	1.529
2385	5	2-Jun-15	9-Jun-15	3.95	5.078	1790	2.30
2390	3.98	20-May-15	27-May-15	3.98	10	511	1.28
2403	3.92	2-Jun-15	9-Jun-15	3.92	10	510	1.301
2410	4	19-May-15	26-May-15	4.0	10	507.3	1.27
2415	4	20-May-15	27-May-15	3.92	5		1.99
2429	4	1-Jun-15	8-Jun-15	4	10	328	0.82
2432	4	25-May-15	1-Jun-15	3.67	10	16613.4	4.528
2442	3	27-May-15	2-Jun-15	3.42	10	349.120	1.02
2459	4	26-May-15	2-Jun-15	3.913	4	941	0.962
2468	3.78	28-May-15	4-Jun-15	3.78	10	0.981	2.596
2475	3.97	25-May-15	1-Jun-15	3.97	10	388.04	0.98
2482	4	19-May-15	26-May-15	4.0	40	107.95	1.0795
2485	10	2-Jun-15	9-Jun-15	3.84	20	171.59	0.891
2488	9	18-May-15	25-May-15	3.72	10	527.37	1.417
2489	4	4-Jun-15	11-Jun-15	3.81	10	102.19	1.07
2492	4.0	3-Jun-15	10-Jun-15	3.95	25.0	277.9	1.747

Analytical details for samples #15075-1, continued:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
2495	4.00	27-May-15	3-Jun-15	3.830	10.00	1023	-----
2497	5	21-May-15	28-May-15	3.906	4.9	517.8	1.332
2504		28-May-15	4-Jun-15	3.919	10.0	503.072	1.284
2511	4.0	8-Jun-15	15-Jun-15	3.90	10	379.82	0.973
2514	4.0	10-Jun-15	17-Jun-15	3.80	10	604	1.59
2515		20-May-15	27-May-15	3.91	10	0.415	1.062
2516	3.8	21-May-15	28-May-15	3.8	10	0.000731521	1.925055
2546	3	28-May-15	4-Jun-15	3.8461	5	1295.50	1.684
2549	5	21-May-15	28-May-15	3.85	50	175.86	2.28
2560	4	2-Jun-15	9-Jun-15	3.8426	25	215	1.39
2563	15-17	19-May-15	26-May-15	3.923	20	1.26	1.256
2566	4.5	22-May-15	29-May-15	3.96	5	1.17	1.47
2567	4.0	4-Jun-15	11-Jun-15	3.902	10	426.40	1.09
2590	10	22-May-15	29-May-15	3.95	10	594.4	1.502
2605	4.0	2-Jun-15	9-Jun-15	4.0	25	0.232	1.450
2613	4	7-Jun-15	14-Jun-15	3.95	5	1.015	1.015
2618	4	20-May-15	27-May-15	3.903	5	1.231	1.231
2622	3	3-Jun-15	10-Jun-15	3.87	25	593	3.831
2624	4	29-May-15	6-Jun-15	3.91	10	321.825	0.834
2637	4	3-Jun-15	10-Jun-15	3.84	4	5200	5.36
2643	4.0	27-May-15	3-Jun-15	3.8	25	151	0.9934
2649	6	26-May-15	2-Jun-15	3.9	10	677.7	1.74
2651	4	1-Jun-15	8-Jun-15	3.84	5	1.1868	1.5453
2653	6	2-Jun-15	9-Jun-15	3.91	6.2	1130	1.7918
2654	3.5	29-May-15	5-Jun-15	3.50	10	309.0	0.88
2657	4	29-May-15	5-Jun-15	3.9	10	390	1.01
2668	3.7	28-May-15	4-Jun-15	3.70	25	0.2593	1.7520
2674	4.00	26-May-15	2-Jun-15	3.99	20	177.316	0.8888
3100	4.0	18-May-15	25-May-15	4.0	10	0.468	1.170
3116	4	22-May-15	29-May-15	3.92	10	578	1.474
3118							-----
3124							-----
3145	15	21-May-15	28-May-15	3.84	25	267.2	1.740
3146	3.9	21-May-15	28-May-15	3.91	10	530	1.36
3153	3.9	1-Jun-15	8-Jun-15	3.927	10	445	1.13
3160	7.5	21-May-15	28-May-15	3.96	20	329.4	1.66
3172	4.0			3.88			0.66
3176	8	22-May-15	29-May-15	3.93	25	588	2.743
3182	4.0	26-May-15	2-Jun-15	3.91	10	539.737	1.380
3185	3.9	3-Jun-15	10-Jun-15	3.9	10	393	1.007
3190	4.0	1-Jun-15	8-Jun-15	4.0	25	0.19	1.19
3197	3.9	25-May-15	1-Jun-15	3.9	10	429	1.12
3200	3.91	8-Jul-15	15-Jul-15	3.91	10	368	0.941
3210	10	29-May-15	5-Jun-15	3.14	10	1240	3.95
3214	3.85	25-May-15	1-Jun-15	3.85	10	465	1.21
3218	3.9	2-Jun-15	9-Jun-15	3.9	10	412	1.06
3220	10	26-May-15	1-Jun-15	0.28224	10	0.433	1.533
3225	4.0	4-Jun-15	11-Jun-15	3.913	10	1.42	1.42
3228	3.94	21-May-15	25-May-15	3.94	10	0.33	0.835
3233	3.9	27-May-15	3-Jun-15	3.9	3.9	0.0002	1.000
3237	8	28-May-15	4-Jun-15	3.95	10	38.838	0.983
3242	5	18-May-15	25-May-15	3.34	25	262	1.96
3243	3.842	26-May-15	2-Jun-15	3.8418	3.84	1380	1.38
3248	4	2-Jun-15	9-Jun-15	3.93	25	183	1.16

Analytical details for sample #15075-2:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
110	3.85	6-Jun-15	12-Jun-15	3.85	10	1345.1	3.4938
310	4	4-Jun-15	11-Jun-15	3.68	10	8837	3.79
330				3.94	4		-----
339	10	8-Jun-15	16-Jun-15	3.6	20	905.50	2.447
362	3.90	2-Jun-15	9-Jun-15	3.9		1143	1.143
551	25	3-Jun-15	10-Jun-15	3.9363	50	100	0.635
623							-----
840	4	29-May-15	5-Jun-15	3.95	10	474	1.19
1051	4	3-Jun-15	10-Jun-15	4	10	1319.276667	3.298
2115	3.85	28-May-15	4-Jun-15	3.84	10	451.111	1.173
2121	4	5-Jun-15	12-Jun-15	4.06	15	545.3	2.014
2129	2.5	20-May-15	27-May-15	3.95	3	1189.257	0.903
2137	4.0	2-Jun-15	9-Jun-15	3.7	25.0	301	2.026
2139	5	5-Jun-15	12-Jun-15	3.84	50	1.621	2.11
2156	5	2-Jun-15	9-Jun-15	3.9144	5	781.0	0.998
2165	4.0	1-Jun-15	1-Jun-15	3.86	5.0	895.88	1.160
2172	4	26-May-15	2-Jun-15	3.89	10	716.0	1.841
2184	4	19-May-15	26-May-15	4.03	10	479	1.186
2190	3.8	29-May-15	5-Jun-15	3.82	10	17400	45
2201	3.9	28-May-15	4-Jun-15	3.9	25	118.3	0.7582
2229	4	20-May-15	27-May-15	3.84	4	1430.0	1.49
2230	5	21-May-15	28-May-15	3.86	5	361.036	0.4676
2232	10.0	19-May-15	26-May-15	3.93	25.0	0.251	1.596
2235	8	22-May-15	29-May-15	3.96	10	30.9	1.95
2236	3.93	3-Jun-15	10-Jun-15	3.93	10	523	1.3308
2238	3.9	25-May-15	1-Jun-15	3.9	10	511	1.31
2241	3.95	1-Jun-15	8-Jun-15	3.95	10	343.3	0.871
2247	4	27-May-15	2-Jun-15	3.92	10	453.0	1.156
2255	4	9-Jun-15	16-Jun-15	3.9	10	423	1.08
2256				3.903	10	552	1.414
2271	4.0			3.91	5.0	0.7682	0.98
2285	8.0	2-Jun-15	9-Jun-15	3.9332	10.00	512.4	1.30
2289	4.0	28-May-15	4-Jun-15	4.0	10.0	313.4	0.784
2290	3.9	1-Jun-15	8-Jun-15	3.9	10	761.2	1.952
2295	5	26-May-15	2-Jun-15	3.91	5	1572	0.936
2296	2	3-Jun-15	10-Jun-15	3.93	50	91	1.156
2300	4	14-May-15	21-May-15	3.89	5	740.79	0.937
2301	4	1-Jun-15	7-Jun-15	3.78	5	0.9940	1.3149
2309	4	4-Jun-15	11-Jun-15	3.89	10	448	1.15
2310	5	22-May-15	29-May-15	3.71	10	364	0.98
2311	5	28-May-15	4-Jun-15	3.9	10	448.489	1.15
2320	3.854	29-May-15	5-Jun-15	3.854	5.0		2.257
2347	3.96	26-May-15	2-Jun-15	3.96	10	381.76	0.9640
2352	3.95	26-May-15	2-Jun-15	3.95	10	0.4481	1.1344
2357	4	28-May-15	4-Jun-15	3.88	10	4036	1.040
2362	4	29-May-15	5-Jun-15	3.94	10	426	1.081
2363	3.90	22-May-15	29-May-15	3.93	4.88	830	1.031
2365	3.91	28-May-15	4-Jun-15	3.91	10	391	1.000
2366	4.0	26-May-15	2-Jun-15	3.90	5.0	784	1.004
2370	4	21-May-15	28-May-15	3.936	10	0.392	0.996
2375	3.84	27-May-15	3-Jun-15	3.84	10	341.18	1.12
2379	3.84	20-May-15	27-May-15	3.84	5	939	1.22
2380	4.00	1-Jun-15	8-Jun-15	3.90	10.00	417	1.069
2385	5	2-Jun-15	9-Jun-15	3.92	5.078	2120	2.75
2390	3.98	26-May-15	2-Jun-15	3.98	10	330	0.83
2403	3.92	2-Jun-15	9-Jun-15	3.92	10	502	1.280
2410	4	19-May-15	26-May-15	4.0	10	451.8	1.13
2415	4	20-May-15	27-May-15	3.92	5		1.25
2429	4	1-Jun-15	8-Jun-15	4	10	316	0.79
2432	4	25-May-15	1-Jun-15	3.67	10	86.192x20x10	4.697
2442	3	27-May-15	2-Jun-15	3.42	10	436.244	1.27
2459	4	3-Jun-15	10-Jun-15	3.913	4	913	0.933
2468	3.78	28-May-15	4-Jun-15	3.78	10	0.582	1.540
2475	3.97	25-May-15	1-Jun-15	3.97	10	757.14	1.91
2482	4	19-May-15	26-May-15	4.0	40	103.22	1.0322
2485	10	2-Jun-15	9-Jun-15	3.84	20	234.05	1.217
2488	9	26-May-15	2-Jun-15	3.71	10	596.94	1.609
2489	4	4-Jun-15	11-Jun-15	3.81	10	102.61	1.08
2492	4.0	3-Jun-15	10-Jun-15	3.93	25.0	212.5	1.340
2495	4.00	27-May-15	3-Jun-15	3.830	10.00	1679	-----
2497	5	21-May-15	28-May-15	3.906	10	535.1	1.373
2504		28-May-15	4-Jun-15	3.919	10.0	500.216	1.276
2511	4.0	8-Jun-15	15-Jun-15	3.90	10	382.30	0.980
2514	4.0	10-Jun-15	17-Jun-15	3.80	10	577	1.52

Analytical details for samples #15075-2, continued:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
2515		20-May-15	27-May-15	3.91	10	0.359	0.918
2516	3.8	21-May-15	28-May-15	3.8	10	0.000592582	1.559426
2546	3	28-May-15	4-Jun-15	3.8461	5	935.97	1.217
2549	5	21-May-15	28-May-15	3.85	50	152.95	1.98
2560	4	2-Jun-15	9-Jun-15	3.8426	25	280	1.82
2563	15-17	20-May-15	27-May-15	3.923	20		2.01
2566	4.5	22-May-15	29-May-15	3.96	5	1.15	1.45
2567	4.0	4-Jun-15	11-Jun-15	3.902	10	460.20	1.18
2590	10	22-May-15	29-May-15	3.95	10	579.1	1.473
2605	4.0	2-Jun-15	9-Jun-15	4.0	25	0.193	1.206
2613	4	7-Jun-15	14-Jun-15	3.95	5		0.982
2618	4	20-May-15	27-May-15	3.903	5		1.334
2622	3	3-Jun-15	10-Jun-15	3.87	50	277	3.579
2624	4	29-May-15	6-Jun-15	3.91	10	319.825	0.829
2637	4	3-Jun-15	10-Jun-15	3.84	4	5400	5.42
2643	4.0	27-May-15	3-Jun-15	3.8	25	174	1.1447
2649	6	26-May-15	2-Jun-15	3.9	10	666.9	1.71
2651	4	1-Jun-15	8-Jun-15	3.84	5	1.1707	1.5244
2653	6	2-Jun-15	9-Jun-15	3.91	6.2	1130	1.7918
2654	3.5	29-May-15	5-Jun-15	3.52	10	319.6	0.91
2657	4	29-May-15	5-Jun-15	3.9	10	440	1.14
2668	3.7	28-May-15	4-Jun-15	3.70	25	0.2696	1.8126
2674	4.00	26-May-15	2-Jun-15	3.99	20	209.298	1.0491
3100	4.0	18-May-15	25-May-15	4.0	10	0.485	1.212
3116	4	22-May-15	29-May-15	3.93	10	643	1.636
3118							-----
3124							-----
3145	15	21-May-15	28-May-15	3.84	25	384.8	2.505
3146	3.9	21-May-15	28-May-15	3.91	10	436	1.12
3153	3.9	1-Jun-15	8-Jun-15	3.934	10	533	1.35
3160	7.5	21-May-15	28-May-15	3.96	20	438.1	2.21
3172	4.0			3.88			1.22
3176	8	22-May-15	29-May-15	3.93	25	586	2.683
3182	4.0	26-May-15	2-Jun-15	3.91	10	481.428	1.231
3185	3.9	3-Jun-15	10-Jun-15	3.9	10	448	1.149
3190	4.0	1-Jun-15	8-Jun-15	4.0	25	0.14	0.89
3197	3.9	25-May-15	1-Jun-15	3.9	10	546	1.41
3200	3.91	8-Jul-15	15-Jul-15	3.91	10	372	0.951
3210	10	29-May-15	5-Jun-15	3.14	10	985	3.14
3214	3.85	25-May-15	1-Jun-15	3.85	10	416	1.08
3218	3.9	2-Jun-15	9-Jun-15	3.9	10	421	1.08
3220	10	26-May-15	1-Jun-15	0.28224	10	0.495	1.753
3225	4.0	4-Jun-15	11-Jun-15	3.916	10		1.40
3228	3.94	21-May-15	25-May-15	3.94	10	0.42	1.065
3233	3.9	27-May-15	3-Jun-15	3.9	3.9	0.000189	0.945
3237	8	28-May-15	4-Jun-15	3.95	10	40.242	1.019
3242	5	18-May-15	25-May-15	3.34	25	263	1.97
3243	3.842	26-May-15	2-Jun-15	3.8418	3.84	1540	1.54
3248	4	2-Jun-15	9-Jun-15	3.93	25	190	1.21

Analytical details for sample #15075-3:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
110	3.85	6-Jun-15	12-Jun-15	3.85	10	1480.8	3.8462
310	4	4-Jun-15	11-Jun-15	3.68	10	8400	1.95
330				3.94	4		-----
339	10	8-Jun-15	16-Jun-15	3.6	20	428.04	2.378
362	3.90	2-Jun-15	9-Jun-15	3.9		1222	1.222
551	25	3-Jun-15	10-Jun-15	3.9363	50	90	0.571
623							-----
840	4	29-May-15	5-Jun-15	3.95	10	511	1.28
1051	4	3-Jun-15	10-Jun-15	4	10	**1214.771667**	3.037
2115	3.85	28-May-15	4-Jun-15	3.84	10	369.350	0.960
2121	4	5-Jun-15	12-Jun-15	4.06	15	772.1	2.852
2129	2.5	20-May-15	27-May-15	3.95	3	1145.951	0.870
2137	4.0	2-Jun-15	9-Jun-15	3.7	25.0	149	1.003
2139	5	5-Jun-15	12-Jun-15	3.84	50	1.122	1.46
2156	5	2-Jun-15	9-Jun-15	3.9196	5	894.8	1.141
2165	4.0	1-Jun-15	1-Jun-15	3.86	5.0	728.94	0.944
2172	4	26-May-15	2-Jun-15	3.89	10	848.3	2.181
2184	4	19-May-15	26-May-15	4.03	10	371	0.918
2190	3.8	29-May-15	5-Jun-15	3.82	10	19900	52
2201	3.9	1-Jun-15	8-Jun-15	3.9	25	118.0	0.7562
2229	4	20-May-15	27-May-15	3.84	4	1247.5	1.30
2230	5	21-May-15	28-May-15	3.86	5	357.007	0.4624
2232	10.0	19-May-15	26-May-15	3.93	25.0	0.252	1.602
2235	8	22-May-15	29-May-15	3.92	10	30.9	1.97
2236	3.93	3-Jun-15	10-Jun-15	3.93	10	479	1.2188
2238	3.9	25-May-15	1-Jun-15	3.9	10	554	1.42
2241	3.94	1-Jun-15	8-Jun-15	3.94	10	300.0	0.763
2247	4	27-May-15	2-Jun-15	3.92	10	452.4	1.154
2255	4	9-Jun-15	16-Jun-15	3.9	10	554	1.42
2256				3.899	10	547	1.403
2271	4.0			3.91	5.0	0.8024	1.03
2285	8.0	2-Jun-15	9-Jun-15	3.9360	10.00	519.2	1.32
2289	4.0	28-May-15	4-Jun-15	4.0	10.0	326.7	0.817
2290	3.9	1-Jun-15	8-Jun-15	3.9	10	793.5	2.035
2295	5	26-May-15	2-Jun-15	3.91	5	2640	0.924
2296	2	4-Jun-15	11-Jun-15	3.93	50	78	0.986
2300	4	14-May-15	21-May-15	3.89	5	749.51	0.963
2301	4	1-Jun-15	7-Jun-15	3.78	5	1.1600	1.5345
2309	4	4-Jun-15	11-Jun-15	3.89	10	448	1.15
2310	5	22-May-15	29-May-15	3.71	10	369	0.99
2311	5	5-Jun-15	12-Jun-15	3.9	10	440.803	1.13
2320	3.854	29-May-15	5-Jun-15	3.854	5.0		1.892
2347	3.96	26-May-15	2-Jun-15	3.96	10	410.18	1.0358
2352	3.95	26-May-15	2-Jun-15	3.95	10	0.3455	0.8747
2357	4	28-May-15	4-Jun-15	3.88	10	5012	1.292
2362	4	29-May-15	5-Jun-15	3.94	10	466	1.183
2363	3.90	22-May-15	29-May-15	3.93	4.88	765	0.950
2365	3.91	28-May-15	4-Jun-15	3.91	10	458	1.171
2366	4.0	26-May-15	2-Jun-15	3.90	5.0	982	1.256
2370	4	21-May-15	28-May-15	3.922	10	0.412	1.05
2375	3.84	27-May-15	3-Jun-15	3.84	10	500.10	1.30
2379	3.84	20-May-15	27-May-15	3.84	5	947	1.23
2380	4.00	1-Jun-15	8-Jun-15	3.90	10.00	479.9	1.230
2385	5	2-Jun-15	9-Jun-15	3.98	5.078	1450	1.85
2390	3.98	27-May-15	3-Jun-15	3.98	10	734	1.84
2403	3.92	2-Jun-15	9-Jun-15	3.92	10	496	1.265
2410	4	19-May-15	26-May-15	4.0	10	664.2	1.66
2415	4	20-May-15	27-May-15	3.92	5		2.05
2429	4	1-Jun-15	8-Jun-15	4	10	336	0.84
2432	4	25-May-15	1-Jun-15	3.67	10	87.459x20x10	4.766
2442	3	27-May-15	2-Jun-15	3.42	10	484.108	1.41
2459	4	3-Jun-15	10-Jun-15	3.913	4	1052	1.075
2468	3.78	28-May-15	4-Jun-15	3.78	10	0.940	2.487
2475	3.97	25-May-15	1-Jun-15	3.97	10	401.06	1.01
2482	4	19-May-15	26-May-15	4.0	40	145.42	1.4542
2485	10	2-Jun-15	9-Jun-15	3.84	20	159.03	0.826
2488	9	28-May-15	4-Jun-15	3.66	10	493.19	1.347
2489	4	4-Jun-15	11-Jun-15	3.81	10	100.58	1.05
2492	4.0	3-Jun-15	10-Jun-15	3.95	25.0	354.2	2.229
2495	4.00	27-May-15	3-Jun-15	3.830	10.00	1202	-----
2497	5	21-May-15	28-May-15	3.906	10	526.1	1.349
2504		28-May-15	4-Jun-15	3.919	10.0	490.131	1.251
2511	4.0	8-Jun-15	15-Jun-15	3.90	10	348.07	0.892
2514	4.0	10-Jun-15	17-Jun-15	3.80	10	524	1.38

Analytical details for samples #15075-3, continued:

lab	initial volume (ml)	date sample in solution	date sample out solution	area (cm^2)	final volume (ml)	measured Ni-conc. (µg/l)	reported (µg/cm^2/week)
2515		20-May-15	27-May-15	3.91	10	0.377	0.963
2516	3.8	1-Jun-15	8-Jun-15	3.8	10	0.000374495	0.985513
2546	3	28-May-15	4-Jun-15	3.8461	5	1171.02	1.522
2549	5	21-May-15	28-May-15	3.85	50	152.22	1.97
2560	4	2-Jun-15	9-Jun-15	3.8426	25	220	1.43
2563	15-17	21-May-15	28-May-15	3.923	20		1.451
2566	4.5	22-May-15	29-May-15	3.96	5	1.16	1.46
2567	4.0	4-Jun-15	11-Jun-15	3.902	10	393.10	1.01
2590	10	22-May-15	29-May-15	3.95	10	622.7	1.582
2605	4.0	2-Jun-15	9-Jun-15	4.0	25	0.211	1.325
2613	4	7-Jun-15	14-Jun-15	3.95	5		0.977
2618	4	2-Jun-15	9-Jun-15	3.903	5		1.101
2622	3	3-Jun-15	10-Jun-15	3.87	25	743	4.800
2624	4	29-May-15	6-Jun-15	3.925	10	447.70	1.152
2637	4	3-Jun-15	10-Jun-15	3.84	4	5300	5.56
2643	4.0	27-May-15	3-Jun-15	3.8	25	202	1.3289
2649	6	26-May-15	2-Jun-15	3.9	10	703.5	1.81
2651	4	1-Jun-15	8-Jun-15	3.84	5	1.1666	1.5190
2653	6	2-Jun-15	9-Jun-15	3.91	6.2	908	1.4397
2654	3.5	4-Jun-15	11-Jun-15	3.45	10	366.2	1.05
2657	4	29-May-15	5-Jun-15	3.9	10	410	1.06
2668	3.7	28-May-15	4-Jun-15	3.70	25	0.3147	2.1263
2674	4.00	26-May-15	2-Jun-15	3.99	20	254.106	1.2737
3100	4.0	18-May-15	25-May-15	4.0	10	0.496	1.240
3116	4	22-May-15	29-May-15	3.91	10	598	1.529
3118							-----
3124							-----
3145	15	1-Jun-15	8-Jun-15	3.84	25	225.7	1.469
3146	3.9	21-May-15	28-May-15	3.91	10	837	2.14
3153	3.9	1-Jun-15	8-Jun-15	3.932	10	536	1.36
3160	7.5	21-May-15	28-May-15	3.94	20	303.8	1.54
3172	4.0			3.88			0.99
3176	8	26-May-15	2-Jun-15	3.93	25	525	2.778
3182	4.0	26-May-15	2-Jun-15	3.91	10	546.859	1.399
3185	3.9	3-Jun-15	10-Jun-15	3.9	10	473	1.213
3190	4.0	1-Jun-15	8-Jun-15	4.0	25	0.11	0.67
3197	3.9	25-May-15	1-Jun-15	3.9	10	780	2.03
3200	3.91	8-Jul-15	15-Jul-15	3.91	10	375	0.959
3210	10	29-May-15	5-Jun-15	3.14	10	1730	5.51
3214	3.85	25-May-15	1-Jun-15	3.85	10	437	1.14
3218	3.9	2-Jun-15	9-Jun-15	3.9	10	469	1.20
3220	10	26-May-15	1-Jun-15	0.28224	10	0.451	1.6
3225	4.0	4-Jun-15	11-Jun-15	3.907	10		1.43
3228	3.94	21-May-15	25-May-15	3.94	10	0.34	0.863
3233	3.9	27-May-15	3-Jun-15	3.9	3.9	0.000154	0.770
3237	8	28-May-15	4-Jun-15	3.95	10	42.543	2.154
3242	5	18-May-15	25-May-15	3.4	25	266	1.96
3243	3.842	26-May-15	2-Jun-15	3.8418	3.84	1120	1.12
3248	4	2-Jun-15	9-Jun-15	3.93	25	193	1.23

APPENDIX 4

Test method procedure details for sample #15075:

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?
110	Glass	NO	Yes. with 0.5% Dodecylbenzene sulfonic acid. sodium salt solution	5-Jun-15	6.50	0,2mL DL-lactic acid, 1,0 g Sodium Chloride, 0,2 g, urea, 200mL De-ionized water	ICP-MS	NO
310	Glass	NO	Yes, with detergent	4-Jun-15	6.45	Ureum, NaCl Lactaat, NaOH, HCL PH6,5 geconserveerd met Nitric Acid	ICP-MS	NO
330							ICP-MS	NO
339	Glass	NO	NO	8-Jun-15	between 6,45-6,55	0,1%m/m Lactic Acid, 0,5% Sodium Chloride, 1M and 0,1M Sodium Hydroxide, 0,1%m/m Urea, Mili-Q water solvent	ICP-OES	YES
362	Glass	Yes, with 5% Nitric Acid for 24 hours		2-Jun-15	6.5	acc. To EN1811	ICP-OES	YES
551	Glass		NO	3-Jun-15	6.5		ICP-OES	
623								
840	Glass	Yes, with 10% Nitric Acid solution for 4 hours	Yes, degrease solution	29-May-15	6.5	Sodium Chloride, Urea, DL-Lactic Acid	ICP-OES	YES
1051	Glass	Yes, with 10% Nitric Acid for 40 hours	Yes, with Sodium dodecylbenzene sulfonate	3-Jun-15	6.50	Sodium Chloride, Urea, Lactic Acid	ICP-OES	NO
2115	Polypropylene	NO	NO	28-May-15	6.49	1g/L Urea, 5g/L Sodium Chloride, 1g/L Lactic Acid	ICP-MS	NO
2121	Glass	Yes, with 2% Nitric Acid for 8 hours	Yes, with Sodium dodecylbenzene sulfate	5-Jun-15	6.54	0,5%NaCl, 0,4% Lactic Acid, 0,1% Urea	ICP-MS	YES
2129	Polysterene	NO	Yes, with dish soap	20-May-15	6.517	1,00g Urea, 5,00g Sodium Chloride, 1.00g Lactic Acid in 1000mL H2O	ICP-MS	NO
2137	Glass	Yes, with 5% Nitric Acid for 24 hours	NO	2-Jun-15	6.5	0,5% sodium chloride, 0,1%m/m lactic Acid, 0,1%m/m Urea, 1M and 0,1M sodium hydrocide solution	ICP-OES	YES
2139	Polypropylene	NO	Yes, with Sodium dodecylbenzene sulfate	5-Jun-15	6.47	Deionized water, 0,5% Sodium Chloride, 0,1% Lactic Acid, 0,1% Urea, 1M and 0,1M Sodium Hydroxide solution	ICP-OES	YES
2156	Polypropylene	Yes, with 5% Nitric Acid for 18 hours	Yes, Commercial Detergent	2-Jun-15	6.50	Urea, Sodium Chloride, Lactic Acid, Deionized water	ICP-OES	NO
2165	Polypropylene	NO	Yes, with 0,5% Sodium dodecylbenzene sulfate	1-Jun-15	6.48	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea	ICP-OES	NO
2172	Glass	Yes, with 4M Nitric Acid for 4 hours	Yes, 5g sodium dodecylbenzene sulfonate in 1L deionized water	26-May-15	6.48	1,00g Urea, 5,00g Sodium Chloride, 1,00g Lactic Acid in 1L deionized water. Adjust the solution to PH 6.50 with 0,1M Sodium Hydroxide solution	ICP-OES	YES
2184	Polypropylene	Yes, with 5% Nitric acid for 24 hours	Yes, with 0,5% Sodium dodecylbenzene sulfate	19-May-15	6.5	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, 1M and 0,1M Sodium Hydroxide Solution	ICP-OES	YES

lab	Test vessel	pretreat Vessel,	sample degreas.	date of test	pH	Composition test solution	analysis	use

	material	(if yes which solution)	(if yes which solution)	sol. prep.			technique	replicates?
2190	Glass	NO	NO	29-May-15	6.5	1g Urea,5g NaCl, 1g Lactic Acid in 1L H2O, PH adjust with NaOH	ICP-OES	NO
2201	Polypropylene	Yes, with 5% Nitric Acid for 24 hours	Yes, with 5% Dodecylbenzene Acid, SodiumSalt	28-May-15	6.49	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea	ICP-OES	YES
2229	Polytetrafluoreth ylene	Yes, stored in a 25% dilute nitric acid solution for 4 hours	Yes, Appropiately-diltuted, neutral, commercially available detergent	20-May-15	6.509	0,5% Sodium Chloride, 0,1%m/m DL Lactic Acid, 0,1%m/m Urea	ICP-OES	NO
2230	Glass	Yes, diluted Nitric Acid for 4 hours	Yes, dodecylbenzene Sulfonate	21-May-15	6.51	0,1% Urea, 0,5% Sodium Chloride, 0,1% Lactic Acid	ICP-OES	YES
2232	Glass	Yes, diluted Nitric Acid for 4 hours	NO	19-May-15	6.5	1,00g Urea, 5,00g Sodium Chloride, 1,00g Lactic Acid	ICP-OES	YES
2235	Glass	Yes, Nitric Acid for 1 hour	Yes, diluted neutral detergent	29-May-15	6.52	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea	ICP-OES	YES
2236	Polytetrafluoreth ylene	NO	Yes, with 5g Dodecylbenzene Sulfonic Acid/1000mL Deionized H2O)	3-Jun-15	6.52	0,5001g NaCl, 0,1011g Urea, 0,1004g Lactic Acid, diluted to final volume with 25mL deionized H2O	ICP-MS	YES
2238	Polypropylene	Yes, with 5%v/v Nitric Acid for 24 hours	Yes, with 0,5% Sodium Dodecylbenzene Sulfate	25-May-15	6.49	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, sodium hydroxide solution	ICP-OES	YES
2241	Polypropylene	NO	Yes, 5g Sodium Dodecylbenzene Sulfonate in 1000mL water	1-Jun-15	6.47	1g Urea, 1g Lactic Acid, 5g Sodium Chloride	ICP-OES	YES
2247	Polypropylene	NO	Yes, with 0,5% aquous commercial detergent	27-May-15	6.49	0,5008g Urea, 2,5009g NaCl, 0,5004g Lactic Acid, 500mL water	ICP-OES	YES
2255	Glass	Yes, Dilute nitric acid (approx 5%) for 4 hours	Yes, degreasing solution for 2 min (anionic surface active agent)		6.5	0,5% sodium chloride+0,1%m/m lactic acid+0,1%m/m urea	ICP-MS	YES
2256	Glass	Yes, with 10% Nitric Acid for 24 hours	Yes, with Sodium Dodecylbenzene Sulfonate		6.50	Urea, Sodium Chloride, Lactic Acid	ICP-OES	YES
2271	Glass	Yes, dilute nitric acid for 5 hours	Yes, with Sodium Dodecylbenzene Sulfate	5-Jun-15	6.50	Deionized water, 0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, 1M and 0,1M sodium hydroxide solution	ICP-OES	NO
2285	Polypropylene	Yes, dilute approx 5% nitric acid for 5 hours	Yes, with 0,5% Sodium Dodecylbenzene Sulfate	26-May-15	6.51	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, 1M and 0,1M Sodium Hydroxide Solution	ICP-MS	YES
2289	Polypropylene	Yes, 5% Nitric Acid for 12 hours	Yes, with 0,5% Sodium Dodecylbenzene Sulfonate	28-May-15	6.5	0,5% NaCl, 0,1% Lactic Acid, 0,1% Urea	ICP-OES	YES
2290	Glass	Yes, 10% Nitric Acid for 24 hours	Yes, with 0,5% Sodium Dodecylbenzene Sulfonate	1-Jun-15	6.49	5,0g Sodium Chloride, 1,0g Urea, 1,0g DL-Lactic Acid, 1000mL Deionized water	ICP-MS	YES

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?
2295	Polypropylene	NO	Yes, Dodecylsulfate sodium salt	26-May-15	6.5	5g NaCl, 1g Lactic Acid, 1g Urea	ICP-MS	YES
2296	PTFE	Yes, with Nitric Acid for 4 hours	NO	1-Jun-15	6.5	As per EN1811:2011	ICP-OES	YES
2300	Other	Yes, with 5% Nitric Acid for 4 hours	Yes, with anionic surface-active agent solution	14-May-15	6.5	1g Urea+5g Sodium Chloride+1g Lactic Acid, dilute to 1L with deionized water	ICP-OES	YES
2301	Glass	Yes, Soak with 5% Nitric Acid for 8 hours	NO	1-Jun-15	6.51	Urea, Sodium Chloride, Lactic Acid, pH adjusted to 6.5	ICP-OES	NO
2309	Polypropylene	NO	NO	5-Jun-15	6.50		ICP-OES	YES
2310	Glass	Yes, with 5% Nitric Acid solution for 4 hours	Yes, with 0,5% Sodium Dodecylbenzene sulfonic acid	22-May-15	6.5	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, maintained pH to 6,5 with 1M/01M Sodium Hydroxide Solution	AAS	YES
2311	Glass	Yes, with 5% Nitric Acid solution for 4 hours	NO	28-May-15	6.48	0,5% Sodium Chloride, 0,1%m/m Lactic Acid, 0,1%m/m Urea, maintained pH to 6,5 with 1M/01M Sodium Hydroxide Solution	ICP-MS	YES
2320	Glass	Yes, dilute Nitric Acid for 3 hours	Yes, with 5g Dodecylbenzene Sulfonic Acid sodium salt in 1000mLwater)	29-May-15	6.5	1,00g Urea, 5,00g Sodium Chloride, 940µl DL Lactic Acid, made up to 1000mL deionized water	ICP-OES	YES
2347	Polypropylene	NO	Yes, with 5% Sodium Dodecyl Sulfonic salt solution	26-May-15	6.54	Sodium Chloride, UREA, DL-Lactic Acid,Grade 1 water	ICP-OES	NO
2352	Glass	NO	Yes, with 0.5% Dodecylbenzene Sulfonic Acid Sodium salt solution	26-May-15	6.52	0.1% Urea, 0.5% Sodium Chloride, 0.1% DL-Lactic Acid	ICP-OES	YES
2357	Polypropylene	NO	Yes, with 5% Dodecylbenzene Sulfonic Acid Sodium salt	28-May-15	6.50		ICP-OES	NO
2362	Polystyrene	Yes, with 1% Nitric Acid for 8 hours	Yes, with0.5% Sodium Dodecylbenzene Sulfate	20-May-15	6.51	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide	FAAS	YES
2363		NO	Yes, with 5% Sodium Dodecyl Sulfate	22-May-15	6.51	0.1% Urea, 0.5% Sodium Chloride, 0.1% DL-Lactic Acid	ICP-OES	NO
2365	Polypropylene	NO	Yes, with 5% Sodium Dodecyl Sulfate salt	28-May-15	6.51	Urea, Sodium Chloride, Lactic Acid	ICP-OES	YES
2366	Polyethylene	Yes, with 5% Nitric Acid for 4 hours	Yes, with Sodium Dodecylbenzene Sulfate	26-May-15	6.50	5g Sodium Chloride, 1g Urea, 1g DL-Lactic Acid made up to 1000mL with Deionized water	ICP-OES	YES
2370	Polypropylene	Yes, with 10% Nitric Acid for 1 hour	Yes, with Extran MA02 neutral	21-May-15	6.49	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide	ICP-OES	YES
2375	Polypropylene	NO	Yes, with Sodium dodecylbenzene sulfonate	27-May-15	6.5	Sodium Chloride, Urea, Lactic Acid, Sodium Hydroxide(for adjust pH)	ICP-MS	YES

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?
2379	Glass	Yes, with 20% Nitric Acid for 12 hours	Yes, with detergent	20-May-15	6.54	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, Matrix = DI water	ICP-OES	YES
2380	Glass	Yes, with 5% Nitric Acid solution for 5 hours	Yes, with degreasing solution (5g Dodecylbenzene Sulfonic Acid Sodium salt in 1000mL DI water)	1-Jun-15	6.52	1.00g Urea, 5g NaCl, 1g DL-Lactic Acid, pH adjust with 1.0M and 0.1M NaOH and 0.1M HCL	AAS	YES
2385	Polypropylene	NO	Yes, with 0.1% Na-Dodecylsulfate	2-Jun-15	6.5	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, Adjust pH to 6.5	ICP-OES	YES
2390	Polypropylene	NO	NO	20-May-15	6.5	5g Sodium Chloride, 1g Urea, 1g DL-Lacti Acid	ICP-OES	YES
2403	Polypropylene	Yes, with 5% Nitric Acid for 4 hours	NO	2-Jun-15	6.51	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, 1M and 0.1M Sodium Hydroxide	ICP-OES	YES
2410	Polypropylene	NO	Yes, with Sodium Dodecylbenzene Sulfate	19-May-15	6.40	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea	ICP-OES	YES
2415	Glass	NO	NO	20-May-15	6.52	Urea, Lactic Acid, NaCl	ICP-OES	YES
2429	Polypropylene	Yes, with 5% Nitric Acid for 12 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	1-Jun-15	6.5	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, Sodium Hydroxide solution, Milli-Q water	ICP-OES	YES
2432	Polypropylene	Yes, with 10% Nitric Acid overnight and test solutions for 10 min	Yes, with commercially available detergent	25-May-15	6.5	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, 1M and 0.1M Sodium Hydroxide, Deionized water	ICP-MS	YES, by spiking
2442	Polypropylene	NO	NO	27-May-15	6.15	1g Urea, 5g Sodium Chloride, 1g Lactic Acid in 1L deionized water	ICP-MS	NO
2459	Glass	Yes, with Sodium Dodecylbenzene Sulfonate for 1/2 hour	Yes, with Sodium Dodecylbenzene Sulfonate	26-May-15	6.52	Deionized water acc. to EN ISO 3696:1195. Grade II, Sodium Chloride, DL-Lactic Acid, Urea, Sodium Hydroxide	ICP-OES	YES
2468	Polypropylene	NO	Yes, with 5g/L Sodium Dodecylbenzene	28-May-15	6.5	5g Sodium Chloride, 1mL Lactic Acid, 1g Urea, Sodium Hydroxide to adjust pH, made up to 1L with Deionized water	ICP-OES	YES
2475	Glass	Yes, wih 5% Nitric Acid for 5 hours	Yes, with Sodium Dodecylbenzene Sulfonate	25-May-15	6.52	0.5% Sodium Chloride, 0.1% Urea, 0.1% Lactic Acid, 1M and 0.1M Sodium Hydroxide	ICP-MS	YES
2482	Glass	NO	Yes, with Ethanol	19-May-15	6.5	According to EN 1811:2011	ICP-MS	YES
2485	Polypropylene	Yes, with 5% Nitric Acid for 4 hours	Yes, with 5g dishwashing detergent per liter water	2-Jun-15	6.5	1.0g Urea, 5.0g Sodium Chloride, 1.0g Lactic Acid per liter H2O	ICP-OES	YES
2488	Glass	NO	NO	18-May-15	6.45	1g Lactic Acid, 1g Urea, 5g Sodium Chloride	ICP-MS	YES
2489	Polypropylene	NO	Yes, with 0.5% Aquous commercial detergent	4-Jun-15	6.50	1.0006g Urea, 5.0008g NaCl, 940µL Lactic Acid, 1000mL water	ICP-MS	YES
2492	Polypropylene	NO	Yes, with 0.5% Aquerous solution of Sodium Dodecylbenzene Sulfate	3-Jun-15	6.522	Sodium Chloride, Lactic Acid, Urea	ICP-MS	YES
2495	Polypropylene	NO	Yes, with 0.5% Sodium Alkylaryl Sulfate	27-May-15	6.50	5g NaCl, 1g Lactic Acid, 1g Urea to 1L with MilliQ water, pH adjusted with 1M and 0.1M NaOH	ICP-OES	NO

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?
2497	Polypropylene	Yes, with 4% diluted Nitric Acid for 4 hours	Yes, with commercial soap	21-May-15	6.5	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide, Deionized water	ICP-MS	YES
2504	Glass	Yes, with Sodium Dodecyl Sulfate for 30 min	Yes, with Sodium Dodecyl Sulfate	28-May-15	6.50	5g NaCl, 1g Urea, 1g Lactic Acid in 1000mL, pH adjusted to 6.5 with NaOH	ICP-MS	NO
2511								
2514	Glass	Yes, with 5% diluted Nitric Acid for 4 hours	Yes, with degreasing solutionfor 2 min (anioninc surface active agent)	10-Jun-15	6.5	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea		YES
2515	Polypropylene	NO	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	20-May-15	6.51	1g Urea, 5g NaCl, 1g Lactic Acid, 900mL Deionized water, pH adjusted to 6.5, transfer to 1L volumetric flask	ICP-OES	YES
2516	Glass	Yes, with 5% Nitric Acid for 4 hours	Yes, with neutral detergent	21-May-15	6.48	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide solution	ICP-OES	YES
2546	Polypropylene	NO	Yes, with Neutral commercially detergent	28-May-15	6.50	1g Urea, 5g NaCl, 1g Lactic Acid in 1Liter	ICP-OES	YES
2549	Polypropylene	Yes, for 4 hours	No	21-May-15	6.51	1g Urea, 5g NaCl, 1g Lactic Acid in 1L deionazed water	ICP-MS	YES
2560	Glass	NO	Yes, with Sodium Dodecylbenzene Sulfonate	2-Jun-15	6.50	1g Urea, 5g NaCl, 1g Lactci Acid in 1L. pH adjusted with 0.1M NaOH	ICP-MS	YES
2563	Glass	Yes, with 2% Nitric Acid for 1/30 hour	Yes, with Natrium Dodecyl Sulfate	19-May-15	6.59	1g Urea, 5g NaCl, 826µL Lactic Acid + H2O add to 1L	ICP-MS	YES
2566	Polypropylene	NO	Yes, with commercial detergent solution (Dodecylbenzene)	22-May-15	6.5	Lactic Acid, Sodium Chloride, Urea	ICP-OES	YES
2567	Glass	NO	Yes, with Sodium Dodecylbenzene Sulfonate	4-Jun-15	6.5	Urea, Sodium Chloride, Lactic Acid, DI water, pH adjusted with NaOH	ICP-MS	NO
2590	Polypropylene	NO	NO	22-May-15	6.48	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, water solution	ICP-MS	YES
2605	Glass	Yes	Yes	2-Jun-15	6.50	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide solution	ICP-OES	YES
2613	Glass	Yes, with diluted Nitric Acid for 24 hours	Yes, with Dodecylbenzene sulfonic acid	7-Jun-15	6.49	1g Urea, 5g NaCl, 940µl Lactic Acid in 1L DI water	ICP-OES	YES
2618	Glass	Yes, with 5% Nitric Acid for 4 hours	Yes, with Sodium Dodecylbenzene Sulfonate	20-May-15	6.50	1.0g Urea, 5.00g Sodium Chloride, 940µL Lactic Acid, Deionized water to make 1L solution	ICP-OES	YES
2622	Polypropylene	NO	Yes, with 5g/L Sodium Dodecylbenzene sulfate	3-Jun-15	2.82	0.5% NaCl, 0.1% Urea, 0.1% Lactic Acid	ICP-OES	YES
2624	Polypropylene	Yes, with 5% Nitric Acid for 4 hours	Yes, with 5% Sodium Alkyl Sulfonate	29-May-15	6.54	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea in 1000mL Deionized water, 1M and 0.1M Sodium Hydroxide solution to adjust pH to 6.5	ICP-OES	YES

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?

2637	HD-PE	NO	NO	3-Jun-15	6.5	5g/L NaCl, 1g/L Lactic Acid, 1g/L Urea	ICP-MS	YES
2643	Glass	Yes, dilute Nitric Acid for 4 hours	Yes, with Sodium Dodecylbenzene Sulfate	27-May-15	6.5	1g Urea, 5g Sodium Chloride, 1g Lactic Acid	ICP-OES	YES
2649	Glass	NO	NO	26-May-15	6.5	1g Urea, 5g NaCl, 1g Lactic Acid in 1Liter	ICP-MS	YES
2651	Glass	Yes, with 10% Nitric Acid for 24 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	1-Jun-15	6.50	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide solution	ICP-OES	NO
2653	Glass	NO	Yes, with household detergent	2-Jun-15	6.55	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, 0.1M Sodium Hydroxide, H2O	ICP-OES	YES
2654	Glass	Yes, with 5% Nitric Acid for 4 hours	Yes, with Sodium Dodecylbenzene Sulfonate	29-May-15	6.5	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1% Ammonia solution	ICP-OES	NO
2657	Glass	Yes, with 5% diluted Nitric Acid for 24 hourt	Yes, with 0.5% Sodium Lauryl Sulfate	29-May-15	6.51	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, 1M and 0.1M Sodium Hydroxide Solution, 0.1M HydroChloric Acid Solution	ICP-OES	YES
2668	Polypropylene	Yes, with diluted Nitric Acid for 4 hours	No	28-May-15	6.54	NaCl, Urea, Lactic Acid	ICP-OES	YES
2674	Polypropylene	NO	NO	26-May-15	6.51	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, Deionized water	ICP-OES	YES
3100	Glass	Yes, with diluted Nitric Acid for 4 hours	Yes, with degreasing solution	18-May-15	6.50	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxide solution	ICP-OES	NO
3116	Glass	Yes, with diluted Nitric Acid for 4 hours	Yes, with degreasing solution	22-May-15	6.5	1g Urea, 5g Sodium Chloride, 1g Lactic Acid in 1L volume	ICP-OES	YES
3118								
3124								
3145	Glass	Yes, with 5% Nitric Acid for 4 hours	Yes, with 5g/L Sodium Dodecyl Sulfate	21-May-15	6.49	250mL test Solution with 1.25g NaCl, 0.25g Lactic Acid, 0.25g Urea	ICP-OES	YES
3146	Polypropylene	NO	Yes, with Sodium Dodecyl Sulfate	21-May-15	6.47	Sodium Chloride, Lactic Acid, Urea	ICP-OES	YES
3153	Glass	Yes, with 5% Nitric Acid for at least 4 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	1-Jun-15	6.53	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, Sodium Hydroxide solution for pH adjustment	ICP-OES	YES
3160	Glass	Yes, with 5% Nitric Acid for 2 hours	Yes, with Anionic Surfactant	21-May-15	6.49	0.5% NaCl, 0.1%m/m Lactic Acid, 0.1%m/m Urea, pH adjust with NaOH to 6.5	ICP-OES	NO
3172								
3176	Polypropylene	NO	Yes, with neutral detergent	22-May-15	6.5	Urea, Sodium Chloride, Lactic Acid	ICP-MS	YES
3182	Polypropylene	NO	NO	26-May-15	6.5	1gUrea, 5g NaCl, 1g Lactic Acid	ICP-MS	NO
3185	Glass	Yes, with 30% Nitric Acid for 24 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	3-Jun-15	6.54	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea	ICP-OES	YES
3190	Glass	Yes, with 20% Nitric Acid for 24 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	1-Jun-15	6.51	0.5% Sodium Chloride, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M Sodium Hydroxyde solution	ICP-OES	YES

lab	Test vessel material	pretreat Vessel, (if yes which solution)	sample degreas. (if yes which solution)	date of test sol. prep.	pH	Composition test solution	analysis technique	use replicates?
3197	Polypropylene	Yes, with 5% diluted Nitric Acid for 4 hours	Yes, with degreasing solution for 2 min (surface active agent)	25-May-15	6.49	0.5% NaCl, 0.1%m/m Lactic Acid, 0.1%m/m Urea	ICP-MS	YES
3200	Polypropylene	Yes, with 5% Nitric Acid for 24 hours	Yes, with sodium dodecyl benzene sulfonate	8-Jul-15	6.50	1.0 g urea, 5.0 g sodiumchloride, 1.0 g lactic acid, 0.1 water	ICP-OES	NO
3210	Polysterene	NO	Yes	28-May-15	6.55		ICP-OES	NO
3214	Glass	Yes, with 5% Nitric Acid for 18 hours	Yes, with Acid detergent (Citranox)	25-May-15	6.49	0.5% Sodium Chloride, 0.1%m/m DL-Lactic Acid, 0.1%m/m Urea, Deionized water, 1M and 0.1M Sodium Hydroxide solution	ICP-OES	YES
3218	Polypropylene	Yes, with 5% Nitric Acid for 12 hours	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	2-Jun-15	6.48	0.5% Sodium Chloride, 0.1% Lactic Acid, Sodium Hydroxide solution, MillQ water	ICP-OES	YES
3220	Glass	NO	NO	26-May-15	6.5	0.1g Urea, 0.5g NaCl, 0.1Lactic Acid make up with 100ml	ICP-MS	NO
3225	Polypropylene	NO	Yes, with 0.5% Sodium Dodecylbenzene Sulfonate	4-Jun-15	6.53	0.1% DL-Lactic Acid, 0.1% Urea, 0.5% Sodium Chloride	ICP-OES	YES
3228	Polypropylene	NO	Yes, with 0.5% Sodium Dodecylbenzene Sulfate	21-May-15	6.46	1.00g Urea, 5.00g Sodium Chloride, 1.00g Lactic Acid, to 1L, pH 6.45-6.55	ICP-OES	YES
3233	Polystyrene	NO	Yes, with DECON 90	27-May-15	6.48	0.1g Urea, 0.5g NaCl, 94µL Lactic Acid in 100ml, adjust pH to 6.5 with 1M NaOH	ICP-OES	YES
3237	Polypropylene	NO	Yes, with 5g/L degreasing solution (commercial detergent) in deionized water	28-May-15	6.52	5g NaCl, 1g Urea, 1g Lactic Acid fill to 1000 mL deionized water	ICP-MS	YES
3242	Polypropylene	Yes, with 5% diluted Nitric Acid for 4 hours	Yes, with Sodium Dodecylbenzene Sulfonate	18-May-15	6.53	0.5% Sodium Chloride, 0.1% Lactic Acid, 0.1% Urea, 1M and 0.1M Sodium Hydroxide for pH adjustment	ICP-OES	NO
3243	Polypropylene	NO	NO	26-May-15	6.52	0.5% NaCl, 0.1%m/m Lactic Acid, 0.1%m/m Urea, 1M and 0.1M NaOH solution	ICP-OES	YES
3248	Glass	Yes, with artificial sweat solution	Yes, with degreasing solution	2-Jun-15	6.53	Urea, Sodium Chloride, Lactic Acid	ICP-MS	NO

APPENDIX 5

Number of participants per country

8 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA, Kingdom of
1 lab in	DENMARK
7 labs in	FRANCE
8 labs in	GERMANY
9 labs in	HONG KONG
11 labs in	INDIA
3 labs in	INDONESIA
9 labs in	ITALY
1 lab in	JAPAN
4 labs in	KOREA
1 lab in	MALAYSIA
1 lab in	MOROCCO
28 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	SINGAPORE
1 lab in	SPAIN
1 lab in	SRI LANKA
1 lab in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
3 labs in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
6 labs in	TURKEY
2 labs in	U.S.A.
3 labs in	UNITED KINGDOM
4 labs in	VIETNAM

APPENDIX 6

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's GESD outlier test
R(0.05)	= straggler in Rosner's GESD 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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

2 Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning (REACH) under entry 27 of Annex XVII, 2012

3 EN1811:2011 + AC2012

4 Horwitz. Journal of AOAC International Vol. 79 No.3. 1996

5 P.L. Davies. Fr Z. Anal. Chem. 351. 513. (1988)

6 W.J. Conover. Practical; Nonparametric Statistics. J. Wiley&Sons. NY. p.302. (1971)

7 ISO 5725 (1986)

8 ISO 5725 parts 1-6. (1994)

9 ISO13528:2005 Statistical methods for use in proficiency testing by interlaboratory comparisons

10 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)

11 Analytical Methods Committee Technical brief, No4 January 2001.

12 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/).

13 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), pp. 165-172, (1983)